Erbium and Hydrogen Implantation into Glass for Optical Device Applications

David John Kyle.
1996.

Thesis submitted for the degree of Doctor of Philosophy to the Department of Electronic and Electrical Engineering, University of Surrey.
Abstract

A material which exhibits a refractive index change on exposure to ultra violet light is obtained when germania doped silica is heated in a hydrogen atmosphere. This phenomena is called photosensitivity. Bragg gratings, which are useful for the fabrication of feedback mirrors in optical amplifiers and as optical filters, can be readily fabricated by exposing a photosensitive waveguide to an ultraviolet interference pattern.

Ion implantation, which allows localised substrate doping, is a useful means of introducing hydrogen when compared to thermal annealing. In this thesis, it is demonstrated for the first time that hydrogen implantation into heated germania doped silica yields a photosensitive material.

The optical properties of arsenic doped silica (ASG) waveguides allow the fabrication of single mode waveguides of height 2 μm, a depth which implanted erbium ions may readily penetrate. Erbium implanted ASG waveguides are therefore a suitable candidate for use as the amplifying region in waveguide optical amplifiers. In this thesis, ion implantation is used to introduce erbium atoms into ASG waveguides, and the optical characteristics of the implanted waveguides assessed.

The implanted waveguide optical propagation loss, the 1550 nm fluorescent lifetime decay, and the optical propagation loss of erbium implanted ASG waveguides was measured as a function of erbium dose. These experiments are required to determine the suitability of erbium implanted ASG waveguides for the fabrication of optical amplifiers. It was demonstrated that erbium fluorescence lifetimes of up to 10 ms and an optical absorption in the region of 2 dB/cm to 4 dB/cm at a wavelength of 1550 nm could be obtained whilst maintaining a low waveguide optical propagation loss. These results suggest that erbium implanted into ASG waveguides is suitable for the fabrication of the active region of optical amplifier devices.
Acknowledgements

The author wishes to acknowledge Mr B J Ainslie and Dr G Maxwell of BT Laboratories for useful discussions and sample fabrication; the UK Science and Engineering Research Council and BT Laboratories for the support of this project; Dr P D Prewett of Rutherford Appleton Laboratory for the excimer laser exposure; Dr G Buist of the University of Surrey for help with the absorption measurements; Dr R Wilson for the implantation of samples, and Dr B L Weiss for supervision and guidance during the course of this project.
Contents

Introduction 8

Chapter 1  A Review of Erbium in Amorphous Silica.
  1.0  Introduction 12
  1.1  Rare Earth Elements in Amorphous Silica 13
  1.2  The Role of Glass Composition 15
  1.3  Factors Effecting Amplifier Performance 17
      1.3.1  Co-operative Up-conversion 17
      1.3.2  Amplified Spontaneous Emission 19
      1.3.3  Excited State Absorption 19
      1.3.4  Optimisation of Pump Conditions 21
  1.4  Erbium Doped Waveguides and Optical Fibres 21
      1.4.1  Waveguide Fabrication 22
      1.4.2  Properties of Erbium Doped Planar Waveguides 23
  1.5  Erbium Implanted Planar Waveguides 25
  1.6  Summary 27
  1.7  References 35

Chapter 2  A Review of Photosensitivity in Germania Doped Silica Glass
  2.0  Introduction 39
  2.1  The Mechanism of Photosensitivity 40
  2.2  Implantation 42
  2.3  Grating Formation 44
  2.4  Summary 47
  2.5  References 55
Chapter 3  Experimental Methods

3.0  Introduction
3.1  Sample Fabrication and Structure
3.2  Ion Implantation
    3.2.1  Post Implantation Sample Cleaning
3.3  Ultraviolet Absorption
3.4  Waveguide Propagation and Insertion Loss
3.5  Waveguide White Light Transmission Spectra
3.6  Erbium Fluorescence Lifetime Decay
3.7  Conclusions
3.8  References

Chapter 4  Modelling of the Implanted Ion and Waveguide Mode Profiles

4.0  Introduction
4.1  Computer Simulations
4.2  Rutherford Backscattering Analysis
4.3  ASG Waveguide Fundamental Optical Mode Intensity Profiles
4.4  Comparison of Waveguide Optical Mode Profiles and Implantation Profiles
4.5  Comparison of Optical Mode Profiles
4.5  Conclusions
4.5  References

Chapter 5  The Photosensitivity of Proton Implanted Germania Doped Planar Silica Structures

5.0  Introduction
5.1  Procedure
5.2 Results and Discussion

5.2.1 Effect of Room Temperature Implantation as a Function of Implantation Dose 105

5.2.2 Ion Beam Heating 107

5.2.3 The Effect of Sample Temperature During Implantation 108

5.2.4 Multiple Energy Implantation 109

5.2.5 Optical Exposure 110

5.3 A Comparison of Thermal Hydrogenation and Implantation 111

5.4 Summary of Results 111

5.5 References 119

Chapter 6 The Optical Properties of Erbium Implanted ASG Waveguides

6.0 Introduction 120

6.1 Procedure 121

6.1.1 Implantation Parameters 121

6.1.2 Post Implantation Annealing 123

6.2 Implantation Induced Waveguide Damage 124

6.2.1 Scattered Light Technique 124

6.2.2 Fibre In/Fibre Out Technique 125

6.3 Fluorescence Characteristics 127

6.3.1 Material Fluorescence 127

6.3.2 Erbium Fluorescence Lifetime and the Effect of Annealing 128

6.3.3 Fluorescence as a Function of Pump Power 132

6.3.4 Multiple Implantation 135

6.3.5 Comparison of Er in ASG and Phosphorous Waveguides 135

6.4 Optical Transmission Spectra 135

6.5 Summary of Results 137

6.6 References 144
Introduction

Various components are required for the development of modern optical fibre communication networks, of which the erbium doped fibre amplifier (EDFA) is of particular importance. With the introduction of the EDFA it was theoretically possible to deploy a single optical fibre spanning an entire ocean that could carry over 500,000 simultaneous telephone conversations without any signal loss. This is because the EDFA allows zero loss signal propagation without the need for complicated repeater electronics to regenerate reduced signals.

Together with optical fibre technology, the glass optical integrated circuit plays an ever increasing role in handling telecommunications data. Typical devices include splitters/combiners, optical switches and the wavelength division multi/demultiplexer. The use of an optical amplifier as a standard component, operating in the same manner as the EDFA, is extremely useful to the continuing development of the glass optical integrated circuit.

To date various groups have achieved amplification in erbium doped stripe waveguides using a variety of fabrication techniques. However, in the majority of cases it was required that the entire substrate on which the device was based must be erbium doped, a problem that cannot be easily solved when conventional sample fabrication is considered. In this project, ion implantation is considered as a means of localized erbium doping.

Various groups, including Polman et al, have reported on the optical properties of erbium implanted into silica based glasses, including phosphosilica glass. In these materials, a singlemode 1550 nm waveguide is approximately 7 μm deep. Since the penetration depth of the implanted atom is proportional to the implantation beam energy, high implantation energies are required to penetrate the waveguide to the 7 μm depth. It would therefore be useful if this problem could be solved to allow implantation at lower, more commercially viable, energies.
Arsenic doped silica glass (ASG), fabricated by the BT laboratories, has a high Δn (refractive index difference) between the core and the cladding of the waveguide. This is achieved by varying the arsenic content between the waveguide core and cladding layer. Using this material system, 1550 nm single mode optical waveguides have a core thickness of 2 μm, and hence lower implantation energies are required to achieve a close match between the implanted erbium profile and the waveguide fundamental optical mode profile. In this project, arsenic doped silica planar waveguides are implanted with erbium and the optical properties of the structures characterised. From the data obtained, the suitability of erbium implanted arsenic doped silica as a material capable of optical amplification is assessed.

In addition, the fabrication of Bragg gratings in silica waveguides is important as they are useful for wavelength filters and components in optical amplifiers. Various groups have produced high quality Bragg gratings which are written into silica integrated circuits simply by exposure to interfering ultraviolet laser beams. The exposure to the ultraviolet light modifies the infrared refractive index of the material and this process is called photosensitivity. To fabricate the photosensitive material, immersion at a high temperature in a reducing atmosphere is required, a process which may be harmful to other devices on the same substrate. One of the most promising photosensitive materials is germania doped silica. In this project, the enhancement of the photosensitivity in germania doped silica by hydrogen implantation, as an alternative to the thermal indiffusion of hydrogen, is investigated.

Chapters 1 and 2 contain a literature review of erbium doped waveguides and photosensitivity in silica. Chapter 3 describes the experimental techniques used in the project. Following this, chapter 4 gives details of the implantation parameters. The experimental procedure and results of the hydrogen and erbium implantation are described in chapters 5 and 6. Finally the conclusions reached and possible future work are discussed in chapter 7.
References


Chapter 1

A Review of Erbium in Amorphous Silica

1.0 Introduction

This chapter reviews the optical characteristics of erbium in silica and erbium in silica waveguides. When incorporated into a silica matrix, the rare earth elements remain optically active, and most importantly they can behave as the amplification medium of a laser. The basics of the optical amplifier are described in section 1.1.

The performance of erbium amplifiers is dependant on several factors, probably the most significant of which is the host glass composition. Together with erbium, various co-dopants can have profound effects on improving amplifier characteristics. This is described in section 1.2.

Several mechanisms play an important role in determining amplifier performance and are described in section 1.3. The more important mechanisms include co-operative up-conversion, amplified spontaneous emission, excited state absorption and the pump light configuration.

Sections 1.1 to 1.3 of this review discuss the optical properties of erbium in silica on an atomic level. The remainder of this chapter is devoted to describing results achieved by various groups. To begin with, the properties of various erbium doped silica waveguides and the fabrication techniques involved are briefly reviewed in section 1.4. However, as this project is primarily concerned with erbium implantation, the properties of erbium waveguides fabricated by ion implantation are reviewed in more detail in section 1.5. Finally, a summary of this chapter is presented in section 1.6.
### 1.1 Rare Earth Elements in Amorphous Silica

The fifteen elements in the lanthanide series ranging from lanthanum (La, atomic number 57) to lutetium (Lu, atomic number 71) all share the same outer electron configuration. With increasing atomic number, electron shell filling takes place within the inner 4f electron shell. The atomic configuration of the lanthanides is shown in Table 1.1 where [Xe] denotes the configuration of xenon.[1]

<table>
<thead>
<tr>
<th>Atom</th>
<th>Electron Configuration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lanthanum</td>
<td>[Xe] (6s (^2) 5d)</td>
</tr>
<tr>
<td>Cerium</td>
<td>[Xe] (6s (^2) 4f (^5) 5d)</td>
</tr>
<tr>
<td>Praseodymium</td>
<td>[Xe] (6s (^2) 4f)</td>
</tr>
<tr>
<td>Neodymium</td>
<td>[Xe] (6s (^2) 4f)</td>
</tr>
<tr>
<td>Promethium</td>
<td>[Xe] (6s (^2) 4f)</td>
</tr>
<tr>
<td>Samarium</td>
<td>[Xe] (6s (^2) 4f)</td>
</tr>
<tr>
<td>Europium</td>
<td>[Xe] (6s (^2) 4f)</td>
</tr>
<tr>
<td>Gadolinium</td>
<td>[Xe] (6s (^2) 4f (^5) 5d)</td>
</tr>
<tr>
<td>Terbium</td>
<td>[Xe] (6s (^2) 4f)</td>
</tr>
<tr>
<td>Dysprosium</td>
<td>[Xe] (6s (^2) 4f(^6))</td>
</tr>
<tr>
<td>Holmium</td>
<td>[Xe] (6s (^2) 4f(^5))</td>
</tr>
<tr>
<td>Erbium</td>
<td>[Xe] (6s (^2) 4f(^8))</td>
</tr>
<tr>
<td>Thulium</td>
<td>[Xe] (6s (^2) 4f(^8))</td>
</tr>
<tr>
<td>Ytterbium</td>
<td>[Xe] (6s (^2) 4f(^8))</td>
</tr>
<tr>
<td>Lutetium</td>
<td>[Xe] (6s (^2) 4f(^8) 5d)</td>
</tr>
</tbody>
</table>

Table 1.1. The Structure of the lanthanides.

Ionization of the rare earth elements normally takes place to form the trivalent state (i.e. Er\(^{3+}\)), whereby two of the 6s and one of the 4f electrons are removed while the outer 5p shell remains intact.[2] Electronic transitions taking place within the 4f shell are partially shielded from external electric fields by the 5p shell. This is why rare earth elements incorporated into glasses show similar optical properties to the free element. However, Stark splitting, whereby the residual electric field from the host lattice penetrates the inner electron orbitals results in a broadening of the observed 4f transition energies.[3]
An excited electron can decay either by the emission of a phonon or by the transfer of energy to another electron or by the emission of a photon. In the majority of optical transitions, an excited electron will almost immediately return to the ground state via the emission of a photon of wavelength corresponding to the electron transition energy, i.e. spontaneous emission. However, an excited electron can interact with an incident photon of energy corresponding to the electron excitation energy, resulting in the decay of the electron and the emission of a photon identical in phase and momentum to the first photon. This is called stimulated emission. The process of stimulated emission allows optical amplification, hence the term LASER: "Light Amplification by the Stimulated Emission of Radiation".\textsuperscript{[4]}

For the operation of a laser, it is necessary that a high concentration of excited electrons in an energy level corresponding to the laser transition wavelength can be maintained. When the electron density in the excited state is higher than the electron density in the ground state a condition called population inversion exists. Population inversion is commonly achieved by either electrical excitation (i.e. HeNe and Ar lasers) or optical absorption (ruby lasers). As mentioned previously, an excited electron will return to the ground state almost immediately. However, the decay time from specific energy levels, termed metastable levels, is relatively long. The longer the decay lifetime the more efficient the laser, because less energy is required to maintain population inversion.

The Er\textsuperscript{3+} ion has a metastable energy level corresponding to a transition wavelength of 1554 nm,\textsuperscript{[2]} which coincides with the low loss telecommunications wavelength in silica optical fibres. The incorporation of Er\textsuperscript{3+} ions into a silica glass matrix has led to the erbium doped fibre amplifier (EDFA). The EDFA is operated by passing the pump beam, which is used to excite the erbium atoms, through the same fibre as the signal beam to be amplified. The pump beam is absorbed to produce a high concentration of excited electrons (i.e. population inversion), thereby amplifying the signal beam by stimulated emission. The basic configuration of the erbium doped fibre amplifier is shown in figure 1.1.
Energy level diagrams are a convenient way of describing the various electron states possible in an excited atom. Figure 1.2 represents the main energy levels within the Er$^{3+}$ atom. Spectroscopic notation is used to describe the state of the atom, and is given by $^2S_{2L}$, where $S$ is the spin quantum number, $J$ the total angular momentum quantum number, and $L$ the orbital angular momentum quantum number. At pump wavelengths other than 1480 nm, the EDFA operates as a three level laser system. The pump light excites the 4f electrons to a higher energy level where they subsequently relax to the metastable $^4I_{15/2}$ level by non-radiative transitions. The electrons then return to the ground state ($^4I_{15/2}$) either by the emission of a photon or by a non-radiative process. Optical transitions may take place with either the creation or annihilation of a phonon. This accounts for the difference in the excitation and de-excitation wavelengths of the ground state to $^4I_{15/2}$ transition. The white light transmission spectra of a typical erbium doped waveguide shows optical absorption at wavelengths corresponding to the erbium atom transition energies, the strength of the absorption is dependant on the level of erbium doping. The major absorption wavelengths of erbium in silica are 980 nm, 1480 nm and 1550 nm. Figure 1.3 shows a typical optical absorption spectra of an erbium doped optical fibre.

1.2 The Role of Glass Composition

Of crucial importance to EDFA design is the host glass composition. The high softening temperature of amorphous silica (1940 °C) can be reduced by adding network formers (e.g. sodium ions) to the covalently bonded amorphous SiO$_2$ (an amorphous structure shows no repeatability in the crystal lattice; glass is a supercooled liquid). For example, soda glass, with a softening temperature of 1003 K has a composition of 73% SiO$_2$ and 27% Na$_2$O. Sodium forms ionic bonds with SiO$^-$ groups breaking up the glass structure and reducing the required processing temperatures.

Various co-dopants have been used to modify the properties of the high quality glasses used for the manufacture of optical fibres. In pure silica there is a lack of SiO$^-$ groups which leads to the poor solubility and clustering of dopant erbium ions.
In particular $\text{GeO}_2$ and $\text{Al}_2\text{O}_3$ are used to raise the core refractive index in silica based fibres. While germanium oxide has little effect on rare earth solubility, $\text{Al}_2\text{O}_3$ forms an aluminum complex around the rare earth ion (a solvation cell) which is readily incorporated into the silica structure. The benefit of incorporating $\text{Al}_2\text{O}_3$ is the higher rare earth concentrations which can be introduced before the onset of clustering. $\text{P}_2\text{O}_5$ and $\text{Al}_2\text{O}_3$ are used as co-dopants to significantly increase the solubility of erbium in silica.\cite{7} Optimization of the glass composition allows up to several weight percent of erbium to be incorporated in the glass. The more tightly bonded erbium in $\text{Al}_2\text{O}_3$ co-doped glasses also prevents Er diffusion during device manufacture.

Optical amplifiers operating at 1550 nm are now an established technology. However, an efficient means of amplification at 1300 nm still remains an elusive goal. Praseodymium doping (praseodymium has an optical emission peak at 1300 nm) is the most promising solution identified to date. Unfortunately, the small energy difference between the $^1\text{G}_4$ and $^3\text{F}_4$ levels and the high phonon energy of silica leads to a complete quenching of the metastable level. A promising solution to this problem is offered by fluoride glass\cite{8} (a mixture of $\text{ZrF}_4$, $\text{BaF}_2$, $\text{LaF}_3$, $\text{AlF}_3$, and $\text{NaF}$ commonly abbreviated to ZBLAN glass) which has a lower phonon energy than silica because of the heavier masses of the constituent elements.

Lanthanum fluoride also allows higher rare earth doping concentrations.\cite{9} Erbium doped fluoride glasses show radiative transitions at 2700 nm, a region described as the fourth telecommunications window.\cite{10} The spectral width of the 1550 nm Er optical absorption peak in erbium/fluoride glasses is also broadened when compared to silica host glasses.\cite{11}

Ytterbium may be used as a co-dopant to increase the range of suitable pump wavelengths. The broad absorption band of Yb (800 - 1100 nm) is utilized by allowing cross relaxation to take place between Yb and Er, i.e. Yb returns to the ground state via a transfer of energy to promote Er to an excited state. Er/Yb amplifiers operating at 1550 $\mu$m have been reported with gains of up to 51 dB using...
a 1064 nm pump source. The amplifier efficiency is limited by a significant energy loss from the Yb radiative transitions and cross relaxation between neighbouring Yb atoms.[12]

High concentrations of optically inactive lanthanum (it has no 4f electrons) have been used to increase amplifier efficiency. If a high enough La concentration is present, clustering will preferentially involve La and not Er. High gain La/Er fibre amplifiers with gains of 30 dB have been fabricated using only 2 m to 4 m of doped fibre. The first fabricated Er/La doped fibre had an Er concentration of $2.3 \times 10^{18}$ ions/cm$^3$ with an La to Er ratio of 14:1. Erbium lifetimes of the order 10 ms to 11 ms were measured and gains of 30 dB were achieved over only 4 m of fibre, compared to about 20 m that would be required without La co-doping.[13]

1.3 Factors Effecting Amplifier Performance

In addition to the host glass composition there are a number of factors which affect the optical characteristics of erbium doped silica waveguides, and the important ones are discussed in the following section.

1.3.1 Co-operative Up-conversion

The pump power dependence of the fluorescence decay lifetime of the Er $^4I_{13/2}$ metastable level increases with increasing erbium concentration, a phenomena that has been attributed to co-operative up-conversion. Although it is not a problem in fibre amplifiers, up-conversion remains a serious obstacle in waveguide amplifiers where the much shorter optical path length (of the order of cm) necessitates a significantly higher erbium concentration.

The effect of erbium concentration on the quantum efficiency (QE) and fluorescence decay lifetime was described by Ainslie.[2] $\text{Al}_2\text{O}_3-P_2\text{O}_5-\text{SiO}_2$ fibres were fabricated with erbium concentrations ranging from 0.2 wt. % to 18 wt. % using the solution
doping method. QE measurements were performed by pumping the fibre with 514 nm light to populate the $^4_{132}$ state and collecting the fluorescence from the fibre with a Ge detector. The QE was calculated using the solid angle of the acceptance of the detector, the detector efficiency and the measured absorbed pump power. Fluorescence lifetime measurements were calculated by pumping the fibre with mechanically chopped 807 nm light at powers between 1 mW and 80 mW. A very short fibre length was used to avoid inaccuracies due to amplified spontaneous emission; a phenomena discussed in section 1.3.2 below. The erbium fluorescence decay curve was found to consist of a single exponential component for optical fibres with low doses of erbium while for higher erbium doses the decay curve was the sum of two exponential decays, the original slow decay and a fast decay component whose magnitude was proportional to the erbium dose and the pump power. Table 1.2 summarizes the results obtained, listing the $1/e$ lifetime of both the fast and slow decay components and the quantum efficiency as a function of erbium concentration.

<table>
<thead>
<tr>
<th>Er wt%</th>
<th>Quantum Efficiency</th>
<th>Fast (ms)</th>
<th>Slow (ms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.07</td>
<td>-</td>
<td>None</td>
<td>10.3</td>
</tr>
<tr>
<td>0.15</td>
<td>0.8</td>
<td>8.7</td>
<td>9.8</td>
</tr>
<tr>
<td>0.20</td>
<td>-</td>
<td>8.7</td>
<td>9.8</td>
</tr>
<tr>
<td>1.50</td>
<td>1.0</td>
<td>4.5</td>
<td>9.5</td>
</tr>
<tr>
<td>2.30</td>
<td>0.9</td>
<td>1.0</td>
<td>8.5</td>
</tr>
<tr>
<td>14</td>
<td>-</td>
<td>4.0</td>
<td>8.5</td>
</tr>
<tr>
<td>18</td>
<td>-</td>
<td>4.0</td>
<td>8.8</td>
</tr>
</tbody>
</table>

Table 1.2. The effect of erbium concentration on the fluorescent lifetime decay and amplifier quantum efficiency.
Co-operative up-conversion can be explained as follows.\cite{14} As the $^4I_{13/2}$ laser level becomes heavily populated non-radiative energy transfer takes place between neighbouring excited erbium atoms. The accepting ion is promoted to the $^4I_{9/2}$ level followed by an immediate decay to either the $^4I_{13/2}$ level, producing a green fluorescence, or the ground state and the donor ion returns to the ground state. The process is shown schematically in figure 1.4. The erbium atoms involved in the cross relaxation must be close enough for the interaction to take place, and there must be a high probability for the neighbouring atoms to be simultaneously excited to the $^4I_{13/2}$ level. Consequently, up-conversion is only observed in highly doped, strongly pumped structures.\cite{11}

1.3.2 Amplified Spontaneous Emission

If an excited erbium atom returns from the metastable state to the ground state via the spontaneous emission of a photon and, providing the photon is within the numerical aperture of the fibre, it will be guided and amplified. This leads to the concept of amplified spontaneous emission (ASE) which results in a backward and forward propagating signal that is independent of the original input signal. ASE is responsible for the noise level of the EDFA and is wasteful of pump energy.\cite{15} ASE also reduces the efficiency of the optical amplifier.

1.3.3 Excited State Absorption

When an incident photon interacts with an erbium atom in the ground state it can promote an electron to a higher level, this is called ground state absorption (GSA). In the same way an electron in the long lived $^{15}I_{13/2}$ metastable level can be further exited to higher energy levels, this is called excited state absorption (ESA).\cite{16}

Initially it appears that the 800 nm pump band is ideal for amplifier operation as high power semiconductor lasers are available at this wavelength. Unfortunately, the energy difference between the metastable level and the $^4I_{9/2}$ level corresponds to 800
nm. This leads to ESA promoting an electron in the metastable level to a higher energy level followed by an immediate decay to the ground state, limiting the device efficiency.\textsuperscript{[17]} Although ESA restricts EDFA performance when pumped in the region of 800 nm, this pump band has still been a subject of interest due to the possibility of laser diode pumping (e.g. 820 nm GaAlAs laser diodes).

A more suitable choice of pump wavelength is the 980 nm absorption band where the ESA cross section for absorption is relatively low. ESA at 980 nm promotes the electron from the $^4I_{11/2}$ level to the $^4F_{7/2}$ level, followed by a fast 550 nm radiative transition to the ground state. In this case the ESA is strongly dependant on the $^4I_{11/2}$ lifetime, which is 1 μs, and is only visible in strongly pumped amplifiers. However, as the ESA cross section is relatively low this minimises any detrimental effects.\textsuperscript{[18]}

The typical EDFA transmission loss between 1.0 μm and 1.3 μm is approximately 0.1 dB/m. However, the possibility that this loss is dependant on pump power was investigated by Farries.\textsuperscript{[17]} When an amplifier is populated to the lower laser level a new set of absorption bands is generated. The $^4I_{13/2}$ to $^4F_{9/2}$ ESA transition results in a broad band absorption centred at 1.15 μm, which may affect 1.3 μm signal propagation in the device. Farries measured the ESA absorption in two ways; by subtracting the energy of the metastable level from all other energy levels, and by pumping the EDFA with an argon laser whilst simultaneously monitoring the fibre transition spectrum of a white light signal. The two ESA spectra were found to compare favourably. Three main ESA peaks were detected; the first at 800 nm giving rise to the low 800 nm pump efficiency; the second at 1.15 μm extending to 1.25 μm and the third between 1.5 μm and 1.75 μm which may have a small affect on amplifier performance at 1.55 μm. The ESA strength at 1.2 μm was found to be unlikely to effect 1.3 μm signal propagation.

In fluorophosphate (FP) glasses, ESA is considerably smaller than GSA over the wavelength range 800 nm to 827 nm. Computer models have been developed to simulate the performance of FP EDFA’s with a fibre core diameter of 3.2 μm and
doping concentrations of 100 ppm. The results predicted optimum fibre lengths of 15 m and 22 m for pump wavelengths of 801 nm and 815 nm respectively, with a 4 dB stronger signal gain when compared to a phosphate/silica glass EDFA of similar characteristics.\textsuperscript{19}

1.3.4 Optimisation of Pump Conditions

Co-operative up-conversion, ESA and ASE all affect the properties of the erbium optical amplifier on an atomic level. By minimising these effects and by choosing the most suitable glass composition, the optical properties of the erbium atoms are optimised, although, the design of the amplifier on a larger scale is just as important.

When the signal and pump beams propagate through an optical amplifier, the intensity of the pump beam is gradually reduced by erbium optical absorption and background scattering. After a specific propagation length which is dependant on the optical waveguide characteristics, the pump light will be unable to sustain population inversion. This is represented schematically in figure 1.5. For longer fibre lengths there is no further signal gain. Another consequence of this effect leads to a difference in amplifier performance depending on the propagation direction of the pump beam. An EDFA will operate more efficiently if the pump beam is propagating in the opposite direction to the signal beam so that the intensity of the pump and signal beams are more closely matched, i.e. population inversion, and hence gain will be highest where the signal level is the highest.\textsuperscript{11}

1.4 Erbium Doped Waveguides and Optical Fibres

Sections 1.1 to 1.3 described the behaviour of erbium in silica and the mechanisms that determine amplifier characteristics. This section briefly reviews both the fabrication and reported characteristics of erbium doped structures.
1.4.1 Waveguide Fabrication

Flame hydrolysis\(^{(20)}\) is a useful technique for fabricating planar waveguide structures as precise thicknesses of glass can be grown on a suitable substrate. In this process \(\text{SiCl}_4\) is passed through delivery lines into an oxy-hydrogen burner which is directed onto a silicon substrate to deposit an SiO\(_2\) soot. After deposition the substrate is heated to 1250 °C in an electric furnace to consolidate the deposited film. Various dopants are easily added to the glass film by feeding, for example \(\text{AlCl}_3\) or rare earth compounds into the burner and using helium as a carrier gas. Following deposition, reactive ion etching and photolithography are used to form the required waveguide structure.

EDFA manufacture was described by Ainslie.\(^{(2)}\) Unlike the halide materials used in glass manufacture, i.e. silicon chloride, the rare earth halides must be heated to several hundred degrees to achieve a suitable working vapour pressure. Figure 1.6 shows a representation of the vapour phase technique used for EDFA manufacture. A silica rod is modified upstream from the deposition by adding a dopant chamber in which the rare earth halide is deposited. During core deposition, as the \(\text{SiCl}_4\), \(\text{GeCl}_4\) and \(\text{O}_2\) are fed into the tube where the glass layer is to be deposited, a stationary burner is used to heat the dopant chamber and drive off the rare earth halide into the reaction mixture. Once deposition is complete, the rod can be drawn into a fibre. This basic method can be improved upon by replacing the dopant chamber with a rare earth impregnated silica sponge. Useful EDFA's with Er concentrations down to 1 ppm have been grown by this technique. These amplifiers demonstrate unity gain at 1550 \(\mu\)m for a 10 km fibre length using a pump power of only 15 mW at a pump wavelength of 1480 \(\mu\)m.

The liquid phase technique of erbium doping can also be used for EDFA fabrication, a schematic representation of the process is shown in figure 1.7. A cladding layer is deposited inside the silica substrate in the normal way but the core deposition takes place at a lower temperature leaving behind a porous layer. The porous layer is then
soaked in an aqueous or alcohol solution of the rare earth salt before the layer is dried and fused, leaving behind a clear glass. Germanium can be used as a co-dopant to modify the refractive index of the glass. The radial profiles of the germanium and erbium concentrations show a central dip, which is attributed to burn-off of the dopant during fabrication. This problem is not seen when aluminium is used as a dopant. This technique is useful to achieve a close match between the erbium profile and the fundamental optical mode profile of the pump beam.

### 1.4.2 Properties of Erbium Doped Planar Waveguides

Phosphate glass containing 0.25 wt. % erbium was manufactured and polished to produce 1.5 mm thick glass plates.\[^{21}\] Stripe waveguides with widths between 7 μm and 30 μm were fabricated in the glass using a silver ion exchange process. The waveguides were 8 mm long. The 7 μm waveguide was single mode at 1.528 μm and the white light absorption spectrum of the waveguide showed an attenuation of 20 dB/cm at 1.535 μm which was attributed to the presence of erbium.

Kitagawa et al.\[^{22}\] used the flame hydrolysis process to fabricate a waveguide laser in erbium doped phosphorus/silica glass, the dimensions of which were 25 μm wide, 6 μm high and 45 mm long. The erbium doping concentration was 8000 ppm. The measured optical loss spectrum of the waveguide showed absorption peaks of 0.25 dB/cm at 0.98 μm and 0.72 dB/cm at 1.54 μm. The background scattering loss of the guide was less than 0.1 dB/cm. The waveguide was excited using a Ti:sapphire laser (λ=980 nm) to reveal fluorescence peaks at 1.535 μm and 1.552 μm. Oscillation of the laser was found to occur at wavelengths of 1.598 μm and 1.604 μm, and the wavelengths were independent of pump power. The threshold pump power of the laser was 49 mW with a 0.81 % slope efficiency (the slope efficiency is defined as the derivative of the laser output power as a function of the laser pump power). The quantum efficiency was calculated to be 5.9 % (the ratio of absorbed photons to emitted photons).
A potassium ion-exchange planar waveguide laser operating at a wavelength of 1540 nm was fabricated in 0.5 wt % erbium doped glass. The glass was formed by melting small pieces of glass with \( \text{Er}_2\text{O}_3 \) and then leaving the glass to cool for several hours. The glass was sliced into samples with dimensions of approximately 50x20x1 mm and then polished to produce samples with suitable input and output edges. An aluminium layer was deposited onto the surface and channels between 1.5 µm and 7.5 µm wide were opened in the aluminium using standard photolithographic techniques. The glass was then immersed into molten KNO\(_3\) for eleven hours to form stripe waveguides. The waveguide ends were then polished and dielectric mirrors deposited onto them. The mirrors were 90% reflective at 1540 nm and 90% transmissive at the pump wavelength. The optical absorption spectrum of the glass was found to have a value of 0.2 dB/cm at 980 nm and 1.4 dB/cm at 1540 nm with a background waveguide scattering loss of 0.5 dB/cm. When the devices were pumped at 980 nm using a Ti:sapphire laser, the fluorescence intensity was found to peak at 1538 nm and 1544 nm. Laser threshold pump power was found to be between 150 mW and 300 mW with lower thresholds for small waveguide widths. No laser emission was obtained from waveguide widths below 3.5 µm, which was attributed to imperfections in the manufacturing process. For the 7.3 µm waveguide with a 150 mW threshold pump power, the slope efficiency was found to be 0.55% with a maximum output power of 0.40 mW for a pump power of 270 mW. The laser emission spectrum showed a peak at 1538 nm and a second peak at 1544 nm.

Shmulovich et al. fabricated a waveguide optical amplifier using RF sputtering to create 1.5 µm high sodium-calcium silica waveguides. Spectral absorption measurements showed propagation losses of 8.5 dB/cm at 1.53 µm with a background optical propagation loss of 1 dB/cm. The fluorescence lifetime of the erbium was 9 ms. By pumping the waveguide with 120 mW at 975 nm a gain of 21 dB was obtained for a 1.53 µm signal.
1.5 Erbium Implanted Planar Waveguides

The previous section reported the properties of erbium doped waveguides manufactured by various groups using a wide range of techniques. Rather than a general review of erbium doped silica, this section is specifically concerned with the properties of erbium implanted silica. Although ion implantation is a useful method for the controlled introduction of atoms into specific locations within a sample, careful post implantation sample processing is required. The post implantation annealing that is required to remove the implantation induced damage is of particular importance, and the benefits of which are described below.

Poleman et al studied 10 μm high amorphous SiO$_2$ films which were implanted with 3.5 MeV Er ions with a dose of 5x10$^{15}$ ions/cm$^2$. Rutherford backscattering showed that the implanted Er profile had a peak depth of 1.25 μm and a full width at half maximum of 0.56 μm. The peak erbium concentration was 0.11 at. %. Post implantation annealing in vacuum had no measurable effect on the erbium distribution. An argon laser operating at 488 nm was used as the fluorescence pump source and the luminescence was detected using a monochromator and a liquid nitrogen cooled germanium photo-detector. At a temperature of 4.2 K the photoluminescence (PL) spectra of the as-implanted sample showed a broad band luminescence, which correlated with a peak in optical absorption centred at a wavelength of 1.537 μm. The broad band luminescence, which is attributed to implantation induced defects, was removed by annealing at 1000 °C for 1 hour and was not present in room temperature PL spectra. Annealing at 800 °C increased the room temperature PL intensity, in comparison to the as implanted sample, by a factor of ~ 2. At higher anneal temperatures the PL intensity decreased. Transmission electron microscopy (TEM) of the high temperature annealed samples revealed precipitates of diameters between 10 μm and 20 μm.

The fluorescence lifetimes of Er implanted into phosphosilicate glass (formed by low pressure chemical vapour deposition), amorphous silica (formed by high pressure
steam oxidation HIPOX) and two types of fused SiO₂ (Suprasil 1: 100 ppm OH and 100 ppm Cl and Suprasil W1: 0.4 ppm OH and 200 ppm Cl) were measured by Lidgard et al. After implantation the samples were annealed in a furnace at pressures below 10⁻⁷ mbar over a range of temperatures. The fluorescence excitation source was the 514 nm line of an argon laser.

Characterization of the as-implanted HIPOX samples (the implantation dose was 5.0x10¹⁵ ions/cm²) showed a fluorescent decay time of ~ 7 ms, which rose to 12.5 ms after annealing at 700 °C, while increasing the anneal temperature further had no effect. Room temperature implantation followed by annealing at 500 °C and implantation into samples heated to 500 °C with no further annealing produced similar results. Lifetime measurements of the erbium doped phosphosilicate glass implanted with a dose of 3.4x10¹⁵ ions/cm² showed an as-implanted lifetime of 9.8 ms. This remained the same after annealing for 1 hour at 500 °C.

<table>
<thead>
<tr>
<th>Glass Type</th>
<th>Fluence (ions/cm²)</th>
<th>Implantation temp (°C)</th>
<th>Anneal temp (°C)</th>
<th>Lifetime (ms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Suprasil 1</td>
<td>3.4x10¹⁵</td>
<td>20</td>
<td>No Anneal</td>
<td>4.2</td>
</tr>
<tr>
<td>Suprasil 1</td>
<td>3.4x10¹⁵</td>
<td>20</td>
<td>900</td>
<td>9.5</td>
</tr>
<tr>
<td>Suprasil 1</td>
<td>3.4x10¹⁵</td>
<td>500</td>
<td>No Anneal</td>
<td>5.2</td>
</tr>
<tr>
<td>Suprasil 1</td>
<td>3.4x10¹⁶</td>
<td>20</td>
<td>No Anneal</td>
<td>1.6</td>
</tr>
<tr>
<td>Suprasil 1</td>
<td>3.4x10¹⁶</td>
<td>20</td>
<td>900</td>
<td>6.7</td>
</tr>
<tr>
<td>Suprasil W1</td>
<td>3.4x10¹⁵</td>
<td>20</td>
<td>No Anneal</td>
<td>5.2</td>
</tr>
<tr>
<td>Suprasil W1</td>
<td>3.4x10¹⁵</td>
<td>500</td>
<td>No Anneal</td>
<td>9</td>
</tr>
</tbody>
</table>

Table 1.3. Fluorescence Decay lifetimes of Implanted Silica. The composition of Suprasil 1 is 100 ppm OH and 100 ppm Cl. Suprasil W1 is 0.4 ppm OH and 200 ppm Cl.
The measured fluorescence lifetimes as a function of anneal temperature, ion dose and glass composition for the suprasil glasses are shown in Table 1.3. Post implantation annealing at 900 °C and implantation at 500 °C increased the fluorescence lifetime. The samples with lower OH concentrations had longer lifetimes, possibly because of coupling between the rare earth states and overtones of the OH⁻ vibrations.

Amorphous SiO₂ (suprasil 1) was implanted with 2.9 MeV Er ions with either 3.4x10¹⁵ ions/cm² or 3.4x10¹⁶ ions/cm² and annealed at 900 °C for 1 hour. The high dose sample showed an approximate 10 fold increase in photoluminescence intensity and had a broader photoluminescence peak when compared to the low dose sample. The photoluminescence spectrum of the as-implanted samples showed a similar structure but much reduced intensity in comparison to the annealed samples; 4 times lower peak intensity for the low fluence sample and 7 times lower for the high fluence sample. UV absorption was used to quantify the beam induced defects. The highest dose sample showed the greatest UV absorption. Annealing of the low dose sample restored the UV absorption to the pre-implantation value, indicating removal of the implantation induced defects.

1.6 Summary

The rare earth doping concentration is one of the most significant factors in determining the characteristics and performance of the erbium doped optical waveguide amplifier. The white light spectral scan of a typical erbium doped waveguide reveals the characteristic erbium atom absorption peaks, the strongest of which are situated at wavelengths of 980 nm, 1480 nm, and 1550 nm. Increasing the erbium concentration increases the magnitude of these absorption peaks. However, as the erbium concentration rises a corresponding fall in the 1550 nm fluorescent decay lifetime is observed. This effect is caused by the onset of clustering and up-conversion, resulting in a drop in the measured erbium fluorescent decay lifetime.
To achieve optimum erbium amplifier efficiency it is desirable that the erbium fluorescent decay lifetime is maximized to allow a lower optical pump power to be used to maintain population inversion. Achieving a gain greater than unity requires the erbium related signal amplification to exceed the waveguide propagation loss. The signal propagation loss of modern single mode optical fibres can be as low as 0.1 dB/km, so that for amplification in such a fibre the gain need only exceed 0.1 dB/km. The amplification that can be achieved is proportional to the erbium content of the fibre so that only low erbium concentrations are therefore required to realize amplification in these optical fibres. Gains in excess of 45 dB have been reported using device lengths of 100 m and erbium concentrations as low as 100 ppm. In these devices the erbium fluorescence lifetime does not present an efficiency problem because the erbium concentration is low enough for up-conversion and clustering not to play a significant role in determining the fluorescent decay lifetime.

In comparison to optical fibre amplifiers, the length of stripe waveguide optical amplifiers are considerably shorter. A limit is placed on the physical dimensions of the device by the size of the substrate it is grown upon and by the high waveguide aspect ratios required. Partial solutions to this problem involve the use of feedback mirrors and coiling the waveguide to achieve device lengths in the region of tens of cms, although the device lengths still remain significantly shorter than lengths attainable with optical fibre. Typically, a 7 µm single mode phosphorus doped silica waveguide, produced by the flame hydrolysis technique shows a 1550 nm signal propagation loss of ~0.5 dB/cm.

Both the short path length and high optical propagation loss creates a significant problem in the production of stripe waveguide amplifiers. To overcome this problem erbium doping concentrations of up to several wt. % are required to achieve gain. At these high erbium concentrations up-conversion and clustering can significantly effect the fluorescence decay lifetime thus reducing the device performance. It is clear that when designing an erbium doped stripe waveguide amplifier a trade off between the erbium fluorescent lifetime and erbium related optical absorption is required. The
erbia related optical absorption must be larger than the waveguide optical propagation loss to achieve amplification, but the erbium concentration must be kept low to maximize the erbium fluorescent decay lifetime. Typically, reported waveguide devices have required erbium concentrations in excess of 8000 ppm.[22]

Erbium doping of waveguides is achieved using a variety of techniques, including solution doping and flame hydrolysis, which result in a continuous distribution of dopant erbium ions across the waveguide core. In the case of ion implantation, a single energy erbium implant results in a dopant depth profile approximating to a Gaussian distribution. A more uniform dopant distribution can be achieved by implanting at several different beam energies into the same sample since the implantation depth is proportional to beam energy. During implantation the structure of the implanted material is damaged due to the creation of defects by the action of the implantation beam. These defects significantly affect the properties of the waveguide. The interaction between erbium atoms and implantation induced damage may also affect the erbium optical properties. Thermal annealing can however be used to remove the implantation induced damage and restore the waveguide loss to values close to that of the pre-implantation value, and to enhance the erbium fluorescent lifetime.[33][37]
Figure 1.1. The basic configuration of an erbium doped fibre amplifier.

Figure 1.2. Main erbium transition wavelengths. From [5].
Figure 1.3. Typical erbium optical absorption spectra. The peak height is dependant on the erbium concentration. From [2].
Figure 1.4. Co-operative up-conversion. The numbers 1 to 3 represent the three stages of the up-conversion process. From [1].

Figure 1.5. A representation of the intensity of the pump and signal beams as they enter and leave the EDFA. The diminishing volume in the centre of the waveguide represents the region of population inversion.
Figure 1.6. EDFA fabrication. From [2].

Chapter 1 : Page 33
Figure 1.7. Solution Doping. (a) Deposition of the porous core layer. (b) Impregnation of the porous layer by the rare earth salt solution. (c) Drying the porous layer. (d) Fusing the porous layer and collapsing the tube. From [2].


7. B. Ainslie, S. Craig, "The Fabrication, Assessment and Optical Properties of High-Concentration Nd$^{3+}$ and Er$^{3+}$ Doped Silica-Based Fibres", Materials Letters, Vol. 6, No. 6, 1988, 139-143.


9. Y. Miyajima, "Progress Towards a Practical 1.3 μm Optical Fibre Amplifier",


17. M. Farries, "Excited-State Absorption and Gain in Erbium-Fibre Amplifiers"


1990, 2859-2861.


Chapter 2

A Review of Photosensitivity in Germania Doped Silica Glass

2.0 Introduction

The phenomena of diffraction presents an elegant solution to many problems faced in the design of integrated optical devices and telecommunications networks. Diffraction has been used to realise devices based on the Bragg grating, some of the possible applications of which are described below.

If a permanent periodic refractive index modulation (i.e. a Bragg diffraction grating) can be written into a waveguide or optical fibre then the wavelength corresponding to the grating period will be reflected and all other wavelengths transmitted.\(^1\) Such a device has many uses, the most obvious of which is the extraction of only one signal wavelength from an optical fibre carrying many signals (wavelength division demultiplexing).\(^2\) Other examples of the use of a Bragg grating include the ability to test the integrity of an optical communications network, i.e. if a test wavelength (632 nm) is launched into a 1550 nm communications system it can be reflected back to the source by a Bragg grating to verify the system, whilst communication traffic at 1550 nm remains unaffected. The Bragg grating can also function as a high quality mirror, a useful device in the design of a laser system.\(^3\) As a final example the Bragg grating can be used to detect environmental changes.\(^4\) Any environmental changes, for example thermal expansion or stress will change the period of the Bragg grating, and hence this will change the optical transmission spectrum of the grating.

This review summarizes the methods by which a refractive index change can be readily induced in germania doped glasses by the absorption of laser light which is the
process known as photosensitivity. The review also describes how the phenomenon can be used to produce diffraction gratings within an optically guiding structure. Section 2.1 describes the mechanism by which a refractive index change is induced. Section 2.2 describes the effect of ion implantation on refractive index, and finally, before a summary of this chapter, section 2.3 reviews the fabrication and properties of various reported Bragg gratings.

2.1 The Mechanism of Photosensitivity

The exact mechanism of photosensitivity is not absolutely clear. When the ultraviolet spectrum of a germania doped silica fibre is examined a strong absorption peak is found at 240 nm which has been linked to both the Ge-Ge bond and the Ge-Si bond. After exposure to light of wavelengths shorter than about 500 nm, a change in 240 nm absorption is observed. This change can be either positive or negative depending on the strength of the illumination. The Kramers-Kronig relationship shows that a change in ultraviolet absorption at short wavelengths results in a change in refractive index at visible and infra-red wavelengths. It has been speculated that in the original photosensitivity experiments two photon absorption of 515 nm light into the 240 nm band resulted in the bleaching of Ge-Ge and Ge-Si bonds. This formed Ge-E' defect centres (trapped holes at oxygen vacancies), and hence the refractive index of the glass at higher wavelengths was modified.

If two photon absorption at 515 nm was responsible for the induced index change then the exposure time could be reduced using higher energy ultraviolet photons at wavelengths around 240 nm (i.e. from a KrF or frequency doubled argon ion laser). Schwartz et al exposed a germania doped silica fibre to 254 nm light and simultaneously monitored the ultraviolet absorption and the concentration of Ge-E' centres using electron paramagnetic resonance (EPR). An ultraviolet lamp with a peak output wavelength of 254 nm, which had an intensity at the sample which was estimated to be 2.6 mJ/cm², was used for the exposure. The EPR and ultraviolet spectrum of the sample were measured before and after illumination. Results
demonstrated that the Ge-E' concentration increased linearly with exposure time with a corresponding growth in ultraviolet absorption. However, the results could not prove whether the Ge-E' centres were directly responsible for the ultraviolet absorption or if they were a secondary effect of irradiation. A similar relationship was also demonstrated by Simmons et al.\cite{6}

Later research showed a discrepancy in the optical exposure intensity and total optical dose required to produce equal magnitudes of refractive index change.\cite{5} In general, optical powers of the order $10^3 \text{ J/cm}^2$ produced a refractive index change of about $10^{-5}$, although Malo et al reported a similar induced index change using illumination intensities of only $0.24 \text{ J/cm}^2$ using a KrF excimer laser.\cite{9}

Tsai et al\cite{5} investigated this discrepancy by illuminating samples with both a high pressure xenon arc lamp and a KrF excimer laser ($\lambda=256 \text{ nm}$). The induced defects were studied using electron spin resonance (ESR). The concentration of the Ge-E' centres was found to decrease with KrF laser beam exposure until they were all removed using an optical dose of greater than $0.25 \text{ J/cm}^2$. A second series of samples were exposed to the xenon arc lamp ($10^{-7} \text{ W/cm}^2$). After an exposure of 20 hours, the Ge-E' concentration had increased by a factor of eight. The probability of two photon absorption during Xe-arc irradiation is very small due to the low optical intensity, although in the case of high intensity KrF illumination two photon absorption becomes a significant factor. It was suggested that single photon absorption leads to photogeneration of the Ge-E' defects and two photon absorption leads to photobleaching of the Ge-E' defects present in the sample.

These results explain the differences in the light intensity required for the formation of gratings reported by various groups. Equal magnitudes of refractive index change can be produced by either the photobleaching or photogeneration of Ge-E' defects. Photobleaching occurs via two photon absorption which requires high intensity ultraviolet illumination but with only a low total dose of ultraviolet light. Photogeneration of Ge-E' defects requires high intensity 514 nm argon laser light or
high dose but low intensity ultraviolet light (one photon absorption). Figures 2.1[5] and 2.2[5] show the Ge-E' defect concentration as a function of exposure time to a Xe arc lamp and KrF excimer laser respectively.

Maxwell demonstrated that the photosensitivity of germania doped silica could be increased by the thermal indiffusion of hydrogen.[10] Photosensitivity relies on the presence of Ge-Ge or Ge-Si bonds (giving the 240 nm absorption) which can be photobleached to produce Ge-E' defects, i.e. the removal of the 240 nm absorption band and the formation of the 213 nm band. By the reducing action of hydrogen on germanium oxide, the concentration of the Ge-Ge and Ge-Si bonds is increased, and the material photosensitivity is enhanced.

An alternative mechanism for grating formation was suggested by Payne et al.[11] Photoinduced defects can be photoexcited by a large range of wavelengths, to release electrons. An electron excited into the conduction band will, after a given time be trapped by another defect. If an optical fibre contains a non-uniform E field due to the two interfering counter-propagating laser beams, then free electrons will migrate in the direction of the field. This build up of electrons will result in a sufficiently large E field, of the order $10^5$ V/m, to produce a refractive index modulation (Pockels effect). As Bragg reflection from the grating increases with the magnitude of the refractive index difference, the electric field will grow, which will intensify the grating strength further until saturation is reached.

2.2 Implantation

The use of hydrogenation to significantly increase the photosensitivity of germania doped silica glass shows great potential for the formation of Bragg gratings in silica waveguides.[10] Unfortunately, during hydrogenation the entire area of the sample will be affected by the growth of the intense 240 nm absorption band. The resultant refractive index change could be detrimental to other devices fabricated on the same substrate. A possible solution to this problem is hydrogen implantation. Using this
technique only specific areas of the sample are modified, i.e. made photosensitive.

Presby et al\cite{12} described the effects of hydrogen implantation into silica glass. Several methods exist in order to determine the implantation induced refractive index change. For example, a waveguide will be formed during implantation and measurement of the mode angle using prism coupling allows the refractive index to be determined. However, this method suffers from problems related to surface preparation and the necessity to calibrate the system. Presby used the approach of interference microscopy, whereby the sample was illuminated by two coherent optical beams resulting in the formation of interference fringes. The number of interference fringes was then used to calculate the refractive index of the implanted sample. A fibre was implanted with 1.8 MeV protons over a range of doses from $2.3 \times 10^{16}$ ions/cm$^2$ to $2.03 \times 10^{17}$ ions/cm$^2$. Under both conventional white light and double beam interference microscopy a refractive index change at the projected ion range was clearly visible. Figure 2.3\cite{12} shows the refractive index change as a function of depth.

It can be seen from figure 2.3 that the higher dose implant shows a minimum in refractive index at the top of its peak, which is thought to be due to the build up of internal stress. The refractive index change is centred at a depth of 38 \textmu m, which corresponds to the expected proton implantation depth at an energy of 1.8 MeV. A broadening of the peak is noticeable at higher doses as the index change begins to saturate. The refractive index change due to the electronic energy loss is visible from the sample surface to the nuclear stopping region as a steady increase in refractive index. The implantation induced index change was found to saturate at a dose of $10^{17}$ ions/cm$^2$ with a corresponding refractive index change of $5 \times 10^4$. The energy deposited per \textmu m in the electronic stopping region was about 40 times greater than that of the nuclear stopping region. This showed a factor of 200 in the relative efficiency of the nuclear to electron energy loss which is transformed into a refractive index change. Measurement of the annealing characteristics of the implanted glass showed that the implantation induced refractive index change in the nuclear stopping region was stable at annealing temperatures below 500 °C. However, annealing at
1000°C reduced the implantation induced refractive index in the nuclear stopping region by over 80%. For the electronic stopping region the implantation induced refractive index change was stable at temperatures below 750 °C. Annealing at 1000 °C reduced the implantation induced refractive index change in the electronic stopping region by only 22%. Presby speculated that the annealing observations suggest that the damage mechanism in the electronic stopping region differs from that in the nuclear stopping region.

Germanium was implanted directly into silica glass by Albert et al. using ion energies of 3 MeV and 5 MeV and doses in the range $10^{13}$ ions/cm$^2$ to $5 \times 10^{14}$ ions/cm$^2$ whilst maintaining the sample stage at room temperature. The projected ion range was calculated to be 2.1 μm at 3 MeV and 4 μm at 5 MeV. After implantation the samples were bleached using a KrF excimer laser operating at $\lambda=249$ nm with a pulse duration of 12 ns and an energy of 100 mJ per pulse. Spectral measurements showed a clear absorption peak centred at 240 nm which was removed by exposure to the KrF laser, as shown in figure 2.4. This demonstrated that pure silica could be made photosensitive without the requirement of germanium doping during sample fabrication.

During Ge implantation a waveguide was formed, and the induced refractive index change was calculated using the mode angle found by prism coupling. A refractive index change of $4 \times 10^{-3}$ was measured at 632 nm. The induced refractive index change was completely removed by annealing at 1200°C for 20 minutes. This suggests that the implantation induced defects formed by Ge implantation are removed by annealing.

2.3 Grating Formation

Early attempts at grating fabrication involved coating an optical fibre with photoresist, exposing it to a mask of the desired pattern, and then using reactive ion etching to permanently form the grating. Unfortunately, while the technique produced good quality gratings of reflectivity 92% and 1.8 nm bandwidth, the process resulted in a...
high waveguide loss.\textsuperscript{14}

The major breakthrough in grating formation occurred in 1978 when Hill et al\textsuperscript{6} launched an argon ion laser beam into a germania doped silica optical fibre to form a diffraction grating. The experimental procedure for the formation of a "Hill" grating was described in detail by Lappierre et al.\textsuperscript{15} Argon laser light ($\lambda=514$ nm) at optical powers up to 776 mW was launched into a cleaved germania doped silica optical fibre. Reflected light from the output end of the fibre, i.e. Fresnel reflection, propagated back down the fibre interfering with the forward propagating light. A refractive index modulation formed in the optical fibre of period corresponding to the interference pattern which defined the grating period.

It was observed that grating formation started immediately the laser was switched on. If too high a beam power was used to write the grating, thermal expansion resulted in a continually changing Bragg wavelength until thermal equilibrium was reached. The technique resulted in a typical grating of reflectivity 44 % when exposed to 67 mW for 1 minute. Figures 2.5\textsuperscript{15} and 2.6\textsuperscript{15} show the reflectivity of the grating as a function of exposure time for laser powers of 67 mW and 5 mW respectively.

Hill et al\textsuperscript{6} was the first to show that certain glasses were photosensitive, i.e. the refractive index of a glass was permanently changed by exposure to light of specific wavelength. However, many problems were still to be overcome to achieve the formation of useful Bragg gratings, the most significant of which was to develop a method whereby gratings of any desired wavelength could be produced. The photon energy required for grating production exhibits a threshold around the green region of the spectrum; wavelengths less than $\sim 500$ nm were required. The period of the gratings fabricated by Hill depended only on the wavelength of the writing laser beam, hence the "short wave" gratings that could be fabricated remained useless for telecommunications systems operating at 1300 nm and 1550 nm.

Variable period gratings can be fabricated by the interference of two ultraviolet laser
beams incident on the surface of the waveguide on which the grating is to be written, this process was described by Kashyap et al. A frequency doubled argon laser beam (257.3 nm at 100 mW) was expanded in one dimension, passed through a beam splitter and then recombined in a fused silica prism such that an optical interference pattern formed on the surface of the prism. An optical fibre, in which the grating was to be written, was then placed in contact with the prism to expose it to the interference pattern. The period of the interference pattern could be controlled by varying the intersection angle between the two laser beams. The grating strength was measured by splicing the fibre containing the waveguide to a 50:50 coupler. The first input to the coupler was connected to an LED operating at the peak transmission of the grating. Light reflected from the grating could then be monitored via the second "input" to the coupler. A grating of reflectivity of 0.5 % was fabricated by the above method and then spliced onto an erbium doped fibre amplifier. When energized the laser was found to oscillate at the Bragg wavelength of the grating.

The magnitude of the optically induced refractive index changes were measured by Hand et al. Light from a HeNe laser was launched into the first input of a 50:50 coupler. The two outputs of the coupler were spliced to fibres with highly polished ends in order to reflect as much light as possible. The reflected light recombined in the coupler and was monitored at the second input. The intensity of monitored light was a function of the phase difference between the two beams recombining within the coupler. When a refractive index change occurred in one of the fibres, the change in phase difference between the recombining beams altered the optical output intensity. By monitoring the output light intensity as the refractive index change was occurring, the magnitude of the index change could be calculated. The variation of the induced refractive index change as a function of wavelength for a germania doped silica fibre exposed to 260 nm light with an intensity of 250 mW/cm² is shown in figure 2.7.

Meltz et al. estimated the photo induced refractive index change to be of the order $10^{-5}$. The 244 nm light from a frequency doubled argon laser was split into two beams which were recombined in the core of a germania doped silica fibre. A filtered
mercury arc lamp and monochromator were used to characterize the induced grating which was found to have a reflectivity of 50\% and a bandwidth of 42 \text{ GHz}. The magnitude of the photo induced index change was calculated from a knowledge of the length, the period and the reflectivity of the grating.

The magnitude of the induced refractive index change has been significantly increased by Maxwell et al\cite{Maxwell} using the indiffusion of hydrogen. They used flame hydrolysis to produce Ge doped (10\% by mole) planar silica glass samples. The samples were annealed in a hydrogen atmosphere at 750\degree \text{ C} for two hours. The untreated sample showed little ultraviolet absorption while after hydrogenation significant absorption bands at 200 nm and 240 nm were observed. Figure 2.8\cite{Figure2.8} shows the spectral transmission curve of a planar Ge doped silica substrate as a function of hydrogen anneal duration.

Stripe waveguides were fabricated and subjected to the hydrogenation process described above.\cite{Stripe} Before hydrogenation the transmission spectrum of the waveguide showed a flat spectral response between 1000 nm and 1600 nm with a loss of 0.1 \text{ dB/cm} at 1550 nm. After hydrogenation the waveguide attenuation increased at the shorter wavelengths but was unchanged in the infra-red region. The waveguide was then exposed to an ultraviolet interference pattern with a period of 1536 nm using a frequency doubled argon laser operating at 25 mW. The resultant grating had a peak reflectivity of 44\% and a 1.1 nm bandwidth. The grating was found to be thermally stable for 15 hours at 150\degree \text{ C}. Figure 2.9\cite{Figure2.9} shows the transmission spectrum of the grating, which is suitable for many telecommunications applications.

2.4 Summary

After the initial discovery by Hill that high intensity laser light could be used to permanently alter the refractive index of germania doped glass, many groups have strived for the optimum technique to produce efficient fibre/waveguide Bragg gratings. The mechanism of grating formation is summarized in figure 2.10 and a brief
summary of the method used for grating manufacture is as follows. (i) The germania
doped silica glass waveguide is heated in a hydrogen atmosphere reducing Ge-O bonds
to form either Ge-Ge or Ge-Si bonds. (ii) The Ge-Ge and Ge-Si bonds show a strong
absorption at 240 nm, which modifies the refractive index of the glass, and the extent
of this refractive index modification is described by the Kramers-Kronig relationship.
(iii) On exposure to an ultraviolet interference pattern the Ge-Ge and Ge-Si bonds are
photoexcited which changes the sample ultraviolet absorption spectrum and hence the
sample refractive index.

In the original experiment described by Hill\textsuperscript{[5]} et al the Bragg grating wavelength was
dependant only on the laser wavelength used to write the grating, and the
characteristics of the optical fibre in which the grating was to be written. However,
by using the method described by Kashyap\textsuperscript{[6]} et al gratings of variable period could
be manufactured. An interference pattern was formed on the surface of the optical
fibre in which the grating was to be written. By simply varying the angle between the
two interfering ultraviolet beams, Bragg gratings of any wavelength were feasible.
Using a combination of this technique and the thermal hydrogenation of germania
doped silica, the production of high quality diffraction gratings became possible.
However, hydrogen thermal indiffusion required the entire volume of the sample to
be made photosensitive, possibly to the detriment of other devices on the same sample.
Ion implantation offers the possibility of the modification of selected areas of the
sample surface.
Figure 2.1. Ge-E' concentration as a function of exposure to a Xe arc lamp. From [5].

Figure 2.2. Ge-E' concentration as a function of exposure to a KrF laser. From [5].
Figure 2.3. Refractive index profile vs depth for the following hydrogen doses.
(a) $2.3 \times 10^{16}$, (b) $5.0 \times 10^{16}$, (c) $1.0 \times 10^{17}$ and (d) $2.0 \times 10^{17}$ ions/cm$^2$. From [12].

Figure 2.4. (a) Absorption spectrum of a fused silica sample implanted with a dose of $10^{13}$ Ge ions/cm$^2$ at 3 MeV. (b) The same sample after bleaching for 2 min at 50 pulses per second. (c) the unimplanted substrate. From [13].
Figure 2.5. Growth in reflection at an incident power of 67 mw. From [15].

Figure 2.6. Growth in reflection at incident power 5 mW. From [15].
Figure 2.7. Induced refractive index change as a function of wavelength. From [17].

Figure 2.8. The effect of anneal duration upon the UV transmission spectrum of planar Ge doped silica. From [4].
Figure 2.9. Grating transmission spectra. From [10].
Figure 2.10. The mechanism of photosensitivity.
2.5 References


Chapter 3

Experimental Methods

3.0 Introduction

This chapter discusses the experimental methods and the measurement techniques required for the study of the photosensitivity of germania doped silica and the optical properties of Er implanted ASG waveguides.

The ASG samples used here were fabricated by the British Telecom laboratories (BT Labs). The sample structure and fabrication is described in section 3.1. For both the erbium doped and photosensitive structures the implantation procedures described in section 3.2 were used. The ultraviolet spectral analysis which was used to determine the photosensitivity of hydrogen implanted germania doped silica is described in section 3.3. The measurements used to characterize the optical properties of erbium implanted waveguides are the waveguide optical propagation loss at the operational wavelength of the device (~ 1550 nm), the erbium fluorescence decay lifetime, and the spectral absorption characteristics of the Er implanted waveguide. These measurements are described in sections 3.4 to 3.6.

3.1 Sample Fabrication and Structure

The samples used for the photosensitivity work were produced using chemical vapour deposition (C.V.D)\(^1\) to deposit a 7 wt. % germania doped silica layer onto a silicon substrate. The 7 wt. % Ge concentration is the same Ge concentration used in the thermal hydrogenation experiments reviewed in chapter 2. The samples were cut into dimensions of 1 cm by 1 cm using a diamond saw. The upper layer of the samples, on which the germania doped silica was deposited, was marked in the corner with a
diamond scribe so that the germania silica layer could be identified after processing.

The ASG waveguides were prepared using flame hydrolysis\textsuperscript{[2]} to deposit 8 wt. % ASG onto a silicon substrate to form a planar waveguide structure. At a wavelength of 1500 nm the core and cladding refractive indices were 1.490 and 1.445 respectively. Following deposition, a diamond saw was used to cut channels into the ASG glass to form 25 \( \mu \)m wide waveguides. The waveguides were cut into samples 1 cm by 2 cm along the direction of optical propagation, and the ends polished. A schematic representation of the ASG waveguide is shown in figure 3.1.

### 3.2 Ion Implantation

Ion implantation is used to introduce "impurities" into a sample by the interaction of a charged particle beam with the sample. The charged particles are embedded in the sample and reach a depth which is a function of the energy of the implanted ion. The location of the buried doped layer is controlled by a combination of ion energy and sample masking. The main advantages of ion implantation are the precision of the impurity location, the accuracy of the doping level, and the low processing temperatures required. A schematic representation of the Surrey 2 MeV implanter is shown in figures 3.2 to 3.4.

To generate the high energy ion beam a low energy ion beam of the required dopant is first required. This can be achieved using a variety of techniques, the choice of which is dependant on the desired ion species. In the case of erbium implantation the beam is generated by the vaporization and ionization of the rare earth halide. Halide materials offer high vapour pressures at comparatively low temperatures. The halide compound is vaporized using an electric heater and ionized using a heated cathode and anode assembly. The ion source is situated inside a magnetic field to increase the electron path and hence the ionization probability. As hydrogen is gaseous at room temperature this greatly simplifies the extraction of protons in comparison to the
generation of erbium ions. Hydrogen ions are readily extracted from an electrically sustained hydrogen plasma.

The charged particles from the ion sources (either erbium or hydrogen) are extracted through an aperture using a negative voltage (-20 kV) and passed into the main acceleration tube through a Wen filter. The Wen filter uses crossed electric and magnetic fields so that only particles of a narrow mass and energy band will be able to pass through the exit slits at the end of the filter and into the acceleration tube.

Figure 3.3 shows the acceleration tube which is surrounded by a series of rings. Each ring is in turn connected to a decreasingly negative voltage which is supplied by a 2 MV Van de Graaf generator and a potential divider network. When the ion enters the acceleration tube, it is accelerated to the required energy. The ion leaves the acceleration tube through the last ring which is held at ground potential. A series of magnets are used to prevent electrons, generated by beam collisions with residual gas atoms, travelling back down the acceleration tube and creating X-rays. The whole assembly is mounted inside an SF₆ atmosphere (~ 6 bar) to prevent electrical breakdown.

Following acceleration and before arriving at the main bending magnet the beam is focused using a set of quadrupole magnets. The radius of curvature of a charged particle through a magnetic field is proportional to the particle mass and energy. By varying the magnetic field and adjusting the size of slits after the magnet, the required beam species and in some cases the beam isotope can be selected. After the slits a second quadrupole magnetic lens is used to refine the beam focus.

The beam is scanned over the sample to be implanted using X and Y electric field deflection plates. The ion dose is calculated by integrating the sample current with time. As neutral beam particles will result in dose errors the target chamber is slightly offset from the main beam line and a third set of deflection plates is used to correct for this and steer charged particles onto the target.

Chapter 3 : Page 59
The arrangement of the sample holder is shown in figure 3.4. The area of the target to be implanted is controlled using aperture plates. Metal clips are used to hold the sample onto the sample holder which is connected to earth via a current integrator. To prevent dose inaccuracies secondary electrons emitted from the sample (due to interaction with the ion beam) are deflected back towards the sample using a negative voltage applied to the suppression plates.

Operation of the implanter requires both technical knowledge and experience of the machine. By varying the current supplying the main bending magnet, adjusting the slit width, and measuring the beam current reaching the sample holder a mass spectrum of the beam composition is obtained and the desired species is then selected. In the case of 4 MeV erbium implantation, the beam species used was Er^{++}. The double charge dropped through the acceleration tube gives a doubling of beam energy (i.e. Er^{++} dropped through a voltage of 2 MV gives a beam energy of 4 MeV). Once the correct beam species is obtained, the beam scanning is adjusted until the scanned area of the beam is just larger than the sample holder aperture.

When the correct beam and scanning area have been chosen, the sample holder is isolated from the implantation beam and the sample is moved into position. The current integrator is then reset to zero and the beam switched on until the desired dose is obtained. In the case of implantation into heated samples the sample holder is situated above a graphite strip heater capable of reaching temperatures greater than 1000 °C.

### 3.2.1 Post Implantation Sample Cleaning

After implantation the samples were cleaned to remove deposits of vacuum pump oil. This was done by soaking them in a series of organic solvents (each for five minutes) in the following order: trichloroethane, acetone and methanol. Finally the samples were rinsed in deionized water and inspected using an optical microscope to ensure that the surfaces were free from particulate contamination. The samples were also
cleaned prior to implantation using this same procedure.

3.3 Ultraviolet Absorption

As described in chapter 2 the ultraviolet absorption spectrum of germania doped silica glass indicates the extent of the material photosensitivity. Figure 3.5 shows a simplified diagram of the diode array spectrometer used to measure the ultraviolet transmission spectrum of the germania doped silica samples. The spectrometer operates by passing white light, obtained from a deuterium arc lamp, through the sample. A diffraction grating is then used to split the output beam into a spectrum which is measured using an array of diodes. The resolution of the spectrometer is dependant on the diode spacing. In this case the spectrometer had a resolution of 2 nm over the wavelength range 190 nm to 900 nm.

The spectrometer was designed to study chemical reactions by measuring the white light transmission spectra of test tubes held within the device. To measure the transmission of the silica samples a sample holder was fabricated to replace the test tubes that would normally hold the sample to be measured. It was found that slight changes in the angle of incidence between the input beam and the sample surface had a large effect on the spectral data obtained. To ensure reliable results the transmission spectrum of each sample was measured several times. After each measurement the samples were removed from the sample holder and then replaced before repeating the measurement.

Meticulous cleaning of the samples, using the procedure described in section 3.2.1 above, was found to be necessary since small deposits of grease had large effects on the sample ultraviolet absorption spectra. A comparison of spectral measurements of the unimplanted material and air, i.e. no sample in the machine, was used to check for false peaks in the absorption spectra.
As light propagates through a waveguiding structure the intensity of the propagating beam decreases with distance due to absorption and scattering of the propagating beam. This loss is known as the propagation loss and is expressed in dB/cm. The loss is dependant upon both the characteristics of the waveguide and the concentration of light scattering defects. In the case of the optical amplifier it is important that the propagation loss is less than the available amplification.

Ion implantation can significantly increase the defect concentration by the interaction of the ion beam with the sample. However, these defects can be removed by thermal annealing. To minimise the implantation induced damage it is necessary to measure the waveguide propagation loss as a function of implantation dose and annealing temperature and time. The optimum anneal conditions required to restore the waveguide loss to the pre-implantation value can then be determined. This section describes the measurement technique used to measure the waveguide propagation loss.

The waveguide propagation loss is calculated using equation (3.1) below.

\[
\frac{10 \log \left( \frac{A}{B} \right)}{L} = p
\]

where \( p \) = Propagation loss in dB/cm.

A = Optical power coupled into the waveguide input facet.
B = Optical power emitted from the waveguide output facet
L = Waveguide length between input and output facet in cm.

The determination of the propagation loss is more difficult than would first appear. It is not sufficient to assume that the power emitted from the input source to the waveguide is equivalent to the power launched into the waveguide. The mode mismatch between the input beam and the waveguide optical mode profile as well as the losses due to Fresnel reflections at the waveguide input and output must be taken
into account. An alternative measurement to the waveguide propagation loss is the insertion loss, which is defined as the sum of the propagation loss, mode mismatch loss and Fresnel reflection loss.

The simplest method of measuring the propagation loss is the "cut back method", where the waveguide loss is measured as a function of waveguide length, the waveguide being progressively shortened and remeasured. Since the losses due to Fresnel reflections and the mode mismatch are constant they can be eliminated from the results and the final propagation loss calculated. Unfortunately the cut back method is not ideal because it is destructive and it requires the new end face to be identical to the previous one.

The propagation loss of the ASG waveguides was first measured by the scanning probe technique, which is described as follows. A 50 μm diameter optical fibre cable was mounted at 90° to the waveguide surface and it was scanned above the excited waveguide to collect the scattered light. A photodiode was used to measure the output intensity of the optical fibre. Assuming the scattered light intensity is proportional to the power propagating through the waveguide (i.e. the distribution of the scattering centres is constant), then by scanning the probe transversely across the direction of the propagating beam at distances along the beam path, the propagation loss can be determined. Unfortunately the diamond saw method of waveguide fabrication used for the ASG waveguides created a random distribution of defects on the waveguide surface. This generated a large scattered light signal which varied randomly, preventing the use of this method. However, accurate results were achieved with waveguides manufactured using the flame hydrolysis/plasma etching techniques.

The propagation loss of the ASG waveguides was measured using a fibre in/fibre out technique. One optical fibre is used to couple light into the waveguide, and a second fibre used to collect the light emitted from the waveguide output. By measuring the optical power collected by the output fibre, with and without the waveguide between the two fibres, the waveguide insertion loss can be calculated. The optical mode
mismatch between the singlemode launch fibre and the ASG waveguide was calculated and taken into account when calculating the propagation loss from the measured insertion loss (see section 4.5 for calculation of the mode mismatch). The Fresnel reflections were reduced between the waveguide and the optical fibres by using index matching oil ($n=1.49$). Index matching oil was also used to form the waveguide upper cladding layer. The fibre used to collect the waveguide output had a diameter of 50 $\mu$m, which was significantly larger than the 2 $\mu$m by 25 $\mu$m waveguide output. This eliminated optical mode mismatch problems at the waveguide output.

The experimental system used to measure the waveguide insertion loss is shown in figure 3.6. The system is based around a series of interchangeable fibre optic cables using four light sources. Two of the light sources are pigtailed semiconductor lasers operating at 1526 nm and 1300 nm, each producing an optical fibre output power in the region of 0.50 mW. The white light source, which is used to determine the waveguide spectral absorption curves is discussed in section 3.5 below. The 632.8 nm beam from a HeNe laser, which was launched into a singlemode fibre optic cable using a X 6.5 microscope objective lens, is used for system alignment.

The four optical fibres delivering the various light beams can be connected to either a singlemode or multimode fibre optical cable, which is in turn connected to the waveguide to be assessed. Finally, either a singlemode or multimode optical fibre can be used to collect light from the waveguide output and characterise it as appropriate. The waveguide and launch and collection fibres are mounted upon three axis micrometer controlled alignment stages, to allow alignment of the optical fibres and waveguide. The output fibre can be replaced by a microscope objective lens to focus an image of the waveguide output onto an infrared sensitive video camera for viewing the waveguide output.

The procedure used to measure the waveguide insertion loss is as follows. The singlemode launch fibre is connected to the visible 632.8 nm light source to align the system. A microscope objective lens is used to form an image of the waveguide
output so that the transmitted power can be maximised. To align the fibre with the waveguide, the position of the launch fibre is manipulated relative to the waveguide using the 3-axis micrometer stages until a bright spot can be seen on the projection screen. The microscope objective lens is then replaced by a multimode collection fibre which is positioned to collect the maximum intensity of light leaving the waveguide. An optical power meter monitors the output power emitted from the collection optical fibre. Once initial alignment is complete the 632.8 nm light source is replaced by either the 1526 nm or 1300 nm light sources and the positioning stages are manipulated to maximise the transmitted power. The output optical power of the waveguide can then be measured and the insertion and propagation losses calculated. The input optical power to the waveguide is measured by connecting the launch fibre directly to the power meter via the multimode collection fibre.

3.5 Waveguide White Light Transmission Spectra

Measurement of the waveguide transmission spectra is a logical extension to the measurement of the waveguide insertion loss at specific wavelengths. When the spectral transmission of an Er doped waveguide is measured, an absorption peak around 1520 nm due to the presence of optically active erbium is observed. The white light absorption spectra is therefore useful to assess the erbium content of erbium doped waveguides. The concentration of implanted erbium may not be the same as the concentration of optically active erbium. This may be due to a range of effects including clustering of erbium and implantation induced defects.

The apparatus used to measure the waveguide transmission spectra in the range 1400 nm to 1600 nm is shown in figure 3.7. White light is launched into the waveguide from a singlemode 7 µm diameter optical fibre cable which is butt coupled to the waveguide core. Index matching oil (n=1.49) is used both between the fibre and waveguide and to form the waveguide upper cladding layer. The output of the waveguide is focused onto the entrance slit of the 0.25 m monochromator using a x 6.5 microscope objective lens. Between the waveguide and monochromator a
mechanical chopper with a 30 blade wheel is used to facilitate A.C. signal detection. A 90 % transmission beam splitter is used to allow an infrared video camera to monitor the waveguide output.

An InGaAs diode with a reverse bias of 9 V is used to measure the output optical intensity from the monochromator. The detection system uses a lock-in amplifier. The mechanical chopper control provides a reference output for the lock-in amplifier and a current pre-amplifier, with a gain of $10^7$ V/A, is used to boost the InGaAs diode photodetector signal. A computer is used to control the monochromator scanning and to record the output of the lock-in amplifier.

In setting up the measurement system, chromatic aberration of the microscope objective lens was found to be a problem. As the scanned wavelength increased from 1400 nm to 1600 nm the waveguide output beam moved away from the monochromator entrance slits as the focal length of the lens changed with wavelength. To overcome this problem precise alignment of the waveguide output, the objective lens, and the monochromator was required. The beam incident on the objective lens was adjusted to be normal to the lens surface and to propagate through the centre of the lens.

To achieve optimum system alignment the mounting stages for the waveguide, launch fibre and objective lens were the first items fixed to the optical bench. Apertures with a diameter of 5 mm were attached to each stage and the positioning of the stages optimised so that a 632.8 nm laser beam could propagate through each aperture. At a distance of approximately 1.5 m away from the mounting stages the InGaAs detector was fixed to the bench so that the 632.8 nm laser beam was incident upon the detector. The detector was mounted using a positioning stage that allowed freedom of movement in a plane perpendicular to the alignment beam. Subsequently, the monochromator was adjusted to transmit a wavelength of 632.8 nm and was placed between the detector and waveguide mounting stages in such a position that the beam passed through the monochromator while still remaining incident upon the detector.
The optical fibre white light source used a 50 W halogen bulb. A stabilized power supply was constructed to ensure a constant optical output from the halogen bulb, this was checked by monitoring the variation of the light output with time. Several experiments were conducted to maximize the coupling efficiency between the halogen bulb and the 50 µm multimode collection fibre. Initially a system of parabolic mirrors and lenses were used to couple the light from the halogen bulb into the multimode fibre. However, the simpler system of butting the polished fibre up to the surface of the bulb envelope was found to produce the best results. A white light scan using a commercial spectrum analyzer showed the 50 µm fibre to have an output power of -40 dBm at wavelengths between 1400 nm and 1600 nm. Connecting the multimode fibre to a 7 µm singlemode fibre reduced the intensity to ~50 dBm. The measured power levels were identical to those of a commercial white light fibre source.

The waveguide output with the above white light source passed through a 7 µm diameter singlemode launch fibre and a 2 µm by 25 µm waveguide is of very low intensity. Once the waveguide output light is passed through a monochromator, detection of the light becomes very difficult indeed. To align the system with the waveguide and begin the spectral scan, a careful procedure was followed to ensure the maximum signal level was obtained. Initially the waveguide was excited with the 7 µm singlemode launch fibre connected to the (visible) 632.8 nm light source. The objective lens was then adjusted so that the beam passed through the monochromator, which was set to 632.8 nm with the slits fully open, and then on to the detector. Following this the 632.8 nm source was disconnected and the launch fibre connected to a 1526 nm laser diode source. The InGaAs photodetector was then connected to a pico-ammeter and the monochromator adjusted to pass a wavelength of 1526 nm. The objective lens, photodiode position and launch fibre position were then fine tuned to maximise the photodiode signal strength. The photodiode was reconnected to the lock-in amplifier system, the chopper was placed in the beam and the signal switched on. The chopper was operated at a high frequency (~2.5 kHz) to minimize noise levels.

Initially the lock-in amplifier was adjusted to a time constant of 1 second and to a
minimum gain because the signal was very intense. The phase and zero offset of the 
lock-in amplifier were adjusted until a maximum signal strength was obtained. Then 
the laser was disconnected from the launch fibre and the white light source connected. 
The sensitivity of the lock-in amplifier was increased (from 1 V to 3 mV) and the 
time constant set to 10 seconds. By following this procedure a useful signal from the 
waveguide output was obtained.

The beam splitter was then removed from the optical path and the computer used to 
perform an automated spectral scan. The computer was operated so that each point 
of the wavelength vs. intensity scan was averaged over 50 successive measurements. 
A scan of the output of the launch fibre was carried out to correct for the detector 
response curve, the light source spectral characteristics and aberrations of the image 

The waveguide relative optical absorption, in dB, as a function of wavelength (λ) was 
calculated using equation (3.2), where A(λ) and B(λ) represent the output powers of 
the excited waveguide and the launch fibre as a function of wavelength.

\[
Absorption(\lambda) = 10 \log \left( \frac{B(\lambda)}{A(\lambda)} \right)
\]  

(3.2)

The white light transmission spectra of the Er doped waveguide fabricated by BT Labs 
is shown in figure 3.9. A peak erbium absorption of ~ 10 dB at ~ 1530 nm is clearly 
visible. This agrees with the spectrum obtained by BT Labs using a commercial 
optical spectrum analyzer.

The BT Labs waveguide, from which the spectrum shown in figure 3.9 was obtained, 
is of cross section 7 μm by 7 μm. The coupling efficiency between the singlemode 
launch fibre and the BT Labs waveguide is high due to the similarity of the guiding
layer characteristics. However, for the ASG waveguides which have a 2 µm by 25 µm cross section the total amount of light leaving the waveguide is lower by a factor of at least 10. The spectral attenuation curve shown in figure 3.9 was obtained using a lock-in amplifier sensitivity of 10 mV and time constant of 1 second. A higher lock-in amplifier sensitivity and decay constant was required to repeat the experiment for the ASG waveguides.

The 1 mV lock-in amplifier sensitivity required to obtain an output from the ASG waveguide resulted in a very high signal to noise ratio (~1/4 the signal strength). This was partially compensated for by the use of a long lock-in amplifier decay constant (10 s), although the low frequency noise could not be eliminated. Thermal effects and mechanical instability placed a limit on the total time required to perform the measurement before the alignment of the ASG waveguides drifted significantly, thus preventing increased data averaging or scan time. To give a maximum signal strength the monochromator input and output slits were left fully open, limiting the resolution to 10 nm.

Obtaining the spectral scan of the ASG waveguides proved to be an extremely unreliable process. As many as 30 scans had to be repeated before 3 identical scans could be obtained. Minute differences in the launch fibre coupling conditions were required to obtain the spectra presented. As noise levels were extremely high the results give only a guide to the true characteristics of the spectral absorption curves.

3.6 Erbium Fluorescence Lifetime Decay

The efficiency of the erbium optical amplifier is partially dependant on the 1.55 µm fluorescent decay lifetime of the $^7\text{I}_{13/2}$ to $^4\text{I}_{15/2}$ transition (the laser level to ground state transition). This is because when the decay time increases, less energy is required to maintain population inversion. It is important to determine if the fluorescent decay lifetime of Er implanted ASG is suitable for amplification. Fluorescent decay lifetimes in the approximate range of 3 ms to 11 ms are suitable for use in optical amplifiers.
and have been obtained in silica structures (see chapter 1). As the fluorescent decay lifetime is concentration dependant it is also useful to investigate the decay lifetime as a function of the implanted erbium dose. This section describes the techniques used to measure the erbium fluorescent decay lifetime.

Optically excited erbium doped glass waveguides emit a 1.55 µm fluorescence and when the excitation source is removed, the intensity of the fluorescence decays exponentially to zero. The form of the decay curve may be considered to be the sum of two main exponential curves. The first is due to the fast up-conversion related decay and is of a time constant shorter than the second longer decay which represents the steady state decay as the $^1I_{13/2}$ level depopulates.

Figure 3.10 shows the apparatus used to measure the fluorescent decay curves of erbium implanted ASG. A mechanically chopped argon ion laser operating at 514 nm with a variable output power up to > 1.5 W was used as the excitation source. The waveguide was mounted on a micrometer controlled two axis positioning stage to give freedom of movement in the plane perpendicular to the pump beam direction. Microscope objective lenses (X 6.5 input and X 20 output) were used to launch light into the waveguide and to form an image of the waveguide output to aid alignment. The objective lenses were mounted on 3 axis micrometer positioning stages. A low magnification input lens was used to ensure that the pump beam was launched within the acceptance angle of the waveguide. The coupling efficiency of the 514 nm laser beam with the ASG waveguides was ~ 50 %, which was calculated by measuring the waveguide input and output optical power and taking into account the waveguide propagation loss.

A germanium photodiode was used to monitor the emitted 1.55 µm fluorescence. The distance between the detector and the waveguide was approximately 5 mm above the input end of the waveguide. The fluorescence, which was strongest above the input of the waveguide, was measured above the input of the waveguide to partially account for the varying optical propagation loss of different samples. Two silicon wafers,
placed between the waveguide and detector, were used to filter out the 514 nm pump light from the Ge detector. Slight variations in the detector to waveguide distance were possible during measurement of different samples, which prevented direct fluorescent intensity comparisons.

The detector output was connected to a current to voltage pre-amplifier, which had a gain of $10^7$ V/A, and was monitored using a digital storage oscilloscope triggered by the reference output of the mechanical chopper.

The fluorescence lifetime decay measurements were performed as follows. The waveguide was first aligned with the 10 mW, 514 nm laser beam so that the waveguide output beam could be seen on the image screen. The photodiode and silicon filter were then positioned above the waveguide to collect the 1550 nm fluorescence. With the 514 nm laser beam operating at maximum power, the precise alignment of the waveguide and launch lens were then adjusted until the strongest 1550 nm fluorescence signal intensity could be measured.

Once the alignment was complete the pump beam was switched off with the chopper still running and the zero offset of the oscilloscope corrected. The pump beam was then switched on again and the chopper speed adjusted to give a pulse width of between 30 ms and 40 ms. The oscilloscope was used to average > 200 decay curves to remove noise and uncertainty from the measurement. The output signal of the photodetector was found to be dominated by signals induced by the stray 50 Hz electrical field by as much as three times the magnitude of the fluorescence related signal. By careful adjustment of the chopper frequency away from harmonics of the 50 Hz mains frequency, unwanted stray signals were removed by the averaging feature of the oscilloscope.

Decay curves were obtained for a range of pump powers in 100 mW steps from 100 mW to the maximum available laser power. The exponential decays recorded on the storage oscilloscope were transferred to a computer for later analysis. Figure 3.11
shows both the decay and rise of the fluorescence signal of a typical Er implanted waveguide. The apparatus produced identical results to those obtained using a similar system at BT Labs.

A measurement of the laser output power was given by the laser control panel. The accuracy of this display was checked using a photodiode to measure the beam power. A slight non-linearity was observed which was corrected in all the results presented here.

Analysis of the initial 2 ms of the fluorescence decay can be used to evaluate the level of the fast decay component arising from the up-conversion process. To capture the data, it is necessary to run the two blade chopper at the highest speed so that the pump beam on/off transition time is as short as possible. A series of experiments were carried out on a test sample provided by BT Labs with the chopper running at a pulse width of 3 ms.

The exponential decay given by equation (3.3) can be manipulated to give equation (3.5), where \( I \) is the fluorescent intensity, \( t \) is time, \( 1/-b \) is the 1/e decay time, and \( a \) and \( c \) are constants. (assuming \( c \) is small)

\[
I = ae^{-bt} + c
\]

\[
\ln I = -bt + \ln a
\]

\[
\frac{d(\ln I)}{dt} = -b
\]

By plotting the differential \( \ln(I) \) as a function of time for a single exponential decay, a straight line graph is obtained from which the decay time is calculated. If the decay curve consists of more than one exponential the decay time as a function of time can be calculated, and the magnitude of the fast decay component determined. Comparison of the results provided by the apparatus described above and apparatus
The differentiation process is shown in equation (3.6), where \( n \) represents a data point and \( n+c \) a second data point which is \( c \) time steps away from \( n \).

\[
\frac{dI}{dt} = \frac{I_n - I_{n+c}}{t_n - t_{n+c}} \tag{3.6}
\]

This differentiation process greatly exaggerated the signal to noise ratio. When the data obtained from the implanted ASG waveguides was differentiated, the noise level was found to be too high for reliable analysis of the data; the luminescence from the implanted ASG waveguides is significantly weaker than the luminescence from the BT Labs test waveguide. A second method was therefore developed to calculate the fluorescence decay time and to assess the magnitude to which up-conversion was taking place. Up-conversion is dependant on both the laser pump power and the average erbium concentration. As the pump power and erbium concentration is increased the fluorescence decay lifetime decreases. Plotting the \( 1/e \) decay time as a function of laser pump power can therefore show to what extent up-conversion is taking place, although the magnitude of the fast decay time can not be easily evaluated. To calculate the \( 1/e \) decay time curve fitting software was used to fit the captured decay curves to equation (3.3).

3.7 Conclusions

The experimental methods described have been used successfully to characterise the photosensitivity and fluorescence of the glass structures. However, the difficulty in obtaining the spectral absorption curve was extreme and represents a considerable effort in setting up the measurement system.
Figure 3.1. ASG waveguide dimensions.

Figure 3.2. The 2 MeV implanter.
Figure 3.3. The acceleration tube.

Figure 3.4. A schematic representation of the target sample holder.
Figure 3.5. A schematic representation of the diode array spectrometer.

Figure 3.6. Waveguide optical loss assessment apparatus.
Figure 3.7. Measurement of waveguide white light transmission spectra.
Figure 3.8. InGaAs photodiode response curve.
Figure 3.9. The white light spectra of a 2 cm, 0.01 M Er waveguide.
Figure 3.10. Lifetime assessment apparatus.
Figure 3.11. Typical erbium fluorescent decay curve.
3.7 References.


Chapter 4

Modelling of the Implanted Ion
and Waveguide Mode Profiles

4.0 Introduction

The main purpose of this chapter is to estimate the implantation parameters that are required to match the depth distribution of implanted erbium atoms with the vertical intensity distribution of the fundamental optical modes of the ASG waveguides.

In conventionally fabricated devices (i.e. flame hydrolysis) uniform erbium concentrations as a function of depth are possible, this is however not the case for ion implantation. Measurement of the concentration profile with depth of implanted ions shows a distribution that can be approximated to a Gaussian curve. The mean distance an implanted ion travels is called the range and the statistical distribution around the range is called the straggle. The range and straggle depend on the implantation beam species, the beam energy and the composition of the target material.\(^1\)

This project uses the ASG waveguides, described in section 3.1, which have a 2 \(\mu\)m thick guiding layer. The vertical optical intensity distribution of the propagating modes in these waveguides, as with other singlemode waveguides, takes the form of a Gaussian curve. It is desirable that the implantation energy is chosen so that the depth of the implanted erbium ions corresponds to the peak in the vertical intensity of the propagating optical modes. If for example the Gaussian distribution of the implanted erbium atoms peaked at 0.1 \(\mu\)m whilst the Gaussian distribution of the pump beam optical mode profile peaked at a depth of 1 \(\mu\)m, then the interaction
would be extremely inefficient.

Section 4.1 of this chapter is concerned with the TRIM computer software which has become a standard tool used in the modelling of implantation profiles. The ion profiles calculated by TRIM were verified using a sample analysis technique called Rutherford backscattering and this is described in section 4.2. The fundamental vertical optical mode intensity profiles of the ASG waveguides were calculated and are given in section 4.3. Section 4.4 compares the ASG waveguide optical mode profiles and the implantation profiles. To measure the ASG waveguide optical propagation loss, the optical mode mismatch between the launch optical fibre and the ASG waveguide, which is calculated in section 4.5, is required. Finally a summary of this chapter is given in section 4.6.

4.1 Computer Simulations

On entering the target the implanted ion gradually loses momentum through two main energy loss mechanisms. Random collisions with the target nuclei cause both deflection of the incident particle and dislodge the target nuclei. This process is referred to as nuclear stopping. The second loss mechanism, known as electronic stopping, is the Coulombic interaction of the incident ion with the cloud of electrons surrounding the targets atoms. This leads to ionization and excitation of the target atoms. The implantation processes are modelled by the TRIM 92\(^{[2]}\) (Transport of Ions in Matter) computer software to predict the profile of implanted ions given a series of input parameters. These include the ion species, the ion energy and the target composition. The output of the program is of the form of implanted ion concentration and target damage distribution as a function of depth. From this the ion range, the straggle and full width at half maximum (FWHM) of the ion distribution can be calculated.

To model the erbium implantation profile amorphous silica of density 2.2 g/cm\(^2\), as opposed to the ASG material, was defined as the target.\(^{[3]}\) This was because the
density of the ASG material was not precisely known. It was found that a 25 % density increase and the addition of 8 wt. % arsenic to the silica modeled by TRIM gave a 15 % change in the predicted erbium implant depth. The ASG glass is composed of 92 wt. % silica and 8 wt. % arsenic oxide. The density of the ASG glass is therefore unlikely to differ significantly from that of pure silica.

Figure 4.1 shows the TRIM 92 predicted erbium implant depth and straggle (approximately equal to sigma of a Gaussian distribution) as a function of the implantation beam energy. The TRIM calculations suggest that the depth of the erbium profile increases linearly with ion beam energy by 0.29 μm per MeV. Calculations show this to be true at lower energies. Although at higher energies particles may not have sufficient time to interact with the target atoms to achieve efficient energy transfer. Figure 4.2 shows the TRIM 92 predicted erbium implantation profile in silica for an implantation energy of 2.7 MeV.

For the photosensitivity work (see chapters 2 and 5) TRIM 92 was used to predict the depth and profile of implanted hydrogen into silica; the results of which are presented in figure 4.3. A 7 μm germania doped silica layer was deposited onto a silica substrate (see section 3.1) to form the samples for implantation (typically single mode waveguides fabricated in this material are 7 μm high). The TRIM 92 software predicted that a 400 keV hydrogen beam would penetrate silica by 3.8 μm (with a straggle of 1.2 μm). This corresponds to approximately half the height of the germania/silica layer. An energy of 400 keV was therefore used so that the hydrogen would not penetrate past the 7 μm Ge layer.

4.2 Rutherford Backscattering Analysis

Rutherford backscattering (RBS) is a useful technique for non-destructive material analysis. The composition of a sample can be measured from the surface to a depth of several microns. An ion beam, usually hydrogen or helium, is incident on the sample and as the ion beam passes through the sample it loses energy. A small
proportion of the ion beam is backscattered out of the target material and its energy spectrum measured. Since the energy of the backscattered particle is a function of both the distance it has travelled through the sample and the mass of the target atom involved in the backscattering collision, the composition of the target may be deduced.

In this section, Rutherford backscattering is used to measure the depth profile of implanted erbium. This is so that the results of the TRIM modelling can be compared to experimental data and then modified if necessary. A silica wafer was implanted with 1 MeV Er$^+$ to a dose of 5x10$^{13}$ ions/cm$^2$. To determine the implant profile a 1.5 MeV He$^+$ beam normal to the sample surface was used as the RBS probe beam. The angle between the backscattered particle detector and sample surface was 160°.

Figure 4.4 shows the results of the RBS analysis. The channel number (the x-axis) is proportional to the energy of the backscattered particle - the lower the energy the deeper into the target the channel represents. The y-axis is proportional to the number of backscattered particles counted at each energy or channel. The energy of each backscattered particle was measured by a semiconductor detector. The detection electronics was used to convert the particle energy into a channel number proportional to the energy of that particle.

When a backscattering event takes place, the energy of the recoiled ion is proportional to the mass of the target atom. Collisions with erbium will therefore produce the highest energy backscattered particles, i.e. the target is composed of silica, oxygen, and erbium - erbium is the heaviest of these. If a backscattering event takes place on the sample surface then the energy of the recoiling probe atom is easily calculated since the analysis involves only the kinematics of backscattering, i.e. energy loss through the material is irrelevant. Facilities available on the Surrey RBS analysis computer software were used to find the channel numbers corresponding to He atoms scattered from erbium, oxygen and silicon located at the sample surface, these channels are called the surface channels. The surface channels are shown in table 4.1 and are indicated in figure 4.4. To determine the depth of implanted erbium the RBS
channel numbers must be calibrated to an energy value.

<table>
<thead>
<tr>
<th>Element</th>
<th>Surface Channel</th>
<th>Stopping Power ($10^{-15}$/cm$^2$ eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Erbium</td>
<td>435</td>
<td>84</td>
</tr>
<tr>
<td>Silicon</td>
<td>275</td>
<td>91</td>
</tr>
<tr>
<td>Oxygen</td>
<td>175</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 4.1. Element vs. RBS surface channel and material stopping power.$^{[4]}$

The energy of the recoiling probe beam from backscattering collisions at the sample surface may be calculated using equation (4.1) where $I$ is the input beam energy, $U$ the output beam energy and $K$ is a constant. $K$, which is obtained from reference [4], is dependant on the target mass and the detector to target angle.

$$\frac{U}{I} = K \tag{4.1}$$

Given an input helium beam energy of 1.5 MeV, and with $K=0.5721$ and 0.3708 for silicon and oxygen respectively this gives the backscattered helium energy from surface oxygen as 0.56 MeV (channel number 175) and 0.85 MeV from surface silicon (channel number 275).
Given equation (4.2) \( E= \text{energy, } C= \text{channel number, } e = \text{energy per channel and } k = \text{constant} \)

\[
E = C e + k
\]  

then for silicon :-

\[
0.858 = 274 e + k
\]

and for oxygen :-

\[
0.556 = 176 e + k
\]

Solving equations (4.3) and (4.4) gives an energy per channel of 3.08 keV (and an offset from zero of 0.013 keV - in theory \( k=0 \)). Examination of the RBS spectrum in figure 4.4 shows that the peak in the indicated erbium profile is 70 channels deep from the erbium surface edge. This corresponds to a probe beam energy loss of 215.7 keV (i.e. 70 \times 3.08). This means that the probe beam has lost 215.71 keV in travelling from the sample surface to the region of peak erbium concentration. From this the depth of the erbium can be calculated.

For an erbium dose of \( 1\times10^{15} \text{ ions/cm}^2 \) (into silica) the average ratio of erbium to silica to oxygen is approximately \( 1:10^4:2\times10^4 \). These parameters were used by the RBS computer software to calculate the stopping powers shown in table 4.1. The calculated stopping powers assume a uniform erbium distribution. This is a valid approximation providing the erbium is close to the sample surface and is in weak concentration. The stopping powers represent the energy loss per unit concentration.

The calculated stopping powers for erbium show that the probe beam will lose 0.84 eV per \( 10^{15} \) erbium atoms per cm\(^2\). An energy loss of 215.7 keV therefore corresponds to \( 2.57\times10^{18} \) erbium atoms per cm\(^2\).
The ratio of the atomic density of silica ($6.6 \times 10^{22}$ atoms per cm$^3$) to the RBS measured erbium concentration of $2.57 \times 10^{14}$ atoms per cm$^2$ gives a peak erbium implantation depth (at an energy of 1 MeV) of 0.37 μm. Using a similar procedure the full width at half maximum (FWHM=20 channels) of the 1 MeV implanted erbium profile was calculated to be ~ 0.1 μm.

As well as silica the ASG waveguides were also implanted with erbium and analyzed using the RBS technique. This was done to determine the difference in erbium implantation depths in pure silica and in the ASG material. However, the high arsenic signal masked the erbium signal and prevented RBS observation of the implanted erbium in ASG waveguides.

The TRIM 92 software predicted that erbium implanted into silica would penetrate to a depth of 0.29 μm per MeV of implantation energy. A value 0.37 μm per MeV was obtained using RBS analysis. Published values$^{[5,6]}$ of implantation depth against energy shows that the RBS figure of 0.37 μm per MeV is correct and correlates well with that obtained by others, i.e. Poleman et al demonstrated that a 3.5 MeV erbium beam penetrated silica by 1.25 μm.$^{[5]}$ This would indicate an inaccuracy in the TRIM 92 calculations. The implants described in chapter 6 therefore use the figure of 0.37 μm per MeV to calculate the implantation depth. Discrepancies with experimental data and the TRIM modelling of MeV heavy ion implantation are discussed in reference [7], problems in the TRIM calculations include errors due to dose effects and the trade off between simulation accuracy and computational speed.

Comparison of the TRIM 92 FWHM predictions with published results and the RBS analysis shows a large discrepancy. TRIM 92 predicted the FWHM for a 1 MeV erbium implant to be 0.06 μm, in comparison to the RBS measured value of 0.10 μm. Using an erbium implantation energy of 3 MeV, TRIM 92 predicted the FWHM of the erbium profile to be ~ 0.25 μm. This compares to published experimental values (found by RBS) of 0.56$^{[6]}$ μm and 0.46$^{[3]}$ μm which were obtained using implantation energies of 3.5 MeV and 2.9 MeV respectively. Again TRIM is seen not to be
accurate for these implant parameters. A best fit line to the published data of implantation depth as function of implantation dose and the RBS results was therefore used to predict the erbium implantation profile FWHM.

4.3 ASG Waveguide Fundamental Optical Mode Intensity Profiles

The fundamental optical mode intensity profiles of the 2 μm thick guiding layers of the ASG waveguides were calculated using Maxwell's equations.[9] To simplify the calculations the 2 μm by 25 μm ASG stripe waveguides were approximated to a planar structure. The ASG waveguides had a large aspect ratio, i.e. the structure is approaching a planar waveguide, and only the vertical optical mode profiles of the waveguides were required. The approximation to a planar structure is therefore valid as the ASG waveguides are highly multimode in the horizontal direction, but single mode in the vertical. The mode profiles were calculated at wavelengths of 514 nm and 1500 nm. These wavelengths correspond to the regions of the pump and signal wavelengths. At wavelengths of 514 nm and 1500 nm the FWHM of the vertical fundamental optical modes in the 2 μm thick guiding layers were calculated to be 0.7 μm and 2.3 μm respectively. This data was calculated using core and cladding refractive indices of 1.440 and 1.445.

4.4 Comparison of Waveguide Optical Mode Profiles and Implantation Profiles

This section compares the implanted erbium profile to the profile of the waveguide vertical fundamental optical modes. This was achieved by calculating the ratio of the FWHM of the Er profile to the FWHM of the ASG waveguide vertical optical mode intensity profile. However, because the implanted ion concentration profiles and the fundamental optical mode intensity profiles represent separate physical quantities a direct comparison of the two profiles is difficult.

The calculated fundamental optical mode profiles of 2 μm high planar ASG waveguides at 514 nm and 1500 nm are shown in figures 4.5 and 4.6, together with
the implanted erbium profile at energies of 1.35 MeV, 2.00 MeV and 3.00 MeV. The optical mode profiles are indicated by lines (D) while lines (A) to (C) represent the implantation profiles. The scaling of the implantation and optical mode profiles are based on arbitrary units. At these implantation energies the predicted erbium profiles peak at depths of 0.5 μm, 1 μm and 1.5 μm. The FWHM of the profiles are 0.13 μm, 0.23 μm and 0.29 μm (see section 4.2).

It is important that there is a good overlap between the dopant erbium and the fundamental waveguide optical mode profiles. If this is not the case some of the signal and pump light may not be able to interact with the implanted erbium. In conventionally grown samples (e.g. flame hydrolysis) this is not a problem as the entire volume of the waveguide can be uniformly erbium doped. However, in the case of implantation the erbium is introduced at a specific depth. Figures 4.5 and 4.6 show that a single energy implant covers only a small section of the waveguide. Any light propagating through the waveguide outside the implanted region does not interact with the implanted erbium and introduces inefficiencies.

The optimum overlap between the profile of implanted erbium and the waveguide optical mode intensity profile occurs when the erbium is implanted to a depth corresponding to the maximum in the optical mode intensity profile. Given an erbium implantation depth of 0.37 μm per MeV it can be calculated that an implantation energy of 2.7 MeV gives a peak erbium distribution at a depth of 1 μm. This coincides with the peak in intensity of the waveguide vertical fundamental optical mode profile. The FWHM of the erbium profile at an implantation energy of 2.7 MeV is 0.54 μm. The FWHM of the fundamental optical mode profiles at 514 nm and 1500 nm are 0.70 μm and 2.31 μm respectively. To compare the overlap between the implanted erbium profile and the waveguide optical mode profiles, the ratio of the FWHM of the erbium profile to the FWHM of the two optical mode profiles was calculated. This gave overlap ratios between the implanted erbium profile and the vertical optical mode profiles of 77 % and 23 % at optical propagation wavelengths of 1550 nm and 514 nm respectively. As previously stated, the implantation profile
and the optical mode profiles represent different physical quantities making absolute comparisons difficult.

To improve the ratios of the implanted erbium profile and the waveguide optical mode profile, multiple energy implantation can be used. By using three separate implants at energies of 1.35 MeV, 2.7 MeV and 4 MeV into the same sample the volume of implanted waveguide can be increased significantly. By summing the FWHM of the three implants (0.1 μm, 0.23 μm and 0.25 μm) and taking into account any region of implant overlap the width of the implanted sample was calculated to be 1.42 μm. This represents an overlap with the 1500 nm fundamental optical mode profile of 61 % and a complete overlap with the 514 nm optical mode profile (see figures 4.5 and 4.6 for a graphical representation).

4.5 Comparison of Optical Mode Profiles

To calculate the waveguide optical propagation loss from the measured insertion loss (measured by the fibre in/fibre out loss assessment apparatus - section 3.4), it is necessary to determine the optical mode mismatch between the launch fibre, the waveguide and the collection fibre. The launch fibre is single mode of core diameter 6 μm, the ASG waveguide is of dimensions 2 μm by 20 μm and the multimode collection fibre has a core diameter of 100 μm. All of the light emitted from the ASG waveguide will be launched into the multimode collection fibre giving 100 % optical overlap at the waveguide output (because the collection fibre is bigger than the waveguide). However, in the vertical plane, the ASG waveguide is significantly smaller than the core diameter of the single mode launch fibre.

The fundamental optical modes at a wavelength of 1500 nm of both the launch optical fibre (using manufactures data) and the ASG waveguides were plotted. The ratio of the integral of the ASG waveguide vertical optical mode intensity profile, to the integral of the single mode launch fibre optical intensity profile was then measured from the two intensity profiles. The results indicated that the mode overlap between
the launch optical fibre and the ASG waveguide was 48 % at a wavelength of 1500 nm.

4.6 Conclusions

Using a combination of RBS analyses and the computer modelling of the implantation profiles of erbium into silica it is estimated that erbium penetrates the ASG material by 0.37 \( \mu \text{m} \) per MeV of implantation energy. A 2.7 MeV implant will therefore generate an erbium profile with an approximate depth of 1.00 \( \mu \text{m} \), and the FWHM of the implant profile is estimated to be 0.52 \( \mu \text{m} \). At this implant energy the overlap between the implanted region of sample and the ASG waveguide optical mode intensity profile is 23 % at a propagation wavelength of 1500 nm. The overlap is 76 % at a propagation wavelength of 514 nm. Multiple implantation into the same ASG waveguide at energies of 1.35 MeV, 2.0 MeV and 4.0 MeV improves the overlap ratio to 100 % at 514 nm and 61 % at 1500 nm.

Comparison of the optical mode intensity profiles of the single mode optical fibre used to launch light into the waveguide and the ASG waveguide at a wavelength of 1500 nm gives a mode overlap of 48 %. The results of this chapter are also indicated in tables 4.2 and 4.3.
Table 4.2. The depth, FWHM and straggle of erbium implant profiles at 1.35 MeV, 2.7 MeV and 4.0 MeV. The FWHM of the multiple implant represents the total width of the implanted region. The FWHM of the ASG waveguide fundamental optical mode profiles are also given.

<table>
<thead>
<tr>
<th>Subject</th>
<th>Depth (μm)</th>
<th>FWHM (μm)</th>
<th>Straggle (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.35 MeV Implant</td>
<td>0.50</td>
<td>0.30</td>
<td>0.13</td>
</tr>
<tr>
<td>2.70 MeV Implant</td>
<td>1.00</td>
<td>0.52</td>
<td>0.23</td>
</tr>
<tr>
<td>4.0 MeV Implant</td>
<td>1.50</td>
<td>0.68</td>
<td>0.29</td>
</tr>
<tr>
<td>Multiple Implant</td>
<td>1.00</td>
<td>1.42</td>
<td>-</td>
</tr>
<tr>
<td>1500 nm Optical Mode</td>
<td>-</td>
<td>2.31</td>
<td>-</td>
</tr>
<tr>
<td>0514 nm Optical Mode</td>
<td>-</td>
<td>0.71</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 4.3. The percentage overlap between the waveguide fundamental vertical optical modes and the implant profiles, together with the overlap between the single mode optical fibre and the ASG waveguide.

<table>
<thead>
<tr>
<th>Subject</th>
<th>% Overlap</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.7 MeV implant + 1500 nm optical mode</td>
<td>23</td>
</tr>
<tr>
<td>2.7 MeV implant + 0514 nm optical mode</td>
<td>76</td>
</tr>
<tr>
<td>Multiple implant + 1500 nm optical mode</td>
<td>61</td>
</tr>
<tr>
<td>Multiple implant + 0514 nm optical mode</td>
<td>100</td>
</tr>
<tr>
<td>Waveguide optical mode + single mode fibre optical mode profile (both at 1500 nm)</td>
<td>48</td>
</tr>
</tbody>
</table>
Figure 4.1. TRIM predicted implantation depth and straggle of erbium implanted silica.
Figure 4.2. Trim predicted profile of 2.7 MeV Er implant into silica.
Figure 4.3. TRIM predicted implantation depth and straggles of hydrogen implanted silica.
Figure 4.4. RBS energy spectrum of 1 MeV Er implanted Silica.
Figure 4.5. Mode profile of (A) 4.00 MeV Er implant, (B) 2.7 MeV Er implant, (C) 1.35 MeV Er implant, (D) 514 nm ASG waveguide fundamental optical mode.
Figure 4.6. Mode profile of (A) 4.00 MeV Er implant, (B) 2.7 MeV Er implant, (C) 1.35 MeV Er implant, (D) 1500 nm ASG waveguide fundamental optical mode.
4.7 References.


Chapter 5

The Photosensitivity of Proton Implanted Germania Doped Planar Silica Structures

5.0 Introduction

The degree of photosensitivity in germania doped silica can be significantly enhanced by thermal annealing in a hydrogen atmosphere\(^{[1,2]}\). The degree of photosensitivity is considered to be the magnitude of the optically induced refractive index change. The thermal indiffusion of hydrogen requires the entire sample to be subject to the thermal hydrogenation process which may be detrimental to other devices sharing the same substrate. If only localized sample treatment is required this problem must be overcome. Ion implantation offers a solution to this problem by allowing discrete areas of the sample to be hydrogen doped. This chapter describes experiments designed to promote the growth of the Ge-Ge and Ge-Si bonds which are responsible for the photosensitivity of germania doped silica by the use of hydrogen implantation.

The 7 μm high 7 wt. % germania doped planar silica samples fabricated by the BT Labs, which were used in the experiments, are described in section 3.1. The level of the germanium concentration is similar to the doping level demonstrated previously to be suitable for the thermal hydrogenation indiffusion process\(^{[3]}\).

The germania doped silica samples were implanted with a range of hydrogen doses and implantation temperatures, the details of which are described in section 5.1. The optical absorption due to the Ge-Ge/Si defects in the implanted samples was measured using the diode array spectrometer and the experimental techniques described in section 3.3 to evaluate the photosensitivity of the material. These spectral absorption measurements give an indication of the extent of the Ge-Ge or Ge-Si defects present in the samples because the Ge-Ge and Ge-Si bonds have a characteristic optical absorption.
absorption spectra which shows peaks at 240 nm and 190 nm. By measuring the strength of these optical absorption peaks the photosensitivity of the implanted samples can be estimated.

At a wavelength of 1500 nm singlemode optical waveguiding requires a guiding layer thickness of approximately 7 µm which is the value used here. Initially 400 keV was used as the hydrogen implantation energy since this energy gives a hydrogen implantation profile which peaks at approximately half the depth of the germania/silica layer (see section 4.1). However, as the sample transmission spectrum was measured using normal incidence, the exact depth of the implanted hydrogen was not critical. The only energy requirement was that all of the implanted ions were retained in the germania/silica layer, i.e. they did not penetrate into the silica substrate.

5.1 Procedure

The germania/silica structures were implanted at room temperature with 400 keV hydrogen using ion doses ranging between $5 \times 10^{14}$ ions/cm$^2$ and $5 \times 10^{17}$ ions/cm$^2$. The equivalent peak concentrations of these doses are 0.002 at. % and 2.4 at. %, the upper dose range was chosen to be similar to the magnitude of the level of germania doping. Referring to section 4.1, an implantation energy of 400 keV has a predicted ion range ($R_p$) of 3.8 µm which corresponds to half the depth of the guiding layer. Each implant was carried out twice; once with atomic hydrogen (H) and once with molecular hydrogen ($H_2$) using the same ion energy and total proton dose. In comparison to atomic hydrogen, the implantation of molecular hydrogen doubles the implantation dose rate because there are twice the number of hydrogen atoms for the same beam current. The two different beam species were implanted to investigate if either proton or molecular hydrogen implantation produced a greater change in the spectral absorption curves of the germania silica samples.

The enhancement of photosensitivity by the thermal indiffusion of hydrogen requires the germania/silica samples to be at high temperatures: typically 800°C. To determine if this is also the case for hydrogen implanted samples four germania silica
samples were implanted with protons at a dose of $1 \times 10^{17}$ ions/cm$^2$ and at temperatures between 700 °C and 1000 °C. During implantation the samples were mounted on a graphite strip heating element. Before and after the implantation of a single sample a time of approximately 1 hour was required for the sample holder to reach the desired temperature or cool down to room temperature. The heating/cooling cycle took place in the ion implanter vacuum chamber at a typical vacuum of $10^{-5}$ mbar. The temperature of the heated sample stage could be kept constant to ± 20 °C by monitoring the sample temperature with a thermocouple positioned underneath the sample which was linked to the heater control circuitry.

Figure 4.3 shows that the width (straggle) of a 400 keV hydrogen implanted layer is 1.2 μm. The guiding layer thickness is 7 μm. Approximating the hydrogen implant to a uniform doping density of width 1.2 μm gives a ratio of unimplanted to implanted material (using a 400 keV implant) of 18 %. To increase the implantation profile width of the implanted ions in the guiding layer multiple energy implantation was used. Three implants at beam energies of 200 keV, 300 keV and 400 keV were carried out into the same heated sample (800 °C). A dose of $1 \times 10^{17}$ ions/cm$^2$ was used for each implanted ion energy. This technique of multiple energy implantation gave an approximate three fold increase in the width of the implanted ion profile in the germania doped silica guiding layer.

To measure the implantation induced changes in the Ge-Ge and Ge-Si defect concentrations, the ultraviolet transmission spectra (190 nm to 400 nm) of all the germania samples were measured before and after implantation.

Experiments were carried out to investigate if the growth in the Ge-Ge/Si bond concentration as a result of thermal annealing in a hydrogen atmosphere could be enhanced by hydrogen implantation. Following measurement of the optical transmission spectra, the hydrogen implanted samples were annealed in a 10 % hydrogen and 90 % nitrogen atmosphere for 2 periods of 60 minutes at temperatures ranging between 600 °C and 1000 °C. This is the same temperature range that has been demonstrated to be most suitable for the thermal hydrogenation process.[1] The
optical spectra of the samples were then remeasured.

To determine the photosensitivity of the implanted samples, those with the strongest implantation induced 240 nm absorption were exposed to an excimer pulse laser. The laser operated with an energy of 0.5 J per pulse, a pulse frequency of 50 Hz and a pulse duration of 10 ns. The peak output wavelength of the laser was 249 nm. The samples were exposed to the excimer laser beam for two minutes before the optical spectra were remeasured. The procedure was repeated until no change in the sample optical transmission spectra could be observed. The results were compared to those obtained from samples fabricated by the thermal hydrogenation process.

5.2 Results and Discussion

5.2.1 Affect of Room Temperature Implantation as a Function of Implantation Dose

Interactions between the ion beam and the sample create defects which can affect the optical properties of the sample. This can be due to either the physical interaction of the sample material with the ion beam or by a chemical interaction between the implanted ions and the sample. In the case of germania doped silica, hydrogen implantation can change the concentration of the Ge-Ge bonds, the Ge-Si bonds and the Ge-E' defects, which are the bonds and defects that are responsible for photosensitivity. The thermal hydrogenation process promotes the growth of an optical absorption peak centred at 240 nm which is the characteristic absorption wavelength of the Ge-Ge and Ge-Si bonds. Exposure to UV light can break these bonds and form Ge-E' defects. These defects are characterised by a peak optical absorption at 213 nm. The strength of both the 240 nm and the 213 nm absorption peaks influence the refractive index of the sample at infrared wavelengths. A more detailed explanation of this process is given in chapter 2. Characterisation of the optical absorption spectra of the implanted germania doped silica structures is therefore a useful method for indicating the level of photosensitivity within the sample.
The optical absorption spectrum (line A) of the as-grown germania/silica structure is shown in figure 5.1 together with the spectrum of the same sample after a room temperature implant with 400 keV protons (atomic hydrogen) using a dose of $1 \times 10^{17}$ ions/cm$^2$ (line B). The spectral data is presented only in the region 190 nm to 300 nm since no spectral features were found between 300 nm and 900 nm.

The optical absorption spectrum measured from the implanted sample is larger than that measured from the as-grown sample, see figure 5.1. The magnitude of the implantation induced change in absorption is seen to decrease in an exponential manner with increasing wavelength. Together with the increase in background absorption at all wavelengths, the spectrum of the room temperature implant sample also shows an absolute increase of 10% in absorption at 213 nm. Subtracting the estimated strength of the implantation induced background absorption gives a peak absorption of only 4% (i.e. the 213 nm absorption peak is considered to be superimposed onto an exponentially decaying absorption curve with increasing wavelength).

The growth in 213 nm absorption with proton dose is shown in figure 5.2. The increasing absorption with dose was found to saturate at dose levels higher than $1 \times 10^{17}$ ions/cm$^2$. The formation of the 213 nm absorption peak suggests that room temperature proton implantation leads directly to the Ge-E' defect, which has a peak optical absorption at 213 nm. In the case of samples exposed to the thermal hydrogenation process, the 240 nm absorption peak, which is linked to the Ge-Ge (or Ge-Si) bond is the strongest optical absorption peak that is observed, in these samples the 213 nm absorption does not dominate until after optical exposure. In the case of the room temperature implanted samples, the strongest absorption was measured at 213 nm (the Ge-E' defect) and not 240 nm (the Ge-Ge bond). To form a strong photosensitive material growth of the 240 nm absorption peak, which corresponds to the Ge-Ge defect, is required. This 240 nm absorption is produced by thermal hydrogenation which did not occur for room temperature implantation.

Implantation of molecular hydrogen gives twice the proton dose when compared to
proton implantation. After this was taken into account, the results showed that the induced 213 nm optical absorption following molecular hydrogen implantation was less than half of that obtained after the equivalent proton dose. At present no explanation can be offered for this. It would be expected that on impact with the sample the 400 keV molecular hydrogen molecule would dissociate into two 200 keV protons which would act in the same way as two separately implanted 200 keV protons.

After annealing for up to 2 hours at 800 °C in the 10 % hydrogen and 90 % nitrogen atmosphere, comparison of the spectral transmission of all the room temperature implanted samples and an as-grown sample showed no difference. The 213 nm absorption peak was removed from the implanted samples, which was also observed after annealing in pure nitrogen, and all the samples showed an identical growth in absorption at 240 nm (19.5%). This indicates formation of the Ge-Ge bond with no preference for the implanted material and that the Ge-E' defects are easily removed by thermal annealing.

5.2.2 Ion Beam Heating

Four samples were placed adjacent to each other on the same stainless steel sample holder and implanted sequentially, each with $1 \times 10^{17}$ H$^+$ ions/cm$^2$ at room temperature. The sample implanted first showed the largest growth in 213 nm absorption, see figure 5.3. Sample (A) was implanted first and sample (D) last. The total time between the first and last sample to be implanted was approximately 5 hours. For the majority of this time the beam was on.

With an implantation energy of 400 keV and typical ion beam current of $\sim 5$ µA approximately 2 W of power is incident on the sample during implantation. This power is dissipated as heat which leads to a significant increase in the temperature of the sample holder. The results presented in figure 5.3 suggest that beam heating of the samples prior to implantation modifies the spectral absorption curve while beam heating after implantation has no significant effect. This effect was found to be a
function of the time the samples were heated in a vacuum of \(< 10^{-5}\) mbar. The fourth sample, which was implanted last was heated for about 5 hours and its spectral absorption curve is shown in figure 5.3. It is suggested that the variation between samples could be due to vacuum degassing of the sample surface removing impurities. This may affect the optical absorption of the sample and the chemical interaction of the sample with the hydrogen beam although the details of this process are not known.

5.2.3 The Effect of Sample Temperature During Implantation

The absorption spectrum of a heated stage implanted sample is shown in figure 5.1. The sample was heated to a temperature of 800 °C and held at that temperature while implanted with a proton dose of \(1 \times 10^{17}\) ions/cm\(^2\). In comparison to the room temperature implanted sample, use of the heated sample stage results in a 240 nm peak in the spectral absorption curve, as opposed to 213 nm in the case of room temperature implantation. The magnitude of the absorption peak is 21 % which is almost twice the magnitude of the 213 nm absorption peak observed in the room temperature implanted sample.

The germania doped silica structures were implanted with protons using a dose of \(1 \times 10^{17}\) ions/cm\(^2\) and at sample stage temperatures of 700 °C, 800 °C, 900 °C and 1000 °C. Visual examination of the sample implanted at the temperature of 1000 °C showed a brown discoloration which was attributed to ultraviolet absorption (caused by implantation induced defects) extending to the blue end of the spectrum. The optical absorption of the sample was > 95 % at all scanned wavelengths (no 240 nm peak was found). The optical absorption spectra of the samples implanted at the other temperatures are shown in figure 5.4. Line (A) represents the 700 °C implant, line (B) 800 °C and finally line (C) shows the 900 °C implant.

Using a sample temperature of 700 °C during implantation results in a 240 nm absorption peak of similar magnitude but less well defined than the absorption peak produced by 800 °C implantation. However, at 900 °C the absorption spectrum showed a similar absorption value at 240 nm to the sample implanted at 800 °C, but
gave an increased absorption at all other scanned wavelengths. Increasing the implanted ion dose to $5 \times 10^{17}$ ions/cm$^2$ had no further effect on the magnitude of the induced 240 nm absorption peak.

These experiments demonstrate that the optimum sample implantation temperature for the production of a photosensitive material is $\sim 800^\circ$C. The growth of the 240 nm absorption peak during heated stage implantation represents an increase in the concentration of Ge-Ge bonds, the same defects which are created by the thermal hydrogen indiffusion process. This compares to a growth in the concentration of the Ge-$E'$ defects, which have a peak optical absorption of $\lambda = 213$ nm, which were formed in samples implanted at room temperature. These results show that heated stage implantation is necessary to promote the chemical reactions necessary to form the Ge-Ge and Ge-Si bonds required for photosensitivity.

5.2.4 Multiple Energy Implantation

Three implants, each of dose $1 \times 10^{17}$ protons/cm$^2$ at energies of 200 keV, 300 keV and 400 keV were carried out into the same sample. The highest energy implant was carried out first and the lowest energy last. During implantation the sample was heated to $800^\circ$C.

Measurement of the 240 nm absorption peak of the $800^\circ$C multiple energy implant sample showed identical results to the single energy $800^\circ$C implant sample of dose $1 \times 10^{17}$ protons/cm$^2$. At $800^\circ$C a proton implanted into the sample is unlikely to remain in the nuclear stopping region since at these elevated temperatures the implanted protons can diffuse through the sample.$^{[5]}$ Consequently, the use of three different implantation energies will be the equivalent to a single energy implant with three times the dose. The growth in 240 nm absorption was seen to saturate with hydrogen doses greater than $1 \times 10^{17}$ ions/cm$^2$. It can therefore be expected that multiple energy implants using total doses higher than this will not increase the 240 nm absorption further.
5.2.5 Optical Exposure

The sample implanted at 800 °C using 400 keV protons with a dose of $1 \times 10^{17}$ ions/cm$^2$ was exposed to an excimer laser. The excimer laser operated with a pulse duration of 10 ns, a pulse repetition frequency of 50 Hz and a pulse energy of 0.5 J. The operating wavelength of the laser was 249 nm which is close to the major absorption peak of the Ge-Ge (or Ge-Si) bonds. Absorption of the 249 nm laser beam due to the 240 nm absorption peak can break the Ge-Ge (or Ge-Si) bonds to form Ge-$E'$ defects which show a characteristic optical absorption at 213 nm. If the optical exposure intensity is sufficiently high the trapped hole orbiting the Ge-$E'$ can be photo-ionised. This process changes the infrared refractive index of the sample.$^{[6,7]}$

After two minutes of exposure to the pulsed optical beam, the absorption peak at 240 nm was found to decrease by approximately 5%. Figure 5.5 shows the absorption spectra of the sample before and after optical exposure. Further exposure to the excimer laser had no measurable effect.

The reduction in 240 nm optical absorption suggests that (i) the Ge-Ge (or Ge-Si) bond concentration is reduced by optical exposure and (ii) the Ge-$E$(or Ge-Si) bonds are broken by the 249 nm light and hence that the sample is photosensitive.$^{[6]}$ No strong absorption peak at 213 nm was observed in the sample following optical exposure. This would suggest that in this case either the Ge-$E'$ bonds are not formed during the exposure process or that they are immediately bleached by the intense 249 nm laser beam. The slight increase in absorption at 213 nm could be attributed to a small increase in the concentration of Ge-$E'$ defects.
5.3 A Comparison of Thermal Hydrogenation and Implantation

The optical absorption spectra of a 7 wt. % germania silica sample (identical to the implanted samples) which was annealed in a hydrogen atmosphere for a period of 1 hour at 800°C is shown in figure 5.6. The optical absorption spectrum of this sample shows absorption levels of greater than 95 % at wavelengths of 240 nm and 190 nm. The optical absorption at both 190 nm and 240 nm, which is attributed to the Ge-Ge or Ge-Si defect, indicates a significantly larger Ge-Ge (or Ge-Si) concentration than that formed in the hydrogen implanted samples.

The concentration of available hydrogen in the thermal hydrogenation process is many orders of magnitudes greater than the hydrogen content of the implantation beam. In other words, at any instant in time the concentration of hydrogen incident on the sample surface from the implantation beam is very small compared to the hydrogen available by annealing the sample in a hydrogen atmosphere. If the chemical reaction required to reduce the germanium to form Ge-Ge bonds is unfavourable then a higher concentration of hydrogen will improve the yield of Ge-Ge bonds. It is suggested that this reason could account for the difference in the optical absorption spectra obtained from the hydrogen implanted sample and the sample annealed in hydrogen.

5.4 Summary of Results

This chapter details experiments, the results of which are discussed below, to determine if germania doped silica can be made photosensitive using hydrogen implantation rather than by the thermal indiffusion of hydrogen. This novel procedure would allow the fabrication of locally photosensitive regions, and hence Bragg gratings on integrated optical circuits.

Protons (H⁺) were implanted into 7 wt. % germania doped silica with the sample stage, neglecting beam heating effects, at room temperature. The implantation was carried out with a beam energy of 400 keV and with ion doses ranging between 5x10¹⁴ ions/cm² and 5x10¹⁷ ions/cm². To measure the relative changes in the Ge-E' defect
concentration and the Ge-Ge bond concentration, the ultraviolet transmission spectra of the implanted samples was compared to that of the as grown samples. A growth in optical absorption at 213 nm was observed in all of these samples. The strongest 213 nm absorption peaks were observed in the samples implanted with proton doses \( \geq 1 \times 10^{17} \text{ ions/cm}^2 \). A 213 nm optical absorption of 10% was measured in the \( 1 \times 10^{17} \) protons/cm\(^2\) sample. No further growth in optical absorption was observed as the proton dose was increased to \( 5 \times 10^{17} \) ions/cm\(^2\).

The as grown 7 wt. % germania doped silica structures were then implanted with proton doses of \( 1 \times 10^{17} \) ions/cm\(^2\) whilst maintaining the sample stage at temperatures between 700°C and 1000°C. After implantation, the samples heated to a temperature of 800°C showed the largest increase in 240 nm optical absorption of 21%. Increasing the proton dose higher than \( 1 \times 10^{17} \) ions/cm\(^2\), and using multiple energy implantation had no further effect on the measured increase of 21% in the 240 nm optical absorption.

All of the implanted samples were annealed in a 10% hydrogen - 90% Nitrogen atmosphere at temperatures of up to 800°C and for periods of up to 2 hours. Any 213 nm optical absorption present in the samples was removed and all the samples showed an identical post annealing 240 nm optical absorption.

The samples implanted with a dose of \( 1 \times 10^{17} \) protons/cm\(^2\) at implantation stage temperatures of 800°C and room temperature were exposed to an excimer laser. The 249 nm excimer laser operated with a pulse frequency of 50 Hz and a pulse energy of 0.5 J. The samples were exposed for periods of one minute and after each exposure the sample optical transmission spectra was measured. Optical exposure had no effect on the 213 nm optical absorption levels. However, after an exposure time of 2 minutes, a reduction of 2% was observed in the 240 nm optical absorption level in the high temperature implant sample, indicating a reduction in the concentration of Ge-Ge and Ge-Si bonds.
Figure 5.1. The absorption spectra for (A) the as-deposited sample, (B) a sample implanted at room temperature $(1 \times 10^{17} \text{ H/cm}^2)$, and (C) a sample implanted at a temperature of $800^\circ \text{C} (1 \times 10^{17} \text{ H/cm}^2)$.
Figure 5.2. The effect of proton dose on the amplitude of the 213 nm absorption peak for samples implanted at room temperature.
Figure 5.3. The absorption spectra of several samples which were mounted on the same sample stage and implanted sequentially in the order A to D.
Figure 5.4. The absorption spectra of samples implanted at temperatures of (A) 700 °C, (B) 800 °C and (C) 900 °C.
Figure 5.5. Absorption spectra showing the 240 nm absorption peak of samples implanted at 800 °C before and after exposure to the excimer laser.
Figure 5.6. Optical absorption spectra of a 7 wt. % germania sample annealed in hydrogen for a period of 1 hour.
5.5 References.


The Optical Properties of Erbium Implanted ASG Waveguides

6.0 Introduction

The fabrication of the erbium optical amplifier in ASG planar glass waveguides by ion implantation is potentially a useful process. Before this device can be fabricated it is important to characterise the optical properties of erbium implanted ASG. The work described in this chapter is concerned with the optimisation of the implantation process required to introduce optically active Er into the ASG waveguides described in section 3.1. Ultimately the data presented may be used in the design of an Er doped optical amplifier in planar waveguides fabricated by ion implantation.

To achieve gain in the erbium optical amplifier it is important that the fluorescence decay lifetime due to the implanted erbium and the 1550 µm erbium related optical absorption are sufficiently high. The results presented here address the questions that are to be answered before Er implantation can be used successfully to fabricate optical amplifiers. To summarize, the following problems are considered:

[1] In the case of a single energy erbium implant, how does the fluorescence decay lifetime vary with erbium dose? Is the fluorescence decay time enhanced by thermal annealing?

[2] How does up-conversion affect the fluorescence decay lifetime?

[3] Can erbium be implanted in sufficient quantity to produce a good spectral absorption curve? At the erbium doses required what is the measured fluorescence lifetime?
6.1 Procedure

6.1.1 Implantation Parameters

To assess the optical properties of Er implanted ASG waveguides, several samples with different erbium doses were required. The ASG waveguides described in section 3.1 were implanted with erbium doses ranging from $5 \times 10^{14}$ ions/cm$^2$ ($\sim 0.01$ at. %) to $5 \times 10^{17}$ ions/cm$^2$ ($\sim 13$ at. %). The implantation energy used was 2.7 MeV which was obtained using an acceleration voltage of 1.35 MeV and extracting doubly charged erbium ions. The range of the 2.70 MeV implant in the ASG material is $\sim 1$ $\mu$m. The FWHM (full width at half maximum) of the implantation profile is 0.54 $\mu$m. The peak erbium ion concentrations were obtained using equation (6.1), where $C$ represents the percent atomic concentration, $D$ the ion dose in ions/cm$^2$, $S$ the implantation profile straggle in cm and $P$ the atomic density of silica in atoms/cm$^3$. 
To achieve good amplifier efficiency it is desirable that the profile of implanted erbium is closely matched to the waveguide optical intensity mode profile. This is not possible in the case of a single energy implant. The FWHM of a 2.7 MeV implant is 0.54 µm and the FWHM of the fundamental vertical optical mode profile of the ASG waveguides is 2.3 µm at a wavelength of 1500 nm (see chapter 4, figure 4.6). The percentage overlap between the optical and erbium profile is only 23%. At a wavelength of 514 nm the vertical optical mode width of the ASG waveguide is 0.70 µm. The corresponding overlap with 2.7 MeV implanted erbium is 77%. Erbium implanted at 2.7 MeV penetrates silica to an estimated depth of 1 µm. This corresponds to the centre of the waveguide and the peak in optical power density.

A useful method of increasing the width of the implanted erbium profiles is the technique of multiple energy implantation. A single sample is implanted with the same ion species at several different beam energies to increase the volume of implanted material. In this case an ASG waveguide was implanted three times using beam energies of 1.35 MeV, 2.70 MeV and 4.00 MeV. The ion doses were 0.5x10^{16} ions/cm^{2}, 1.0x10^{16} ions/cm^{2} and 0.5x10^{16} ions/cm^{2} respectively. The total erbium fluence implanted into the sample was 2x10^{16} ions/cm^{2}. Using multiple implantation the local erbium concentrations are reduced when compared to a single energy implant of the same ion dose. This may be beneficial when considering cooperative up-conversion. The peak erbium concentrations of the three implants were 0.23 at. % (1.35 MeV), 0.26 at. % (2.70 MeV) and 0.10 at. % (4.00 MeV). Comparison of the 1.35 MeV and 4.00 MeV implants shows a discrepancy in the peak erbium concentration even though the implantation doses are the same. This is because the FWHM of the implant profile increases with beam energy. At energies of 1.35 MeV, 2.70 MeV and 4.00 MeV the implantation depths are 0.5 µm, 1.0 µm and 1.5 µm with FWHM of 0.30 µm, 0.54 µm and 0.68 µm. The FWHM of the implanted region is 1.42 µm, the overlap with the 514 nm vertical optical mode profile

\[ C = \frac{40D}{SP} \]
(FWHM=0.70 μm) is 100 % and with the 1500 nm optical mode profile (FWHM=2.31 μm) it is 61 %. This compares to an overlap of 23 % between the 1500 nm vertical optical mode profile and the erbium concentration profile for a single energy 2.7 MeV implant. Multiple energy implantation was carried out in the order of the highest energy implant first and lowest energy implant last. This was done to avoid implanting through the first and second implanted layers. The mode overlap ratios and the implantation FWHM stated were calculated in chapter 4. Chapter 4 also contains graphical representations of the implantation and optical mode profiles.1

For all the implants described the implanted ion dose accuracy was within ± 10 % of the stated dose. A direct comparison between implanted erbium concentrations and conventionally doped erbium concentrations (e.g. flame hydrolysis, solution doping) is not always possible. Conventional doping results in a uniform erbium distribution throughout the sample volume which is not the case for ion implantation where only a local area of the sample is doped.

6.1.2 Post Implantation Annealing

To measure the effect of post implantation annealing on the fluorescence lifetimes of erbium implanted samples, they were implanted with erbium ion doses up to and inclusive of 4.75x10^15 ions/cm^2. The samples were then annealed in 100 °C intervals in the temperature range 500 °C to 900 °C. At each temperature the samples were annealed for two periods of 30 minutes. At erbium ion doses greater than 2x10^16 ions/cm^2, including the multiple energy implant sample, the samples were annealed at temperatures of 800 °C and 900 °C for up to three periods of 30 minutes at each temperature. The waveguides were not annealed at lower temperatures as annealing in the range 500 °C to 900 °C showed that the maximum fluorescence lifetime was obtained at the anneal temperature of ~ 800 °C, see section 6.3.2. A similar behaviour in silica glass was observed by Lidgard.1

The samples were annealed in a preheated furnace containing a free flowing nitrogen

1When comparing implantation concentration profiles to optical mode profiles, the overlap is the ratio of the full width at half maximum (FWHM) of the waveguide vertical optical mode profile to the FWHM of the implantation concentration depth profile. In this work the centre of the two profiles are coincident. When the FWHM of the implantation profile is less than the FWHM of the optical mode profile, the overlap is greater than 100%.
atmosphere at slightly greater than atmospheric pressure. Before timing of the anneal period began, the samples were placed into the furnace and allowed to reach greater than 97% of maximum temperature, which took ~2 minutes. On cooling, the samples took ~2 minutes to reach temperatures <100 °C. The furnace temperature was constant to an accuracy of ~±1% and was monitored using a thermocouple located immediately beneath the silica sample holder. The temperatures quoted are those of the thermocouple; the sample temperature possibly being slightly lower. Before annealing the samples were cleaned using the procedure described in section 3.2.1.

To monitor any change in optical characteristics, the fluorescence decay lifetime, the spectral absorption and the waveguide insertion loss of the samples were measured before and after implantation and following each anneal.

6.2 Implantation Induced Waveguide Damage

6.2.1 Scattered Light Technique

Prior to the use of the fibre in/fibre out waveguide insertion loss measurement apparatus (section 3.4 and 6.2.2), a scanning probe was used to measure the waveguide propagation loss using 632.8 nm light from a HeNe laser as the excitation source. Using this technique samples implanted with erbium doses ranging from 5x10^14 ions/cm^2 to 4.75x10^15 ions/cm^2 were shown to have an increased optical propagation loss following implantation. The optical propagation loss was returned to the pre-implantation level after annealing at 800°C for one hour.

The ASG waveguides were manufactured using a diamond saw to form the guiding channels. The cutting process may leave scratches on the waveguide surface which could account for a random distribution of scattering centres, which would prevent the reliable use of this technique. The uncertainty in the results was ±25 dB/cm. This was calculated by repeated measurements of identical waveguides. The results that were obtained indicate only that the waveguide propagation loss was increased by
implantation and that it was restored by annealing, although this result is not clear
with such a large margin of error.

6.2.2 Fibre In/Fibre Out Technique

Both prior to implantation, and before and after annealing, the optical insertion loss
of the ASG waveguides was measured using the fibre in/fibre out launch technique
(see section 3.4). Measurements were carried out at 1526 nm and 1300 nm.

The optical mode mismatch between the launch optical fibre and the ASG waveguide,
which was calculated in section 4.4, was used to calculate the waveguide optical
propagation loss from the measured insertion loss by multiplying the waveguide input
power by the percentage mode mismatch. The Fresnel loss was not significant as
index matching oil was used between the optical fibres and the ASG waveguide. It
is the waveguide propagation loss values that are presented.

The propagation loss of the pre-implanted ASG waveguide was measured to be less
than 1 dB/cm which agrees with results obtained by the BT Labs. After implantation
the optical propagation loss of the sample implanted with a dose of 5x10^{14} ions/cm^2
increased to ~ 5 dB/cm. For the higher dose of 4.75x10^{15} ions/cm^2 the implantation
induced waveguide loss was too high to measure since no optical output was reliably
detected from the excited waveguide. After annealing at 600 °C for one hour the
propagation loss of these samples was restored to the pre-implantation value and
annealing at higher temperatures had no further effect on the measured propagation
loss. Measurement of the insertion loss of these samples at wavelengths of 1526 nm
and 1300 nm gave identical results.

Paradoxically, the higher erbium dose waveguides showed a smaller increase in
implantation induced optical propagation loss than the 2x10^{16} ions/cm^2 dose
waveguide. Implantation with erbium doses between 5x10^{15} ions/cm^2 and 5x10^{17}
ions/cm^2 had no measurable effect on the waveguide optical propagation loss.
Although, for the erbium dose of $5 \times 10^{17}$ ions/cm$^2$ the optical propagation loss measured at a wavelength of 1300 nm was between 0.25 dB/cm to 0.50 dB/cm lower than that measured at a wavelength of 1526 nm. The difficulty in measuring propagation loss values lower than 1 dB/cm and experimental error prevented verification of this. Following implantation the propagation loss of the multiple implant sample rose to 22.8 dB/cm with no measurable difference at wavelengths of 1300 nm and 1526 nm. The propagation loss was restored to the pre-implantation value of $< 1$ dB/cm after annealing at 800 °C for 1 hour, as was found for all the samples.

The presence of erbium is revealed by peaks in the white light transmission spectra of erbium doped waveguides, although it may not be visible at low dopant levels since the optical absorption increases with erbium concentration. Optical absorption of 1526 nm light into the strong absorption peak situated at approximately 1550 nm, which corresponds to the main Er laser level, could account for the difference in the waveguide optical loss measured at 1526 nm and 1300 nm which was observed in the higher dose sample ($5 \times 10^{17}$ ions/cm$^2$).

The kinematic interaction between the implanted Er ion beam and host crystal lattice damages the target material. Defects created by the implantation process can have a profound effect upon the guiding characteristics of the waveguide. However, these defects may be partially or completely removed by thermal annealing, the exact anneal requirements will vary with the material and the extent of the implantation damage. This effect is demonstrated by the increase in excess waveguide loss as the implantation dose is increased from $5 \times 10^{14}$ ions/cm$^2$ to $4.75 \times 10^{15}$ ions/cm$^2$. The results show that moderately low temperatures (500 °C for 1 hour) are required to remove the implantation damage and that higher temperature annealing, which may be required to optimise the erbium fluorescent properties, has no detrimental effect on the waveguide optical propagation loss.

At the higher implantation doses the negligible effect of implantation on waveguide
loss is in direct contradiction to the results that would be expected. Sample heating takes place during implantation due to the dissipation of an average ion beam power of 1.3 W for an average beam current of 0.5 µA. If 1% of the supplied heat is retained in the sample (it is likely to be higher as the sample is mounted in vacuum) and given the specific heat of silica is 600 J kg\(^{-1}\) K\(^{-1}\), then in the first hour of implantation the sample temperature will increase by over 100°C. In achieving high implantation doses, continuous implants for successive periods of over 5 hours were not unusual. In effect this means that the higher dose samples are annealed during the implantation procedure. It is likely that this process accounts for the low as-implanted optical propagation loss measured in the higher dose samples and suggests that the implantation induced damage can be removed by annealing.

Using the fibre in/ fibre out launch system, measurements of loss values lower than 1 dB/cm were extremely difficult. Repeated measurements in determining the waveguide loss showed an uncertainty of approximately ± 0.5 dB/cm.

6.3 Fluorescence Characteristics

6.3.1 Material Fluorescence

An as-grown ASG waveguide was characterised using the fluorescence lifetime measurement system, as described in section 3.6. A weak fluorescence of lifetime decay constant < 1 ms was observed. The measured lifetime was found to be invariant with laser pump power (laser wavelength=514 nm) in the pump power region 500 mW to 1500 mW. The fluorescence signal was too weak to measure below 500 mW, and was unaffected by thermal annealing at temperatures up to 900°C for one hour. The fluorescence spectrum of the material, which was measured by the BT Labs, consisted of a broad band fluorescence centred at ~1100 nm with a width of ~100 nm. The lifetime of the ASG fluorescence was found to be < 0.5 ms. The response time of the fluorescence lifetime decay measurement system using a mechanical chopper pulse width of 50 ms was less than 0.5 ms. Lower lifetimes than
this could therefore not be quantified. The absolute value of the fast decay ASG fluorescent lifetime therefore remains in question.

The measured fluorescence decay curve is the sum of all the luminescence with wavelength greater than 1000 nm, which is determined by the transmission wavelength of the silicon filter. The luminescent intensity of the higher dose (> 4.75x10^{15} ions/cm^2) samples after annealing for 1 hour at 800°C shows a more than ten fold increase in the fluorescence over the unimplanted ASG fluorescence intensity. When considering the higher erbium dose samples it is therefore unlikely that the ASG material fluorescence significantly contributes to the measured 1550 nm fluorescence. If this had not been the case, the silicon filter between the waveguide and detector could have been replaced with a filter of higher cut off wavelength, i.e. > 1200 nm. This would allow measurement of only the 1550 nm erbium fluorescence.

### 6.3.2 Erbium Fluorescence Lifetime and the Effect of Annealing

The fluorescence decay lifetimes of samples implanted with ion doses up to and including 4.75x10^{15} ions/cm^2 are shown in figure 6.1. The lifetimes were measured with a pump wavelength of 514 nm and pump power of 1.5 W. As previously described, the samples were annealed at temperatures between 500 °C and 900 °C for two periods of 30 minutes at each temperature. Lines (A), (B), (C) and (D) of figure 6.1. show the fluorescence lifetimes obtained with erbium doses of 5x10^{14} ions/cm^2, 1x10^{15} ions/cm^2, 2x10^{15} ions/cm^2 and 4.75x10^{15} ions/cm^2 respectively. At each temperature the samples were annealed twice which is indicated by two data points for each temperature. Prior to annealing and after implantation no fluorescence was observed from any of these samples.

The results presented in figure 6.1 show that following the first anneal step of 500 °C fluorescence was obtained from all of the samples indicated. The measured fluorescence lifetime was seen to increase with both the annealing temperature and the erbium dose. For the lowest dose sample (line (A) - 5x10^{14} ions/cm^2) the measured
lifetime increased from 1.8 ms to 4.0 ms with increasing anneal temperature (from 500 °C to 900 °C). At the higher ion dose of $4.75 \times 10^{15}$ ions/cm$^2$, shown by line (D), the measured lifetime increased from 6.8 ms to 8.8 ms after annealing at 500 °C and 800 °C respectively.

It has been shown above in section 6.2.2 that the pre-anneal implantation induced waveguide loss increased from < 1 dB/cm in the unimplanted waveguide to 5 dB/cm for an erbium dose of $5 \times 10^{14}$ ions/cm$^2$ and subsequently to a value that was too high to measure following implantation with a dose of $4.75 \times 10^{15}$ ions/cm$^2$. At doses up to and including $4.75 \times 10^{15}$ ions/cm$^2$ neither the ASG material fluorescence or the 1550 nm erbium fluorescence was measurable in the as implanted samples. It is likely that the low pump power density in the waveguide resulting from the high post implantation optical loss partially accounted for this, particularly in the case of the sample in which the optical loss was too high to measure. Secondly, implantation induced defects may create alternative non-radiative transitions by which excited erbium can return to the ground state. When the probability of non-radiative decay increases, the concentration of excited erbium is depleted, thereby shortening the fluorescence decay lifetime and lowering the fluorescence intensity. This may also contribute to the absence of fluorescence from the as-implanted samples.

Comparison of the optical loss of the as-annealed and as-implanted waveguides in section 6.2 shows that thermal annealing removes implantation induced defects. The increase in the fluorescence decay time with increasing annealing temperature may also be attributed to the removal of these defects, which has been observed previously. At temperatures of 900 °C and greater, which is similar to the decomposition temperature of the material, defects may form which involve the precipitation of erbium. This could account for the slight drop in the measured fluorescence decay lifetimes following 900 °C annealing.

It has been shown that the erbium fluorescence decay lifetime decreases with increasing erbium ion dose. This is because the interaction probability between
neighbouring erbium ions increases with erbium ion density.\textsuperscript{[4]} It is therefore likely that the observed apparent increase in fluorescence decay lifetime with increasing ion dose, from $5 \times 10^{14}$ ions/cm$^2$ to $4.75 \times 10^{15}$ ions/cm$^2$, is a manifestation of the 1100 nm ASG material fluorescence and not a true result. The level of the luminescence intensity obtained from the unimplanted waveguide and the annealed $5 \times 10^{14}$ ions/cm$^2$ dose waveguide are of a similar magnitude. This suggests that the fluorescence signal from the low dose sample contains a significant contribution from the broad band ASG fluorescence. Increasing the erbium dose to $4.75 \times 10^{15}$ ions/cm$^2$ resulted in an approximate ten fold increase in luminescence intensity. The measured fluorescence decay curve is the sum of both the short lifetime ASG fluorescence and the long lifetime erbium related fluorescence. It is probable that the low dose, $5 \times 10^{14}$ ions/cm$^2$, fluorescence decay curve is dominated by the fast decay ASG material fluorescence. This explains the short measured lifetime. However, as the erbium ion dose increases so does the erbium related luminescent intensity. Hence the ASG material fluorescence become less significant in determining the measured lifetime, i.e. the long lifetime erbium fluorescence begins to dominate the ASG fluorescence. This is probably the reason why the apparent fluorescence decay lifetime increases with implanted erbium dose (up to $4.75 \times 10^{15}$ ions/cm$^2$).

An erbium dose of $4.75 \times 10^{15}$ ions/cm$^2$ corresponds to a peak erbium concentration of 0.12 at.%. At this level up-conversion does not play a major role in determining the fluorescence decay lifetime.\textsuperscript{[4]} It is therefore likely that the true erbium fluorescence decay lifetime is approximately constant for ion doses of $5 \times 10^{14}$ ions/cm$^2$ to and $4.75 \times 10^{15}$ ions/cm$^2$.

The results of figure 6.1 suggest that annealing at 800 °C for 1 hour optimizes the erbium fluorescence decay lifetime. It has been shown in section 6.2.2 that the implantation induced waveguide loss is returned to the pre-implantation value following this anneal. The samples implanted with erbium doses ranging between $2 \times 10^{16}$ ions/cm$^2$ and $5 \times 10^{17}$ ions/cm$^2$ were annealed for one hour at 800 °C. The post anneal fluorescence decay lifetimes of all the single energy implant samples are
shown in figure 6.2. Pre-anneal fluorescence was obtained from the samples implanted with erbium doses ranging from $2 \times 10^{16}$ ions/cm$^2$ to $5 \times 10^{17}$ ions/cm$^2$; the lifetimes of these samples are also indicated in figure 6.2. All lifetimes shown in figure 6.2 were obtained using a pump power of 1.5 W and a pump wavelength of 514 nm.

For an erbium dose of $2 \times 10^{16}$ ions/cm$^2$, the as-implanted fluorescence lifetime was measured to be $\sim 6.5$ ms which increased to 8.1 ms after annealing at 800 °C for 1 hour. Annealing for further periods and at higher temperatures (up to 900 °C) had no effect on the measured fluorescence lifetime. For an ion dose of $5 \times 10^{16}$ ions/cm$^2$ annealing the sample at 800 °C for one hour increased the as implanted fluorescence decay lifetime from 3.5 ms to 6.7 ms. Annealing further at 800 °C had little effect, while annealing at 900 °C for a further hour increased the measured decay lifetime by 1 ms. Samples implanted with erbium ion doses of $1 \times 10^{17}$ ions/cm$^2$ and $5 \times 10^{17}$ ions/cm$^2$ showed as-implanted fluorescence decay lifetimes of 5.0 ms and 4.5 ms respectively. These values increased to 5.9 ms and 5.3 ms respectively following an 800 °C one hour anneal. Annealing further at 800 °C and at higher temperatures had no significant effect.

As described previously, ion implantation creates defects in the implanted structure, which possibly affect the available erbium transition energies and hence the fluorescence decay lifetimes. The increase in lifetime with annealing, in conjunction with the removal of the implantation induced excess waveguide loss in the lower dose samples, indicates that this is the case and that these defects are at least partially removed during the anneal process.

The annealing of higher dose samples during implantation could account for the observed as-implanted sample fluorescence. The increase in the fluorescence lifetime of these samples with doses $\geq 2 \times 10^{17}$ ions/cm$^2$ after further annealing suggests that implantation induced annealing was not sufficient to minimize the concentration of beam induced defects.
The increasing fluorescence decay lifetime after the 800 °C anneal, with ion doses up
to an erbium level of 0.12 at. % (4.75x10^15 ions/cm²) are shown in figure 6.2. At
erbium doses lower than 4.75x10^15 ions/cm² up-conversion is not significant. As
previously explained, the increase in lifetime may be due to the increasing dominance
of the erbium fluorescence over the ASG material fluorescence. However, as the dose
rises to 5x10^17 ions/cm², the measured lifetime falls from 8.8 ms to 5.3 ms whilst the
luminescent intensity remains significantly higher (by at least a factor of 10) when
compared to the unimplanted material. This suggests that this is a true effect related
to the erbium concentration and not an effect resulting from the ASG material
fluorescence. A dose of 5x10^17 ions/cm² corresponds to a peak erbium concentration
of 13 at. %, a concentration where up-conversion is thought to play a significant role
in reducing the erbium fluorescence decay lifetime. The effect of up-conversion is
discussed further in section 6.3.3.

Although relative luminescent intensities between samples have been discussed, no
numerical comparisons are given. This is because no provision was made to keep the
detector to waveguide separation constant, which was approximately 5 mm. Hence
there is an element of inaccuracy in comparing the absolute luminescent intensity of
the different samples.

Comparison of identical waveguides showed the measured fluorescence decay lifetime
to vary by no more than < 0.5 ms. The response time of the system, which is
dependant on the electronics, the chopper and the detector, was measured to be < 0.5
ms.

6.3.3 Fluorescence as a Function of Pump Power

On excitation, a visible green fluorescence was emitted from the erbium doped
waveguides. The intensity of this green fluorescence is representative of the
magnitude of the up-conversion process. The fluorescence, which was viewed
through a narrow band stop filter to remove the 514 nm pump light, was present to
a varying extent in all of the implanted samples indicating that up-conversion was taking place. Although the intensity of this fluorescence was not measured, it gives a useful representation of the significance of the up-conversion process and is also a good indication that the waveguide is correctly aligned. In both the annealed and the as-implanted samples with the lowest dose \(5 \times 10^{16}\) ions/cm\(^2\), the fluorescence was barely visible. It could be clearly seen in the annealed and as-implanted sample with an erbium dose of \(5 \times 10^{17}\) ions/cm\(^2\). This visual check confirms that with increasing ion dose, up-conversion takes an increasingly predominant role in determining the erbium fluorescence lifetime.

The log\(_e\) differential of the fluorescence lifetime decay curve, as shown in section 3.6, can be used to measure the fast up-conversion lifetime decay component. The intensity of the fluorescence signal emitted from even the highest dose erbium implanted sample was very low, giving a low signal level and high signal to noise ratio. Differentiation of this data amplified the noise to a level of similar magnitude to the signal data, preventing the reliable use of this method.

The erbium fluorescence decay lifetime as a function of 514 nm pump power in the range 100 mW to 1500 mW was measured in 100 mW steps for all the implanted samples. The fluorescence decay lifetime as a function of the pump beam optical power gives a useful measurement of the level of up-conversion. In conjunction with the fluorescence decay lifetime the relative sample luminescence intensity as a function of pump power was also measured. The sample luminescence intensity was found to vary linearly with optical pump power suggesting that the fluorescence output was not saturated. To check for stable waveguide alignment, abrupt changes in the fluorescence intensity as a function of pump power were used as an indication that waveguide misalignment had occurred. If this was found to be the case the experiment was repeated.

The measured fluorescence decay lifetime as a function of 514 nm pump power is shown in figure 6.3. Lines (A) to (D) represent the fluorescence decay time as a
function of beam power for the indicated erbium doses. The gradient to the best straight line fit to each data set has been plotted in figure 6.4 and is represented as a function of the erbium dose, with the error in gradient being ± 10%. The data shown in figure 6.4 covers the ion dose range $2 \times 10^{16}$ ions/cm$^2$ to $5 \times 10^{17}$ ions/cm$^2$. Below this dose level no variation in fluorescence intensity with laser pump power was observed. This suggests that up-conversion does not take place to a measurable extent in samples implanted with ion doses less than $2 \times 10^{16}$ ions/cm$^2$.

The up-conversion component of the 1550 nm fluorescence decay time as a function of erbium concentration was described by Ainslie et al. The data reported in [4] was based on phosphorus doped silica glass which may not show the same optical behaviour as ASG glass. At Er doping levels approximately equivalent to $4.75 \times 10^{15}$ ions/cm$^2$, the fast up-conversion related component of the fluorescence decay was reported to be 8.7 ms. The slow component was 10.3 ms. This suggests that at doping levels of $4.75 \times 10^{15}$ ions/cm$^2$ up-conversion is not dominant in determining the fluorescence lifetime. It is therefore not surprising that the fluorescence lifetime of the low dose Er implanted ASG waveguides is independent of the optical pump power.

Referring to the data presented by Ainslie et al. the higher Er dose samples ($\geq 2 \times 10^{16}$ ions/cm$^2$) contain erbium concentrations likely to promote up-conversion. The fast up-conversion related fluorescence decay component reported with erbium concentrations equivalent to a dose of $\sim 1 \times 10^{16}$ ions/cm$^2$ was 4.5 ms, while the slow component was 9.5 ms. Figure 6.4 shows the trend of increasing fluorescence lifetime pump power dependence with ion dose observed in the Er implanted ASG samples. It is possible that the lower pump power dependence of the $1 \times 10^{16}$ ions/cm$^2$ sample can be attributed to experimental error. This would conform with the conclusion that the fall in measured decay lifetime from 8.1 ms at a dose of $2 \times 10^{16}$ ions/cm$^2$ to 5.3 ms at a dose of $5 \times 10^{17}$ ions/cm$^2$ is related to co-operative up-conversion.
6.3.4 Multiple Implantation

Figures 6.3 and 6.4 also show data corresponding to the multiple energy implant sample (see section 6.1.1.). For a pump power of 1500 mW, the fluorescence decay time of the $2 \times 10^{16}$ ions/cm$^2$ sample is 8.1 ms. This compares to the 10 ms lifetime of the multiple energy implant sample. Figure 6.4 shows that up-conversion is significantly less dominant in the multiple implant sample when compared to the equivalent dose single energy implant sample. This is indicated by the longer fluorescence lifetime of the multiple implant sample and the low dependence of lifetime on the pump power level. This could be because multiple implantation gives a lower localized erbium concentration in comparison to a single energy implant of the same total dose. The decrease in erbium concentration reduces the interactions between neighbouring erbium atoms and hence increases the fluorescence decay lifetime. The erbium profile of the multiple energy implantation sample is the sum of the three implant profiles shown in figure 4.5.

6.3.5 Comparison of Er in ASG and Phosphorus Waveguides

The fluorescence decay lifetime of a phosphorus doped silica waveguide of Er concentration 2 at. % over the entire volume of the waveguide which was produced by the BT Labs was found to be ~ 4 ms for a pump power of 1500 mW. The lifetime measured from an ASG waveguide of implantation dose $1 \times 10^{17}$ ions/cm$^2$ (peak concentration 2.6 at. %) was 5.9 ms (pump power = 1500 mW). These results would indicate that longer lifetimes are obtainable from Er implanted ASG in comparison to conventional phosphorus doped silica waveguides.

6.4 Optical Transmission Spectra

The reported 1540 nm spectral absorption of a 0.72 at. % silica waveguide was ~ 0.8 dB/cm. Measurement of a 2 at. % Er doped phosphorus doped silica waveguide supplied by the BT Labs showed a 1550 nm absorption of 2.4 dB/cm. Assuming that
at these Er doses the strength of the 1550 nm absorption is proportional to the erbium content of the waveguide, then this data indicates that the 1550 nm optical absorption of an erbium doped silica waveguide is approximately 1.1 dB/cm per 1 at. % of Er dopant. This is only a rough approximation and may not be valid over large variations in Er concentration. The overlap between the implanted Er profile and the ASG waveguide fundamental vertical optical mode is 23 % at λ=1500 nm. This means that the measured optical absorption of an implanted waveguide will be only 23 % of the absorption measured in a uniformly doped guide, i.e. those doped during growth. This data would indicate an expected 1540 nm erbium absorption in the region of 3.3 dB/cm for the highest dose implant (5x10^{17} ions/cm^2), while at the lower dose implant of 1x10^{17} ions/cm^2 the expected absorption would be 0.66 dB/cm. Given the relatively high noise level of the system which is in excess of 1 dB/cm, it is unlikely that the absorption spectra of the waveguide implanted with the dose of 1x10^{17} ions/cm^2 will be visible.

The white light absorption spectra of the sample implanted with 5x10^{17} erbium ions/cm^2 is shown in figure 6.5. No erbium related optical absorption that could be distinguished from the background noise was visible for lower dose samples. The baseline of the spectra is adjusted to zero and the absorption levels are given in dB/cm. Figure 6.5 shows an erbium absorption level of between 2 dB/cm and 4 dB/cm, a result that is consistent with the above estimations.

The measurement of the absorption spectra required very careful alignment between the waveguide and launch fibre. Small differences in alignment could mean the difference between obtaining the spectra and a flat line scan. This was due to the small guiding layer thickness of 2 μm which gives a small mode overlap, as discussed above. It is probable that precise adjustment of the launch fibre was required to achieve a maximum interaction with the region of implanted erbium and the 1500 nm optical mode profile. When performing the spectral scans it could not be determined when, or if, the optical launch conditions were optimized until the scan was complete. Although in figure 6.5 an absorption is clearly present, the results can only be
considered an indication of the true spectrum due to the high noise levels and difficulty in obtaining the data.

6.5 Summary of Results

The ASG waveguides were implanted with 2.7 MeV Er\textsuperscript{**+} ions. The erbium doses ranged between $4.75\times10^{13}$ ions/cm\textsuperscript{2} (0.013 at. %) to $5\times10^{17}$ ions/cm\textsuperscript{2} (13 at. %). A single sample was implanted with erbium doses of $0.5\times10^{16}$ ions/cm\textsuperscript{2}, $1.0\times10^{16}$ ions/cm\textsuperscript{2} and $0.5\times10^{16}$ ions/cm\textsuperscript{2} using ion energies of 4.00 MeV, 2.70 MeV and 1.35 MeV respectively. This increased the width of the implanted erbium profile from 0.54 µm to 1.42 µm when compared to a single energy 2.70 MeV erbium implant. Following implantation, the waveguides were annealed at temperatures between 500°C and 900°C for periods of up to two hours. The waveguide propagation loss, the 1550 nm fluorescence decay lifetime, and the waveguide white light transmission spectrum were measured in the as grown, the implanted, and the annealed waveguides.

The 1526 nm optical propagation loss of the as grown waveguide was measured to be < 1 dB/cm. Following erbium implantation using a dose of $5\times10^{14}$ ions/cm\textsuperscript{2} the optical propagation loss increased to 5 dB/cm. The optical propagation loss following an erbium dose of $4.75\times10^{15}$ ions/cm\textsuperscript{2} could not be measured. This was due to the low level of the waveguide output optical power. However, implantation using erbium doses of $5\times10^{16}$ ions/cm\textsuperscript{2} and $5\times10^{17}$ ions/cm\textsuperscript{2} had no measurable effect on the waveguide propagation loss. After an anneal period of 1 hour at a temperature of 800°C the waveguide optical propagation loss for all the implanted waveguides was restored to that of the as grown waveguide.

Using a pump wavelength of 514 nm, the unimplanted waveguide showed a broad band 1100 nm florescence of lifetime < 1 ms. At erbium ion doses up to $4.75\times10^{15}$ ions/cm\textsuperscript{2}, reliable determination of the erbium related fluorescence was prevented by the domination of the 1100 nm ASG florescence. No fluorescence was observed for the as implanted samples with doses ≤ $4.75\times10^{15}$ ions/cm\textsuperscript{2}. The measured erbium
fluorescence lifetime of the as implanted $2 \times 10^{16}$ ions/cm$^2$ dose sample was 6.5 ms, this decreased to 4.5 ms for an implantation dose of $5 \times 10^{17}$ ions/cm$^2$. The maximum lifetimes were measured following an 800 °C anneal. The fluorescence lifetimes of the annealed $4.75 \times 10^{15}$ ions/cm$^2$ and $5.0 \times 10^{17}$ ions/cm$^2$ dose samples were 8.8 ms and 5.35 ms respectively. Using a single energy 2.7 MeV implant, the fluorescence lifetime of the annealed $2 \times 10^{16}$ ions/cm$^2$ dose sample was 8.1 ms. The measured lifetime of the annealed multiple energy implant sample, of total dose $2 \times 10^{16}$ ions/cm$^2$, was $\sim 10$ ms. All of these lifetimes were measured using a 514 nm optical pump power of 1500 mW. Decreasing the pump power to 50 mW increased the lifetime of the $5 \times 10^{17}$ dose sample by $\sim 1.5$ ms, the lifetime of the $2 \times 10^{16}$ ions/cm$^2$ dose sample by 0.9 ms and the lifetime of the multiple energy implant sample by $\sim 0.1$ ms. The characteristic erbium optical absorption spectra was only visible in the 800 °C annealed, $5 \times 10^{17}$ ions/cm$^2$ dose sample. The results indicated an approximate 1550 nm optical absorption of between 2 dB/cm to 4 dB/cm.
Figure 6.1. The fluorescence decay lifetime as a function of anneal temperature and duration for Er doses of
(A) 5x10^{-14} ions/cm^2, (B) 1x10^{-15} ions/cm^2, (C) 2.5x10^{-15} ions/cm^2 and (D) 4.75x10^{-15} ions/cm^2.
Figure 6.2. The fluorescent lifetime of Er implanted silica before and after annealing. The doses are (A) 5x10^{14}, (B) 1x10^{15}, (C) 2.5x10^{15}, (D) 4.75x10^{15}, (E) 2x10^{16}, (F) 5x10^{16}, (G) 1x10^{17} and (H) 5x10^{17} ions/cm².
Figure 6.3. Erbium fluorescent lifetime as a function of pump power for doses of (A) $2 \times 10^{16}$, (B) $5 \times 10^{16}$, (C) $1 \times 10^{17}$, (D) $5 \times 10^{17}$ and (E) the multiple implantation sample of total erbium dose $= 2 \times 10^{16}$ ions/cm$^2$. 

Chapter 6 : Page 141
Figure 6.4. Pump power dependence of fluorescent lifetimes for erbium doses of (A) the multiple implant sample, (B) $2 \times 10^{16}$, (C) $5 \times 10^{16}$, (D) $1 \times 10^{17}$ and (E) $5 \times 10^{17}$ ions/cm$^2$. 

Chapter 6 : Page 142
Figure 6.5. The white light transmission spectra of the ASG waveguide implanted with a 2.7 MeV erbium dose of $5 \times 10^{17}$ ions/cm$^2$. 

Chapter 6 : Page 143
6.6 References.


Conclusions and Future Work

7.0 Introduction

Section 7.1 contains the conclusions reached and sections 7.2 and 7.3 give details of future work required to develop the project further.

7.1 Conclusions

7.1.1 Photosensitivity in Germania Doped Hydrogen Implanted Silica

The thermal hydrogenation of germania doped silica structures promotes the growth of an optical absorption band at 240 nm. This is due to the reducing action of hydrogen on germanium oxide to form Ge-Ge and Ge-Si bonds. The bleaching effect of UV light can then be used to break the Ge-Ge and Ge-Si bonds to form the Ge-E' defect, which has a peak optical absorption wavelength centred at 213 nm. As the material undergoes changes in the ultraviolet optical absorption spectrum the infrared refractive index of the material also changes, which is known as photosensitivity. This is because the Kramers-Kronig relationship shows that the infrared refractive index of a material is partially dependant on the ultraviolet transmission spectrum of the material.

The results of room temperature proton implantation highlight two important conclusions. Firstly, room temperature proton implantation does not result in the formation of the desired Ge-Ge bonds. During the thermal hydrogenation process, oxygen is removed from the germanium oxide molecules allowing Ge-Ge bonds to form. In contrast to this, room temperature implantation leads directly to the formation of Ge-E' defects. This would suggest that the high temperatures required
by the thermal hydrogenation process, which are needed to form the Ge-Ge bonds, are also required during proton implantation, as was found. Secondly, the growth in 213 nm optical absorption observed in the sample implanted with a proton dose of \(1 \times 10^{17}\) ions/cm\(^2\) is comparatively small when compared to the change in 240 nm optical absorption observed in the thermal hydrogenation sample. This result would also suggest that higher sample temperatures are required to encourage the growth in the optical absorption levels.

The heated stage implants show that high temperature implantation promotes the same process as observed during the thermal hydrogenation process. The observed growth in the concentration of Ge-Ge defects suggest that the sample will be photosensitive. However, the level of the 240 nm optical absorption in the hydrogen implanted sample is significantly lower than the 240 nm optical absorption in the thermal hydrogenation sample, which was measured to be in excess of 95%. It is possible that varying the concentration of the Ge doping in the implanted sample may increase the measured growth in the 240 nm optical absorption.

Annealing of the implanted and as-grown samples in the nitrogen/hydrogen atmosphere demonstrates that the Ge-\(E'_1\) defects are removed by annealing, and that proton implantation prior to thermal hydrogenation offers no advantage. A consequence of this result is that Bragg gratings fabricated in these materials are not suited to high temperature applications.

These experiments suggest that the fabrication of a photosensitive material in hydrogen implanted germanium doped silica is possible. This was demonstrated by the exposure of the hydrogen implanted sample to the excimer laser, a 2% drop in the sample 240 nm optical absorption levels was observed following exposure to the laser beam. However, the photosensitivity observed in the hydrogen implanted samples is likely to be significantly less than that observed in samples heated in a hydrogen atmosphere. This is because the results have demonstrated that significantly less Ge-Ge bonds are formed during hydrogen implantation when compared to hydrogen thermal indiffusion.
Finally, it has been demonstrated that the optimum growth in 240 nm optical absorption in 7 wt. % germania doped silica is obtained using proton implantation doses of $1 \times 10^{17}$ ions/cm$^2$ into samples heated to temperatures of approximately 800°C.

7.1.2 The Optical Properties of Erbium Implanted Arsenic Doped Silica

To a varying extent the optical propagation loss of the ASG waveguides was increased following implantation. However, in all cases, thermal annealing at 800°C for 1 hour restored the 1536 nm optical propagation loss to the pre-implantation value of $< 1$ dB/cm. This indicated the formation of defects during implantation which were subsequently removed by thermal annealing. In samples with Er doses $\geq 5 \times 10^{16}$ ions/cm$^2$ the waveguide optical propagation loss was unaffected by implantation and it is likely that beam heating of the samples during implantation accounted for this.

In the case of all the implants, the measured waveguide optical propagation loss was the same in the 800°C annealed implanted samples as the optical loss in the as-grown samples. This is an important conclusion as it shows that implantation induced damage can be removed and the waveguide loss returned to the pre-implantation value. In the optical amplifier the available amplification must be greater than the waveguide optical propagation loss, without this condition gain is not possible. It is therefore important that the waveguide loss is low and is not significantly affected by implantation.

The unimplanted ASG material was found to fluoresce at a peak wavelength of 1100 nm with a fluorescence decay lifetime of $< 1$ms. The ASG waveguides were implanted with erbium doses ranging between $4.75 \times 10^{15}$ ions/cm$^2$ (0.013 at. Er %) and $5 \times 10^{17}$ ions/cm$^2$ (13 at. Er %). At erbium doses $\leq 4.75 \times 10^{15}$ ions/cm$^2$ (0.12 at. %) the ASG material fluorescence was significantly more intense than the 1550 nm erbium fluorescence. This prevented reliable determination of the erbium fluorescence decay lifetime for erbium doses $\leq 4.75 \times 10^{15}$ ions/cm$^2$. For the highest implanted erbium dose ($5 \times 10^{17}$ ions/cm$^2$) the 1550 nm erbium fluorescence was $> 100$ times more intense.
intense than the ASG fluorescence. Practical stripe waveguide amplifier applications require erbium concentrations in excess of 4.75x10^{15} ions/cm^2 because at doses lower than 4.75x10^{15} ions/cm^2 the erbium related spectral absorption, and therefore any available gain, is very low. The difficulty in obtaining the erbium fluorescence lifetimes at doses lower than this was therefore not important.

Before annealing, no fluorescence was observed in samples implanted with erbium doses less than 2x10^{16} ions/cm^2. This was not the case for the higher dose samples. For these higher dose samples, post implantation annealing increased the measured fluorescence decay lifetime by as much as 3 ms. It is possible that implantation induced defects may interact with the erbium fluorescence decay mechanism by offering alternative non-radiative decay mechanisms. The removal of these defects by annealing can account for the observed increase in the fluorescence decay lifetime. The effect of ion beam heating, which anneals the samples during implantation may account for the fluorescence observed in the higher dose as-implanted samples.

For the samples annealed at 800°C for 1 hour, increasing the ion dose from 4.75x10^{15} ions/cm^2 to 5x10^{17} ions/cm^2 resulted in a fluorescence lifetime decrease from 8.8 ms to 5.3 ms. Variation of the optical pump power was found to have no effect on the erbium fluorescence lifetime for samples implanted with doses \leq 4.75x10^{15} ions/cm^2. In the case of the sample implanted with 5x10^{17} ions/cm^2, the fluorescence decay lifetime was seen to decrease from 6.8 ms to 5.3 ms when the optical pump power was increased from 50 mW to 1500 mW. A green fluorescence, which was attributed to up-conversion, was visible from all the samples and the intensity of which increased with increasing ion dose; the green 514 pump beam was removed by filtering. These results demonstrate the increasing dominance of up-conversion with increasing ion dose.

To increase the overlap between the implanted erbium and the waveguide vertical fundamental optical mode profile, multiple energy implantation was used. A triple energy implant with a total dose of 2x10^{16} ions/cm^2 was carried out into the same
sample. Comparison of the multiple energy implant sample with the single energy implant sample of the same total erbium dose, showed a lifetime increase of 2 ms for the multiple energy implant sample. This is because of the lower erbium concentration in the multiple energy implantation sample when compared to the single energy implant sample. The 514 nm pump power dependence of the erbium fluorescence lifetime in this sample was over three times lower than the lifetime pump power dependence of the 2×10^{16} ions/cm^2 single energy implant sample. These results demonstrate that the technique of multiple energy implantation significantly reduces the effect of co-operative up-conversion.

The results show that implantation of erbium into ASG yields fluorescence lifetimes that are suitable for the fabrication of optical amplifiers. The measured erbium fluorescence lifetimes in the annealed samples ranged from 10.1 ms to 5.3 ms. These values are comparable to, if not slightly better than, those reported in glasses other than ASG. For example, Lidgard et al demonstrated an erbium fluorescence lifetimes of 6.7 ms from annealed silica glass implanted with an erbium dose of 3.4×10^{16} ions/cm^2. The measured erbium fluorescence lifetime of the annealed ASG implanted with a dose of 2×10^{16} ions/cm^2 was 8.1 ms.

Measurement of the white light transmission spectra of the sample implanted with an erbium dose of 5×10^{17} ions/cm^2 indicated a 1550 nm optical absorption of magnitude between 2 dB/cm and 4 dB/cm.

Reported optical amplifiers have operated with an erbium 1550 nm optical absorption level in the region of 8 dB/cm. In comparison, the ASG waveguide implanted with a single energy implant erbium dose of 5×10^{17} ions/cm^2 showed a 1550 nm optical absorption level of between 2 dB/cm to 4 dB/cm. Increasing the single implant erbium dose further in the ASG waveguides to reach absorption levels of 8 dB/cm is not a practical proposition. This is because the fluorescence lifetime will be decreased by co-operative up-conversion. In the ASG samples, co-operative up-conversion decreased the lifetime from over 10 ms at lower doses to 5.3 ms at a
dose of $5 \times 10^{17}$ ions/cm$^2$. A more favourable option is the use of multiple energy implantation. By implanting the same sample with three or more different ion energies, each with a dose of $5 \times 10^{17}$ ions/cm$^2$, a uniform erbium distribution could be achieved throughout the waveguide core. This would allow high 1550 nm Er optical absorption levels whilst still maintaining the longer fluorescence decay lifetimes.

In summary, it has been demonstrated for the first time that erbium implanted ASG is a possible candidate for the fabrication of waveguide optical amplifiers. It has been shown that useful 1550 nm fluorescence decay lifetimes are obtained from the implanted erbium doped ASG structures and that the fluorescence lifetimes remain useful at high erbium doses. This is an important conclusion as the results also suggest that high erbium doses are required before the 1550 nm erbium related optical absorption reaches a useful level. If the high doses required to achieve a good 1550 nm optical absorption reduced the erbium fluorescence decay lifetimes below a useful threshold then amplification in ASG waveguides would not be a practical proposition. The results also show that the technique of multiple energy implantation is beneficial. By implanting erbium into the ASG structures at several different beam energies, a lower erbium density whilst maintaining a higher total dose, in comparison to single energy implantation, is possible. It has also demonstrated that the annealing cycle required to optimise the erbium fluorescence decay lifetime is sufficient to remove any measurable effect of implantation damage on the implanted waveguide optical propagation loss. This is extremely important as a high waveguide propagation loss leads to an inefficient amplifier device.

Finally, during implantation it is important that the penetration depth of the implantation beam is comparable to the waveguide height. This is so that a good match can be achieved between the implanted erbium profile and the waveguide optical mode profile. Due to the tight optical confinement available from ASG waveguides, the waveguide height required for single mode 1550 nm waveguiding is 2 μm. In comparison, single mode silica waveguides are typically 7 μm high. Consequently, the potential for the ion beam fabrication of optical amplifiers in
ASG is extremely useful. This is because erbium implantation to a depth of 2 μm in ASG requires a lower beam energy, and hence is a more commercially viable process, than implantation to a depth of 7 μm in silica glass.

7.2 Future Work

7.2.1 Photosensitivity

The 7 wt. % germania doped silica samples fabricated by the BT laboratories were used for the work described above. Although they are suitable for the thermal indiffusion of hydrogen, 7 wt. % Ge may not be ideal in the case of hydrogen implantation. It would therefore be useful to vary the germanium content of the glass and observe the effect on the absorption spectra of hydrogen implanted samples.

The experiments discussed above were carried out on planar waveguides. It would be useful to implant stripe waveguides under the same conditions and measure the implantation induced optical propagation loss. It is important that the implantation induced loss can be removed. However, this is unlikely to present a problem as it is probable that the high temperature required (800 °C) to enhance the sample photosensitivity will remove the implantation induced waveguide propagation loss.

In the work described it is assumed that changes in the sample UV absorption are representative of changes in the sample infrared refractive index. As this has been assumed and not proven it would be worthwhile to measure the sample refractive index before and after implantation and after optical exposure.

Using the optimum glass composition and implantation parameters it would be useful to write a Bragg grating into a stripe waveguide structure, and to compare the characteristics of the grating to those in samples made photosensitive by the thermal indiffusion of hydrogen.
7.2.2 Erbium Implantation

Although multiple energy implantation was shown to be of benefit, further experiments are required to gain the maximum advantage from this technique. Implantation was described using energies of 1.35 Mev, 2.7 MeV and 4.0 MeV. This gave a 61% overlap between the implanted region of waveguide and the waveguide vertical optical mode profile ($\lambda$=1500 nm). By implanting at both higher and lower energies the volume of implanted sample could be further enhanced. For the multiple energy implant sample the implanted erbium doses were $5 \times 10^{15}$ ions/cm$^2$, $2 \times 10^{16}$ ions/cm$^2$ and $5 \times 10^{15}$ ions/cm$^2$ (at beam energies of 1.35 MeV, 2.0 MeV and 4.00 MeV). The peak ion concentration obtained using an implantation dose of $2 \times 10^{16}$ ions/cm$^2$ was 0.5 at. %. It would be useful to increase the dose of each implant since results from the single energy implant samples showed that useful erbium fluorescent decay lifetimes could be obtained at higher doses. Together with modifying the implantation energies (as described above) this technique may result in a stronger erbium spectral absorption and therefore more useful optical amplifier structures.

In obtaining the ASG waveguide spectral absorption curve the main difficulty encountered was the low optical intensity from the monochromator output. Exchange of the InGaAs photodetector for a cooled photodetector to improve noise and sensitivity is one possible solution. Secondly it may be useful to consider a brighter white light source. For example would an LED be more useful than a halogen bulb and if so are LED's available with the required spectral range?

Together with 514 nm, other possible pump wavelengths used for assessing the erbium fluorescent decay lifetime include 800 nm, 980 nm and 1480 nm. The behaviour of the fluorescent lifetime decay may vary with the pump wavelength. It would therefore be useful to perform measurements at 800 nm, 980 nm and 1480 nm. These pump wavelengths are available from semiconductor lasers and are preferentially chosen for driving erbium amplifiers.
The use of the diamond saw, by the BT Labs, to fabricate of the ASG stripe waveguides was chosen here for convenience rather than quality. Since these waveguides were required only to assess the erbium fluorescent lifetime, the erbium absorption spectrum and the effect of implantation on waveguide optical loss, the quality of the guides was not important. However, to produce amplifier devices, the fabrication of high quality waveguides using flame hydrolysis and ion etching would be required.

7.3 References
