Characterisation of Stripe Optical Waveguides Fabricated by Silicon Impurity Induced Disordering of GaAs/AlGaAs MQW Material

A.C. Wismayer

1991

A thesis presented in partial fulfilment of the requirements of the degree of Doctor of Philosophy in the Department of Electronic and Electrical Engineering, University of Surrey, Guildford, Surrey, U.K.

© A.C. Wismayer 1991
ABSTRACT

Impurity Induced Layer Disordering (IILD) of a GaAs/AlGaAs Multi Quantum Well (MQW) structure is known to produce a change in the refractive index dependent on the polarisation of the propagating mode. This index change can be used to provide lateral confinement in stripe optical waveguides, which are an essential component in any integrated optical circuit.

Silicon implantation has been used to disorder GaAs/AlGaAs MQWs and the effects of implantation dose, encapsulant and annealing conditions on the disordering process have been investigated using Photoluminescence (PL). It was observed that deep level emissions accompanied the disordering and the results suggest that the degree of intermixing and the deep level emissions were determined by several competing processes. Calculation of the deep level/band-edge integrated intensity ratio for these emissions, provided an indication of the suitability of the process for the fabrication of disorder delineated stripe waveguides, where a correlation between the propagation loss of the waveguides and the integrated intensity ratio was observed.

Buried stripe optical waveguides fabricated using Si IILD has been demonstrated for the first time in this work. Waveguide assessment using end-fire coupling was performed with the propagation losses and modal dimensions determined. The lowest loss of a waveguide fabricated by IILD presented in this thesis is 21.9dB/cm and it is suggested that the deep level states observed using PL are a significant source of attenuation of the propagating mode in the side walls of the waveguides.
ACKNOWLEDGEMENTS

This work was supported by SERC (U.K.).

The author wishes to express his gratitude to his supervisor, Dr. B.L. Weiss, for his guidance and support throughout the course of this research; to the members of the Solid State Devices and Ion Beam Technology Group for the numerous useful discussions; to the staff of the D.R. Chick Accelerator Laboratory and to the technical staff of the Department of Electronic and Electrical Engineering at the University of Surrey who have helped in the progress of this work.

Finally, the author would like to express his gratitude to Linda for her help and encouragement during the course of this study.
PUBLICATIONS

Parts of this work have been published elsewhere.

A.C. Wismayer and B.L. Weiss. 'The Etching of Al$_{0.3}$Ga$_{0.7}$As Using KI-I$_2$', Mat. Lett. 6, 284, 1988


B.L. Weiss, A.C. Wismayer and J.S. Roberts. 'Disorder-Induced Buried Stripe Optical Waveguides in GaAs/AlGaAs MQW Material', Electron. Lett. 25, 653, 1989

## CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Abstract</td>
<td>i</td>
</tr>
<tr>
<td>Acknowledgements</td>
<td>ii</td>
</tr>
<tr>
<td>Publications</td>
<td>iii</td>
</tr>
<tr>
<td>Contents</td>
<td>iv</td>
</tr>
<tr>
<td><strong>Chapter 1: Introduction</strong></td>
<td>1</td>
</tr>
<tr>
<td>1.1 Introduction</td>
<td></td>
</tr>
<tr>
<td>1.2 Integrated Optics</td>
<td></td>
</tr>
<tr>
<td>1.2.1 III-V Semiconductors in Integrated Optics</td>
<td></td>
</tr>
<tr>
<td>1.2.2 Applications of Multi-Quantum- Wells in Integrated Optics</td>
<td></td>
</tr>
<tr>
<td>References</td>
<td>9</td>
</tr>
<tr>
<td><strong>Chapter 2: Review</strong></td>
<td>12</td>
</tr>
<tr>
<td>2.1 Introduction</td>
<td></td>
</tr>
<tr>
<td>2.2 Properties of GaAs and AlGaAs</td>
<td></td>
</tr>
<tr>
<td>2.3 Multi-Quantum-Well Structures</td>
<td></td>
</tr>
<tr>
<td>2.4 MQW Waveguiding</td>
<td></td>
</tr>
<tr>
<td>2.4.1 Free Carrier Effects</td>
<td></td>
</tr>
<tr>
<td>2.5 IILD in GaAs/AlGaAs MQW Structures</td>
<td></td>
</tr>
<tr>
<td>2.6 Ion Implantation</td>
<td></td>
</tr>
</tbody>
</table>
2.7 Photoluminescence Characterisation of MQW Structures

2.8 Optical Waveguide Theory

References

Chapter 3: Sample Preparation Techniques

3.1 Introduction

3.2 Material Specification

3.3 Sample Processing Procedures

3.3.1 Sample Cleaning

3.3.2 Ion Implantation

3.3.3 Encapsulation Procedure

3.3.3.1 Silicon Nitride

3.3.3.2 Aluminium Nitride

3.3.3.3 Dual Encapsulant: Si₃N₄/AlN

3.3.4 Annealing Techniques

3.3.4.1 Furnace Annealing

3.3.4.2 Dual Graphite Strip Annealing

3.4 MQW Stripe Waveguide Fabrication

3.4.1 Photolithography

3.4.2 Thinning and Cleaving

3.4.3 Characterisation of Etchants

References
Chapter 8: Conclusions and Recommendations
for Future Work

8.1 Introduction
8.2 Conclusions
8.3 Recommendations for Future Work

References
CHAPTER 1

INTRODUCTION

1.1 Introduction

The concept of developing a communication system where signals are transmitted by propagating light through optically transparent mediums as opposed to electrical impulses down wires is not a recent one. Indeed as early as 1880 Alexander Graham Bell reported the use of a light beam in speech transmission.\(^{[1,4]}\) It was not until the 1960's however that the idea started to gain today's present momentum with the development of the ruby laser\(^{[2,3]}\) and the glass optical fibre.\(^{[3,4]}\) The laser provided a source of intense coherent light with low beam divergence, whilst the optical fibre, whose losses were initially prohibitively high, have been reduced to the point where they now provide a low loss propagating medium.

With the realisation of low loss optical communications there was a resurgence of interest in the area of integrated optics (IO), with the aim of developing a microfabrication technology for single mode optical devices, initially using dielectrics and later semiconductors.
The work undertaken in IO can be divided into two broad categories. Firstly the possible hybrid integration of semiconductor lasers and detectors with dielectric switches and modulators, and secondly the monolithic integration of lasers, detectors and modulators using III-V semiconductors. More recently the operating wavelength of optical communications has moved from the visible to the near infrared part of the spectrum, due to the improved low loss performance of optical fibres at these wavelengths. This trend has led to IO development towards the monolithic integration offered by III-V semiconductors.

Considerable progress has been achieved in the use of III-V semiconductors using bulk layers for the fabrication of circuit elements. However, a substantial degree of additional flexibility has been achieved by the use of multiple thin layers of alternating low and high bandgap III-V semiconductors. This allows the "tailoring" of the bandgap, and hence the electrical and optical properties of the material to specific requirements. In such a structure the low and high bandgap layers are referred to as wells and barriers respectively, and the structure is known as a Multi-Quantum-Well (MQW). It has also been shown that it is possible to mix these wells and barriers, providing a further degree of flexibility, by the introduction of impurity atoms followed by annealing. This process may be combined with photolithographic techniques to achieve selective area modification of the material for device fabrication.

An essential component of an IO circuit is the waveguide which acts as the link between the source, eg. laser, and the detector. Ideally this link should be a low loss connection at the emission wavelength of the laser and research effort is being devoted to the design and fabrication of these components for optimum performance.
1.2 Integrated Optics

The advent of the idea of IO can be traced back to Anderson[1] who in 1965, proposed a technology for the integration of optical components along the same lines as that which had occurred for electronic devices. However, it was not until 1969 that the term "integrated optics" was first coined by Miller,[2] whilst reviewing the long term prospects for this developing technology, and this was the beginning of intensive effort to develop a reliable technology for the purpose of optical communications. Today, as then, the majority of data processing is achieved by electronic means, where the source signal is converted to an electrical output via a transducer with subsequent processing and transmission being achieved electronically.

Considerable progress has been made in electronic VLSI technology and the possibility of integrating miniaturised optical components is expected to offer the same advantage of lower cost due to batch fabrication, with the further advantages of an optical system of an increased data processing speed, higher frequency transmission and larger bandwidth communication systems.

Two types of optical integrated circuits (OIC) have emerged. Firstly the hybrid circuit in which two or more suitable materials are interfaced during integration, these often comprise a laser and detector fabricated in a suitable semiconductor with the switching or modulation being performed using a dielectric. These have the advantage of being able to be fabricated using existing technology and materials which can be chosen for optimum performance for the desired circuit component. The second type is the monolithic circuit which offers the possibility of integration on one substrate. Monolithic integration is only possible using materials from which
laser sources can be fabricated, such as the III-V semiconductors. It is the monolithic approach to fabricating OICs that has emerged as the forerunner, due in part to the elimination of the problems associated with the optical and mechanical coupling of various devices on a single substrate.

The majority of the initial work in the 1970’s using semiconductors was based on the GaAs and AlGaAs systems working at a wavelength ($\lambda$) of 0.85μm since this corresponded to a minimum in the optical absorption for the glasses commonly used in optical fibres. More recently effort has been concentrated on the development of semiconductor systems working at the longer wavelengths of 1.3μm and 1.55μm, the latter corresponding to the low loss optical windows in modern silica optical fibres.

The fundamental element in any optical integrated circuit is the optical waveguide. These structures have been fabricated in III-V semiconductors using a variety of techniques. The most popular device structure uses vertical confinement provided by compositional changes and lateral confinement by wet or dry etching to produce a ridge structure. This process being non planar has the disadvantage of being more sensitive to surface contamination than structures such as buried waveguides where lateral confinement can be achieved by ion implantation which, being an established microelectronics process, offers precision process control.

A more recent development in the field of III-V semiconductor optical system integration has been the use of MQWs, which have provided the opportunity to "engineer" the bandgap of the material, and hence its optical and electronic properties, by varying the layer thickness and composition. Further modification of the material parameters can be achieved by the mixing of these layers by long time, high temperature annealing, or impurity induced layer disordering (IILD), where the
impurity can be introduced during growth, by diffusion or ion implantation. This mixing of the quantum well layers has been seen to provide refractive index changes between the mixed and unmixed regions of up to 2.4% and therefore offers considerable potential for the fabrication of buried optical waveguides.

1.2.1 III-V Semiconductors in Integrated Optics

As previously stated, monolithic integration can only be achieved in materials in which a laser source can be fabricated. This has limited the choice to the III-V semiconductors, which are particularly useful because of the ability to vary the energy gap over the emission wavelengths of interest eg. 0.85µm - 1.55µm. To date, the most extensively investigated IOCs have been in the GaAs - Gaₙ,ₐAl,ₐAs system, where compositional variations can be achieved by changing the Al concentrations (value of x), between 0 and 1. This material system has an emission wavelength in the range of 0.65µm - 0.91µm, and is transparent between 0.6µm - 12µm, and because the lattice constants of GaAs and AlAs are very close there is negligible mismatch and interfacial strain.

Due to the early interest, numerous devices have been fabricated in the GaAs-AlGaAs system, which include low loss optical waveguides, directional couplers, lasers and modulators which include many of the devices required for optical integration. However, the number of devices that have been integrated onto one chip is very small in comparison to that achieved for electronic devices.

Initially the optical fibres used were glass based and had a minimum attenuation at 0.85µm, which suited the emission range of the GaAs-AlGaAs system. However,
another attenuation window also existed at 1.3\(\mu\)m with the added benefit that at this longer wavelength it coincided with a minimum in the dispersion, so that suitable sources were sought at these wavelengths. This has lead to the development of other III-V compound systems, such as InGaAsP/InP, where lasers which can operate at 1.3\(\mu\)m and 1.55\(\mu\)m have been fabricated.\(^{[149]}\)

A further increase in desired operating wavelength to 1.55\(\mu\)m has accompanied the introduction of the modern silica based optical fibres, where loss due to water absorption has been virtually eliminated and the principal loss mechanism is Rayleigh scattering which decreases according to \(\lambda^4\). Therefore, increasing the operation wavelength of the system from 1.3\(\mu\)m to 1.55\(\mu\)m halves the fibre loss. The last remnant of OH absorption occurs at 1.4\(\mu\)m and this basic absorption mechanism in silica fibres restricts the wavelength to less than 1.6\(\mu\)m. Consequently, losses in modern fibres of 0.2\(\text{dB/Km}^1\) have been achieved at 1.55\(\mu\)m\(^{[20]}\) and semiconductor circuits, which can operate at these wavelengths, are being developed.

### 1.2.2 Applications of Multi-Quantum-Wells in Integrated Optics

As stated previously, the sequential growth of thin layers of alternating high bandgap and low bandgap semiconductors causes a modification of the materials electrical and optical properties, from those of the bulk materials, when these layers are made sufficiently thin for them to be considered as quasi-two-dimensional (~100\(\AA\)), that is dimensions approaching the Bohr radius, with abrupt interfaces, they exhibit a number of unique optical and electrical characteristics. The initial research effort was confined to the GaAs/AlGaAs system, where the growth of single crystal multi-layers was first reported in 1976.\(^{[21]}\) Subsequent research has shown that MQW structures
have large room temperature nonlinearities\cite{1,23} and large electroabsorption effects.\cite{1,23}

These effects have lead to the development of various optoelectronic devices for ultrafast switching speeds, such as the bistable optical switch,\cite{1,24} the quantum confined stark modulator\cite{1,25} and the self electro-optic effect device.\cite{1,26}

Recently further developments have been made in the area of disordering of MQWs to produce intermixing of the layers. The mixing of a GaAs/AlGaAs MQW produces a region of AlGaAs whose Al concentration is the average for that of the MQW. This mixed region therefore has a larger energy gap and modified optical properties. It is these properties that have made this technique of particular interest and importance to IO. The ability to selectively modify portions of a MQW was quickly realised with buried heterostructure lasers receiving the most attention so far.\cite{1,27,1,28}

Studies of MQW structures in other III-V compound semiconductors has been limited primarily to material characterisation, where the effects of introducing interfacial strain due to the mismatch in lattice constants is being investigated. It is expected that the effects of interfacial strain, once characterised, will provide an additional degree of flexibility in the design of optical components.

The work described in this thesis is concerned with the fabrication and characterisation of buried stripe optical waveguides in GaAs/AlGaAs MQWs, by impurity induced layer disordering using ion implantation, and the characterisation of the disordered MQW using photoluminescence (PL) and waveguide measurements.

This thesis is comprised of four parts. Part one consisting of Chapter 1 and Chapter 2 which contain an overview of the work undertaken in the topic of this thesis and related fields, and also reviews the relevant PL and optical waveguide theory,
necessary to understand the results presented here. Part two, which comprises of
Chapter 3 and Chapter 4, contain the experimental techniques used in the sample
preparation and in the experimental techniques used to obtain the PL spectra and
optical waveguide results. The third part, which consists of Chapter 5 and Chapter 6,
contains the results and discussion for both the optical waveguide measurements and
PL results, obtained using the techniques described in Chapter 4. Finally, part four,
brings together the work undertaken, draws relevant conclusions, and suggests areas
for future investigation.
REFERENCES

1.1 A.G. Bell. The Electrician, 214, 215, 220, 221, 1880


1.4 A. Werts. L’Onde Eletrique, 46, 967, 1966


1.7 D.B. Anderson. 'Optical and Electrooptical Information Processing', 221, MII Press, 1965

1.8 S.E. Miller. Bell Syst. Tech. J. 48, 2059, 1969


1.10 W.A. Gambling. Elect. and Power, 29, 777, 1983


CHAPTER 2

REVIEW

2.1 Introduction

In this chapter a review of the work relevant to the area of investigation of this thesis is presented. Initially, physical constants for the GaAs - AlGaAs system, including those that were used during the course of this work are listed and discussed briefly. Section 2.3 contains an introduction to multi-quantum-well (MQW) structures which is followed by a review of waveguiding in GaAs/AlGaAs MQW structures, where the refractive indices of such structures and the effects of free carriers are discussed. A review of Impurity Induced Layer Disordering (IILD) and its application to waveguide fabrication is presented in Section 2.5. Reviews of ion implantation, which was used for the introduction of the impurity atoms into the MQW, and photoluminescence, which was used as a characterisation technique, are also presented. Finally, a section is included on optical waveguide theory which contains the calculations used for the determination of the refractive index difference between the guiding layer and the confining regions in the waveguides fabricated in this work.
2.2 Properties of GaAs and AlGaAs

In this section the bulk material physical parameters relating to the GaAs/AlGaAs MQW system are presented. A schematic diagram of the wafer design used in this work, is shown in Chapter 3. On complete intermixing of this structure, an AlGaAs region of 14.4% Al concentration was expected and data concerning this composition has also been included below. The mechanical and thermal parameters are given in Table 2.1 and the physical constants used in the photoluminescence and optical waveguiding calculations in Table 2.2 and Table 2.3 respectively. The parameters for AlAs have also been included to enable the values for any AlGaAs composition to be determined where permissible, using Vegard’s law, which uses linear interpolation. Where this has been done no reference has been included.

It can be observed from Table 2.3 that the refractive indices for AlGaAs cannot be determined by linear interpolation between the values for GaAs and AlAs. This is due to the nonlinear variation of the refractive index of Al<sub>x</sub>Ga<sub>1-x</sub>As for x>0.45, where the band structure is known to become indirect.<sup>[21]</sup> However, for Al<sub>x</sub>Ga<sub>1-x</sub>As compositions where x<0.45, and has a direct bandgap, it is known that the refractive indices can be modelled as varying linearly with composition,<sup>[21]</sup> and this relationship was used.
Table 2.1. Mechanical and Thermal Properties of Bulk Al<sub>x</sub>Ga<sub>1-x</sub>As for Compositions Relevant to the MQW Structure Used in this Study.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>GaAs</th>
<th>Al&lt;sub&gt;x&lt;/sub&gt;Ga&lt;sub&gt;1-x&lt;/sub&gt;As &lt;br&gt;x=0.144</th>
<th>Al&lt;sub&gt;x&lt;/sub&gt;Ga&lt;sub&gt;1-x&lt;/sub&gt;As &lt;br&gt;x=0.24</th>
<th>AlAs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lattice Const. (Å)</td>
<td>5.6533&lt;sup&gt;a&lt;/sup&gt;</td>
<td>5.6544</td>
<td>5.6552</td>
<td>5.6611&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>5.360&lt;sup&gt;a&lt;/sup&gt;</td>
<td>5.130</td>
<td>4.976</td>
<td>3.760&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>Specific Heat (J/g deg)</td>
<td>0.335&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.353</td>
<td>0.365</td>
<td>0.461&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>Therm. Rest' ty. (deg cm/W)</td>
<td>2.27&lt;sup&gt;a&lt;/sup&gt;</td>
<td>2.246</td>
<td>2.229</td>
<td>1.10&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>a</sup>Data from [1].
<sup>b</sup>Data from [2].
<sup>c</sup>Data from [3].
<table>
<thead>
<tr>
<th>Parameter</th>
<th>GaAs</th>
<th>Al_{x}Ga_{1-x}As (x=0.144)</th>
<th>Al_{x}Ga_{1-x}As (x=0.24)</th>
<th>AlAs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bandgap (E_g) (eV) 300K</td>
<td>1.424(^{a})</td>
<td>1.604(^{b})</td>
<td>1.723(^{c})</td>
<td>2.168(^{d})</td>
</tr>
<tr>
<td>Bandgap (E_g) (eV) 80K</td>
<td>1.507(^{i})</td>
<td>1.693(^{i})</td>
<td>1.816(^{i})</td>
<td>2.231(^{k})</td>
</tr>
<tr>
<td>Elect. Eff. Mass (m^*)</td>
<td>0.0665(^{i})</td>
<td>0.0712(^{a})</td>
<td>0.0782(^{a})</td>
<td>0.15(^{m})</td>
</tr>
<tr>
<td>Heavy Hole Mass (m_{hh})</td>
<td>0.340(^{p})</td>
<td>0.400</td>
<td>0.441</td>
<td>0.760(^{v})</td>
</tr>
<tr>
<td>Light Hole Mass (m_{lh})</td>
<td>0.094(^{p})</td>
<td>0.102</td>
<td>0.107</td>
<td>0.150(^{v})</td>
</tr>
</tbody>
</table>

Table 2.2. Electronic Properties of Bulk Al_{x}Ga_{1-x}As for Compositions Relevant to the MQW Structure Used in this Study.
### Table 2.3. Optical Physical Parameters of Bulk $\text{Al}_x\text{Ga}_{1-x}\text{As}$ for Compositions Relevant to the MQW Structure Used in this Study.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>$\text{GaAs}$</th>
<th>$\text{Al}<em>x\text{Ga}</em>{1-x}\text{As}$</th>
<th>$\text{AlAs}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Refract.</td>
<td>1.15μm</td>
<td>3.471r</td>
<td>3.3998</td>
</tr>
<tr>
<td>Index</td>
<td></td>
<td>3.3998</td>
<td>3.352r</td>
</tr>
<tr>
<td>Refract.</td>
<td>1.3μm</td>
<td>3.450r</td>
<td>3.3794</td>
</tr>
<tr>
<td>Index</td>
<td></td>
<td>3.3794</td>
<td>3.332r</td>
</tr>
<tr>
<td>Refract.</td>
<td>1.54μm</td>
<td>3.432r</td>
<td>3.3608</td>
</tr>
<tr>
<td>Index</td>
<td></td>
<td>3.3608</td>
<td>3.313r</td>
</tr>
</tbody>
</table>

2.3 Multi-Quantum-Well Structures

Studies of quantisation effects in ultrathin semiconductors have increased considerably with the advances made in the control and quality of material growth, with alternating layers of low and high bandgap semiconductors with well defined interfaces now being grown routinely. The band diagram of a typical $\text{GaAs}/\text{AlGaAs}$ MQW structure is shown in Figure 2.1, where the layers of high and low bandgap semiconductors are referred to as barriers and wells respectively.
Figure 2.1: Band-Energy Diagram for a GaAs/AlGaAs Multiple-Quantum-Well Structure.
When the layer thicknesses approach the dimensions of the Bohr radius of the semiconductor (≈100 Å), the free motion of the carriers is restricted to the two directions perpendicular to the growth direction, and such structures are referred to as quasi-two-dimensional. Structures where the barriers are sufficiently thin (<50 Å) to permit electron coupling between the wells are generally referred to as superlattices or coupled-multi-quantum-wells. All these periodic structures have optical and electronic properties which differ from the bulk semiconductors of which they are comprised. It has been reported that GaAs - AlGaAs MQW structures, unlike the bulk semiconductors, exhibit well-resolved excitons at room temperature, which result in enhanced nonlinear effects. One such effect is the quantum confined Stark effect (QCSE), where the application of an electric field perpendicular to the plane of the quantum wells results in a strong electro-absortive effect not present in bulk semiconductors.

The two properties of MQW structures of particular interest to the work presented here were the band-energy and the refractive indices. It is known that reducing the thickness of the low bandgap semiconductor in an MQW structure increases the band energy, thereby permitting the "engineering" of the semiconductor bandgap to specific requirements. The theory relevant to this effect and the work presented here is described in more detail in Section 2.7. The calculation of the refractive indices of MQW structures is discussed in the following section.
2.4 MQW Waveguiding

The potential of MQW structures for novel device fabrication in integrated optics was quickly realised due to additional design flexibility and enhanced nonlinear optical properties. One area of considerable investigation has been the characterisation of the enhanced optical (linear, nonlinear and electrooptic) properties for use in optical signal processing. Furthermore, these studies have been concentrated at propagation wavelengths where the photon energy is close to that of the material bandgap. Consequently, comparatively little work has been published on the characterisation of waveguides at propagation wavelengths which are well above that of the band-edge of the material, as presented in this thesis. For this reason, previous work on optical waveguides has been included in the review of IILD presented in Section 2.5. In the past, as shown in Figure 2.2, vertical confinement of the propagating modes was achieved by the use of reduced refractive index AlGaAs cladding layers, with the MQW providing the high refractive index guiding region, providing a planar waveguiding structure. Lateral confinement for stripe waveguiding was usually achieved using either wet chemical or dry etching processing, to produce either ridge or rib optical waveguiding structures as shown. In the work presented here where lateral confinement was achieved by means of IILD, a buried stripe waveguide structure was formed as shown. In waveguide design one of the most important parameters to be known accurately is the refractive indices of the guiding and cladding layers.
Figure 2.2: Schematic Diagrams of Typical Waveguide Structures.
GaAs/AlGaAs MQW structures are known to be birefringent, where the refractive index is sensitive to the polarisation of the propagating mode. Using the model proposed by Ohke et al., it was suggested that the guiding layer (MQW region), for the transverse electric (TE) polarisation, could be replaced with a uniform material whose refractive index is equal to the root-mean-square value of the refractive indices of the wells and barriers. This model was applicable for the propagation of near-infrared wavelengths, and cases where the dimensions of the layers (wells and barriers) were less than approximately 200Å and large N(>20), where N is the number of well/barrier pairs. It was assumed that the refractive indices of the individual wells and barriers could be expressed by their bulk values.

Theoretically, the refractive index of a MQW region can be considered as being due to two effects. The first is due to the presence of strong excitonic resonances at the absorption edge and the second due to the dielectric nature of the MQW region. When operating at energies below the band-edge (<1.3eV) the effects due to the first can be neglected and the second becomes predominant. It has been reported that the dielectric effect can also be considered to be due to two effects and these are, a) its multilayer nature and b) the two-dimensional density of states. Furthermore, from the resultant anisotropic nature of the two contributions to the dielectric nature of a MQW, it is always seen that \( n_{\text{eff}} > n_{\text{TM}} \). Modelling these terms, where the well and barrier dimensions are restricted to approximately the same values as used by Ohke et al., Van der Ziel and Gossard, using a thin film approximation, derived the following equations:

\[
\eta_{\text{TE}}^2 = \frac{[a.n_a^2 + b.n_b^2]}{[a + b]} = n_{\text{RMS}}^2
\]
Where \( n_w, n_b \) and \( a, b \) are the well and barrier refractive indices and widths respectively. It was seen that the equation for the TE polarisation corresponds precisely with the model proposed by Ohke et al.\(^{2,11} \) An equation for the TM refractive index also derived by Ohke et al.\(^{2,11} \) was also seen to correspond precisely with equation 2.2.

Suzuki et al.\(^{2,13} \) demonstrated experimentally that for GaAs/AlGaAs MQW structures with barrier widths of less than approximately 50 Å, that is smaller than the previous cases, the refractive indices of the layers can be approximated by an AlGaAs layer of average Al mole fraction for the MQW. This is possibly due to expected electron coupling between the wells when barriers of this dimension are used. Such a structure, as mentioned previously, where electron coupling occurs between the adjacent wells, is usually referred to as a coupled-multi-quantum-well or a superlattice.

Using equation 2.1 for the material structure shown in Chapter 3, refractive indices for the MQW guiding layer of 3.4003, 3.3799 and 3.3613 were calculated for the TE polarisation at the propagating wavelengths of 1.15 μm, 1.3 μm and 1.54 μm respectively. Using the refractive indices for the fully mixed material in Table 2.3, it was seen that a positive refractive index difference (\( \delta n \)) of approximately 0.015% would exist between the MQW and the fully mixed region for the TE polarisation at all wavelengths. However, from equation 2.2, refractive indices of 3.3983, 3.3779 and 3.3593 were calculated for the TM polarisation at the propagating wavelengths of 1.15 μm, 1.3 μm and 1.54 μm respectively. Again, using the refractive indices for the
fully mixed material in Table 2.3 it was seen that a negative index difference of approximately 0.044% exists at all wavelengths. This suggests that for the TE polarisation, the MQW has a higher refractive index than the mixed region thereby providing lateral confinement of the propagating mode. However, for the TM polarisation, the mixed region has a higher refractive index than the unmixed MQW, suggesting that no lateral confinement would be observed.

Using equation 2.1 to calculate the TE refractive index of a GaAs/AlGaAs MQW structure, it was seen that the refractive index difference between a GaAs/AlGaAs MQW waveguide and a fully mixed region, for propagation wavelengths whose photon energy is smaller than the material bandgap energy, was a maximum when the wells and barriers were of the same dimension, and the well and barrier materials were GaAs and AlAs respectively. However, for the work undertaken here, where the absorption mechanisms in a buried waveguide fabricated by IILD were investigated, a lower index difference was used so that losses due to absorption of the propagating mode in the side walls of the waveguide could be determined.

2.4.1 Free Carrier Effects

The presence of free carriers in a semiconductor has two effects on the optical properties of the material. They are:- a) change in the refractive index and b) free carrier absorption for $\lambda > \text{band-energy wavelength}$. The former effect has been discussed by Hunsperger\textsuperscript{[24]} and shown to be described by the equation:-

$$\delta n = \frac{[N_S - N_L]e^2}{2n_2\varepsilon_m\omega^2}$$  (2.3)
where $\delta n$ is the change in refractive index, $N_3$ and $N_2$ are the free carrier concentrations in the optical waveguide core and cladding respectively, $e$ the electronic charge, $n_2$ the refractive index of the guiding region and $\epsilon_0$, $m^*$, and $\omega$ the permittivity of free space, the electron effective mass and optical frequency respectively.

For the work presented in this thesis, where the implantation of silicon has been used to produce IILD, intermixing has been reported\cite{213} to occur at a threshold Si concentration of approximately $3 \times 10^{18}$ cm$^{-3}$. Assuming a 15% electrical activation\cite{210} of the implanted impurity atoms and a free carrier concentration of $4.5 \times 10^{19}$ cm$^{-3}$ in the disordered material, this would produce refractive index changes of approximately -0.03%, -0.04% and -0.06% for the fully mixed region at the propagating wavelengths 1.15µm, 1.3µm and 1.54µm respectively. These values were calculated for an Al$_x$Ga$_{1-x}$As layer, where $x=0.144$ which is equivalent to a fully mixed MQW region for the structure used in this work. This would suggest that the free carrier effect on the refractive index for the material is of the same order of magnitude as that of the mixing process and increases with increasing wavelength. Both these effects will enhance lateral optical confinement in the waveguide.

The second effect of increased absorption which would result in an increased waveguide attenuation ($\alpha_{fe}$) was also derived by Hunsperger\cite{217} and shown to be described by the equation:

$$\alpha_{fe} = \frac{[Ne^2\lambda_0^2]}{4\pi^2 n [m^*] \epsilon_0 c^3}$$ (2.4)
Where $\mu$ is the free carrier mobility and $c$ the velocity of light in free space respectively, with the other symbols being as defined previously. Using the same free carrier concentration as before and assuming a carrier mobility of 1000 cm$^2$/Vsec would result in a waveguide propagation loss of approximately 6.88dB/cm, 8.85dB/cm and 12.49dB/cm at the propagation wavelengths ($\lambda_o$) of 1.15$\mu$m, 1.3$\mu$m and 1.54$\mu$m respectively.

Due to the absence of published data on the percentage electrical activation and free carrier mobilities in Si implanted MQWs, the values used for the previous calculations were estimated. Consequently, the results obtained were used only as an indication of the magnitude of the effects expected.

It should be noted that free carrier absorption in GaAs at near-infrared wavelengths for p-type dopants such as Ge, is approximately a factor of four times larger than for n-type, as reported by Sergent et al. The results reported showed that the propagation loss due to free carriers in p-type material doped to $1 \times 10^{16}$cm$^{-3}$ was 2.6dB/cm, at a propagating wavelength of 1.064$\mu$m. This p-type concentration was equal to that in the as-grown material used in this work. From equation 2.4 it can be seen that the free carrier loss is proportional to ($\lambda_o^2$) and by scaling for the propagating wavelengths of 1.15$\mu$m, 1.3$\mu$m and 1.54$\mu$m, respective propagation losses of 3.0dB/cm, 3.88dB/cm and 5.45dB/cm were calculated.

### 2.5 ILD in GaAs/AlGaAs MQW Structures

It was reported by Chang and Komá that semi-insulating GaAs/AlGaAs MQW structures were thermally stable at temperatures up to 850°C, where Ga/Al
Interdiffusion coefficients of $1 \times 10^{-18} \text{cm}^2/\text{sec}$ were recorded. Samples containing sequentially grown GaAs/AlGaAs layers, with varying Al compositions, were annealed over the temperature range 850°C to 1100°C in an As atmosphere. It was observed that the interdiffusion coefficient was dependent upon the Al composition of the high bandgap layer with increased interdiffusion at lower Al compositions. To produce significant intermixing, an undoped AlAs/GaAs structure was annealed at 900°C for 10 hours and a Ga/Al interdiffusion coefficient of approximately $8 \times 10^{-18} \text{cm}^2/\text{sec}$ recorded.

In 1981 Laidig et al., using Zn from a surface diffusion source, were the first to report that the introduction of an impurity species into a MQW structure enhanced the Ga/Al interdiffusion. Significant intermixing was reported for samples annealed between 500°C and 600°C for 4 hours. Similar undoped structures were annealed between 800°C and 1000°C to produce the same extent of intermixing. In the work that followed, it became apparent that IILD could be produced in a variety of III-V MQW systems using a wide selection of impurity atoms.

Intermixing in MQW structures was also achieved using gallium vacancy sources, where an arsenic over pressure or a SiO$_2$ encapsulant was used during annealing, which allowed Ga outdiffusion from the sample surface. Deppe et al. were the first to report the use of a SiO$_2$ capping layer as a group III vacancy source in the fabrication of stripe geometry quantum well lasers, where stripes of Si$_x$N$_{4-x}$ were patterned onto the surface of an MQW structure prior to the deposition of a SiO$_2$ layer which was then annealed at 875°C for 10 hours. Transmission Electron
Microscopy (TEM) was used to show that complete intermixing of the layers had occurred under the SiO\textsubscript{2} cap, whilst no mixing was evident in the regions under the Si\textsubscript{x}N\textsubscript{y} stripes.

Four techniques have been reported for the introduction of the dopant species for IILD. They are diffusion from a surface source,\textsuperscript{[2,20]} ion implantation,\textsuperscript{[2,23]} doping during epitaxial growth\textsuperscript{[2,23]} and more recently laser assisted annealing,\textsuperscript{[2,24]} where the required dopant was deposited on the sample surface and then annealed with a pulsed laser beam, which caused the sample surface to melt and, on regrowth, the dopant species were incorporated into the lattice. The most controllable of these techniques is ion implantation, which allows a more precise introduction of the impurity species with regard to the atomic concentration and its depth distribution, the latter being especially useful for buried layer structures. Furthermore, it allows the introduction of impurity species for which there are no surface diffusion or epitaxial growth sources. Although ion implantation offers these advantages, it introduces a complication in the characterisation of the impurity induced layer disordering, as implantation damage is known to modify the mixing behaviour.\textsuperscript{[2,23]}

MQW layer disordering, using ion implantation followed by annealing, was observed for a number of ion species. Not only was the effect observed for common dopants such as Si,\textsuperscript{[2,22]} Zn,\textsuperscript{[2,26]} Mg,\textsuperscript{[2,27]} and Se,\textsuperscript{[2,27]} but also for the lattice constituent ions Ga,\textsuperscript{[2,28]} Al\textsuperscript{[2,29]} and As\textsuperscript{[2,30]} and other ions such as Kr\textsuperscript{[2,29]} and F.\textsuperscript{[2,30]}

Silicon was one of the most extensively investigated ions used for IILD and chosen as the impurity species for the work reported here. Despite the extensive investigation, the mechanisms by which the mixing occurs are still not fully understood. It is apparent from the literature that the diffusion mechanisms associated with IILD are
complex which was highlighted by work published by Venkatesan et al. where the presence of Be was shown to suppress the disordering produced by Ge. However, it was also observed that implantation of the individual species resulted in the disordering of a GaAs/AlGaAs MQW. This situation was further complicated when suppression of the disordering was found to be dependent upon the implantation order of the two ions. This suggested that several mechanisms were operating simultaneously. The presence of several mechanisms which affect the disordering process was also observed for Si, as reported below. Therefore it is apparent that much more work is required before these mechanisms responsible for layer intermixing are fully understood.

ILD of GaAs/AlGaAs MQW structures using Si as a diffusion source was first reported by Meehan et al. in 1984 and using Si implantation by Coleman et al. in 1982. Since that time considerable progress has been made on the characterisation of Si impurity induced disordering. Kawabe et al. reported that after annealing an as-grown Si modulation doped MQW structure at 800°C for 2 hours using face to face contacting with a GaAs wafer in an H₂ atmosphere, that a threshold Si concentration of approximately $4 \times 10^{18}$ cm⁻³ was required to produce appreciable intermixing, whilst a concentration of approximately $7 \times 10^{18}$ cm⁻³ was required for complete intermixing. This result suggested a strong interdependence of Ga/Al intermixing with Si dopant concentration. Further work revealed that Si diffusion, and consequently Ga/Al interdiffusion, was suppressed for Si concentrations $>1 \times 10^{19}$ cm⁻³. Analysis was performed using transmission electron microscopy which revealed that the annealed, heavily doped, as-grown samples had a high defect density, and the presence of these
defects was believed to suppress the diffusion. In contrast to this process, several authors\textsuperscript{2,5,27,28} suggested that implantation damage resulted in enhanced diffusion of the impurity species and increased Ga/Al interdiffusion.

The dependence of Ga/Al interdiffusion with Si implantation dose was first reported by Hirayama et al.\textsuperscript{22,29} who studied the effects of implantation at 200KeV for Si doses ranging from $2 \times 10^{16}$ ions cm$^{-2}$ to $1 \times 10^{17}$ ions cm$^{-2}$. They observed that Ga/Al interdiffusion increased with an increasing ion dose, which suggested a concentration dependence on the disordering. Kobayashi et al.\textsuperscript{22,29} using secondary ion mass spectrometry (SIMS), reported that a threshold Si concentration of approximately $3 \times 10^{19}$ cm$^{-3}$ was required for the onset of fast diffusion and intermixing in samples implanted at 80 KeV to doses ranging from $3 \times 10^{14}$ ions cm$^{-2}$ to $1 \times 10^{16}$ ions cm$^{-2}$. The Si concentration required for the onset of fast diffusion was the same as that reported for the as-grown material.\textsuperscript{22,29} Using SIMS, Schwarz et al.\textsuperscript{22,31} reported that 180 KeV implantation to a dose of $3 \times 10^{15}$ Si cm$^{-2}$, where the sample was capped and subsequently annealed at 850°C for 3 hours, produced intermixing to depths up to four times the projected range of the ions. However, in the region of maximum implantation damage, the post thermal mixing effect was inhibited. The depth to which mixing was obtained by Schwarz et al suggested that the mixing of thick MQW structures was possible.

Matsui et al.\textsuperscript{22,38} reported the relationship between implantation ion energy and the extent of disordering after the post implantation anneal. It was observed that for the same ion doses, increasing the ion energy resulted in a suppression of the disordering in the near surface regions and that the extent of intermixing in the deeper regions was reduced.
Silicon was seen to segregate into the GaAs layers, which had been preferentially damaged during implantation. As mentioned previously, these effects were associated with Si diffusion inhibition in as-grown Si doped samples.

It was reported by Ishida et al. that the type of encapsulant employed during annealing had a significant effect on the extent of the observed mixing. The GaAs/AlGaAs superlattices (SLs) were implanted with Si at 80KeV to a dose of $3 \times 10^{14}$ ions cm$^{-2}$ and three capping conditions were used for annealing at 850°C for 1 hour anneals. These were: 1) face to face 2) As overpressure and 3) a SiO$_2$ encapsulant. SIMS analysis showed that no intermixing occurred for the sample annealed using the face to face technique, and while some trace of disordering was found for the sample annealed in an As overpressure, whilst complete disordering was only obtained using the SiO$_2$ encapsulant.

It therefore became apparent that the Ga/Al intermixing, induced by the annealing of Si implanted GaAs/AlGaAs MQW structures, was enhanced by the fast diffusion of Si and was inhibited by high damage levels. Furthermore, the literature revealed that the intermixing process was sensitive to the encapsulant used during the annealing.

The potential of IILD disordered was quickly realised for the selective modification, via masking, of MQW structures for device fabrication. In 1981, Holonyak et al. reported the monolithic integration of infrared lasers and "Yellow Gap" cavities in AlGaAs superlattice lasers by selective area Zn diffusion. Further applications were reported for laser devices where the absorption at the output facet was minimised by Zn diffusion impurity induced disordering, which created a wide bandgap output facet window. Thornton et al. reported an output power increase from 1.2W to 2.1W for lasers incorporating a wide bandgap window at the output facet. This
technique was found to be limited by the absorption due to the free carriers introduced by the electrically activated impurity species. Impurity induced layer disordering was also applied in the fabrication of optical detectors and optical waveguides, although the largest effort so far has been devoted to the fabrication of lasers.

There is little information available from the literature on the characterisation of optical waveguides delineated using IILD. In 1986 Ralston et al. reported the fabrication of grating couplers by selective area Se implantation in a GaAs/AlGaAs MQW slab waveguide for use at a propagation wavelength of 1.15 μm.

Propagation losses were measured through a disordered single GaAs/AlGaAs quantum well structure by Werner et al. Disordering was achieved by Si implantation to a dose of 2x10^{15} ions cm⁻² and annealing at 925°C for 5mins in an Ar atmosphere. Propagation losses of 39dBcm⁻¹ were recorded at the propagating wavelength of 0.843 μm. O'Neill et al. reported propagation loss measurements through a GaAs/AlGaAs MQW waveguide disordered using either B or F implantation. Boron implantation was undertaken at 200KeV and 100KeV to provide an approximately uniform impurity distribution to a depth of 0.7 μm at doses ranging from 1x10⁴ cm⁻² to 3x10⁵ cm⁻². Fluorine implantation was performed to the same doses at 300KeV. The samples were capped with SiO₂ and annealed in an As rich atmosphere at 890°C for 2 hours and 4 hours. Propagation loss measurements were undertaken at wavelengths 0.78 μm to 0.880 μm and losses of approximately 50 dBcm⁻¹ were recorded at wavelengths far from the band-edge for both implantation species.

Julien et al. observed an average refractive index difference of 0.9% between the unmixed and disordered regions of an AlAs/GaAs superlattice. They used Zn diffusion from a surface source and annealing at 564°C for 2.5 hours to fabricate
linear waveguides with small abrupt bends. Using a simple plane-wave model, the refractive index step required to support a mode through a bend was calculated. Propagation through 7.5° bends were observed and calculated to correspond to a refractive index change of 0.9%. Optical modes were also observed to be transmitted though 12.5° bends, which corresponds to a MQW order/disorder refractive index change of 2.4%. These measurements were performed at wavelengths between 0.8µm and 0.886µm.

Wolf et al., using the cutoff behaviour of lateral modes, investigated the refractive index step between diffusion disordered and unmixed regions of a GaAs/AlGaAs MQW waveguide. Waveguide fabrication was achieved by the surface diffusion of Zn at 630°C for 70mins for one set of samples, and by diffusion of dopants incorporated during growth by annealing in an As overpressure at 850°C for 4 hours. For the waveguides fabricated by surface diffusion, it was reported that refractive index steps of 4x10⁻³ and 2x10⁻³ were observed at the propagation wavelengths of 0.875µm and 1.06µm respectively. It was also observed that the index step was sensitive to the polarisation of the propagating mode, with a larger index step observed for the TE polarisation. The larger index step for photon energies close to the bandgap was attributed to quantisation size and bandgap bowing effects. Refractive index steps for the waveguides fabricated by annealing in an As overpressure were also observed to be larger at propagation wavelengths close to the band-edge and the refractive index values were also seen to be polarisation sensitive. However, for the samples annealed using an As overpressure, it was observed that the refractive index difference between the MQW and disordered region was smaller than for the waveguides fabricated by the surface diffusion of Zn. This was believed to be due to the reduction in the free carrier concentration and their consequential effect on
the refractive index. Measurements performed on the samples annealed in an As overpressure, at propagation wavelengths well below the band-edge, revealed that the TM polarisation was not supported. It was suggested that this effect was result of the reduced contribution of the free carriers to the refractive index of the material.

2.6 Ion Implantation

Ion implantation is the bombardment of a target by high energy ions of a specific atomic species. The primary advantage of implantation is the ability to precisely control the number of dopant ions introduced into the target and their depth profile. This technique has found widespread application in silicon device fabrication where dopant depth distribution has to be accurately controlled.

The ions are produced by either a hot wire or plasma source which is held at a high positive potential and then extracted through an aperture which is held at a negative potential of a few KV relative to the source. After focusing, the ion beam is accelerated and mass analysed to provide the required isotope of the atomic species. Mass analysis is achieved by passing the charged beam through a perpendicular magnetic field where it is deflected according to the relationship given by the equation:

\[ R.B = \left[ \frac{2MV^2}{Q} \right] \]

(2.5)

where an ion of mass \( M \) and charge \( Q \) is accelerated by a potential \( V \) when passing in a direction which is normal to a magnetic field of intensity \( B \) describes a curve of radius \( R \). The required isotope is selected by varying the current through the coils of
the magnet. Prior to impinging on to the target the beam is electrostatically scanned, providing a uniform implant distribution across the sample surface which is held at ground potential.

When an energetic ion enters a solid, it gradually releases all of its energy to the nuclei and electrons of the target until it finally comes to rest. The depth (projected range) to which the ions penetrate the target is a function of the acceleration potential and the masses of the ion and target atoms. Lindhard et al. \cite{Lindhard1958} predicted a Gaussian distribution of the implanted ions when implanted into an amorphous target. This Gaussian distribution of implanted species may be described by the most probable range normal to the surface, which is called the projected range ($R_p$), and its standard deviation ($\delta R_p$). It was shown that:

$$N(x) = \frac{N_0}{(2\pi)^{1/2} \delta R_p} \exp \left(-\frac{1}{2} \left( \frac{x - R_p}{\delta R_p} \right)^2 \right)$$

where $x$ is the depth normal to the target surface and $N_0$ and $N(x)$ are the ion dose incident on the target and the ion dose at a depth $x$ from the surface respectively.

The wafers were tilted by approximately $7^\circ$ from any major crystal axis to reduce the effects due to channelling, since the semiconductor is a single crystal which has channels in certain crystal orientations down which the incident ions could pass without interacting with the target lattice. Furthermore, the disorder resulting from ion implantation is dependent on the mass, energy and dose of the ion and target temperature during implantation. Post-implantation annealing was required to reduce the electronic damage (point defects) and, in the work presented here, to promote diffusion of the implanted ion and consequently the intermixing of the MQW layers.
As stated previously, on entering the target material the implanted ions release their energy and come to rest at a projected range \( R_p \). This release of energy results in implantation damage of the target material. Implant species and damage profiles have been modelled using SUSPRE developed at the University of Surrey, and typical profiles for the implantation of Si at 500KeV to a doses of \( 3 \times 10^{13} \) ions cm\(^{-2} \), \( 3 \times 10^{14} \) ions cm\(^{-2} \) and \( 1 \times 10^{15} \) ions cm\(^{-2} \) in \( \text{Al}_{0.34} \text{Ga}_{0.66}\text{As} \) are shown in Figure 2.3, Figure 2.4 and Figure 2.5 respectively. From this model, it can be seen that the region of maximum implantation damage increases with increasing dose. It will be suggested from the results presented in Chapter 5 that this implantation damage has a significant effect on the disordering process.

Due to the problem of decomposition of GaAs and AlGaAs at elevated temperatures resulting from the evaporation of Ga and As,\(^{23,24}\) annealing is usually performed with an encapsulant.

During implantation, two potential sources of error which may result in an inaccurate measurement of the implanted ion dose are:- a) the presence of molecules within the beam which have the same mass as the required implant species and b) a neutral component in the beam current. These potential sources of error and the procedures used to overcome them are discussed in detail in Chapter 3.

2.7 Photoluminescence Characterisation of MQW Structures

Photoluminescence is a useful technique for the direct measurement of a semiconductor bandgap and the ionisation energies associated with impurity species present. In this study, PL was used for the characterisation of the as-grown MQW
Figure 2.3: Silicon Ion and Implantation Damage Profiles

Modelled for $3 \times 10^{13} \text{ Si cm}^{-2}$ in $\text{AlGaAs}$.
Figure 24: Silicon Ion and Implantation Damage Profiles
Modelled for 3×10^{14} Si cm^{-2} in Al, Ga, As.

X = Implant Profile
O = Disorder Profile

Depth (Å)

Disorder %
Figure 2.5: Silicon Ion and Implantation Damage Profiles

Modelled for $1 \times 10^{15} \text{ Si cm}^{-2}$ in Al$_{0.24}$Ga$_{0.76}$As.
material and as a means of determining the degree of intermixing.

The technique requires the irradiation of a sample using a laser source whose photon energy is greater than the energy-gap of the semiconductor so that electrons are excited from the valence band to the conduction band, thereby creating electron-hole pairs. These excited electrons eventually recombine with holes in the valence band emitting a photon. A number of possible transitions may take place and those that are relevant to this work are shown in Figure 2.6. Transition (a) is an intrinsic emission whose energy corresponds closely to the energy-gap of the material whilst those of (b), (c), (d) and (e) involve chemical impurities or physical defects, where transition (b) is a conduction band to acceptor, (c) a donor to valence band, (d) a donor to acceptor and (e) deep level transitions. It should be noted however that not all possible transitions result in the emission of a photon and in such cases, the energy of the electron is released to the crystal lattice via a phonon.

In a quantum well system, recombination of the electron-hole pairs preferentially occurs in the semiconductor well, since it is at a lower energy than that of the barrier. The most probable band to band transition being that of the lowest conduction band level (cc1) to the lowest heavy hole level (hh1). When the well material becomes sufficiently thin (≈100Å) the bandgap energy of the semiconductor well is modified. This is shown in Figure 2.7 as a shift from \( E_g \) (GaAs) to the energy levels marked 1, and is seen as an increase in the bandgap energy. Therefore, by the use of PL, it is possible to determine the change in the bandgap energy from the bulk value of the material with the lowest energy-gap. This change in energy can be modelled by the solution of the Schrödinger wave equation, where the boundary conditions are a finite potential step.
Figure 2.61 - Examples of Electron-Hole Recombinations Resulting in Photoemission in Semiconductors.
Figure 2.7: Band-Energy Diagram of an As-Grown GaAs Quantum Well (Dashed-line), and Following Ga/Al Interdiffusion (Solid-Line).
The Schrödinger wave equation is given by:

\[ i \frac{\hbar}{2\pi} \frac{\partial \psi}{\partial t} = -\frac{\hbar^2}{8\pi^2m} \nabla^2 \psi + V(r,t)\psi \]  

(2.7)

where \( \psi \) is the wave function, \( \hbar \) Planck's constant, \( m \) the particle mass, \( r \) the position vector, \( t \) time, \( i \) the \( \sqrt{-1} \) and \( V \) the potential energy of the barrier. A simplified solution of this equation for a finite potential step boundary condition,\(^{25} \) was used to theoretically determine the shift in bandgap energy for the MQW system used during in this work. A shift of 29meV towards higher energies was predicted for the ec1 to hh1 transition in the GaAs well with the ec1 to light hole (lh1) transition energy 10meV larger.

When Ga/Al intermixing between the wells and barriers occurs, the bandgap energy of the system is further modified as shown in Figure 2.7, with a shift from energy levels 1 to an increased energy-gap between levels ec1 and hh1. This shows that the well/barrier interfaces become less well defined and, with increased intermixing, a region of AlGaAs is formed whose aluminium concentration is given by the average composition of the wells and barriers. Observation of this process using PL will show an emission peak which moves to higher energies until the layers are completely intermixed, and an energy corresponding to the AlGaAs region of average composition is reached. Typical PL spectra for the work undertaken in this thesis which show this shift in emission energy are presented in Chapter 5.

Photoluminescence has been used extensively in the characterisation of MQW structures and as a means of determining the extent of layer disordering. Laidig et al.\(^{27} \) used PL in the first published work on IILD to determine when the
layers of an MQW structure were completely intermixed. This was evident when the emission energy from the sample corresponded to the emission from an AlGaAs layer whose Al composition was equal to the average value of the MQW.

It was reported that the enhanced Ga/Al interdiffusion caused by silicon diffusion gave rise to emissions associated not only with the change in well energy gap, as described previously, but also with both shallow and deep level emissions.\[^{[25]}\] Similar levels were also reported by Henning et al.\[^{[25]}\] whilst investigating photoluminescent emissions of Si doped AlGaAs. The shallow emission is generally attributed to a substitutional donor atom, whereas the deep levels have been attributed to donor-vacancy complexess\[^{[25]}\] in Si doped GaAs, nearest-neighbour or Si-Si pairs,\[^{[25]}\] and donor-acceptor transitions.\[^{[25]}\] As we shall see later, the presence of these deep levels appears to play a significant role in determining the loss mechanisms in the optical waveguides, fabricated by Si implantation and subsequent annealing.

### 2.8 Optical Waveguide Theory

The planar waveguide is the simplest form of optical waveguide and is shown schematically in Figure 2.8. It consists of a high refractive index guiding region ($n_1$) with top and bottom cladding layers of lower refractive index ($n_2$).

A ray of light will propagate along the region of high refractive index provided the criteria for total internal reflection are observed ($\theta_i > \theta_c$), where $\theta_c$ and $\theta_i$ are the critical angle and angle of incidence respectively.
Figure 2.8 - Planar Waveguide Ray Diagram.
The spatial variation of the electric field along the propagation direction of the beam can be written as:

\[ E(r) = E_0 \exp[-iK.r] \]  

(2.8)

When the ray is split into its constituent vectors in the \( y \) and \( z \) directions and substituted into equation 2.8, the equation becomes:

\[ E(z,y) = E_0 \exp \left[ \frac{-2\pi n_1}{\lambda_0} [z \sin \theta + y \cos \theta] \right] \]  

(2.9)

For a mode to propagate, a wave crossing the guiding layer twice must have the same phase otherwise destructive interference will occur. Therefore, the phase change must be zero or an integer multiple (\( m \)) of \( 2\pi \). The solution of equation 2.9 for \( m \) has been derived elsewhere\(^{22,25} \) and shown to be described by the equation:

\[ m \leq \frac{2dn_1}{\lambda_0} \left[ 1 - \left( \frac{n_2}{n_1} \right)^2 \right]^{\frac{1}{2}} \frac{\phi}{\pi} \]  

(2.10)

where \( \lambda_0 \) is the wavelength in free space and \( \phi \) is equal to twice the phase change on reflection. Rewriting equation 2.10 yields:

\[ m \leq \frac{V}{\pi} \frac{\phi}{\pi} \]  

(2.11)

where \( V \) is the normalised film thickness and is defined by:

\[ V = \frac{2\pi d}{\lambda_0} \left[ n_1^2 - n_2^2 \right]^{\frac{1}{2}} \]  

(2.12)
It can be seen from the definition that $\phi$ can never be larger than $\pi$ and therefore $\phi/\pi$ can never be greater than 1. By assuming $\phi/\pi$ to be negligible and rewriting equation 2.11 and equation 2.12 for the refractive index step gives:

$$\left[n_1^2 - n_2^2\right] \geq \left[\frac{m\lambda_0}{2d}\right]^2$$

(2.13)

By applying equation 2.13 for the lateral confinement observed in the waveguides fabricated using IILD, taking the limits $0 < m < 1$, the refractive index step between the disordered and unmixed regions were calculated and these results are presented in Chapter 6.

In this Chapter, reviews of the literature and the experimental techniques relevant to the work presented in this thesis have been presented. An overview of the theory relevant to these techniques and the material constants used in any future calculations have also been included.
REFERENCES

GaAs AlGaAs Constants

a: C.M.H. Driscoll, A.F.W. Willoughby, J.B. Mullin and B.W. Straughan. Int. Conf. on Gallium Arsenide and Related Compounds, Deauville, 275, 1975


g: P.D. Maycock. Solid-State Electron. 10, 161, 1967

h: M.A. Afromowitz. J. Appl. Phys. 44, 1292, 1973

i: S. Adachi. J. Appl. Phys. 58, R1, 1985


2.1 S. Adachi. J. Appl. Phys. 58, R1, 1985

2.2 R. Dingle. 'Semiconductors and Semimetals: Applications MQW Selective Doping and Superlattices', 24, 2, 1987


2.11 J.P. van der Ziel and A.C. Gossard. J. Appl. Phys. 49, 2919, 1978


2.14 R.G. Hunsperger. 'Integrated Optics: Theory and Technology' 2nd Ed. 55, Springer-Verlag, 1985


2.16 R. Gwilliam. Private Communication. SERC Central Facility for Ion Implanatation, University of Surrey.

2.17 R.G. Hunsperger. 'Integrated Optics: Theory and Technology' 2nd Ed. 75, Springer-Verlag, 1985


2.20 W.D. Laidig, N. Holonyak, Jr., J.J. Coleman and P.D. Dapkus. J. Electron. Mat. 11, 1, 1982


2.52  L.I. Schiff. 'Quantum Mechanics', 3rd Ed. 37, Mc-Graw Hill, 1985


3.1 Introduction

In this chapter the experimental techniques used in sample preparation for the photoluminescence study of impurity induced layer disordering and waveguide fabrication are presented. During the course of this work, samples were prepared for PL measurements and for the fabrication of buried stripe optical waveguides. The processes that were common to both the PL study and waveguide fabrication which include ion implantation, encapsulation and annealing are presented in Section 3.3. The processes specifically required for stripe waveguide fabrication which included photolithography, sample thinning and cleaving are presented in Section 3.4. As ridge waveguides fabricated using wet etchants were used for characterisation of the as-grown material, the characteristics of these etchants are presented in Section 3.4.3.
3.2 Material Specification

The material used in this work was a GaAs/AlGaAs MQW structure, a schematic diagram of which is shown in Figure 3.1. The MQW consisted of twenty nine $\text{Al}_{0.33}\text{Ga}_{0.67}\text{As}$ barriers 182Å thick and thirty GaAs wells 122Å thick. Vertical confinement was provided by top and bottom $\text{Al}_{0.33}\text{Ga}_{0.67}\text{As}$ cladding layers with thicknesses of 0.5μm and 1.84μm respectively. The structure was capped with approximately 50Å of GaAs to prevent degradation of the top AlGaAs cladding layer. The material was grown 2° off the <100> crystal axis by Metal Organic Vapour Phase Epitaxy (MOVPE) at the SERC central facility for III-V material growth at the University of Sheffield. All layers were undoped and had typical residual p-type carrier concentrations of $1\times10^{16}\text{cm}^{-3}$. The physical constants relevant to this structure have been discussed previously in Chapter 2.

This material structure was designed to be a buried symmetrical waveguide, which supported a single mode in the vertical direction, at a propagation wavelength of 1.15μm. The top $\text{Al}_{0.33}\text{Ga}_{0.67}\text{As}$ cladding layer thickness was chosen to permit the implantation of 500KeV Si which have a range of 0.52μm, into the first few layers of the MQW stack. Reducing the upper cladding layer thickness to 0.5μm was not thought to have a significant effect on the characteristics of the evanescent field in this layer.

3.3 Sample Processing Procedures

During the course of this study several different processing procedures were used to provide impurity induced layer dis ordering and, as in the case of the encapsulants
Figure 3.1: GaAs/AlGaAs MQW Structure used in this Study.
used during annealing, a comparison of their effects was undertaken and these results are presented in Chapter 5 and Chapter 6.

3.3.1 Sample Cleaning

Sample cleanliness was known to be of particular importance in providing reproducibility of the fabrication process. For this purpose, prior to each processing stage the samples were sequentially immersed in baths of hot trichloroethylene, acetone and methanol before finally being rinsed in deionised water and blown dry with filtered nitrogen. The solvents used were high purity analytical reagents (AR), and the order in which the solvent baths were used ensured that each solvent was soluble in the following bath, thereby reducing the risk of solvent staining. After this cleaning procedure, the samples were examined under an optical microscope and if any staining was evident the process was repeated.

All glassware used in the processing of samples was initially cleaned in Micron glassware cleaning fluid and rinsed in distilled water prior to being used for sample processing.

3.3.2 Ion Implantation

All implantation was performed in the SERC central facility for ion implantation at the University of Surrey using the 500kV ion accelerator, a schematic diagram of which is shown in Figure 3.2.
After cleaving the samples into approximately 1cm x 1cm squares and cleaning using the above procedure, the samples were mounted onto a stainless steel sample holder using aqua-dag, to ensure good electrical and thermal contact. Initially, samples were mounted using silver-dag but it was found that this material was difficult to remove after implantation, consequently aqua-dag was used instead. The sample holder was then loaded into the target chamber of the implantation system which was then evacuated to 10^-5 Torr. This pressure was achieved by a two stage process consisting of a rotary pump, which evacuated the chamber to approximately 10^-3 Torr, with the final pressure being achieved using an oil diffusion pump. Each sample in turn was processed by aligning it with the aperture which was used to determine the area exposed to the incident ion beam. The samples were implanted at room temperature using 500KeV silicon ions to doses ranging from 3x10^{13} ions cm^-2 to 7x10^{13} ions cm^-2. These ions had a projected range (R_p) using the PRAL model of 0.52μm, causing the peak impurity concentration to lie at the top of the MQW region. The beam current was kept in the range 0.1μAcm^-2 to 0.8μAcm^-2 to ensure that implantation times greater than 1min were required for ease of control of the exposure time, and to minimise the heating effect of the samples. A uniform coverage of the sample surface during implantation was ensured by raster scanning the beam across the sample, as described below.

During implantation the ion beam was monitored at two stages. Firstly, by placing a vibrating wire detector before the mass analysing magnet, the beam shape was monitored and this allowed the beam focusing to be optimised. Secondly, during implantation, a current is generated between the sample holder and earth and this current was measured. This current flowed as a result of a charged beam being incident on the sample and was used to determine the implant dose as described.
below. When a positively charged ion enters the target sample, an electron is required to maintain the sample at earth potential. Therefore, by measuring this current and integrating to determine the total charge required, the number of ions to enter the sample may be determined and the total dose calculated. Once the total charge reached a predetermined level corresponding to the required dose, the beam was isolated from the sample by means of a pneumatically operated shutter. To ensure that the implant conditions did not alter during implantation the beam current was constantly monitored.

Because the mass analyser was unable to distinguish between an ion and a molecule of the same mass, molecular ions of CO and N₂ were potential beam contaminants, since they have the same mass as Si²⁺. Consequently, the majority of this work was performed using Si²⁺, although this resulted in a drop of available beam current from typical values of several microamps to approximately a few tenths of a microamp. Where high doses i.e. >3x10¹⁶ ions cm⁻² were required, Si²⁺ was used and a mass spectrometer of the beam was taken using the mass analysing magnet to ensure that C, O, and N were not present in any significant quantities.

During transport of the ion beam down the flight tube, charge exchange of the ions with any residual gas molecules in the accelerator results in a neutral component to the incident beam which would cause an error in the dosimetry measurement. This problem was overcome during the system setup, where the beam was deflected by applying a dc bias across the X scan plates until the beam was blocked by the aperture plate. The magnet current was then readjusted to centralise the beam minus the neutral component onto the sample holder. By also applying triangular waveforms to the X and Y scan deflection plates, the beam was electrostatically scanned before it
passed through the aperture providing a uniform implant distribution across the sample. It is known that on entering a target, an ion beam causes the emission of secondary electrons which may cause inaccuracies in the dosimetry measurements. For this reason, a suppression plate held at a potential of -300V with respect to the sample was situated between the aperture and sample, thereby eliminating this effect. Because highly energetic ions release their energy on entering a sample, heating of that sample will occur, which will vary with ion mass, accelerating potential, dose and dose rate (beam current). This heating effect may result in annealing of the sample during implantation and has been shown to enhance diffusion of the implanted ion species. To minimise the effects of beam heating, the samples were mounted in good thermal contact to a large mass sample holder which acted as a heat sink, thereby conducting the heat away from the sample during implantation. It has been reported that a temperature rise of approximately 50°C occurred for a 1MeV He ion implantation into GaAs with a beam current of 1.2μA after a 100mins implantation time. In this work, the implantation current was kept low to minimise the effects of beam heating.

A source of error which may occur during implantation is drift in the magnet and acceleration voltage power supplies, due to possible heating effects. These have been investigated and found to be 0.01% and 4% respectively. It was seen that these effects would not result in any significant change in the implantation conditions used in this work.
3.3.3 Encapsulation Procedure

After implantation, the samples were annealed to remove implantation damage and to diffuse the implanted silicon through the MQW stack causing intermixing of the layers. Annealing was performed at temperatures ranging from 750°C to 900°C which are considerably greater than the incongruent evaporation temperature of GaAs of approximately 600°C. Consequently it was necessary to use an encapsulant to prevent the preferential evaporation of arsenic and degradation of the sample surface. Although capless and proximity capped samples were annealed for a comparison of the effects of the encapsulants, for the majority of the work presented here, the pyrolitically deposited dielectric encapsulant silicon nitride (Si₃N₄) was used. During the course of the work, two types of silicon nitride encapsulant were used. The first where oxygen was suspected of being incorporated into the layer during growth and had a stochiometry of SiOₓNᵧ, and the second where the oxygen content of the encapsulant was removed and had a stochiometry SiₓNᵧ. A further dielectric encapsulant, aluminium nitride (AlN), was used for comparison studies and the evaporation procedure used is described below.

3.3.3.1 Silicon Nitride

The chemical vapour deposition (CVD) silicon nitride apparatus is similar to the one initially designed by Donnelly et al. From the schematic diagram of the system shown in Figure 3.3, it can be seen that the system contained a deposition chamber which housed a rectangular graphite strip. The chamber was evacuated to <10⁻¹ Torr by a two stage pumping procedure using rotary and diffusion pumps and the
Figure 3.3 - Schematic Diagram of CVD Silicon Nitride System.
rectangular graphite strip was heated by passing a current of between 30Amps and 100Amps through it. An inlet valve to the chamber allowed the introduction of the reagent gasses silane (SiH₄) and ammonia (NH₃). For reasons of safety, bottles of 5% silane in 95% nitrogen were used in this work. The fundamental process that governed the growth of the nitride is described by the equation:-

\[ 3SiH_4 + 4NH_3 \rightarrow Si_3N_4 + 12H_2 \]  

(3.1)

It is known that the reaction rate becomes significant for temperature in excess of 500°C. A silicon nitride layer of approximately 1000Å was deposited in all cases except where a dual encapsulant was used, as described in Section 3.3.3.3, when a layer of approximately 300Å was used. It is known that the colour of the deposited encapsulant can be correlated with its thickness, as shown in Table 3.1. This relationship was used for determining the thickness of the grown layer.

Layers of silicon nitride were deposited using the following procedure. The samples were loaded onto the centre of the graphite strip and the chamber was closed and evacuated to approximately 10⁻⁴ Torr. The chamber was then flushed through with nitrogen for five minutes to remove any remaining oxygen. Silane, ammonia and nitrogen were then introduced at flow rates of 20ml/min, 30ml/min and 150ml/min respectively. The flow rates of these gasses and the deposition time and temperature determined the layer thickness. The samples were then heated to 585°C for typically 8secs to grow approximately 1000Å of silicon nitride.
<table>
<thead>
<tr>
<th>Order</th>
<th>Colour</th>
<th>$\text{Si}_3\text{N}_4$ Thickness Range (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Silicon</td>
<td>0-0.020</td>
</tr>
<tr>
<td></td>
<td>Brown</td>
<td>0.020-0.040</td>
</tr>
<tr>
<td></td>
<td>Golden Brown</td>
<td>0.040-0.055</td>
</tr>
<tr>
<td></td>
<td>Red</td>
<td>0.055-0.073</td>
</tr>
<tr>
<td></td>
<td>Deep Blue</td>
<td>0.073-0.077</td>
</tr>
<tr>
<td>First</td>
<td>Blue</td>
<td>0.077-0.093</td>
</tr>
<tr>
<td></td>
<td>Pale Blue</td>
<td>0.093-0.100</td>
</tr>
<tr>
<td></td>
<td>V. Pale Blue</td>
<td>0.100-0.110</td>
</tr>
<tr>
<td></td>
<td>Silicon</td>
<td>0.110-0.120</td>
</tr>
<tr>
<td></td>
<td>Light Yellow</td>
<td>0.120-0.130</td>
</tr>
<tr>
<td></td>
<td>Yellow</td>
<td>0.130-0.150</td>
</tr>
<tr>
<td></td>
<td>Orange-Red</td>
<td>0.150-0.180</td>
</tr>
<tr>
<td>First</td>
<td>Red</td>
<td>0.180-0.190</td>
</tr>
<tr>
<td></td>
<td>Dark Red</td>
<td>0.190-0.210</td>
</tr>
<tr>
<td>Second</td>
<td>Blue</td>
<td>0.210-0.230</td>
</tr>
<tr>
<td></td>
<td>Blue-Green</td>
<td>0.230-0.250</td>
</tr>
<tr>
<td></td>
<td>Light Green</td>
<td>0.250-0.280</td>
</tr>
<tr>
<td></td>
<td>Orange-Yellow</td>
<td>0.280-0.300</td>
</tr>
<tr>
<td>Second</td>
<td>Red</td>
<td>0.300-0.330</td>
</tr>
</tbody>
</table>

Table 3.1 CVD $\text{Si}_3\text{N}_4$ Colour Thickness Relationship.\textsuperscript{[38]}
A study was undertaken of the high temperature stability of this encapsulant. This was performed using a conventional flow furnace, as described below, by depositing 1000Å of $\text{Si}_3\text{N}_4$ on GaAs and annealing at temperatures up to 950°C for times up to 2 hours. It was found that at temperatures of 850°C and above the encapsulant had failed within 1 hour, due to the presence of numerous "pin-hole" defects on the sample surface. By reducing the temperature to 800°C it was seen that the encapsulant withstood a 2 hour anneal. To provide the long time annealing required in this work, the temperature was further reduced to 750°C and it was observed that at this temperature, the encapsulant withstood annealing for times up to 6 hours. Therefore, this encapsulant permitted long time annealing at a temperature at which Si implantation IILD was known to occur.\(^3\,^9\)

As with all the encapsulation procedures used during this work, the sample surface prior to encapsulation had to be free of any contaminants. It was found that the presence of dust particles resulted in the failure of the encapsulant during annealing.

The presence of oxygen within the chamber during growth of the encapsulant is known to result in the deposition of $\text{SiO}_x\text{N}_y$ films\(^3\,^10\) due to the preferential reaction of silane with oxygen. During the course of this study, work was undertaken to reduce the oxygen content in the deposition chamber during growth by replacing all the vacuum seals in the system. Analysis of the samples annealed with this improved $\text{Si}_3\text{N}_4$ encapsulant were observed to give substantially improved results in the characteristics of the disordered regions. Consequently, a study of the effects of the two encapsulants was undertaken and the results are presented in Chapter 5 and Chapter 6.
It was observed that growth of the improved encapsulant was considerably slower such that a temperature of 630°C for 45 secs was required to grow a 1000 Å layer at the previous gas flow rates.

It was observed by other workers using this improved encapsulant that it remained stable at higher temperatures for longer times. However, in order that a direct comparison of the long time annealing effects could be undertaken between the two encapsulants, all long time annealing reported here, was performed at 750°C. At a later stage, rapid thermal annealing was performed at higher temperatures using the improved encapsulant in an attempt to reduce the deep level emissions seen using PL, as described in Chapter 5.

3.3.3.2 Aluminium Nitride

The apparatus used for the deposition of aluminium nitride films (AlN) is shown in Figure 3.4. The system consisted of a glass chamber which was evacuated to approximately 10⁻⁵ Torr by a pumping arrangement similar to that of the CVD silicon nitride system. The glass chamber housed a tungsten filament which was supplied from a 100Amp power supply. A shutter was mounted between the filament and sample table which prevented aluminium being deposited directly on to the samples during the early stages of the evaporation.

The aluminium nitride deposition procedure was carried out as follows. A 6cm length of 2mm diameter Al wire was mounted on the filament and the samples placed on the sample holder which was 20 cm from the filament. The chamber was then evacuated to 10⁻⁵ Torr. Upon reaching this pressure, ammonia gas was bled into the chamber
Figure 3.4 - Schematic Diagram of Aluminium Nitride Deposition System.
until a pressure of between $10^{-2}$Torr to $10^{-4}$Torr was obtained and had stabilised. Care was taken to ensure the pressure did not rise above $10^{-4}$Torr as this would have resulted in oil from the diffusion pump entering the deposition chamber and contaminating the samples. The tungsten filament was heated until the aluminium wire melted and then started to evaporate. Evaporation was allowed to continue for two or three seconds before the shutter was opened, which ensured surface contaminants of the Al wire were not deposited on the samples. Once the evaporation had started and the shutter was opened, the aluminium vapour, which had reacted with the ammonia gas to form aluminium nitride, was deposited on the samples. After all the Al had been evaporated, the chamber was then pumped down to $10^{-4}$Torr before it was let up to air and the samples removed. This procedure resulted in a 600Å layer of AlN being deposited on the sample surfaces. The reaction producing the AlN is described by the equation:

$$2Al + 2NH_3 \rightarrow 2AlN + 3H_2$$

(3.2)

This novel method for depositing AlN was first reported by Bensalem et al.[11]

3.3.3.3 Dual Encapsulant: Si$_2$N$_4$/AlN

It had been found previously[11] that it was difficult to remove the AlN encapsulants from the sample surfaces after annealing and this was attributed to an Al rich layer being deposited during the early stages of the film growth. To overcome this problem, a double layer encapsulant consisting of 300Å of silicon nitride followed by 600Å aluminium nitride was used and found to be easily removed after annealing.
Consequently, it was this dual encapsulant which was used during this work, when a comparison of the effects of encapsulants on disordering was performed as described in Chapter 5.

3.3.4 Annealing Techniques

To diffuse the implanted silicon through the MQW and cause interdiffusion of the layers, long time annealing was required. Consequently, the majority of the annealing performed during this work was undertaken using a conventional flow furnace. For reasons described in Chapter 5, rapid thermal annealing (RTA) was undertaken using a double graphite strip heater. A description of the systems and the operational procedures used are presented below. In all cases, except where capless and proximity caps were used, the annealing stage followed the deposition of an encapsulant. In the case of samples prepared for analysis using photoluminescence, annealing was the last processing stage and PL analysis was performed as described in Chapter 4.

3.3.4.1 Furnace Annealing

The conventional flow furnace used for the long time annealing is shown in Figure 3.5. A silica quartz tube was heated using the three elements placed along its length. The middle element provided the main source of heating with the elements at each end used to extend the range of the constant temperature plateaux within the furnace. In order to provide an inert atmosphere during the annealing process, nitrogen was passed through the furnace over the samples at a flow rate of 1 ltr/min.
Figure 3.5: Schematic Diagram of Conventional Flow Furnace.
The temperature at the centre of the furnace was monitored using a thermocouple inserted into the furnace using a silica quartz rod, and the required temperature obtained by controlling the power supply to the elements. Once the required temperature was reached, it was allowed to stabilise for 30mins prior to removing the thermocouple and inserting the samples which were placed on a boat. Care was taken to ensure that the samples were placed in the same position within the centre of the furnace as previously occupied by the thermocouple. By moving the position of the thermocouple within the furnace and measuring the temperature change, a gradient of 1°C/cm at 750°C in the centre of the furnace was recorded. As it was believed that the samples were placed with an accuracy of 2cm to 3cm, this effect was negligible.

For reasons described above, all long time anneals were performed at 750°C. Insertion and withdrawal times of the boat containing the samples was set to 30secs in order to maintain a constant rate of rise and fall of temperature for different sample batches. The time taken to insert and remove the samples was set at 30secs to reduce the thermal shock and prevent breakage of the boat. The effect of altering the N₂ flow rate on the furnace temperature was undertaken, but no temperature variation was observed with the flow rate between 0.5ltr/min and 1.5ltrs/min.

To determine the accuracy of the temperatures indicated by the thermocouple, the well defined melting points of aluminium (660°C), silver (962°C) and gold (1064°C) were used to calibrate the thermocouple. These elements were chosen because their melting points were in the temperature range of interest to this work. The furnace was preheated to an indicated temperature approximately 10°C degrees greater than the melting point of the element to be used and allowed to stabilise as before. The metal sample was then placed into the furnace at the same point previously occupied by the thermocouple and left for 15mins to allow the sample to reach the measured
Figure 3.6: Thermocouple Calibration Curve.
temperature. Depending on whether the sample melted or not, the temperature was increased or decreased and the process repeated. The calibration was performed using a nitrogen flow rate of 1 ltr/min and it was seen that the indicated temperature of the thermocouple was always greater than the true temperature by approximately 1%, as shown in Figure 3.6. It was seen therefore that the true and indicated temperatures were in close agreement.

3.3.4.2 Dual Graphite Stripe Annealing

Rapid thermal annealing (RTA) is a high temperature short duration annealing technique which has been employed extensively for the study of electrical activation of implanted ion species where diffusion of the impurity species was to be restricted. In the work undertaken here, RTA provided a means of heating at temperatures where long time annealing would have resulted in failure of the encapsulant, and was undertaken to modify the deep level emissions which accompanied disordering, observed using PL, as described previously. All rapid thermal annealing was performed at 900°C using the double graphite strip heater (DGSH) shown schematically in Figure 3.7. The system consisted of a glass chamber which was evacuated using a rotary pump to a pressure of approximately 10⁻¹ Torr. Within the chamber, there were two graphite strips separated by a gap of approximately 1.5 mm, which were heated by passing a current of typically 100 Amps through them. The sample to be annealed was placed in the centre between the two strips, which ensured uniform heating and a constant temperature across the sample during annealing. The temperature of the graphite strips was monitored using a pyrometer which was focused onto the centre of the graphite strips. The annealing
Figure 3.7 - Schematic Diagram of Dual Graphite Strip Rapid Thermal Annealing System.
procedure used was as follows. After encapsulation the samples were cleaved and placed in the middle region between the graphite strips. The chamber was then evacuated to approximately 10⁻¹ Torr and backfilled with nitrogen to provide an inert atmosphere. A current was then passed through the strips which took approximately 3 secs to reach 900°C, this temperature was maintained for the required time and the current switched off. Cooling of the sample to 100°C took approximately 8 secs, after which time the chamber was again evacuated, to remove any arsenic vapour released during the anneal, backfilled with nitrogen and the sample removed. The accuracy of the pyrometer was known to be within ±16°C which was not significant at the temperature employed in this work.

3.4 MQW Stripe Waveguide Fabrication

Waveguides were fabricated by masked implantation to produce buried disorder delineated waveguides and by masked wet chemical etching to produce ridge waveguides. The processing steps used to fabricate disorder delineated waveguides is shown in Figure 3.8.

3.4.1 Photolithography

The fabrication of stripe optical waveguides by both IILD and wet etching required the selective masking of the sample surface to define the waveguide dimensions. This was achieved using photolithography, where the photoresist was inert to the chemical etchants used and, in the case of ion implantation, was sufficiently thick to stop 500 KeV Si ions. The photoresist used in this work was Shipley 1400/37 which was
a) Photolithography.

b) Implantation.

c) Mask Removal, Encapsulation and Annealing.

d) Thinning and Cleaving.

Figure 3.8: Buried Stripe Waveguide Fabrication Process.
developed by Shipley to provide improved protection in ion beam applications. For
the fabrication of stripe waveguides defined using IILD, the photoresist was applied
and patterned, as described below, prior to implantation, and was removed after
implantation by boiling in acetone. In the case of the waveguides defined using wet
chemical etching, the samples were thinned prior to the photolithographic stage, for
reasons described in Section 3.4.2, and removed, after etching, in acetone. The
procedure for defining the photoresist stripes was as follows. To ensure good
resolution and contamination free films, the photoresist was filtered using a 0.8μm
membrane prior to deposition on the samples, which were then spun at 4000rpm for
30secs giving a uniform 2.8μm thick layer of photoresist. The thickness of the
photoresist was determined by talystep measurement of stripes defined on test
samples of GaAs after the last thermal processing stage described below. The
samples were then baked at 95°C for 30mins in an oven followed by exposure to ultra
violet light for 60secs through a mask to define the stripes, using a Kasper mask
aligner. Developing was performed using a solution of 50% developer, 50%
deionised water for 60secs at room temperature after which the samples were rinsed in
deionised water and blown dry in filtered nitrogen. The exposure time was
determined by exposing a series of samples for times up to 90secs and standardising
on a one minute developing time to remove the exposed photoresist. The final stage
consisted of a 115°C bake for 30mins. After being processed, the photoresist stripes
were examined under an optical microscope to ensure uniformity of width and the
absence of defects. In the event of poor line quality, the photoresist was removed in
acetone, the sample cleaned and the process repeated.
3.4.2 Thinning and Cleaving

To provide reproducible good quality cleaved facets for optical waveguide measurements, the samples were thinned prior to cleaving. It was found that waveguide samples which had not been thinned prior to cleaving often required recleaving due to the poor quality of the cleaved facet, which would result in increased attenuation due to scattering losses. Thinning was performed using a mechanical jig which had a 250μm groove milled in the top surface. The sample was mounted face down in the groove using wax and the back surface of the specimen was mechanically thinned by motion over a rigid abrasive sheet. After thinning, the specimen was removed using trichloroethylene and cleaved using a scalpel blade. Finally the sample was cleaned as described previously prior to waveguide measurements being undertaken. All cleaved facets were inspected under an optical microscope to ensure good quality and, if a damage facet was found, the sample was recleaved. Thinning and cleaving of waveguides fabricated using IILD was performed after annealing, with the encapsulant still on the sample surface for protection. However, because it was necessary to mount the sample face down during thinning, this process was performed prior to photolithography for the wet etched guides.

3.4.3 Characterisation of Etchants

Wet etched waveguides were fabricated to measure the optical loss of the as-grown material. Several etchants were investigated by inspecting the surface roughness of GaAs after etching, using an optical microscope. The etchants, which gave the
smoother etched surfaces, were \(3\text{H}_2\text{SO}_4:1\text{H}_2\text{O}_2:1\text{H}_2\text{O}^{13,14}\) and \(1(1\text{N NaOH}):1\text{H}_2\text{O}_2:10\text{H}_2\text{O}^{13,15}\) and these were used in the subsequent fabrication of waveguides. The latter etchant had been reported to reveal two distinctly different sets of crystal planes depending on whether the etched stripe lay parallel to the \(<110>\) or \(<T10>\) crystal axis. It had been observed that stripe ridges fabricated parallel to the \(<110>\) and \(<T10>\) axis revealed the \(<2210>\) and \(<1\bar{T}1>\) crystal planes respectively. Therefore in the work undertaken here, after cleaving the waveguides, the end sections were inspected using a Scanning Electron Microscope (SEM) and the crystal orientation determined. This provided a means of aligning the as-grown wafers and ensuring the stripe waveguides fabricated by wet chemical etching were parallel to the \(<110>\) axis and consequently, had the same cross-sectional profile.

To fabricate waveguides in the structure used in this study, the etch rates of GaAs and Al\(_{0.3}\)Ga\(_{0.7}\)As had to be determined. This was achieved by partially covering samples of the respective materials in black wax and immersing in the etchant for 15secs which was followed by rinsing in distilled water. The black wax was removed using toluene and the step height determined by talystep measurements. The etch rates for GaAs and Al\(_{0.3}\)Ga\(_{0.7}\)As were found to be 1\(\mu\text{m/min}\) and 0.36\(\mu\text{m/min}\) respectively for the \(3\text{H}_2\text{SO}_4:1\text{H}_2\text{O}_2:1\text{H}_2\text{O}\) etchant and 1\(\mu\text{m/min}\) and 0.96\(\mu\text{m/min}\) respectively for the \(1(1\text{N NaOH}):1\text{H}_2\text{O}_2:10\text{H}_2\text{O}\) etchant. Therefore etching times of 125secs and 80secs were calculated and used to etch through the top cladding layer and MQW region for the \(3\text{H}_2\text{SO}_4:1\text{H}_2\text{O}_2:1\text{H}_2\text{O}\) and \(1(1\text{N NaOH}):1\text{H}_2\text{O}_2:10\text{H}_2\text{O}\) etchants respectively. A typical SEM micrograph of the cross section of a 10\(\mu\text{m}\) wide waveguide fabricated using the \(1(1\text{N NaOH}):1\text{H}_2\text{O}_2:10\text{H}_2\text{O}\) etchant is shown in Figure 3.9 and shows the
Figure 3.9: SEM Micrograph of 10μm Wide Waveguide Fabricated by Wet Etching in 1\( \text{(1N NaOH):1 H}_2\text{O:10 H}_2\text{O} \) for 80secs.
planes, confirming the waveguide orientation to be along the $<110>$ axis. After etching, the photoresist was removed in acetone and the samples cleaned and cleaved as previously described.

In conclusion, the techniques used in the processing of samples for photoluminescence analysis and in the fabrication of stripe waveguides have been presented.
REFERENCES

3.1 J.S. Roberts. SERC Central Facility for III-V Material Growth, University of Sheffield. Private Communication.


3.5 S.T. Picraux. 'Ion Implantation in Semiconductors and Other Materials', Editor B.L. Crowder. 641, Plenum Press, 1973


3.8 J.T. Milek. 'Handbook of Electronic Materials', 3rd Ed. 15, 1971


4.1 Introduction

The two measurement techniques used in this study were photoluminescence and optical waveguide characterisation. Photoluminescence was used to study impurity induced layer disordering of MQW samples using Si implantation, and to determine the optical quality of the as-grown material. Investigation of the as-grown MQW structures was undertaken to ensure consistency in structure and composition between the wafers used in this study. Optical waveguide analysis was used to characterise the properties of waveguides fabricated using wet chemical etching of the as-grown material, thereby providing a measurement of the intrinsic loss of the MWQ structure and the characterisation of buried stripe optical waveguides, which were fabricated using selective area disordering.

In this chapter, these techniques and their configuration used in this work are discussed. Following the descriptions of the equipment and the experimental procedures used, a section is included which discusses how the errors in the
measurements reported have been accounted for in the results presented later in this work. The calculations used to determine the waveguide propagation losses reported in this work are also discussed here.

4.2 Photoluminescence Measurements

4.2.1 Description of Equipment

A schematic diagram of the photoluminescence system used in this work is shown in Figure 4.1. The argon ion laser beam was passed through an optical chopper from which a synchronising electrical output was fed to the lock-in amplifier. By connecting the output of the cooled Ge photodetector to the amplifier and selecting a chopper frequency which was greater than the mains frequency (50Hz) or any associated harmonics, the effects of ambient lighting were reduced. Consequently, a chopper frequency of 330Hz was used during the course of this work. To ensure that the correct laser line emission was selected, the chopped laser beam was then passed through a 514.5nm band-pass filter which had a transmittance of 45%. This wavelength was used to ensure that the laser photon energy was greater than the bandgap energy of the samples analysed and was used to generate electron-hole pairs. The transmittance of the lenses and filters in the beam path were used to determine the incident laser power on the sample surface, as calculated in Section 4.2.2. After filtering, the laser beam was then focused down onto the sample which was held at low temperature in a cryostat which was evacuated to approximately 10⁻⁴Torr. This pressure was achieved by pumping to circulate the cooling gas. The cryostat jacket
Figure 4.1: Schematic Diagram of a Photoluminescence System.
was designed to act as a thermal insulator, and therefore it was evacuated to approximately $10^{-6}$ Torr using a conventional two stage pumping process similar to that used in the Si$_3$N$_4$ CVD system, as described in Chapter 3.

The purpose of cooling the sample was to eliminate the effects due to thermally generated carriers. Furthermore, reducing the temperature and consequently the lattice vibration, is known to cause the energy bands within the semiconductor to constrict, thereby making the radiative recombination between different energy levels sharper and more easily identifiable.\textsuperscript{10-11} A consequence of this is that as the temperature of the sample is reduced, more discrete energy transitions can be observed. Cooling of the sample has normally been achieved using liquid nitrogen at 77K or liquid helium at 4K. In the work presented here, all PL measurements were performed on samples cooled using liquid nitrogen. This was due to the large changes expected in the band structure of the material produced by disordering, which did not require the resolution provided by cooling to 4K using liquid helium.

To maintain the sample at a constant low temperature, liquid nitrogen was pumped into the cryostat and through a heat exchanger where it was heated to 80K and became gaseous. After passing the sample, the temperature of the gas was monitored until an equilibrium temperature, between the sample and the nitrogen gas, of 80K was maintained. The control of the gas temperature was performed automatically and was specified by the manufacturers to be accurate to $\pm 0.1$K, which would have no significant effect on the spectra measured in this study.

Light emitted from the sample was collected and focused into the spectrometer through the slits, as shown in Figure 4.1. Because the spectrum of the emitted light extended into the infrared, the optics used on the output side of the cryostat were
made of fused silica, as were the cryostat windows, this material having a transmittance which was typically greater than 99% at these wavelengths. To stop any of the laser beam reflected from the sample surface entering the spectrometer, a 570nm high pass optical filter was included in the output optics of the system.

Light entering the spectrometer fell upon a diffraction grating which was rotated, thereby selecting the wavelengths of interest for detection. The slit width and wavelength scan range determined the resolution of the output spectrum. Although reducing the slit width increased the resolution of the system, it reduced the emitted power collected by the spectrometer, so that a higher sensitivity setting was required on the lock-in amplifier, thereby increasing the effects of noise. Consequently, the slit width setting was a compromise between these two effects. After passing through the spectrometer, the selected wavelength beams were detected by a liquid nitrogen cooled Ge detector and their intensities recorded.

A problem could arise within the spectrometer due to absorption of the required spectrum from water vapour present in the atmosphere, which is known to absorb in the infrared part of the electromagnetic spectrum. To overcome this problem, the spectrometer was continuously purged with dry nitrogen gas displacing any water vapour.

4.2.2 Experimental Procedure

In the work presented here, an argon ion laser output power of 100mW at a wavelength of 514.5nm was used and focused down to a spot size of 1mm² on the sample to be measured. Accounting for the transmittance of the lenses and filter in
the input path of the system, and that the beam was chopped, an incident input power on the sample surface of approximately $2 \times \frac{\text{W}}{\text{cm}^2}$ was calculated. This input power was sufficient for the emitted spectrum to be conveniently analysed, whilst insufficient to cause problems due to sample heating. As stated previously, all measurements were performed at 80K using nitrogen as the cooling gas. The samples were cooled to a temperature 3K above that of liquid nitrogen because at this temperature, the nitrogen was in a gaseous state and this reduced the possibility of wastage due to the cryostat being filled with liquid nitrogen.

After the temperature in the cryostat had stabilised at 80K from room temperature, which took approximately 90mins, the nitrogen supply pump was switched off and the pressure within the cryostat was allowed to reach atmospheric pressure. This was performed to ensure that air was not drawn into the cryostat when it was opened, and to prevent any water vapour freezing on the cryostat windows. The samples, which had previously been mounted, were then inserted and the nitrogen pump was switched on. The temperature was again allowed to stabilise before measurements were undertaken. To ensure that most of the spectrum emitted from the samples was collected by the output lens, the spectrometer was set at a known emission wavelength and the sample and lens positions optimized. Spectra were taken for wavelengths ranging from 650nm to 1350nm, which covered the emissions wavelengths for a completely disordered region, the unmixed MQW structure and the deep level emissions that were seen to accompany the disordering process. Measurements were not undertaken at longer wavelengths as these coincided with second harmonic emissions from the mixed AlGaAs and top cladding layers. Spectra were taken using slit widths of 200µm for the as-grown material, due to the intensity of the emitted IR spectra and 1000µm for the implanted samples. Prior to the recording of a spectrum,
the position of the diffraction grating was noted and checked against the position indicated by the control computer. This check was also performed following the recording of each spectrum to ensure that the recorded and true position of the diffraction grating were in agreement. If any discrepancy was noted the spectrum was retaken. After a spectrum had been taken, individual peaks were then rescanned across a smaller wavelength range thereby increasing the resolution and providing further detail of the emissions for analysis.

Photoluminescence analysis showed that there were deep level emissions associated with the silicon induced mixing process, as discussed in Chapter 5. To provide an indication of the possible suitability of a mixing process for waveguide fabrication, a ratio of the integrated intensities of the deep level emission to band-edge emission was used. The intensity of the band-edge emission provided an indication of the optical quality of the mixed region, whilst the deep level emission gave an indication of the possible absorption that would occur at the propagation wavelengths of interest in the waveguide analysis.

4.2.3 Experimental Accuracy

The resolution of the PL system was determined by the slit widths and scan range used during the individual measurements. Since all disordered samples were measured using a slit width of 1000μm over the scan range of 650nm to 1350nm, which produced a system resolution of ±3meV, this value has been used as the limit of accuracy in the band-edge energies reported. In the analysis of the as-grown
material, where the slit width was reduced to 200\(\mu\)m and a scan range of 790nm to 830nm used, providing a system resolution of \(\pm 1.2\)meV, this improved value has been used.

4.3 Optical Waveguiding Measurements

4.3.1 Description of Equipment

The optical system used for waveguide characterisation is shown in Figure 4.2. To provide isolation from vibration sources elsewhere in the building, the optical system was supported on a pneumatically mounted bench, with the optical table legs placed on vibration absorbing mats. The majority of the waveguide measurements were undertaken at the propagating wavelength of 1.15\(\mu\)m, although some measurements were performed for comparison at the longer wavelengths of 1.30\(\mu\)m and 1.54\(\mu\)m. The 1.15\(\mu\)m and 0.63\(\mu\)m sources were from helium neon (HeNe) gas lasers whilst two InGaAsP semiconductor lasers were used to provide the 1.3\(\mu\)m and 1.54\(\mu\)m laser lines. In each case the infrared lasers had a maximum output power of 1mW with the 0.63\(\mu\)m lasers supplying an output power of approximately 6mW.

Following the system through, the HeNe laser output contained two laser wavelengths i.e. 1.15\(\mu\)m and 1.16\(\mu\)m. These were separated by means of a 1\(\mu\)m line width, 600lines/mm blaze diffraction grating, which then allowed the 1.16\(\mu\)m beam to be blocked and the 1.15\(\mu\)m line focused onto the input facet of the cleaved sample by means of a X40 objective lens. After propagation through the waveguide, the emitted
Figure 4.2: Schematic Diagram of the Optical Waveguide Characterisation System Used in This Study.
light was then collected by a further X40 lens and focused down onto a Ge photodetector whilst a pellicle beam splitter was used to split off 65% of the beam into an infrared camera.

To enable the focusing of both the laser output on to the input facet of the waveguide and the output of the waveguide on to the IR camera and photodetector, the two lenses were mounted on X-Y-Z-θ translation stages. The sample was mounted between the two lenses on an X-Y translation stage. For the purposes of this work, the Z axis was defined as the direction of propagation and θ was rotation in the horizontal plane. To provide the fine adjustment required to optimize the laser input into the waveguide and to focus the waveguide output, piezoelectric transducers were used to control the X-Y-Z-θ stages. Initial focusing of the lenses on the sample facets was performed at the visible wavelength using the two 0.63μm HeNe lasers and, once used for this purpose were switched off for the remainder of the waveguide measurement process.

With all the HeNe lasers used, the emitted beam could be blocked by sliding an opaque shutter across the laser output which allowed the laser to remain excited and to prevent the laser tube from cooling. This was of particular importance when the 1.15μm HeNe laser beam had to be isolated whilst the samples were changed, so that the output power remained constant during the waveguide measurements.

For the same reasons as stated in the PL study, the laser beam was chopped, and both the Ge output and optical chopper synchronising signals were fed to a lock-in amplifier. Again, the chopping frequency was selected so as not to coincide with the mains or an associated harmonic.
The recording of mode profile data was performed by connecting the output of the infrared camera to a video analyser, which scanned the waveform focused on to the camera tube both horizontally and vertically and supplied the data to a graph plotter.

For measurements at the longer wavelengths, the output fibre pigtail of the semiconductor laser was positioned in front of the optical chopper in position A, the output was then focused using a fused silica lens at position B, on to the back of the X40 input objective, allowing the use of the same focusing and measurement arrangement as before.

The pigtail fibre outputs of the semiconductor lasers were not polarisation maintaining and consequently, the optical loss measurements undertaken at 1.3μm and 1.54μm were for unpolarised propagated modes.

A calibration curve for the Ge detector used in this series of experiments is shown in Figure 4.3. The output beam of the 1.15μm laser was focused down onto the photodetector whose output current was recorded. Neutral density filters of known transmittance, were then inserted in the beam path and the detector output current was remeasured. It was seen that there was a linear relationship between the output current and the incident light intensities used in this work.

As the measurement of 1/e mode widths were to be undertaken, the system was calibrated such that a mode profile, as plotted by the X-Y recorder using the video analyser, could be converted to the true dimensions of the mode. This was achieved by inserting a 50μm mounted pinhole between the two lenses and plotting the focused
Figure 4.3: Calibration Curve of the Ge Photodetector Used for the Characterisation of Optical Waveguides.
transmitted beam, detected by the camera. It was found that a plot dimension of 12mm corresponded to a true dimension of 1µm in both the horizontal and vertical directions.

4.3.2 Experimental Procedure

After switching on the 1.15µm HeNe laser, it was allowed to stabilise, which took approximately one hour, before any measurements were made. This was undertaken to ensure that the input power to the waveguide remained a constant during the characterisation of the waveguide. This procedure was not required for the semiconductor lasers which were found to have stable output powers when switched on.

Prior to waveguide measurements being undertaken, the input laser beam 1/e mode width profiles were recorded. This was performed by bringing the two lenses together and plotting the horizontal and vertical intensity profiles of the incident laser beam using the video analyser and X-Y plotter. Subsequently, a 1/e neutral density filter was inserted into the beam path, before the first objective lens, and the beam profiles were replotted. These dimensions were used in the subsequent calculation of the modal mismatch between the input beam and the propagating waveguide mode, as outlined in Section 4.3.2.1. It was found that, for a given wavelength, the input beam was symmetrical, and did not vary with polarisation. A typical laser beam profile for the 1.15µm wavelength source is shown in Figure 4.4. The 1/e mode widths recorded for all three laser sources at the waveguide input are shown in Table 4.1 with the maximum limits of variation observed during this work.
Figure 4.4 - Typical Output Beam Profile for the 1.15μm Laser Source.
Table 4.1. Input Laser Beam 1/e Mode Widths at the Three Propagation Wavelengths of 1.15μm, 1.3μm and 1.54μm.

<table>
<thead>
<tr>
<th>Wavelength (μm)</th>
<th>Mode Widths (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.15</td>
<td>1.3±0.2</td>
</tr>
<tr>
<td>1.3</td>
<td>1.4±0.2</td>
</tr>
<tr>
<td>1.54</td>
<td>1.7±0.2</td>
</tr>
</tbody>
</table>

It can be seen from Table 4.1 that the 1/e widths of the input beam are similar for the 1.15μm and 1.3μm lasers, but increase at 1.54μm. It is known that the minimum focused spot size of a lens increases with increasing wavelength and these results agree with this effect.\(^{[4,5]}\)

After recording the 1/e input beam mode profiles, the output current of the Ge detector was recorded for both TE and TM polarisation. This provided a measure of the effective input power to the waveguide and was used in the subsequent calculation of the propagation loss of the waveguides, as discussed in the following section. The sample to be measured was then placed on the X-Y sample translation stage and the
0.63\textmu m lasers used to focus the two lenses onto the cleaved facets of the waveguides. Once the sample was at the focal point of the input and output objectives, the opaque shutters were placed across the outputs of the 0.63\textmu m lasers and the required infrared laser turned on. As the working distance of the objectives varied with wavelength, manipulation of the X-Y-Z-\theta lens mountings was required to excite the waveguide and focus the output down onto the photodetector and camera. The optimum coupling of the input beam into the waveguide and focusing of the output of the waveguide, using the piezoelectric transducers, was achieved by monitoring the photodetector output and the mode image on the camera. The Ge photodetector output current was then measured and in the case of the IILD delineated waveguides, the vertical and horizontal 1/e mode profiles were also recorded. Mode profile measurements of the wet etched waveguides were not performed due to their multi-mode nature. The disorder delineated waveguides were found to support a maximum of two modes and, in such cases, the fundamental mode was excited and used for all measurements. Unless stated otherwise, measurements were undertaken for both the TE and TM polarisations.

To change between the TE and TM polarisations when using the 1.15\textmu m HeNe, a half-wave plate was inserted into the path of the input beam prior to it entering the first objective lens. The selected polarisation was confirmed using a polarising sheet which was removed before measurements were undertaken. As stated previously, the pigtail fibres for the longer wavelength semiconductor lasers were not polarisation maintaining, therefore measurements were performed for non-polarised beams.
After the characterisation of each sample the input beam 1/e mode profiles and the detector current were remeasured to ensure that the lenses were not out of alignment and that the laser output had not varied.

4.3.2.1 Waveguide Loss Measurement

During the course of this work waveguide propagation losses were calculated. As stated previously, prior to the characterisation of the waveguides, values were recorded for the 1/e mode profiles and Ge detector currents for the laser output beam. The sample to be investigated was then inserted between the two lenses, and after optimizing the power transmitted by the waveguide, the 1/e mode profiles and detector currents were remeasured for the output beam of the waveguide. These two sets of data were then used to calculate the propagation loss of the waveguide using the following equation:-

\[
\text{Waveguide Loss dB cm}^{-1} = \frac{[\Gamma - F - K]}{L}
\]

(4.1)

where \(\Gamma\) is the insertion loss(dB) of the waveguide; \(F\) the Fresnel reflection loss(dB) at the air/semiconductor interfaces; \(K\) the loss(dB) due to modal mismatch between the input laser beam and the waveguide mode and \(L\) the sample length(cm). The insertion loss \(\Gamma\) was calculated using the equation:-

\[
\Gamma dB = 10 \log_{10}\left(\frac{P_i}{P_o}\right)
\]

(4.2)
where $P_i$ and $P_o$ were the measured detector currents for the input laser beam and waveguide output respectively. To correct for Fresnel loss $F$ at the waveguide input and output semiconductor/air interfaces, the Fresnel reflection coefficient for a single interface was calculated using the equation:

$$R = \left[ \frac{n - 1}{n + 1} \right]^2$$  \hspace{1cm} (4.3)

where $n$ was the refractive index of the MQW waveguide for the particular wavelength and polarisation, as calculated in Chapter 2. Equation 4.3 assumes that the incident input beam is perpendicular to the sample surface and $R$ is defined as the ratio of the reflected beam intensity to the incident beam intensity. To determine the loss in dB the value for $R$ was then inserted into equation 4.4:-

$$F_{dB} = 10 \log \frac{1}{[1-R]}$$  \hspace{1cm} (4.4)

this equation yielded typical losses for the Fresnel reflection of 1.5dB at a single interface, consequently the total loss due to both interfaces was approximately 3dB.

Finally, to determine the waveguide propagation loss, the loss due to modal mismatch $K$, between the input beam and propagating mode in the waveguide had to be determined. This was achieved using the 1/e mode profiles recorded for the input laser beam and propagating waveguide modes. It was observed, as shown in Figure 4.4, that the laser output beams were symmetrical, as were the propagating modes in the IILD delineated waveguides, as shown in Chapter 6. Therefore, the
input beam and propagating waveguide mode were modelled as symmetrical gaussian fields which were inserted into the equation for the normalised overlap integral\textsuperscript{4,5} given by equation 4.5:-

\[
\text{Overlap} = \frac{\int_{-\infty}^{\infty} dy \int_{-\infty}^{\infty} e E dx}{\left[ \int_{-\infty}^{\infty} dy \int_{-\infty}^{\infty} e^2 dx \int_{-\infty}^{\infty} dy \int_{-\infty}^{\infty} E^2 dx \right]^{\frac{1}{2}}} \tag{4.5}
\]

where \(E\) was the gaussian profile for the input beam and \(e\) the equation for the fundamental mode of the waveguide. Solution of the normalised overlap integral yielded the modal mismatch between the input and propagated modes and was performed using the computer model which was developed by based on work performed previously by Reed\textsuperscript{4,5}.

Propagation loss measurements were also undertaken using the technique proposed by Walker\textsuperscript{4,7} for single mode waveguides, which required the heating of the sample and the plotting of the output power as the waveguide went through resonance and antiresonance. The measurement of these oscillations was achieved by the use of the video analyser and X-Y plotter. The equation that was derived for the waveguide propagation loss using this technique was:-

\[
\Gamma_k = -20 \log \gamma = -10 \log \left[ \frac{1}{R} \left[ \frac{T}{T^5} \right]^{0.5} - 1 \right] \tag{4.6}
\]

where \(R\) was the Fresnel reflection, given previously in equation 4.3, and \(T\) the ratio of the transmitted power of the waveguide at resonance and antiresonance.
4.3.3 Experimental Accuracy

Loss measurements and 1/e mode profiles were undertaken for each waveguide on a sample and their average calculated. This average value has been reported during this work. Furthermore, each sample was measured at least twice, with the sample being removed and then replaced on the sample translation stage, to minimise effects due to the misalignment of the laser output beam with the input of the waveguide. To account for any inaccuracies in the optical waveguide measurements, the standard deviation of the results for each sample was used to determine the error bars recorded.

In this chapter, the photoluminescence and optical waveguide characterisation systems and experimental techniques have been presented. A discussion on the accuracies used in the presentation of the results obtained during this work has also been presented.
REFERENCES


4.2 E. Hecht and A. Zajac. 'Optics', 89, Addison-Wesley Publishing Co. Inc., 1974


CHAPTER 5

PHOTOLUMINESCENCE MEASUREMENTS

5.1 Introduction

Photoluminescence is known to be a useful technique for the characterisation of semiconductors, providing information on the band structure and associated energy levels. In this chapter photoluminescence results are presented on the characterisation of the as-grown MQW and impurity induced disordering using silicon implantation and subsequent annealing.

The results described are concerned with the characterisation of the material bandgap before and after various processing steps, to determine the degree of the intermixing produced and the possible suitability of the process for waveguide fabrication.

Prior to the fabrication of stripe waveguides by IILD, implantation was performed on samples without a photoresist mask and these were used to characterise the disordering process. Work was initially undertaken to determine the optimum capping procedure and anneal temperature, as these had been previously reported as having a significant effect on the extent of the induced disordering. Deep level
emissions were found in all samples where significant intermixing occurred, and the relationship between the degree of the intermixing and associated deep levels was investigated for silicon doses initially ranging from \(3 \times 10^{13} \text{ ions cm}^{-2}\) to \(1 \times 10^{14} \text{ ions cm}^{-2}\). Subsequent work was performed on reducing the deep level emissions by improving the encapsulant because, as reported later, they were believed to contribute to the propagation losses seen in the optical waveguides. Work was then undertaken, using the improved cap, to characterise the effects of silicon implantation to doses ranging from \(1 \times 10^{15} \text{ ions cm}^{-2}\) to \(7 \times 10^{15} \text{ ions cm}^{-2}\) on the impurity induced disordering and deep level emission. This allowed the direct comparison of the effect of the improved cap to be achieved and further work on the effects of silicon dose on the degree of IILD.

In an attempt to further reduce the deep level emission, rapid thermal annealing was used and these results are presented, as are the results of experiments performed to determine the source of the deep level emissions.

Photoluminescence also provided a means for determining the efficiency of the photoresist as an ion implantation mask used in the fabrication of stripe waveguides and these results are presented in this chapter.

5.2 Material Characterisation

To ensure that the as-grown wafers used were consistent in quality, structure and composition, PL was performed on the as-grown material. Several samples were
selected from across each wafer so that, in addition to the above, any variations in the MQW structure across the wafer, which may have occurred during growth, could also be determined.

A typical PL spectrum, taken at 80K, for an as-grown sample is shown in Figure 5.1. This shows the main conduction band to heavy hole (ec1-hh1) transition at 806.7nm and conduction band to light hole (ec1-lh1) transition at 801.6nm, which correspond to transition energies of 1.537eV and 1.547eV respectively. The transitions seen at 812.2nm and 814.7nm are believed to be due to residual impurities in the as-grown material, which was known to have a p-type carrier concentration of $1 \times 10^{16}$ cm$^{-2}$, resulting from the presence of carbon impurities which originate from the trimethyl-gallium and trimethyl-aluminium used in MOVPE growth.$^{54}$ The emission seen at 819.9nm is close to that for the value for bulk GaAs of 822.8nm, and work was undertaken to determine the source of this emission, as described in the following section.

By an approximate solution of the Schrödinger equation,$^{53}$ using the material parameters of the constituent layers in the MQW, it was possible to determine the extent of the quantisation of the GaAs wells and consequently to model the emissions for a given material structure. Conversely, by taking the emissions for a given sample it was possible to model the material structure. As an approximate solution was used, the result provided was only used as a guide. It was seen that the quantisation effect was much more sensitive to the thickness of the well than to the composition of the barriers for the structure grown. Typical values calculated for the MQW structure used showed that for a band-energy change of 2meV the Al composition of the
TEMPERATURE 80K
LASER 100mW at 514.5nm
SLITS 200\mu m

Figure 5.1 - Typical P.L. Spectrum of the As-Grown Material.
barriers would have to change by 6%, where a well width variation of only 5Å would have the same effect. Therefore, it was the well thickness parameter that was varied in the modelling.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Wafer CPM 383</th>
<th>Wafer CPM 436</th>
</tr>
</thead>
<tbody>
<tr>
<td>ecl-hh1 (eV)</td>
<td>1.539 ± 0.002</td>
<td>1.522 ± 0.002</td>
</tr>
<tr>
<td>Ihl-hhl (meV)</td>
<td>9.7 ± 0.4</td>
<td>-</td>
</tr>
<tr>
<td>FWHM (meV)</td>
<td>4.5 ± 0.3</td>
<td>8.2 ± 1.1</td>
</tr>
<tr>
<td>Well Width Å</td>
<td>110</td>
<td>170</td>
</tr>
</tbody>
</table>

Table 5.1 Photoluminescence Results for Wafers CPM 383 and CPM 436 Showing Wafer Variations.

The experimental results obtained for the two wafers used in this work are shown in Table 5.1, which shows the main ecl-hh1 peak transition and the full width half maximum (FWHM) energies. The ecl-hh1 energy gave the degree of the quantisation within the GaAs wells, which was then used to determine the theoretical value for the
well widths. The full width half maximum (FWHM) provided an indication of the quality of the well/barrier interfaces. The ec1-lh1 transition energy was only observed for wafer CPM 383 and is also recorded.

It was seen from the results that the growth variation across the individual wafers was characterised by a ±2meV deviation in the ec1-lh1 transition energy. Since this value was close to that of the system accuracy and too small to show a significant change of well width in the model, it suggested that the MQW dimensions and composition were uniform across each wafer. The 17meV decrease in the ec1-lh1 transition energies between the two wafers is indicative of an increase in well width for wafer CPM 436. When these values were modelled, well widths of 110Å and 170Å were obtained for wafers CPM 383 and CPM 436 respectively. This was contrary to the information previously obtained where variations in the layer dimensions during growth were within ±4%. As stated previously, a simplified model was used and the well dimensions were believed to be closer to the ±4% value. A more likely interpretation, of the 17meV variation, was the non-uniform nature of the well/barrier interfaces, which, while reducing the quantisation effect would also account for the observed increase in the full width half maximum (FWHM) of the main transition peak. This increase in the FWHM made the measurement of the ec1-lh1 transition energy, and thereby the lh1-hh1 energy in wafer CPM 436 unobtainable.

5.2.1 Sampling Depth Analysis

As stated in the previous section, an emission in the virgin material at a wavelength of 819.9nm, which is close to that expected for bulk GaAs, was observed in both wafers. A possible interpretation of this emission was that PL was analysing all of the
structure down to the substrate. The only other possible sources for a GaAs emission were those of the top 50Å GaAs cap, grown to prevent the oxidation of the top AlGaAs cladding layer, or the existence of a rogue well within the MQW itself. The latter was thought to be unlikely as this would require such a well to exist in both wafers.

To eliminate the possibility of the emission originating from the GaAs cap, two samples, one from each wafer, were immersed in a solution of $1\text{H}_2\text{SO}_4 : 1\text{H}_2\text{O}_2 : 10\text{H}_2\text{O}$ for one minute, to etch away the GaAs capping layer, this etchant was previously characterised and shown to have an etch rate of 260Å/min for GaAs. The samples were then re-examined using PL and it was found that the 819.9 nm emission was unchanged, suggesting that the GaAs cap was not the source of this emission. The 0.5μm top AlGaAs cladding layer was then removed by immersing both samples for 90 secs in an etchant of 113gKI : 65gI$_2$ : 100mIH$_2$O which had previously been characterised.\cite{55} It was found by use of talystep measurements to determine the depth of etching, that the immersion time had to be increased on that previously reported. This was attributed to the lower Al concentration in the cladding than that contained in the material used for the initial characterisation. Remeasurement of the samples again revealed that the emission at 819.9 nm remained. This series of experiments eliminated the GaAs cap and the upper AlGaAs cladding layer as the source of the emission.

One other possible source of the emission was the existence of an acceptor level whose emission wavelength was coincidentally close to that of bulk GaAs, though this was impossible to determine. This explanation appeared to be more likely due to the
depth and nature of the structure being investigated, where recombination in the lower band gap wells was the most efficient transition mechanism and sampling to a depth of greater than 3.23μm would have to occur for the substrate to be reached.

5.3 Annealing of As-Grown Material

Experiments were undertaken to determine the contribution of thermal processing alone on the degree of the intermixing. So that the effect of Si IILD alone could be determined, this contribution could then be accounted for in the following sections, where the mixing has been enhanced by the introduction of silicon into the MQW by implantation. As several encapsulants were used, and the degree of intermixing is known to be dependent on the nature of the encapsulant used, annealing of as-grown material was performed for the caps employed at the times and temperatures used for IILD.

The capping procedures used during the initial part of this work had, apart for the capless samples, been used in previously reported IILD studies. These were, 1) proximity, where the sample surface is in contact with a similar sample, in this case GaAs, 2) where the sample has a dielectric deposited on the surface, in this case Si₃N₄, and 3) capless, where the sample surface was in direct contact with the ambient nitrogen during annealing. Annealing was performed at 750°C for 5 hours. The shift in the ec1-hh1 transition and FWHM energies for the different samples after annealing at 750°C for 5 hours are shown in Table 5.2.
Table 5.2. Variation in the ecl-hh1 and FWHM Energies Following Annealing at 750°C for 5 hours Using Various Encapsulants.

As seen from Table 5.2, all the band-edge shifts were to higher energies indicating some intermixing had occurred, although in the case of the capless and Si$_3$N$_4$ caps this was small. Significantly more intermixing was observed for the proximity cap and this was believed to be as a result of the arsenic vapour pressure formed between the two samples during annealing. It has been shown previously that the degree of intermixing seen, when using arsenic overpressure during annealing, is sensitive to the vapour pressure that exists at the sample surface. Because of the nature of the capping procedure used here, it was impossible to control this arsenic pressure and consequently the technique would yield inconsistent results. This was seen when this experiment was repeated and intermixing comparable to that observed for the capless and Si$_3$N$_4$ capped samples was observed for the proximity cap.
It was determined therefore, from this work, that the effects of intermixing due solely to the thermal effects were less than 2% of the band-edge energy change expected for a fully mixed sample, and therefore, need not be accounted for in the case where enhanced intermixing was achieved by the introduction of silicon.

5.4 Implantation Mask

The fundamental requirement of the Shipley 1400/37 photoresist mask used during ion implantation was that it must prevent the accelerated ions from entering the surface layers of the sample under the mask. Work was undertaken therefore to demonstrate that the mask used during this study met this criteria.

A piece of as-grown GaAs was cleaved into two samples, one was used as a reference, whilst the other was partially masked using the photolithographic technique previously described. Implantation was then performed on the masked sample to a dose of $3 \times 10^{13}$ ions cm$^{-2}$ using 500KeV silicon. After removal of the mask the samples were analysed using PL. It was found that the band-edge emissions from the reference sample and the masked area of the implanted sample were identical, while no band-edge emission was observed for the material exposed directly to the ion beam. This was due to the implantation damage being non-radiative. This confirmed the suitability of the photoresist as an ion implantation mask for the work undertaken in this study.
5.5 PL Study of Si Ion IILD

5.5.1 Introduction

In the work presented here, the effects of impurity induced layer disordering produced by silicon implantation and subsequent annealing, using a variety of capping techniques and annealing conditions, has been investigated. The purpose of this investigation was to characterise the disordering process, and determine the optimum conditions for later use in the fabrication of buried disorder delineated stripe optical waveguides.

Initially experiments concerned with the determination of the optimum capping and annealing procedures to provide a good quality mixed region were carried out. Once this had been achieved the effects of silicon dose were investigated on the extent of disordering. It became apparent that deep level emissions were produced by the disordering process and that potentially they could result in attenuation of the propagating mode in the waveguide side walls. Therefore, further capping procedures were then investigated with the aim of reducing these deep level emissions and an improved encapsulant was developed. Subsequent work on the effects of dose on the degree of disordering and deep level emission allowed a comparison of the improved cap and previous optimum cap to be performed. In an attempt to further reduce the deep level emission, rapid thermal annealing was investigated and these results are presented here.

Further work to determine the source of emission of the deep levels was undertaken. These results are presented in Section 5.5.8.
To provide an indication of the probable suitability of a mixing process, for the fabrication of disorder delineated waveguides, a ratio of the deep level over band-edge integrated intensities was calculated for each sample. The integrated band-edge emission provided information on the quality of the mixed region, whilst the integrated deep level emission, that of a potential source of attenuation of the propagating mode in the waveguide sidewalls. Therefore, the lower this ratio, the more likely the process was to yield lower loss waveguides.

5.5.2 Encapsulation

To determine the optimum capping procedure to use initially, three capping techniques were investigated, these were proximity capping, the use of a Si$_x$N$_y$ dielectric and where no cap was used, or capless processing.

Silicon implantation into MQW samples at room temperature was performed at 500KeV to a dose of $1 \times 10^{15}$ ions cm$^{-2}$, these samples were then annealed at 750°C for times ranging between 20mins to 5 hours using the three capping procedures mentioned previously. It was observed that layer disordering was achieved for all three capping conditions and that two, broad deep level emissions at approximately 894nm and 998nm, were now evident. A PL spectrum, clearly showing these two peaks, taken from a sample annealed for 3 hours using the proximity cap is shown in Figure 5.2. For most samples however, the more intense deep level emission was seen at 894nm, though the emission at 998nm was still evident, but not as clearly as shown in Figure 5.2. A deep level, corresponding to the 894nm emission seen here, had previously been reported for n-type GaAs$^{[5,8]}$ and AlGaAs$^{[5,10]}$ and had been attributed to a donor gallium-vacancy complex. The emission at 998nm was noted to
Figure 5.2: P.L. Spectrum of a Sample containing $1 \times 10^{15}$ Si cm$^{-2}$ and Annealed for 3hrs at 750°C Using a Proximity Cap.
decrease with the use of a Si$_3$N$_4$ encapsulant. This can be seen by comparing the spectrum in Figure 5.2 with that of a sample containing the same dose and annealed using the Si$_3$N$_4$ encapsulant, shown in Figure 5.5. As the use of a Si$_3$N$_4$ encapsulant is known to restrict arsenic outdiffusion from the sample surface, this emission was tentatively attributed to an arsenic related transition.

For the samples annealed using either the proximity or capless procedures, following a 3 hour and 4 hour anneal respectively, the deep level emissions were more intense than the emission from the mixed region. It was further noted that the emission energy from the mixed regions were approximately 29meV broader than those seen with the Si$_3$N$_4$ capped samples, and had energies corresponding to an AlGaAs layer, where the aluminium concentration was greater than that for the average in the MQW. These results suggested that aluminium was diffusing out of the top cladding layer into the mixed MQW material, and that the disordered region had a higher gallium-vacancy (Ga$_v$) and arsenic-vacancy (As$_v$) density than for the Si$_3$N$_4$ capped samples.

In the case of the Si$_3$N$_4$ capped samples, the emission from the mixed region was seen to decrease in wavelength until a value, equal to that corresponding to the average aluminium concentration in the MQW was reached, thereafter remaining constant for increased annealing times. This suggested that a diffusion inhibiting layer was present at the top cladding/MQW interface which prevented aluminium outdiffusion from the cladding into the mixed MQW material. This diffusion inhibiting layer, which was not seen using the other capping techniques, is known to be sensitive to vacancy concentrations, and therefore capping procedures and the resultant gallium outdiffusion. The ratio of the deep level to band-edge integrated intensities was
also seen to be greatly reduced in comparison to the proximity and capless annealing cases, from an average of 25.6:1 to 2.29:1, and consequently this was the capping procedure adopted in the subsequent experiments.

5.5.3 High Temperature Effects

Having determined the optimum capping procedure, work was undertaken to find the optimum annealing temperature to provide complete intermixing, for the shortest time anneals. The upper limit on the anneal temperature was set by the thermal stability of the Si$_3$N$_4$ cap. This was discussed in Chapter 3, where it was found that the cap failed after an 850°C anneal for 1 hour. Consequently, for longer time annealing 800°C was used and the cap was found to be stable for times up to 3 hours.

Samples were implanted to a dose of 1x10$^{15}$ Si cm$^{-2}$ and annealed at 800°C for times ranging between 1 hour and 3 hours. Analysis of these samples using PL, showed a band-edge emission wavelength which corresponded to a completely mixed layer which was accompanied by a substantial rise in the deep level emission, compared to the samples annealed at 750°C. Furthermore, it was seen that after a 2 hour anneal the emission from the deep levels were more intense than that from the mixed MQW material. This increase was consistent with the suggestion that the source of the deep level emissions at 894nm and 998nm were a donor gallium-vacancy complex and an arsenic related transition, since at an increased temperature the extent of gallium and arsenic outdiffusion would increase. As a compromise between the extent of the intermixing of the MQW and the extent of the deep level emission, a furnace anneal temperature of 750°C was chosen for all further disordering experiments.
5.5.4 Dose Effects

Having determined the optimum annealing and capping conditions, the degree of intermixing with implanted dose was investigated. All implantation was performed with 500KeV silicon to doses of $3 \times 10^{13}$ ions cm$^{-2}$, $3 \times 10^{14}$ ions cm$^{-2}$ and $1 \times 10^{15}$ ions cm$^{-2}$. The samples were then capped with 1000Å Si$_3$N$_4$ and annealed at 750°C for between 1 hour and 5 hours in nitrogen, using a conventional flow furnace.

Samples containing the lower dose of $3 \times 10^{13}$ Si cm$^{-2}$ showed no significant intermixing for anneal times up to 5 hours. A typical spectrum is shown in Figure 5.3. The two peaks at 677.9nm and 696.5nm correspond to emissions from the Al$_{1-x}$Ga$_x$As top cladding and an impurity level which is 50meV below the conduction band of the AlGaAs associated with Si doping, respectively.\cite{543} The emission at 724.5nm corresponds to a mixed layer of an average Al concentration of 15.86%. This increase in the Al composition above that of the average for the MQW suggests the presence of a mixed region at the top cladding/MQW interface, where the extra Al is diffusing into the mixed MQW material from the cladding layer. After annealing for 4 hours this emission from the mixed layer merged with the higher energy emission and became indistinguishable. The two emissions at 677.9nm and 696.5nm remained unchanged with increasing anneal times. Emissions for the deep level only became evident after annealing for 4 hours. The intensity of the emission from the mixed region suggested that only a limited degree of intermixing occurred for this dose and the emission from the unmixed MQW was the predominant feature. Because of the limited intermixing and deep level emissions it was not possible to take FWHM and integrated intensity measurements for this dose.
TEMPERATURE 80K
LASER 100mW at 514.5nm
SLITS 1000µm

Figure 5.3: P.L. Spectrum of a Sample Containing $3 \times 10^{18}$ Si cm$^{-2}$ and Annealed for 2hrs at 750°C Using a Si$_3$N$_4$ Cap.
Samples containing a dose of $3 \times 10^{14}$ Si cm$^2$ showed significant intermixing, with the shift in the band-edge emission wavelength of the mixed region and the integrated intensity ratio for increasing anneal times shown in Figure 5.4. As can be seen, the band-edge emission wavelengths correspond to a region where the Al concentration is greater than that of the average for the MQW. Deep level emissions were observed at 894nm and 998nm and were more significant than those seen for the samples containing $3 \times 10^{13}$ Si cm$^2$, whilst the unmixed MQW emission was reduced. These results suggest that Al was diffusing from the top cladding into the mixed region and that the degree of intermixing and deep level emission are dose dependent. The fall in the integrated intensity ratio suggests that with increased annealing time implantation damage is reduced, and an increase in the band-edge emission occurs due to increased intermixing.

Samples containing the $1 \times 10^{15}$ Si cm$^2$ dose showed a shift in the band-edge emission wavelength towards higher energies, which was associated with the intermixing of the MQW. This band-edge shift reached a maximum value after a 2 hour anneal at 750°C and corresponded to an AlGaAs layer whose Al concentration was equal to that of the average for the MQW, further annealing up to 5 hours showed no further shift in the band-edge emission wavelength. A typical spectrum taken for the sample annealed for 2 hours is shown in Figure 5.5. A plot of band-edge emission wavelength shift for increasing anneal times, with a plot of the ratio of the integrated intensities of the deep levels over band-edge emissions is shown in Figure 5.6. These results suggest the formation of a region between the cladding layer and MQW which restricts the diffusion of Al into the mixed MQW material from the cladding layer, an effect which has been reported previously for similar processing conditions.$^{[3,14]}$ A minimum in the ratio of the integrated intensities was observed for the sample annealed for 2 hours,
Figure 5.4: Band-Edge Emission Wavelength and Integrated Intensity Ratio Shifts with Increasing Anneal Time, for Samples Containing $3 \times 10^{14}$ Si cm$^{-2}$, with the Corresponding % Al conc. of the Disordered Region.
TEMPERATURE 80K
LASER 100mW at 514.5nm
SLITS 1000\(\mu\)m

Figure 5.5 - P.L. Spectrum of a Sample Containing \(1 \times 10^{16}\) Si cm\(^{-2}\) and Annealed for 2hrs at 750°C Using a Si\(_3\)N\(_4\) Cap.
Figure 5.6: Band-Edge Emission Wavelength and Integrated Intensity Ratio Shifts with Increasing Anneal Time, for Samples Containing $1 \times 10^{15}$ Si cm$^{-2}$, with the Corresponding % Al conc. of the Disordered Region.
corresponding to a fully mixed region as seen by the Al concentration. This suggests that annealing for longer than 2 hours increases the deep level emissions, due to progressively greater Ga and As outdiffusion, with a small peak being observed from the unmixed MQW. For times shorter than 2 hours it is suggested that, while implantation damage is being annealed out thereby reducing the deep levels, the emission from the mixed region is increasing due to extended intermixing.

5.5.5 Improved Encapsulation

As stated previously when the Si$_3$N$_4$ cap was used, the predominant deep level emission observed occurred at 894nm and was attributed to a Si-Ga$_x$ complex, with the other deep level at 998nm being tentatively attributed to an arsenic vacancy complex. As the purpose of characterising the mixing process was to ultimately use it to form the side walls of a buried stripe waveguide, these levels could possibly result in the attenuation via absorption of the propagating mode in the side walls of the waveguides. Work was therefore undertaken to find an improved capping procedure where Ga and As outdiffusion were reduced. This work included the investigation of the Si$_3$N$_4$ + AlN dual cap, and the improvement of the existing Si$_3$N$_4$ encapsulant. It has been shown that the inclusion of oxygen in a Si$_3$N$_4$ layer enhances gallium outdiffusion\cite{3,8} which would result in a greater concentration of Ga vacancies and consequently Si-Ga$_x$ complexes in the sample, with the effect of increasing the deep level emission. Work was therefore undertaken to reduce this oxygen content and improve the stochiometry of the Si$_3$N$_4$. 

100
For comparison, samples containing a Si dose of $1 \times 10^{14}$ ions cm$^2$ were annealed at 750°C for times between 1 hour and 5 hours using either the improved Si$_3$N$_4$ cap or the Si$_3$N$_4$ + AlN dual cap. The results for both caps, showed a significant reduction in the deep level emission at 894nm, to a level comparable to that of the As related transition at 998nm, with the improved Si$_3$N$_4$ cap giving an average deep level over band-edge ratio of 1.34:1 compared to 2.2:1 for the dual cap. This indicated that Ga outdiffusion had been reduced. Typical spectra for the improved Si$_3$N$_4$ and dual encapsulated samples are shown in Figure 5.7 and Figure 5.8 respectively. The improved Si$_3$N$_4$ encapsulant was therefore used in all subsequent work. It was seen that a fully mixed region was obtained after a 3 hour anneal at 750°C for both caps and not 2 hours as had been observed previously. This suggested that the reduction in vacancies by reduced outdiffusion, decreased the rate of intermixing. Furthermore, the emission from the unmixed MQW was seen to increase. This could have been due to the decrease in deep level emission and/or a narrower mixed region. Plots of band-edge emission wavelength with anneal time, for the Si$_3$N$_4$ and dual cap, are shown in Figure 5.9 and Figure 5.10 respectively. It was noted that the emission wavelengths remained a constant for increased annealing after 3 hours, again indicating the presence of a diffusion inhibiting layer. However, no significant variation was observed in the integrated intensity ratio with increasing anneal time for either cap. These results suggest that the decrease in deep level emission was followed by a similar reduction in the depth of intermixing, which would be consistent with the reduction in Ga vacancies.
TEMPERATURE 80K
LASER 100mW at 514.5nm
SLITS 1000μm

Figure 5.7 - P.L. Spectrum of a Sample Containing 1x10¹⁶ Si cm⁻² and Annealed for 2hrs at 750°C Using the Improved Si₃N₄ Cap.
TEMPERATURE 80K
LASER 100mW at 514.5nm
SLITS 1000μm

Figure 5.8: P.L. Spectrum of a Sample Containing $1 \times 10^{15}$ Si cm$^{-2}$
and Annealed for 2hrs at 750°C Using a Si$_3$N$_4$ and AlN Dual Cap.
Figure 5.9: Band-Edge Emission Wavelength Shift with Increasing Anneal Time for Samples Containing $1 \times 10^{15}$ Si cm$^{-2}$, and Annealed using the Improved Si$_3$N$_4$ Encapsulant, with the Corresponding % Al conc. of the Disordered Region.
Figure 5.10 - Band-Edge Emission Wavelength Shift with Increasing Anneal Time for Samples Containing $1 \times 10^{15}$ Si cm$^{-2}$, and Annealed using the Dual Encapsulant, With the Corresponding % Al conc. of the Disordered Region.
5.5.6 Dose Effects with Improved Encapsulation

After achieving an improvement in the deep level emissions by using the improved Si₃N₄ cap and comparing the degree of intermixing using samples containing 1x10¹⁵ Si cm⁻², silicon implantation to doses of 3x10¹⁵ ions cm⁻² and 7x10¹⁵ ions cm⁻² was performed. The samples were capped with the improved Si₃N₄ and annealed at 750°C for times between 1 hour and 5 hours. In each case the composition of the intermixed material remained a constant for a given dose with increased anneal time. However the degree of intermixing was reduced for increasing dose, as shown in Figure 5.11. PL analysis showed a layer of average Al concentration of 10.7% and 8.86% for the 3x10¹⁵ Si cm⁻² and 7x10¹⁵ Si cm⁻² dose samples respectively. This suggested that the size of the diffusion inhibiting layer observed previously, increased with dose and restricted the movement of the Si and hence suppressed intermixing. This suggestion was consistent with the damage profiles for increasing implantation dose modelled using SUSPRE in Chapter 3. The intensity of the deep level emissions for these samples was observed to increase, and a typical spectra for a sample containing 7x10¹⁵ Si cm⁻² is shown in Figure 5.12, further suggesting a dose dependence on the deep level emission intensity.

5.5.7 Furnace and RTA Effects

In an attempt to further reduce the deep level emission by breaking up the Ga and As vacancy complexes, an RTA step was included in the processing. A sample containing a dose of 1x10¹⁵ Si cm⁻² was annealed at 750°C for 2 hours followed by an RTA anneal at 900°C for 30 secs. Examination of the sample following the RTA
Figure 5.11: Al Composition of Disordered Region
Observed with Increasing Implant Dose,
After a 5 Hour Anneal At 750 °C Using
the Improved Si₃N₄ Encapsulant.
Figure 5.12: P.L. Spectrum of a Sample Containing 7x10^15 Si cm^{-2} and Annealed for 2 hrs at 750°C Using the Improved Si₃N₄ Encapsulant.
revealed a substantial increase in the emissions at 894nm and 998nm. This suggested that instead of breaking the complexes, the rapid anneal had provided further energy for their formation and this line of investigation was discontinued. The spectrum recorded for this sample is shown in Figure 5.13.

5.5.8 Cladding Effects

A sample containing $1 \times 10^{19}$ Si cm$^2$ and previously annealed at 750°C for 2 hours using the improved Si$_3$N$_4$ cap, was etched in a 113gKI :65gI$_2$ :200mH$_2$O solution for 90secs to remove the top cladding layer. Analysis using PL showed no observable decrease in the deep level emission, suggesting that the majority of deep level emission originated in the mixed material, and was not due to donor Ga-vacancies in the top cladding layer.

5.6 Discussion and Conclusions

The initial work involving the characterisation of the as-grown material, showed that there were no significant variations in the MQW structure across the wafer. A change of 17meV in the ec1-hh1 recombination energies between the two wafers was attributed, in the main, to variations in the quality of the well/barrier interfaces consistent with the observed increase in the FWHM energies.

Annealing of the as-grown material, using the maximum temperature and time condition employed in the work performed on the silicon doped samples, showed
TEMPERATURE 80K
LASER 100mW at 514.5nm
SLITS 1000µm

Figure 5.13: P.L. Spectrum of a Sample Containing 1x10^5 Si cm⁻²
and Annealed for 2hrs at 750°C Followed by an RTA at 900°C for 30 Secs Using the Improved Si₃N₄ Encapsulant.
minimal interdiffusion, due to intrinsic thermal dis ordering. This confirmed that all subsequent interdiffusion observed was as a result of the introduction of silicon via implantation.

The results presented for IILD, suggest a complex relationship between the degree of intermixing observed with implantation dose and capping procedure. It was observed that the degree of intermixing increased for silicon doses between $3 \times 10^{13}$ ions cm$^{-2}$ to $1 \times 10^{14}$ ions cm$^{-2}$ with a diffusion inhibiting layer being observed for the highest dose. This layer was believed to occur at the top cladding/MQW interface, in the region of maximum Si concentration where the Ga and As vacancy concentrations will be a maximum. This suggests that at high Si doping levels or damage levels diffusion is restricted. However, where significant gallium outdiffusion occurred, as with the proximity and capless anneals, no inhibiting layer was formed at this dose and Al diffusion into the mixed region occurred. This contradiction cannot be easily explained and the mechanism of Si IILD is the topic of investigation by other researchers.

Improvement of the Si$_3$N$_4$ cap was seen to reduce the deep level emission and extent of the gallium outdiffusion. Consequently the degree of the intermixing suggested that gallium vacancies were required for mixing to occur. Implantation to higher doses of $3 \times 10^{13}$ ions cm$^{-2}$ and $7 \times 10^{13}$ ions cm$^{-2}$ produced less intermixing than that for the $1 \times 10^{13}$ cm$^{-2}$ samples. It was suggested that this was the result of an increase in the width of the inhibiting layer which prevented an increasing proportion of the Si from diffusing, and therefore less interdiffusion.

By removal of the top cladding layer of a processed sample the PL results suggested that the source of the deep level emission was the mixed region and not the top cladding layer when the improved Si$_3$N$_4$ cap was used.
Finally, the suitability of Shipley 1400/37 photoresist as an implantation mask was determined and found to be suitable for the masking of 500Kev silicon, and was therefore used in the later fabrication of buried stripe optical waveguides using IILD.
REFERENCES

5.1 S.M. Sze. 'Physics of Semiconductor Devices' 2nd Ed. 682, Wiley, 1981

5.2 J.I. Pankove. 'Optical Processes in Semiconductors', 124,125,249, Dover, 1975


5.4 J.S. Roberts. SERC Central Facility for III-V Material Growth, University of Sheffield. Private Communication.

5.5 L.I. Schiff. 'Quantum Mechanics' 3rd Ed. 37, McGraw-Hill, 1985

5.6 A.C. Wismayer and B.L. Weiss. Mat. Lett. 6, 284, 1988


5.10 R.M. Gwilliam. Private Communication. SERC Central Facility for Ion Implantation, University of Surrey.


CHAPTER 6

OPTICAL WAVEGUIDE MEASUREMENTS

6.1 Introduction

In this chapter results are presented on the optical waveguiding characteristics of buried stripe optical waveguides, where lateral confinement was achieved using selective area Impurity Induced Layer Disordering (IILD). Silicon implantation was performed on samples masked with a series of 10μm or 8μm wide stripes, to doses ranging from 3x10<sup>14</sup> ions cm<sup>-2</sup> to 3x10<sup>15</sup> ions cm<sup>-2</sup>, which were subsequently annealed at 750°C using a Si<sub>3</sub>N<sub>4</sub> encapsulant. The doses of 3x10<sup>13</sup> ions cm<sup>-2</sup> and 7x10<sup>15</sup> ions cm<sup>-2</sup>, previously investigated using PL, were not used here due to the limited intermixing observed, as reported in Chapter 5. In parallel with the PL investigation, the properties of the Si<sub>3</sub>N<sub>4</sub> cap were improved during the course of this work and its effects on waveguide characteristics were determined. Loss measurements were performed using end-fire coupling at a propagation wavelength of 1.15μm, which were corrected for Fresnel reflection for insertion loss measurements and for modal mismatch, as detailed in Chapter 4 for the calculation of propagation loss. Later, to investigate the dependence of waveguide loss on the wavelength used, measurements
were undertaken at 1.3μm and 1.54μm in addition to 1.15μm. This work involved the characterisation of the as-grown material, by planar waveguide loss measurements, and the characterisation of ridge waveguides fabricated using chemical wet etching.

6.2 Material and Waveguide Assessment

6.2.1 MQW Planar Waveguides

Two wafers, CPM 383 and CPM 436, were used during the course of this work. To ensure that there were no significant variations in the optical characteristics at 1.15μm, the wavelength at which the majority of this work was performed, planar waveguides were cleaved from both wafers and the end facets examined for quality prior to undertaking insertion loss measurements. Figure 6.1 and Figure 6.2 show the variation in insertion loss for the sequentially cleaved planar samples from wafers CPM 436 and CPM 383 respectively, where a linear regression model has been used to give the best straight line fit to the data points. The loss measurements were corrected for Fresnel reflection and, from the gradients of the lines, propagation losses of 40.8dB/cm and 44.4dB/cm were calculated for wafers CPM 436 and CPM 383 respectively. Furthermore, by extrapolating the lines, both samples showed an interface loss, due to the modal properties of the laser beam and waveguide, of approximately 3.8dB. These results suggested that no significant difference existed in the waveguiding properties of the two wafers at a propagation wavelength of 1.15μm.
Figure 6.1: Variation in Insertion Loss for a Sequentially Cleaved Planar Waveguide from Wafer CPM 436.
Figure 6.2: Variation in Insertion Loss for a Sequentially Cleaved Planar Waveguide from Wafer CPM 383.
6.2.2 MOW Ridge Waveguides

Wet etched stripe waveguides were fabricated to provide a further method of characterisation of the optical waveguiding properties of the as-grown material and to determine the crystal orientation of the two wafers, for reasons to be discussed below. In these waveguides the propagating modes would be confined laterally, unlike the planar samples, by the presence of the large refractive index change at the semiconductor/air interface. However, the presence of the side walls is known to introduce scattering effects which depend on their surface roughness. Consequently two etchants were used which had previously been seen to provide smooth etched surfaces as discussed in Chapter 3. The two etchants used were $3\text{H}_2\text{SO}_4 : 1\text{H}_2\text{O}_2 : 1\text{H}_2\text{O}$ (etchant A) and $1(1\text{N}-\text{NaOH}) : 1\text{H}_2\text{O}_2 : 10\text{H}_2\text{O}$ (etchant B), the latter etchant revealing two distinct sets of crystal planes depending on whether the etched stripe lay parallel to the $<110>$ or $<\overline{1}10>$ crystal axis, also discussed in Chapter 3. This characteristic was used to determine the wafer orientation and to ensure that all waveguides fabricated using wet chemical etching were parallel to the $<110>$ axis. Consequently, the waveguides fabricated parallel to the same axis, using the same etchant, would have the same cross-sectional shape, thereby reducing variations in the interface loss and enabling a more direct comparison of insertion losses to be made. However, it must be noted that whilst the waveguides may have the same cross-sectional profile, this would not eliminate interface loss differences between the waveguides, especially since the wet etched waveguides fabricated were all multi-mode. Therefore, to reduce the effects of variations in the interface loss to a minimum, the insertion loss for each waveguide was measured three times and an average of all the measurements was taken, with the standard deviation shown as the error.
Two samples were cleaved from each wafer and 10μm wide ridge waveguides were fabricated by etching a sample from each wafer in one of the two etchants. Insertion loss measurements were taken at 1.15μm, and corrected for Fresnel reflection. The results for the two sets of guides are shown in Tables 6.1 and Table 6.2.

<table>
<thead>
<tr>
<th>Etchant</th>
<th>Insertion Loss (dB)</th>
<th>Sample Length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Etchant A</td>
<td>3.3±0.7</td>
<td>1.3</td>
</tr>
<tr>
<td>Etchant B</td>
<td>3.8±1.0</td>
<td>2.2</td>
</tr>
</tbody>
</table>

Table 6.1. Insertion Loss Measurements of 10μm Wet Etched Ridge Waveguides, Fabricated from Wafer CPM 383 using 3H₂SO₄ : 1H₂O₂ : 1H₂O (etchant A) and 1(1N-NaOH) : 1H₂O₂ : 10H₂O (etchant B).
Table 6.2. Insertion Loss Measurements of 10μm Wet Etched Ridge Waveguides, Fabricated from Wafer CPM 436 using $3\text{H}_2\text{SO}_4 : 1\text{H}_2\text{O}_2 : 1\text{H}_2\text{O}$ (etchant A) and $1(1\text{N-NaOH}) : 1\text{H}_2\text{O}_2 : 10\text{H}_2\text{O}$ (etchant B).

<table>
<thead>
<tr>
<th>Etchant</th>
<th>Insertion Loss (dB)</th>
<th>Sample Length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Etchant A</td>
<td>4.1±0.4</td>
<td>2.8</td>
</tr>
<tr>
<td>Etchant B</td>
<td>3.9±0.7</td>
<td>2.8</td>
</tr>
</tbody>
</table>

For the two samples fabricated using etchant B, the measurements suggest that within the limits of error shown, for waveguides of similar length, insertion losses were approximately equal. Furthermore, the results for the two samples taken from wafer CPM 436 suggest that there is little difference in the insertion loss for waveguides fabricated using the two etchants. The difference in the measured insertion loss for the samples fabricated using etchant A could be largely accounted for by the large variation in sample lengths. As previously stated, all the guides fabricated were multi-mode and consequently loss measurements using the Thermal Fabry-Perot resonant cavity technique and $1/e$ modal profiles were not attempted. Whilst these
results are not definitive, they do corroborate the previous results of the planar waveguides, in that no significant differences exist between the waveguiding characteristics of the two wafers at a propagating wavelength of 1.15μm.

6.3 MQW Stripe Waveguides by IILD

6.3.1 Effects of Si⁺ Dose and Si₃N₄ Encapsulant

From equation 2.13, discussed in Chapter 2, lateral confinement was expected to be achieved for 10μm wide waveguides at a propagating wavelength of 1.15μm, where the side walls consisted of a fully mixed MQW. Consequently, the initial work was based on 10μm stripe waveguides. All propagation losses in the work presented here were determined by calculating the modal mismatch, unless stated otherwise. The values reported are the average for all the waveguides on each sample. Where the waveguide supported more than one mode, the results refer to the fundamental mode.

A series of 10μm wide stripe waveguides were fabricated by Si⁺ implantation to a dose of 1x10¹⁵ ions cm⁻² and were annealed at 750°C in a flow furnace for times ranging from 1 hour to 4 hours, using the original Si₃N₄ cap. The variation of propagation loss and 1/e width of the propagating mode with increasing anneal time, are shown in Figure 6.3 and Figure 6.4 respectively. It can be seen from Figure 6.3 that the losses obtained for the TM polarisation were consistently higher than those for the TE polarisation, with a minimum TE mode propagation loss of 35.8dBcm⁻¹ being obtained for the sample annealed for 2 hours.
Figure 6.3 - Variation in Propagation Loss with Anneal Time for 10μm Wide Waveguides Fabricated by 1x10^{15} Si cm² Implantation and Annealed Using the Original Si₃N₄ Encapsulant.
Figure 6.4: Variation in $\frac{1}{6}$ Mode Widths with Anneal Time for 10\(\mu\)m Wide Waveguides Fabricated by \(1\times10^{15}\) Si cm\(^2\) Implantation and Annealed Using the Original Si\(_3\)N\(_4\) Encapsulant.
Typical lateral and depth TE output mode profiles for these waveguides are shown in Figure 6.5 and Figure 6.6 respectively. These figures suggest the symmetrical nature of these waveguides and were typical of the waveguides fabricated using IILD in this work.

In an attempt to quantify the contribution to the loss of the propagating mode, due to absorption in the side walls, planar loss measurements were undertaken through the mixed region. The losses obtained, calculated using a 3.8dB interface loss previously determined for planar waveguides in Section 6.2.1, were in excess of 155dB/cm for all anneal times. Consequently, accurate measurements were not possible.

Figure 6.4 shows that the 1/e propagated fundamental mode widths were seen to decrease for increased anneal times. These results suggested the degree of lateral confinement increased with anneal time. Mode profile measurements showed that the waveguides only supported one TM mode for all anneal times, whilst for the TE modes, one mode was found for the annealing time of 1 hour and two modes for times greater than 1 hour. It was further noted that the mode widths for the TM polarisation were consistently larger than those for TE. This trend supported the model of MacBean et al.[5,6] where it was proposed that the refractive index of an MQW structure was dependent upon the polarisation of the propagating mode, with the refractive indices for the TE polarisation always being greater than that for the TM polarisation.

The minimum in the TE mode propagation loss, obtained for the sample annealed for 2 hours, corresponded to an insertion loss of 7.3dB for a sample length of 1.3mm. As stated earlier, these waveguides were observed to support a second mode for the TE polarisation, However, because this second mode was weak and difficult to excite,
Figure 6.5: Typical Lateral Profile of a Propagating Mode for a 10µm Wide waveguide, Fabricated by IILD.
Figure 6.6: Typical Depth Profile of a Propagating Mode for a 10μm waveguide Fabricated Using II LD.
loss measurements were undertaken for this sample using the Thermal Fabry-Perot resonant cavity technique and an average loss of 36.4dBcm\(^{-1}\) with a minimum of 33dBcm\(^{-1}\) were recorded. It was seen that this value compared closely to that determined by calculating the modal mismatch as recorded above. This result served to verify the validity of the modal mismatch calculation. In both cases the modal mismatch was found to be 2.6dB. As this value was of the same order as those obtained for the planar waveguides, and the waveguides fabricated using IILD also had large aspect ratios, it was suggested that the major component of the mismatch was due to the difference in the 1/e depth of the propagating mode, and the 1/e width of the input beam. This assumption appeared to be valid for the case here. When the modal mismatch was calculated for the other waveguides in this set of samples, it was found that, whilst the output 1/e mode width varied but still remained large in comparison to the 1/e input beam width, the mismatch calculated did not vary significantly from the 2.6dB already obtained, with a maximum variation of ±0.5dB calculated.

To confirm the minimum loss seen for a two hour anneal, a second set of waveguides were fabricated from wafer using the same process, with the exception that 8μm wide stripes were used to ensure the waveguide supported a single lateral mode. This sample gave increased propagation losses of 47.0dBcm\(^{-1}\) and 48.9dBcm\(^{-1}\) for the TE and TM polarisations respectively, and now the waveguides only supported a single mode. As observed previously, the TM propagation losses and 1/e mode widths for this sample, were consistently greater than those for the TE case. Furthermore, as expected, it was observed that the overall mode widths were smaller than for the 10μm wide waveguides, with values of 4.7μm and 6.4μm recorded for TE and TM modes respectively. Although an increase in loss was observed for this sample, it was
still smaller than that observed for the 10μm waveguides annealed for 1 hour and 3 hours, suggesting that a minimum in attenuation was possibly achieved after annealing for 2 hours. The increased loss observed was possibly due to an increased percentage of the propagated mode travelling in the mixed region and/or increased scattering at the side walls. These possibilities are discussed in more detail at the end of this chapter.

At this juncture, work was undertaken to improve the Si₃N₄ encapsulant and all subsequent annealing was performed using this improved encapsulant.

A comparison of the effects of the improved Si₃N₄ encapsulant was performed by fabricating 8μm wide stripe waveguides from wafer, to ensure single mode operation, using the same processing conditions as used previously. Silicon implantation was undertaken to a dose of 1×10¹⁵ cm⁻² and the samples subsequently annealed at 750°C for times ranging between 2 hours and 5 hours. The variation of propagation loss with annealing time for these samples is shown in Figure 6.7. It was seen that the losses were lower than those obtained with the original cap, with minimum TE and TM propagation losses of 21.9dBcm⁻¹ and 25.6dBcm⁻¹ respectively, obtained for the sample annealed for 5 hours. However, the trend varies significantly from that previously observed with the lowest losses now being obtained for the samples annealed for the longest time. These results suggested that either the depth to which mixing occurs increases with annealing time, thereby increasing the degree of lateral confinement, or that the optical quality of the mixed region improves with extended thermal processing. Planar loss measurements through the mixed region were seen to decrease for increasing anneal times as shown in Figure 6.8, this indicated that the attenuation of the propagating mode due to absorption in the side walls of the
Figure 6.7: Variation in Propagation Loss with Anneal Time for 8μm Wide Waveguides Fabricated by 1x10^15 Si cm² Implantation and Annealed Using the Improved Si₃N₄ Encapsulant.
Figure 6.8 - Variation in Propagation Loss Through the Disordered MQW Material with Anneal Time, for Samples Containing $1 \times 10^{15}$ Si cm$^{-2}$ and Annealed Using the Improved Si$_3$N$_4$ Encapsulant.
waveguide decreases with longer annealing times, suggesting that absorption in the side walls of the waveguide was a significant source of propagation loss. Mode width measurements showed no significant variation of the 1/e mode widths with increasing annealing time, and average 1/e mode widths of 7.5\(\mu\)m and 8.2\(\mu\)m were measured for the TE and TM polarisations respectively.

To investigate the waveguiding characteristics of buried waveguides fabricated using lower implantation doses, the width of the waveguide was increased to 10\(\mu\)m because of the expected reduction in the extent of intermixing, and consequently, lateral confinement. A series of 10\(\mu\)m wide stripe waveguides were fabricated by silicon implantation to a dose of \(3 \times 10^{14}\) ions cm\(^{-2}\) and were annealed for times ranging from 1 hour to 5 hours. Optical waveguide measurements revealed that planar guiding was still evident for the samples annealed from 1 hour to 4 hours whilst lateral confinement was only observed for the sample annealed for 5 hours. For this sample, TE and TM propagation losses of 40.0dBcm\(^{-1}\) and 41.3dBcm\(^{-1}\) were recorded respectively. Measurement of the fundamental 1/e mode widths for this sample produced average values of 5.3\(\mu\)m and 5.8\(\mu\)m for the TE and TM polarisations respectively. It was also observed that the waveguides supported two modes for the TE polarisation and only one for the TM polarisation.

By contrast, to investigate the waveguiding properties of buried waveguides fabricated using higher implantation doses, 8\(\mu\)m waveguides were fabricated by silicon implantation to a dose of \(3 \times 10^{15}\) ions cm\(^{-2}\) and were annealed at 750°C for times ranging between 2 hours and 5 hours. These single mode waveguides showed no change in the propagation losses with increased anneal time, with average propagation losses of 31.2dBcm\(^{-1}\) and 39.0dBcm\(^{-1}\) being determined for the TE and
TM polarisations respectively. Measurement of the 1/e mode profile dimensions also showed no significant variation with increased annealing time and produced average values of 7.6µm and 7.9µm for TE and TM polarisations respectively. As with the stripe waveguide measurements, no variation was observed for planar loss measurements through the mixed region with the average values for the TE and TM mode losses being 69.7dBcm⁻¹ and 66.7dBcm⁻¹ respectively.

6.3.2 Wavelength Effects

Due to the effects of lateral scattering during implantation and the lateral diffusion of the implanted species during annealing, the interface between the MQW guiding layer and the disordered region may result in significant side wall scattering loss. It is known that at longer propagating wavelengths the effect of scattering is reduced. Consequently, an investigation was undertaken to determine the effect of the wavelength of propagation on the optical characteristics of the stripe waveguides fabricated using IILD. For this purpose two semiconductor laser sources were used, with emission wavelengths of 1.3µm and 1.54µm. As stated in Chapter 4, the output beams from the semiconductor lasers were via a non-polarisation maintaining fibre, therefore, the losses presented here are not for a specific polarisation.

The initial characterisation was performed on the wet etched ridge waveguides fabricated from the two as-received wafers using the 3H₂SO₄ :1H₂O₂ :1H₂O etchant. This was undertaken to reveal variations in the optical waveguiding properties of the as-grown material at these longer wavelengths. The insertion losses at the
propagation wavelengths of 1.3μm and 1.54μm were compared to those previously obtained for 1.15μm and are shown in Table 6.3 and Table 6.4 for wafer CPM 383 and CPM 436 respectively.

<table>
<thead>
<tr>
<th>Wafer</th>
<th>CPM 383</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wavelength (μm)</td>
<td>Insertion Loss (dB)</td>
</tr>
<tr>
<td>1.15</td>
<td>3.2±0.7</td>
</tr>
<tr>
<td>1.3</td>
<td>5.7±0.5</td>
</tr>
<tr>
<td>1.54</td>
<td>10.4±1.0</td>
</tr>
</tbody>
</table>

Table 6.3. Insertion Loss Measurements of 10μm Wet Etched Ridge Waveguides, Fabricated from Wafer CPM 383 using 3H₂SO₄ :1H₂O₂ :1H₂O at the Propagation Wavelengths 1.15μm, 1.3μm and 1.54μm for a 1.3mm Long Sample.
Table 6.4. Insertion Loss Measurements of 10μm Wet Etched Ridge Waveguides, Fabricated from Wafer CPM 436 using $3H_2SO_4 : 1H_2O : 1H_2O$ at the Propagation Wavelengths 1.15μm, 1.3μm and 1.54μm for a 2.8mm Long Sample.

<table>
<thead>
<tr>
<th>Wafer</th>
<th>CPM 436</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wavelength (μm)</td>
<td>Insertion Loss (dB)</td>
</tr>
<tr>
<td>1.15</td>
<td>4.1±0.4</td>
</tr>
<tr>
<td>1.3</td>
<td>3.6±0.5</td>
</tr>
<tr>
<td>1.54</td>
<td>5.4±0.5</td>
</tr>
</tbody>
</table>

Inspection of Table 6.3 showed that a significant increase in the insertion losses occurred with increasing wavelength, for the waveguides fabricated from wafer CPM 383, with an increase of 7.2dB measured between the insertion losses at 1.15μm and 1.54μm. In contrast, the insertion losses for the waveguides fabricated from wafer CPM 436 showed only a small increase over the wavelength range, rising by 1.3dB at 1.54μm. As previously shown in Chapter 4, a 1/e input beam widths of 1.3μm, 1.4μm and 1.7μm were obtained for the 1.15μm, 1.3μm and 1.54μm
wavelengths respectively. This could account for the increase in losses seen at 1.54\(\mu\)m in the waveguides from wafer CPM 436 but not for the increases seen in the waveguides from wafer CPM 383.

To determine whether a similar effect occurred for the buried waveguides fabricated using IILD from wafer CPM 383, propagation loss measurements were undertaken. This also allowed for variation in the modal mismatch, due to the 1/e input beam widths changing between wavelengths, to be eliminated. The sample chosen consisted of a series of 8\(\mu\)m wide waveguides fabricated by silicon implantation to a dose of 1\(\times\)10\(^{12}\) ions cm\(^{-2}\) and annealed for 2 hours using the original Si\(_3\)N\(_4\) encapsulant. This sample had previously been used to confirm the minimum attenuation for these processing conditions. It was observed that only a single mode was supported at all wavelengths and the propagation losses recorded for this sample are shown in Table 6.5.

From the results in Table 6.5 it was seen that the increase in waveguide loss, observed for the wet etched samples, was also evident in the IILD delineated waveguides. This trend for the waveguides fabricated from wafer CPM 383 suggested that any variations which may have been observed due to the nature of stripe waveguides fabricated using IILD i.e. scattering effects, would not be seen. Furthermore, this would invalidate the comparison of samples fabricated from the two wafers at the propagation wavelengths of 1.3\(\mu\)m and 1.54\(\mu\)m. Consequently this line of investigation was terminated.
Table 6.5. Propagation Loss Measurements for 8μm Buried Stripe Waveguides, Fabricated from Wafer CPM 383 by Silicon Implantation to a Dose of 1x10^13 ions cm^-2 and Annealed for 2 hours using the Original Si₃N₄ Encapsulant, at the Propagation Wavelengths 1.15μm, 1.3μm and 1.54μm for a 1.8mm Long Sample.

<table>
<thead>
<tr>
<th>Wavelength (μm)</th>
<th>Loss (dBcm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.15</td>
<td>48±2</td>
</tr>
<tr>
<td>1.3</td>
<td>53±3</td>
</tr>
<tr>
<td>1.54</td>
<td>97±7</td>
</tr>
</tbody>
</table>

6.3.3 Encapsulation Effects

All waveguides fabricated using IILD were annealed with a Si₃N₄ encapsulant on the sample surface to minimise the effects of gallium and arsenic outdiffusion.\cite{65} Because this encapsulant was not removed prior to waveguide characterisation, work
was undertaken to ensure the presence of the Si₃N₄ did not affect the propagation characteristics of the MQW waveguides. A sample was cleaved from an as-grown wafer and planar insertion loss measurements were performed at the propagation wavelength of 1.15μm. Losses of 12dB for a sample length of 2mm were measured for both TE and TM polarisations. This sample was then capped with approximately 1000Å Si₃N₄, cleaved and remeasured. Insertion losses of 9.0dB and 9.2dB were recorded for the TE and TM polarisations respectively, for a sample length of 1.2mm. These results were seen to correspond closely to those obtained during the planar loss measurements obtained for the wafer characterisation, which are shown in Figure 6.1 and Figure 6.2. This suggests that the presence of the Si₃N₄ encapsulant had no significant effect on the waveguiding properties of the MQW structure.

6.4 Discussion and Conclusion

The initial characterisation of planar and wet etched ridge waveguides showed that there was no significant difference in the optical waveguiding properties of the two as-grown MQW wafers, at a propagation wavelength of 1.15μm. By sequentially cleaving the planar waveguides, propagation losses of 40.8dB/cm and 44.4dB/cm were recorded for wafers CPM 436 and CPM 383 respectively, and a value of 3.8dB was calculated for the loss due to the interface loss for both wafers. The deviation of the data points in these graphs from the straight line may be due to damage on the cleaved facets which was not seen using the optical microscope. The modal mismatch was found to be closer to 2.6dB for the disorder delineated stripe waveguides, and was found to not vary significantly for the waveguides fabricated using the IILD processing reported here. This value was calculated from the insertion and
propagation losses measured for buried stripe waveguides, fabricated by 1x10^{15} Si cm² implantation and subsequent annealing for 2 hours. The propagation losses for these waveguides were determined using the Thermal Fabry-Perot resonant cavity technique and by calculation of the modal mismatch between the input beam and propagating mode. The two sets of data were seen to be in close agreement, which served to validate the modal mismatch calculation which was used for all subsequent propagation loss calculations. Because the interface loss for the planar and the IILD waveguides were of the same order of magnitude, and both waveguide cross-sections had a large aspect ratio, it was believed that the major component of the mismatch was due to the difference in 1/e depth of the input beam and the depth of the propagating mode. By assuming a mismatch loss of 3dB for the wet etched ridge waveguides, which lies between the values found for the planar waveguides and the single mode IILD waveguides, average propagation losses of 2.95dBcm⁻¹ and 3.5dBcm⁻¹ were obtained for samples etched in the 3H₂SO₄ :1H₂O₂ :1H₂O (etchant A) and 1(1N-NaOH) :1H₂O₂ :10H₂O (etchant B) respectively. Losses of 2.15dBcm⁻¹ have previously been observed in p-type samples with free carrier concentrations equal to that of the as-grown MQW structure of 1x10^{16} cm³, suggesting that residual p-type doping of the as-grown MQW accounted for the majority of the loss recorded. The remaining 0.8dBcm⁻¹ and 1.35dBcm⁻¹ may be attributed to scattering due to roughness of the side walls. It was seen that the difference in losses between waveguides were within the limits of experimental error, further suggesting that no significant difference existed in the optical waveguiding properties of the two wafers at 1.15μm. TM propagation losses and 1/e fundamental mode profiles for the waveguides fabricated using IILD were seen to be consistently larger than those for the TE polarisation. This indicated that the principle loss mechanisms were either attenuation
of the propagating mode in the laterally confining mixed region and/or scattering from the side walls. It was seen that a minimum TE propagation loss of 35.8dBcm⁻¹ was achieved for the 10μm wide waveguides fabricated by 1x10¹⁵ Si cm⁻² implantation and annealing for 2 hours. Reducing the guide width to 8μm and using the fabrication process as described previously, using the original Si₃N₄ encapsulant, resulted in a decrease of the fundamental mode width as expected, whilst increasing the TE propagation loss to 47dBcm⁻¹. Modelling of the 8μm and 10μm waveguides using the TE refractive indices previously calculated from the model proposed by Ohke et al. revealed that 15.37% and 23.14% of the power in the TE propagating mode travelled in the mixed MQW material, for the 10μm and 8μm wide stripe waveguides respectively. A planar loss measurement through the mixed MQW material of the sample annealed for 2 hours gave a loss of approximately 180dBcm⁻¹. This planar loss, with the amount of power calculated to be propagating in the side walls for 10μm and 8μm wide waveguides, provided an estimation of the maximum attenuation that could be attributed to the absorption in the mixed region. The values obtained were 28dBcm⁻¹ and 42dBcm⁻¹ for the 10μm and 8μm wide waveguides respectively. These are approximately equal to the propagation losses recorded. This tentatively suggests that in these waveguides a significant loss mechanism was due to the absorption of the propagating mode in the mixed MQW material.

The 8μm wide waveguides fabricated using the same dose but annealed using the improved Si₃N₄ encapsulant, showed reducing propagation losses with increasing anneal times, with a minimum TE propagation loss of 21.9dBcm⁻¹ being obtained for the sample annealed for 5 hours. The propagation losses of the stripe waveguides followed the same trend as those for the mixed region, where a minimum loss of 54dBcm⁻¹ was obtained again for the sample annealed for 5 hours. This provided
further evidence that a significant contribution to the waveguide propagation loss was due to the absorption of the propagating mode in the mixed MQW material which formed the side walls of the waveguide. Using the same modelling procedure as before, it was seen that for the sample annealed for 5 hours, a maximum of 12.5dBcm⁻¹ of the 21.9dBcm⁻¹ TE propagation loss could be accounted for by absorption in the side walls, with the remaining 9.4dBcm⁻¹ possibly being due to free carrier absorption in the unmixed MQW material and scattering due to side wall roughness. The difference between the waveguide propagation loss and the estimated losses in the mixed MQW material were largest for the shortest anneal time of 1 hour, with a value of 25.8dBcm⁻¹ calculated, which fell to a minimum for the sample annealed for 5 hours. This difference may also be attributed to losses due to side wall scattering, as the smoothness of the interface would be expected to improve with thermal processing. The fact that no trend was observed for the 1/e mode widths with increased annealing time suggests that the mixed MQW material did not extend to the same depth as that achieved with the original Si₃N₄ encapsulant. This would be consistent with reduced gallium and arsenic outdiffusion from the sample surfaces provided by the improved encapsulant.⁶⁹

The waveguiding characteristics of the samples containing a silicon dose of 3x10⁵ ions cm⁻² showed that the extent of mixing was only sufficient to provide lateral confinement after annealing for 5 hours. This suggests that the rate of intermixing was reduced at this dose, requiring extended anneal times to provide the minimum change in refractive indices to produce lateral confinement in the waveguides. The reduced propagation losses observed may be the result of a greater proportion of the
power in the propagating mode travelling in the unmixed MQW, due to the limited depth of the mixed region, or reduced attenuation in a mixed region fabricated by lower dose implantation.

Propagation loss and 1/e mode width measurements of the waveguides fabricated using a silicon dose of $3 \times 10^{13}$ ions cm$^{-2}$ showed no variation with increasing anneal time. This suggests that a region of reduced refractive index was produced, providing lateral confinement, but that it did not change with increased annealing times.

Measurements undertaken at the longer propagation wavelengths of 1.3$\mu$m and 1.54$\mu$m revealed an increasing propagation loss with increasing wavelength for samples fabricated from CPM 383. As this variation was likely to mask any variations seen with wavelength due to the nature of waveguides fabricated using IILD, these experiments were not pursued. A possible explanation for this effect is the existence of deep level absorption centres in the CPM 383 MQW sample which would cause increased absorption at longer wavelengths, although this was not confirmed.

In Chapter 2 the free carrier effects due to the electrical activation of the implanted Si were calculated. It was seen that a free carrier concentration of $4.5 \times 10^{17}$ cm$^{-3}$ would result in a propagation loss of 6.88 dB cm$^{-1}$ at the propagating wavelength of 1.15$\mu$m.

Calculating the contribution of the free carrier absorption in the side walls of the 10$\mu$m and 8$\mu$m wide waveguides, using the percentage powers of the propagating mode travelling in the side walls, gives propagation losses of 1.1 dB cm$^{-1}$ and 1.59 dB cm$^{-1}$ respectively. Therefore, it can be seen that the contribution of the free carrier absorption to the propagation losses measured through the disordered MQW
material was small. Propagation loss measurements through the disordered MQW material at the longer wavelengths of 1.3\(\mu\)m and 1.54\(\mu\)m were not undertaken due to the increased waveguide propagation losses measured at these wavelengths.

Also in Chapter 2, refractive index steps of 0.015\% and -0.044\% were calculated between the unmixed MQW and disordered MQW material for the TE and TM polarisations respectively, at the propagating wavelength of 1.15\(\mu\)m. Furthermore, the contribution of the free carriers in the disordered MQW material were also calculated and these were seen to result in an index change of 0.03\% at the propagating wavelength of 1.15\(\mu\)m. Using the refractive indices calculated for TE polarisation and inserting into equation 2.13 it can be calculated that 10\(\mu\)m and 8\(\mu\)m wide waveguides support 2 modes and 1 mode respectively. These calculations are consistent with results presented in Section 6.3.1. The negative refractive index difference for the TM polarisation suggested that no lateral confinement would be observed for this polarisation even when the free carrier contribution is considered and the values inserted into equation 2.13. It was calculated that the free carrier concentration required to support a single TM mode would have resulted in the 10\(\mu\)m wide waveguides for the TE polarisation supporting 3 modes. This result is discussed further in Chapter 8. As the 1.3\(\mu\)m and 1.54\(\mu\)m laser outputs were unpolarised the calculation for the refractive index differences were not undertaken.

In summary, a comparison of the results presented in this chapter suggest that the propagation losses of the waveguides fabricated were dependent upon the silicon implantation dose, anneal time and the encapsulant employed. It was seen that lateral confinement improved with anneal time for the samples containing a dose of 1\(\times\)10\(^{14}\) Si cm\(^{-2}\) and annealed using the original Si\(_3\)N\(_4\) encapsulant. However, lower loss
waveguiding could be achieved using the same dose but annealed using the improved encapsulant. Because no variation was observed in the 1/e mode widths for waveguides annealed using this encapsulant, it was suggested that the depth to which the disordering occurred was reduced, thereby limiting the degree of lateral confinement. A correlation was observed between the losses in the mixed regions and the stripe waveguide loss. This suggested that a significant contribution to the propagation loss was due to the absorption of the propagating mode in the side walls of the waveguides. Reducing this loss, using the improved encapsulant, reduced the calculated absorption in the side walls to a point where it was comparable to the expected loss due to side wall scattering.

In conclusion, results have been presented for buried stripe optical waveguides fabricated using a range of silicon implantation doses and annealed for various times using two encapsulants. The waveguide results have shown strong dependence on the processing conditions and these have been investigated.
REFERENCES


CHAPTER 7
SUMMARY AND DISCUSSION

7.1 Introduction

A summary of the work performed in this study is presented in this chapter. This summary includes a discussion of the results and the relationship between the photoluminescence measurements for silicon implantation impurity induced disordering, and the characteristics of the optical waveguides fabricated using this technique. Samples processed for PL analysis and buried stripe optical waveguide fabrication used two types of Si₃N₄ encapsulant, and the correlation between the effect of the encapsulant on the degree of intermixing and deep level emission, is discussed in relation to the waveguide attenuation results.

7.2 Summary and Discussion

The initial PL and optical waveguide measurements were performed on samples cleaved from each wafer for the characterisation of the as-grown material. By undertaking these measurements, it was possible to determine whether any significant
variation existed in the MQW structure either across an individual wafer, or between different wafers. It was seen from the PL measurements that a variation of ±2meV existed in the ec1-hh1 transition for samples cleaved from across the same wafer, and a variation of 17meV was recorded between different wafers. Modelling of the 17meV shift suggested that a 60Å difference in the well widths existed between the two wafers. This was in contrast to information suggesting a maximum variation of ±4%. However, it was noted that non-abrupt well/barrier interfaces in the MQW structure would also result in a similar energy shift with an increase in the FWHM energy, and it was suggested that the variation in the MQW structures between wafers was closer to the ±4% value. Impurity induced disordering of both wafer structures were observed to produce an AlGaAs layer with an average Al concentration of 14.4%, suggesting that the figure of ±4% was closer to the actual variation in well width. In parallel to the PL analysis, planar and wet etched ridge optical waveguides from each wafer were measured at a propagating wavelength of 1.15μm. Propagation losses of 40.8dBcm⁻¹ and 44.4dBcm⁻¹ were calculated by measurement of the insertion losses for the sequentially cleaved planar waveguides. From these results and the insertion losses for the wet etched waveguides, it was observed that no significant variation existed in the attenuation of the waveguides at a propagating wavelength of 1.15μm.

Work was then undertaken to ensure that for the anneal temperature and times used to disorder the samples containing Si, that disordering was due solely to the implantation of Si. This was performed by annealing samples cleaved from the as-grown wafers at 750°C for 5 hours. A shift in the ec1-hh1 transition of ±2.4meV was observed for the
$Si_3N_4$ encapsulated samples, less than 2% of the expected energy shift for the fully disordered MQW structure. Consequently, the interdiffusion observed after the implantation of Si was attributed to Si implantation IILD.

As reported elsewhere, impurity induced layer disordering is known to be sensitive to the type of encapsulant used during the annealing of the samples, and three encapsulation techniques were investigated to determine the most suitable for the work undertaken. Samples containing a Si dose of $1\times10^{15}$ cm$^{-2}$ were annealed using either a proximity cap, no cap or a $Si_3N_4$ encapsulant. It was found that for the proximity cap and capless samples the deep level emission, as discussed below, was more intense than the emission from the mixed region following a 750°C anneal for 3 hours and 4 hours respectively. Consequently, because the $Si_3N_4$ encapsulant resulted in less deep level emission it was selected for the initial Si IILD study. As the work progressed it was suggested that these deep levels were a major source of attenuation of the optical waveguides fabricated by Si IILD and an improved encapsulant was sought. Work was undertaken to reduce the oxygen content of the CVD $Si_3N_4$ and this was compared to a dual encapsulant of AlN and $Si_3N_4$. From this investigation the improved $Si_3N_4$ encapsulant was used for all subsequent work.

Finally, work was undertaken which suggested that the Shipley 1400/37 photoresist was suitable as an implantation mask for the selective area implantation and disordering used in the fabrication of buried stripe optical waveguides.

The PL results revealed that for MQW IILD, produced by silicon implantation and subsequent annealing, a complex relationship existed between the silicon dose, the associated implantation damage and the type of encapsulant used during annealing. The intermixing process was accompanied by two deep level emissions at 894nm and
998nm. The former was previously attributed to a Si-Ga complex and the latter was tentatively attributed to a Si-As complex due to the significant reduction in the intensity of this emission when a Si$_3$N$_4$ encapsulant was used, which is known to restrict As outdiffusion.

It has been shown that stripe optical waveguides can be fabricated using impurity induced disordering, where silicon is introduced into the MQW using ion implantation. The 10µm waveguides, initially fabricated by Si implantation to a dose of 1x10$^{15}$ cm$^{-2}$ and annealed at 750°C for 2 hours using the original Si$_3$N$_4$ encapsulant, showed minimum TE and TM propagation losses of 35.8dBcm$^{-1}$ and 36.4dBcm$^{-1}$ respectively. This corresponded to the minimum in the deep level/band edge integrated intensity ratio and the point at which a fully mixed layer was achieved, as previously observed using PL. This correlation suggested that the deep levels may act as a source of attenuation of the propagating mode in the side walls of the waveguide.

To provide an indication of the attenuation of the propagating mode in the mixed region, loss measurements were performed for planar propagation in the mixed region but it was seen that the losses were all greater than approximately 155dBcm$^{-1}$ and accurate measurements could not be attained. The optical measurements also revealed that, at a propagating wavelength of 1.15µm the waveguides were single mode for the TM polarisation but, for samples annealed for 2 hours and longer, a weak second mode was supported for the TE polarisation. The variation in 1/e mode width profiles for the propagated modes at 1.15µm with increased annealing were seen to decrease from 8.1µm to 5.9µm for the TE polarisation, and 8.8µm to 6.4µm for the TM polarisation. This correlated with an observed decrease in the emission from the unmixed MQW in the PL results, suggesting that, with increased annealing, the depth to which intermixing occurred increased and consequently, the extent of
lateral confinement in the waveguides, where after a 2 hour anneal, two lateral modes were supported. It was also noted that the 1/e mode widths for the TM polarisation were consistently larger than those for TE. This trend supported the model of Ohke et al.,\textsuperscript{74} where it was proposed that the refractive index of a MQW was dependent upon the polarisation of the propagated light, with the refractive indices for the TE polarisation always being greater than that for TM. In relation to the work presented here it would mean a smaller refractive index difference between the MQW and the mixed region for the TM polarisation which would result in reduced confinement in comparison to the TE polarisation, and consequently an increase in 1/e mode width dimension, which was observed here. This would also suggest that a larger percentage power of the mode would propagate in the side walls, and if, as suspected, the attenuation due to deep levels in the mixed side walls was a major component of the insertion loss, an increase in loss would be expected to be seen for the TM polarisation. This situation was also observed from the propagation loss measurements.

A second set of 8\textmu m wide waveguides were fabricated using the process conditions above which had provided the lowest loss waveguides. These single mode waveguides were observed to have higher propagation losses of 47dBcm\textsuperscript{-1} and 48.9dBcm\textsuperscript{-1}, and reduced 1/e mode widths of 4.7\textmu m and 6.4\textmu m for the TE and TM polarisations respectively than those reported above. This increase in the propagation loss was consistent with the increase in the percentage power of the propagated mode travelling in the side walls, due to the reduction in the waveguide width.

It was observed from the PL analysis that silicon implantation to a dose of 3x10\textsuperscript{14} ions cm\textsuperscript{-2} and subsequent annealing may cause the indiffusion of aluminium
from the top cladding layer into the mixed region. This would result in a mixed region where the Al concentration was greater than the average for the MQW, and consequently have a reduced refractive index resulting in increased lateral confinement. However, it was also observed from the PL spectra that the emission from the unmixed MQW had increased for this reduced Si dose, and it was suspected that the depth to which the mixing occurred was less than for the samples containing $1 \times 10^{15}$ Si cm$^{-2}$, thereby providing a thinner confining layer with a larger reduction of refractive index. However, another possible explanation for this increased emission was the observed reduction in the deep level emission with decreasing implantation dose, which may result in less absorption in the disordered region and sampling to a greater depth. The deep level/band edge ratio was seen to decrease with increased annealing time though again not to the levels attained for the samples containing $1 \times 10^{15}$ Si cm$^{-2}$ samples. The 10μm waveguides fabricated by $3 \times 10^{14}$Si cm$^{-2}$ implantation showed lateral confinement only after annealing for 5 hours with propagation losses of 40.0dBcm$^{-1}$ and 41.3dBcm$^{-1}$ and 1/e mode widths of 5.3μm and 5.75μm measured for the TE and TM polarisations respectively. This result suggests that the depth to which mixing occurs was reduced for this lower dose, and that while a region of lower refractive index existed in the side walls, cut off was only attained after increased anneal times. The waveguide insertion loss was smaller than the best achieved for the $1 \times 10^{15}$ cm$^{-2}$ samples and suggests that due to a reduced depth of the mixed region less of the mode propagates through the side walls, which are known to be lossy and consequently the attenuation due to the deep levels in the mixed region was reduced.
It is known that the presence of oxygen in a Si$_3$N$_4$ encapsulant promotes the outdiffusion of Ga,\(^{7,8}\) and in an attempt to reduce the associated deep level emissions which are thought to be due to a Si-Ga complex,\(^{7,8}\) and therefore the waveguide losses, work was performed to reduce the oxygen content in the growth chamber. A comparison was undertaken of the effect on the deep levels between this improved Si$_3$N$_4$ encapsulant and a dual encapsulant of AlN + Si$_3$N$_4$. Samples containing a Si dose of 1x10\(^{15}\) ions cm\(^{-2}\) were annealed at 750°C for between 1 hour and 5 hours and deep level/band edge integrated intensity ratios of 1.34:1 and 2.2:1 were recorded for the improved Si$_3$N$_4$ and dual encapsulants respectively suggesting decreased emission from the deep levels for the new encapsulants. It was also observed for the samples annealed using the improved encapsulant that no significant change in the deep level/band edge integrated intensity ratio occurred for anneal times up to 5 hours. Furthermore, annealing for 3 hours was required to obtain a fully mixed region and an increased emission from the unmixed MQW was observed in comparison to the samples annealed using the original Si$_3$N$_4$ encapsulant. The reduction in the deep level/band edge integrated intensity ratio indicated that reduced Ga outdiffusion had been achieved, and the increase in the emission from the unmixed MQW indicated that either the depth of sampling was greater, due to reduced absorption in the mixed region, and/or the depth to which intermixing occurred was reduced. Because of the reduced deep level/band edge ratio it was suspected therefore, that waveguides fabricated using the improved encapsulant would have lower propagation losses.

To determine the effects of the improved encapsulant on waveguide losses, some 8µm wide waveguides were fabricated using Si implantation to a dose of 1x10\(^{15}\) ions cm\(^{-2}\), and waveguide measurements undertaken at the propagation wavelength of 1.15µm. The propagation losses of these waveguides were seen to reduce with increased
anneal times, and minimum TE and TM propagation losses of 21.9dBcm⁻¹ and 25.6dBcm⁻¹ respectively were recorded for the sample annealed for 5 hours. These losses were lower than those obtained previously using the original encapsulant and, because of the reduced stripe width, all guides were single mode for both polarisations. The losses through the mixed region were also observed to decrease with increased anneal times and were smaller than those recorded for samples annealed using the original Si₃N₄ encapsulant with a minimum of 56dBcm⁻¹ being measured. These results further suggest that absorption in the side walls of the waveguide due to presence of deep levels is a major source of attenuation in the waveguides fabricated using Si-III LD. It was observed for these waveguides that no significant variation in the 1/e mode widths occurred with anneal time, with average mode widths of 7.5μm and 8.2μm being measured for TE and TM polarisations respectively. This result appears to be consistent with the observation from the PL measurements that no variation occurred in the deep level/band edge integrated intensity ratio with increased annealing, suggesting that the depth to which intermixing occurred remained relatively constant for anneal times up to 5 hours.

Photoluminescence analysis of samples implanted to a dose of 3x10¹⁵ Si cm⁻² showed that the degree of intermixing was reduced for this increased dose, with a mixed region of average Al concentration of 10.7% being observed for all anneal times. An increase in the deep level/band edge integrated intensity ratio was also observed, as would be expected with increased Si and lattice vacancies for increased ion dose. Waveguides fabricated with this ion dose were single mode and showed no significant change in the propagation losses with increased anneal time, though the overall waveguide losses were seen to increase, with average TE and TM propagation losses of 31.2dBcm⁻¹ and 39.0dBcm⁻¹ and 1/e mode widths of 7.6μm and 7.9μm being
measured. These results indicate that the degree of lateral confinement was reduced due to the limited intermixing, with increased absorption occurring in the mixed region through which an average loss of 68.2 dB/cm was measured, and which also did not vary significantly with anneal time.

Although PL measurements were undertaken for samples containing Si doses ranging from $3 \times 10^{13}$ ions cm$^{-2}$ to $7 \times 10^{15}$ ions cm$^{-2}$, waveguides were not fabricated using the lowest and highest implantation doses because of the limited intermixing observed. It was suggested from the results that increasing the implantation dose from $3 \times 10^{13}$ ions cm$^{-2}$ to $1 \times 10^{16}$ ions cm$^{-2}$ resulted in extended intermixing with a diffusion inhibiting layer being formed between the top cladding layer and the MQW for the sample containing $1 \times 10^{15}$ ions cm$^{-2}$. These results were consistent with those published previously.$^{1,4}$ From the trend of the deep level/band edge integrated intensity ratios obtained for the samples containing $1 \times 10^{15}$ Si cm$^{-2}$ and annealed using the original Si$_3$N$_4$ encapsulant, it was suggested that the initial decrease in the deep level/band edge integrated intensity ratio was due to the annealing out of the implantation damage, which would reduce the deep level emission, whilst the emission from the mixed region would increase as the intermixing progressed through the MQW. However, with increasing anneal times deep level emission may increase due to the increased Ga outdiffusion and the increased probability of Si-Ga complexes forming. It was observed using the improved Si$_3$N$_4$ encapsulant that the time required to form a fully mixed region increased, and that the deep level/band edge integrated intensity ratio remained relatively constant for anneal times up to 5 hours. This suggested that the improved encapsulant reduced Ga outdiffusion thereby reducing the rate of intermixing and limiting the formation of Si-Ga complexes. By increasing the implantation dose to $3 \times 10^{15}$ Si cm$^{-2}$ and $7 \times 10^{15}$ Si cm$^{-2}$
the average Al concentration of the disordered MQW was reduced, and it was suggested that this was due to an increase in width of the diffusion inhibiting layer, which removed an increasing proportion of the Si thereby restricting the Si diffusion and consequently the disordering process.

Modelling of the power propagated in the side walls for the TE polarisation was performed using the model of Skinner et al.\(^1\)\(^\text{2}\)\(^\text{3}\)\(^\text{4}\) and it was found that 15.37% and 23.14% of the power of the modes were propagated in the side walls of the 10µm and 8µm wide waveguides respectively. By measurement of the losses through the mixed region and correlating these with the percentage power propagated in the side walls, it was seen that the losses due to the side walls were close to the propagation losses calculated. However, this calculation was used only as an indication and suggested that a major source of loss in the waveguides was due to absorption in the mixed region. Using the percentage power calculations above with the free carrier losses in the disordered region calculated in Chapter 2, estimated free carrier losses 1.0dBcm\(^{-1}\) and 1.59dBcm\(^{-1}\) would be expected for the 10µm and 8µm wide waveguides respectively. It is suspected therefore that the free carrier losses resulting from propagation in the side walls were small compared to the overall propagation losses measured.

Using the normalised film thickness equation as defined in Chapter 2 a refractive index difference of up to 0.014% between the mixed region and unmixed MQW would have to exist for lateral confinement of a single mode, at a propagating wavelength of 1.15µm, in a 10µm wide waveguide. Furthermore, this index change would have to increase up to 0.0225% for a single lateral mode to be supported in an 8µm wide waveguide. Using the models proposed by Ohke et al.\(^7\)\(^8\)\(^9\)\(^10\) it was calculated
that a refractive index change of 0.015% would exist between the waveguide and disordered layer for the TE polarisation and -0.044% for the TM polarisation. Therefore, it can be seen that this index change for the TE polarisation would support a weak second mode in a 10μm waveguide, as observed, and no lateral confinement would be expected for the TM polarisation. However, from the experimental results lateral confinement for TM modes was observed in both 10μm and 8μm wide waveguides. This suggests that confinement may have been due to free carrier effects alone however, this would have required a free carrier concentration of 8.8x10^17 cm^-3 which would have resulted in the 10μm wide waveguides supporting 3 lateral modes for the TE polarisation, which was not observed during the characterisation of the waveguides. Consequently, the model proposed by Ohke et al[19] was found to be consistent with the experimental data for the TE polarisation and inconsistent with the experimental data for the TM polarisation.

Waveguide measurements undertaken at the propagating wavelengths of 1.3μm and 1.54μm, using the samples containing 1x10^18 Si cm^-3, were initially expected to produce a decrease in the propagation losses due to reduction of scattering losses from the side walls,[18] and due to the smaller deep level emissions seen at these wavelengths. However, the increased attenuation with wavelength, observed in the as-grown material, was seen to be dominant and no decrease in propagation losses with wavelength was observed.

From the summary and discussion above it was seen that the photoluminescence results for Si implantation IILD and optical waveguides fabricated using this technique were consistent with each other, and it has been suggested that absorption due to deep levels in the side walls of the waveguides is a major source of attenuation.
REFERENCES


7.8 R.G. Hunsperger. 'Integrated Optics: Theory and Technology', 2nd Ed. 71, Springer-Verlag, 1985

CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS
FOR FUTURE WORK

8.1 Introduction

This chapter presents a conclusion of the work undertaken in this thesis. Ideas that have been prompted during the work which would be suitable for further investigation are discussed in Section 8.3.

8.2 Conclusions

The purpose of the work was to investigate silicon implantation impurity induced disordering and its use in the fabrication buried stripe optical waveguides. The results obtained can be divided into two categories. Firstly, those from samples annealed using the Si$_3$N$_4$ encapsulant and secondly, those obtained from samples using the improved Si$_2$N$_4$ encapsulant. Photoluminescence measurements of the samples annealed using the original Si$_3$N$_4$ suggested that the depth to which intermixing occurred increased for increasing implantation dose in the range from $3 \times 10^{19}$ Si cm$^{-2}$ to
$1 \times 10^{13}$ Si cm$^{-2}$, and that for the samples containing $1 \times 10^{13}$ Si cm$^{-2}$ a diffusion inhibiting layer was formed at the interface of the MQW region and the top cladding layer. These observations were found to be consistent with results published by other researchers working in this field.\textsuperscript{[8,18]} It was further suggested, for these samples, that the deep level emissions which accompanied the disordering process also increased with increasing implantation dose, which was consistent with the source of these two emissions being a Si-Ga$_x$ complex and an arsenic related transition as proposed in Chapter 5.

The buried stripe optical waveguides fabricated by implantation to a dose of $1 \times 10^{15}$ Si cm$^{-2}$ and annealed using the original Si$_3$N$_4$ encapsulant showed a minimum TE propagation loss of 35.8 dB cm$^{-1}$ for the sample annealed for 2 hours. This corresponded to the minimum observed in the deep level/band edge integrated intensity ratio which was used to provide an indication of the suitability of the IILD process conditions for the fabrication of waveguides. This suggested that the deep levels were a possible significant source of attenuation of the propagated mode due to absorption in the side walls of the waveguide. By measuring the propagation loss through the mixed region and calculating the percentage power of the TE propagating mode travelling in the side walls, using the model of Skinner et al.,\textsuperscript{[8,3]} it was observed that the majority of the waveguide propagation loss could be attributed to absorption in the side walls of the waveguide.

The $1/e$ fundamental mode width measurements for these waveguides showed two distinct trends. Firstly, it was observed that the mode widths decreased with increased anneal times and it was suggested that this was due to the increased depth of intermixing and consequently an increase in the lateral confinement. This suggestion
was corroborated by the observation that the waveguides annealed for 2 hours and longer supported two modes for the TE polarisation whereas the waveguides annealed for 1 hour only supported a single mode for the same polarisation. Secondly, it was observed that the 1/e fundamental mode widths for the TM polarisation were consistently greater than for the TE polarisation. Furthermore, the waveguides fabricated supported only the fundamental mode for the TM polarisation also suggesting that the lateral confinement for a TM propagating mode was smaller than that for the TE polarisation. This trend was consistent with the model proposed by Ohke et al. where the refractive index of an MQW structure is sensitive to the polarisation of the propagating mode, with \( n_{TE} \) always being greater than \( n_{TM} \) which suggests that the refractive index difference between the guiding MQW and the mixed region was greater for the TE polarisation. Although this model gave values for the TE refractive index of the MQW which, when used to determine the index change between the MQW and the disordered region, were consistent with the number of propagating modes observed, it indicated that no lateral confinement would occur for the TM propagating modes. In an attempt to account for the TM lateral confinement solely by free carrier effects, it was seen that the free carrier concentration required would have resulted in three modes being supported for the TE polarisation. Consequently, the MQW refractive index for a TM propagating mode could not be determined.

As a result of the correlation between the minimum in the propagation loss and the minimum in the deep level/band edge integrated intensity ratio work was undertaken to reduce these deep level emissions in the mixed material. The dominant deep level emission in the disordered MQW had been previously attributed to the Si-Ga \(_x\) complex. Also it had been previously reported that Ga outdiffusion through a Si\(_2\)N\(_4\)
encapsulant was suspected when oxygen was incorporated in the encapsulant during growth, which would result in a higher Ga-vacancy concentration and potentially increase the number of Si-Ga$_x$ complexes and the deep level emission. Therefore work was undertaken to reduce the oxygen content of the Si$_x$N$_y$ encapsulant. It was found that the deep level/band edge integrated intensity ratio was significantly reduced for samples implanted to $1 \times 10^{15}$ Si cm$^{-2}$ and annealed using the improved encapsulant. Furthermore, annealing for an increased time was now required to produce a fully mixed region. As the disordering process has been reported to be dependent on the group III vacancy concentration, these results suggested that the gallium vacancy concentration had been reduced. The samples implanted to doses $3 \times 10^{15}$ Si cm$^{-2}$ and $7 \times 10^{15}$ Si cm$^{-2}$ and annealed using the improved encapsulant showed an increase in the deep level emissions consistent with an increase in the silicon and gallium vacancy concentrations which would result from higher dose implantation. Furthermore, it was observed that the intermixing was inhibited with increasing implantation dose. It was also suggested from these results that the width of the diffusion inhibiting layer increased with increased implantation damage. The buried stripe optical waveguides fabricated by implantation to a dose of $1 \times 10^{15}$ Si cm$^{-2}$ and annealed using the improved Si$_x$N$_y$ encapsulant showed a decrease in the propagation loss with increased anneal times and a minimum TE propagation loss of $21.9 \text{dBcm}^{-1}$ for the sample annealed for 5 hours. Propagation loss measurements through the mixed material were observed to follow the same trend, with a minimum also being obtained for the sample annealed for 5 hours. No significant variation was observed in the 1/e fundamental mode widths with increased anneal times and it was suggested that this was due to a reduction in the depth of intermixing, which was consistent with the suspected reduction in the
gallium-vacancy concentration. However, as with all the waveguides fabricated by Si+ IILD, it was observed that the 1/e fundamental mode widths were greater for the TM than the TE polarisations. Using the model of Skinner et al.\textsuperscript{[8,31]} it was shown that a maximum loss of 12.5 dB cm\textsuperscript{-1} could be attributed to absorption in the side walls and this suggested that the remaining loss was predominantly due to side wall roughness, as the free carrier absorption in the guiding MQW had previously been calculated to be approximately 1 dB cm\textsuperscript{-1}.

The waveguides fabricated by implantation to a dose of 3 \times 10^{15} \text{ Si cm}^{-2} were observed to have an increased average TE propagation loss of 31.2 dB cm\textsuperscript{-1} which was observed not to vary significantly with increased anneal times. This was consistent with an increase in the propagation losses recorded through the mixed region, and the increase observed in the deep level/band edge integrated intensity ratio. The 1/e fundamental mode widths for these waveguides were also observed not to vary with increased anneal time and this was consistent with the suspected reduced intermixing observed using PL.

Free carrier effects resulting from the electrical activation of the implanted Si on the refractive index change between the guiding and disordered MQW layers were calculated. It was found that the refractive index change contribution due to the free carriers was consistent with the number of modes supported for the IILD delineated waveguides.

In concluding, work has been undertaken in the characterisation of Si+ IILD, and it has resulted in the first published results of the fabrication of buried stripe optical waveguides using this technique. It was seen that the PL data for the disordered samples correlated closely with optical waveguide propagation characteristics, and
confirms that PL is a useful technique in assessing the suitability of process conditions for the fabrication of waveguides using Si implantation IILD. It has been suggested from the results that the deep levels which accompany the disordering process contribute significantly to the propagation loss by absorption of the propagating mode in the side walls of the waveguides.

8.3 Recommendations for Future Work

During the course of this work several areas for further investigation became apparent which would provide a natural progression of the work undertaken, and assist in the characterisation of waveguides fabricated using Si implantation IILD. Firstly, due to the lack of information in the literature, it was necessary to estimate the percentage of the implanted Si dose that became electrically active and contributed to the free carrier concentration. As the free carriers contributed to both the absorption and lateral confinement in the waveguide, it is suggested that Hall measurements be undertaken to determine both the carrier profiles and mobilities. This would allow a more accurate assessment of the free carrier effects in the waveguides. Secondly, because PL measurements do not provide an absolute measurement of the depth to which intermixing occurs it is suggested that Transmission Electron Microscopy (TEM) and/or Secondary Ion Mass Spectrometry (SIMS) measurements are undertaken. These measurements would provide an accurate method for determining the depth of the confining layer and permit a more accurate model of the propagating mode in the waveguide to be developed.
It is suggested that future work includes an investigation of the effects of varying the sample temperature during Si implantation, as it has been reported that enhanced disordering occurs when the sample temperature is either raised or lowered during Si implantation. This suggests the possibility of achieving the same depth of intermixing for a room temperature implant with a lower dose and potentially a reduced deep level emission.

In conclusion there is an abundance of work still to be completed before the fabrication of buried stripe optical waveguides using IID can become an established technology, although, with the recent increased interest in this new field, the potential of this technology is likely to be realised in the future.
REFERENCES


