Implant Isolation of InP-based materials

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To my family and my precious friends without whom life will be very boring..
Summary

There has been great interest in using ion implantation for III-V semiconductor device isolation as an alternative to mesa isolation technique. This is attributed to several advantages that implant isolation has over mesa isolation. Mesa isolation exhibits problems such as over/under etching, repeatability issue of etching depth and non-planarity of the surface of the semiconductor. However implant isolation is advantageous as the surface planarity is maintained and in general, less intrusion under the mask edges is observed.

This thesis presents a study on the isolation of both n and p-type InP and InGaAs layers and n-type InGaAsP layers by ion implantation. Several different ion species such as protons, helium, nitrogen and iron were used to isolate these materials. The n and p-type layers were grown by Solid Source Molecular Beam Epitaxy. Conductive n-type InP layers were also formed using multiple energy silicon implantation to create a uniform dopant distribution throughout the n-type region. The effects of ion mass, implantation temperature, damage accumulation, initial carrier concentration of the conductive layer and post-implant annealing temperature were investigated in detail through electrical and structural characterisation. The major part of the work was to develop recipes for the isolation of the individual InP, InGaAs and InGaAsP layers. The effects of implantation temperature and dose were also examined thoroughly. A parallel resistor model was also created to confirm the reliability of the measurements.

Keywords: Ion implantation, InP, InGaAs, InGaAsP, Electrical isolation, Carrier removal, Elevated temperature implants, Hall measurements, Secondary ion mass spectroscopy, Rutherford backscattering spectrometry, Semiconductor
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List of Publications


# Table of Contents

Summary iii  
Acknowledgments iv  
List of Publications vi  
Table of Contents viii  
List of Figures x  
List of Tables xviii  
Glossary of Terms xx  

## 1. Introduction 1  
1.1 Background ................................................................. 1  
1.2 Objectives ................................................................. 2  
1.3 Major contributions of the thesis ................................. 2  
1.4 Structure of the thesis .................................................. 3

## 2. Overview of ion implantation 5  
2.1 Theory of ion implantation ............................................ 5  
2.1.1 Ion stopping and range .......................................... 7  
2.1.2 Channelling ......................................................... 10  
2.2 Radiation damage ...................................................... 12  
2.3 Simulation software .................................................... 13

## 3. Review of implant isolation 15  
3.1 Proton and deuterium implant isolation ......................... 17  
3.2 Helium implant isolation ............................................. 20  
3.3 Boron implant isolation ............................................... 22  
3.4 Oxygen implant isolation ........................................... 23  
3.5 Nitrogen implant isolation .......................................... 25  
3.6 Implant isolation using transition metals ..................... 26  
3.7 Implant isolation using other ions ............................... 28  
3.8 Summary of implant isolation ....................................... 28

## 4. Experimental procedures 32  
4.1 Process steps of implant isolation in InP, InGaAs, InGaAsP .... 32  
4.2 Basic principles of ion implantation ............................ 34  
4.3 Plasma enhanced chemical vapour deposition ................ 36  
4.4 Rapid thermal annealing ............................................ 37  
4.5 Photolithography process ......................................... 39  
4.6 Hall effect measurement ........................................... 42  
4.6.1 The Hall effect .................................................. 42  
4.6.2 Resistivity measurement with temperature ............... 46
4.6.3 Sources of errors related to the measurement of Hall effect and sheet resistivity ............................................... 47
4.7 Secondary ion mass spectroscopy technique ............................................. 48
4.8 Rutherford backscattering spectroscopy technique .......................... 51

5. Implant isolation using protons and helium ............................................... 52
5.1 Implant isolation of n-type InP and InGaAs using hydrogen ions .......................................................... 52
5.1.1 Dose dependence at RT and 200°C .................................................. 52
5.1.2 Effect of post-implant annealing temperature ..................................... 56
5.2 Implant isolation of n-type InP and InGaAs using helium ions .......................................................... 59
5.2.1 Effect of substrate and post-implant annealing temperature ............... 60
5.2.2 Effect of damage accumulation ......................................................... 66
5.2.3 Effect of dose on implant isolation of n-type InGaAs using helium ions .......................................................... 68
5.3 Helium implantation into semi-insulating InP at RT and 200°C .......................................................... 70
5.3.1 Effect of dose ................................................................................ 71
5.3.2 The parallel resistor model ................................................................. 75
5.3.3 Effect of post-implant annealing temperature ..................................... 80
5.4 Summary .................................................................................................... 82

6. Implant isolation using iron and nitrogen ............................................... 85
6.1 Implant isolation of n-type InP and InGaAs using iron ions .................. 85
6.1.1 Effect of substrate and post-implant annealing temperature ..................... 86
6.1.2 Effect of dose at different substrate temperatures .......................... 104
6.2 Implant isolation of p-type InP and InGaAs using iron ions .................. 113
6.2.1 Effect of substrate and post-implant annealing temperature ..................... 114
6.2.2 Effect of dose on implant isolation of p-type InGaAs ......................... 118
6.3 Implant isolation of n-type InGaAsP using nitrogen ions ....................... 119
6.3.1 Effect of dose at 77K and RT .............................................................. 121
6.3.2 Reliability of the sheet resistivity measurements .................................. 122
6.4 Implant isolation of n-type InP using nitrogen ions .................................. 125
6.5 Summary .................................................................................................... 131

7. Comparative discussions of implant isolation using protons, helium, iron and nitrogen ............................................... 133
7.1 General summary of the results ............................................................... 133
7.2 Influence of implantation parameters ..................................................... 138
7.3 Isolation matrix ...................................................................................... 142

8. Conclusion and further work ................................................................. 148
8.1 Conclusion ............................................................................................. 148
8.2 Suggestions for further work .................................................................. 150

References ................................................................................................................. 152
<table>
<thead>
<tr>
<th>Figures</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figure 2.1</td>
<td>Effects of ion on targets; (a) doping/isolation, (b) damage creation, (c) sputtering and (d) mixing [120].</td>
<td>6</td>
</tr>
<tr>
<td>Figure 2.2</td>
<td>Difference between a low and high energy range.</td>
<td>8</td>
</tr>
<tr>
<td>Figure 2.3</td>
<td>Gaussian distribution of doping distribution versus range.</td>
<td>9</td>
</tr>
<tr>
<td>Figure 2.4</td>
<td>Schematic diagram of possible range profiles for ions implanted into a single crystal [120].</td>
<td>11</td>
</tr>
<tr>
<td>Figure 3.1</td>
<td>Simple model to explain behaviour of Proton Bombardment on n-type and p-type InP.</td>
<td>17</td>
</tr>
<tr>
<td>Figure 4.1</td>
<td>Flowchart of the implant isolation process.</td>
<td>33</td>
</tr>
<tr>
<td>Figure 4.2</td>
<td>Schematic diagram of the 2MV Van de Graaff ion implanter.</td>
<td>34</td>
</tr>
<tr>
<td>Figure 4.3</td>
<td>Schematic diagram of DP80+, rf-powered at 13.56MHz.</td>
<td>37</td>
</tr>
<tr>
<td>Figure 4.4</td>
<td>8 Lamps rapid thermal annealer.</td>
<td>39</td>
</tr>
<tr>
<td>Figure 4.5</td>
<td>Photolithography process flow.</td>
<td>41</td>
</tr>
<tr>
<td>Figure 4.6</td>
<td>Typical configuration for Hall effect measurements.</td>
<td>43</td>
</tr>
<tr>
<td>Figure 4.7</td>
<td>Electrical Contact Configuration for a sample.</td>
<td>45</td>
</tr>
<tr>
<td>Figure 4.8</td>
<td>Liquid Nitrogen Cryostat with lid removed.</td>
<td>47</td>
</tr>
<tr>
<td>Figure 4.9</td>
<td>Schematic diagram of CAMECA IMS 6f.</td>
<td>50</td>
</tr>
<tr>
<td>Figure 5.1</td>
<td>Damage distribution resulting from 250keV proton implants into InP and InGaAs as determined by TRIM.</td>
<td>51</td>
</tr>
<tr>
<td>Figure 5.2</td>
<td>Effect of dose on sheet resistance for 250keV proton irradiation in n-type InP layers at RT and 200°C.</td>
<td>54</td>
</tr>
<tr>
<td>Figure 5.3</td>
<td>Evolution of sheet carrier concentration and sheet resistance as a function of dose for 250keV proton irradiation in n-type InGaAs layers at RT and 200°C.</td>
<td>56</td>
</tr>
<tr>
<td>Figure 5.4</td>
<td>Evolution of sheet resistance as a function of dose for 250keV proton irradiation in n-type InP layers at RT and 200°C and annealed at 100°C, 200°C and 350°C.</td>
<td>57</td>
</tr>
<tr>
<td>Figure 5.5</td>
<td>Evolution of sheet resistance as a function of dose for 250keV proton irradiation in n-type InGaAs layers at RT and 200°C and annealed at 100°C, 200°C and 350°C.</td>
<td></td>
</tr>
<tr>
<td>Figure 5.6</td>
<td>The relative position of the atomic distributions of the n-type dopant, the helium implant and the damage resulting from helium ions, as determined by TRIM.</td>
<td></td>
</tr>
<tr>
<td>Figure 5.7</td>
<td>Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in a nitrogen atmosphere for 600keV helium implanted n-type InP layers irradiated with 2x10^{14} cm^{-2} at RT, 100°C and 200°C.</td>
<td></td>
</tr>
<tr>
<td>Figure 5.8</td>
<td>Evolution of sheet resistance as a function of post-implant annealing temperature for 250keV proton and 600keV helium implanted n-type InP layers irradiated with 5x10^{14} cm^{-2} and 2x10^{14}cm^{-2} respectively at RT.</td>
<td></td>
</tr>
<tr>
<td>Figure 5.9</td>
<td>Variation of sheet carrier concentration and sheet electron mobility with annealing temperature for 600keV helium implantation using a dose of 2x10^{14} cm^{-2} at RT, 100°C and 200°C.</td>
<td></td>
</tr>
<tr>
<td>Figure 5.10</td>
<td>Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in a nitrogen atmosphere for 55keV helium implanted n-type InP layers irradiated with 2x10^{14} cm^{-2} at RT, 100°C and 200°C.</td>
<td></td>
</tr>
<tr>
<td>Figure 5.11</td>
<td>Evolution of sheet resistance with different post-implant annealing temperature for n+ samples implanted with 2x10^{14} cm^{-2} helium ions at 55keV and 600keV at RT.</td>
<td></td>
</tr>
<tr>
<td>Figure 5.12</td>
<td>Damage distribution profile for 55keV and 600keV He(^{+}) implants in InP.</td>
<td></td>
</tr>
<tr>
<td>Figure 5.13</td>
<td>Sheet resistance of n+ InP irradiated with 55keV and 600keV He(^{+}) as a function of ion dose at RT.</td>
<td></td>
</tr>
<tr>
<td>Figure 5.14</td>
<td>Evolution of sheet carrier concentration and sheet electron mobility of n+ InP irradiated with 55keV and 600keV He(^{+}) as a function of ion dose at RT.</td>
<td></td>
</tr>
</tbody>
</table>
Figure 5.15  Evolution of sheet resistance as a function of dose for 600keV helium irradiation in n⁺ InGaAs layers at RT, and 200°C.  

Figure 5.16  Evolution of sheet resistance and sheet carrier concentration as a function of dose for 600keV helium irradiation in semi-insulating InP at RT, and 200°C.  

Figure 5.17  Sheet resistance as a function of the reciprocal temperature for 600keV helium bombardment in SI InP at RT for different doses.  

Figure 5.18  Sheet resistance as a function of the reciprocal temperature for 600keV helium bombardment in SI InP at 200°C for different doses.  

Figure 5.19  Parallel resistors representation of sheet resistance measurements.  

Figure 5.20  Sheet resistance of n⁺ InP and semi-insulating InP irradiated with 55keV and 600keV He⁺ as a function of dose for RT implant.  

Figure 5.21  Evolution of depletion width as a function of dose for n⁺ InP irradiated with 55keV and 600keV He⁺ at RT.  

Figure 5.22  Sheet resistance of n⁺ InGaAs and semi-insulating (SI) InP irradiated with 600keV He⁺ as a function of dose for RT and 200°C implants.  

Figure 5.23  Evolution of depletion width as a function of dose for n⁺ InGaAs irradiated with 600keV He⁺ at RT and 200°C.  

Figure 5.24  Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in a nitrogen atmosphere for 600keV helium implanted in SI InP at a dose of 1x10¹⁴ cm⁻² at RT.  

Figure 5.25  Sheet carrier concentration and sheet mobility as a function of post-implant annealing temperature for 600keV helium implanted in SI InP irradiated with 1x10¹⁴ cm⁻² at RT.  

Figure 6.1  The damage distribution resulting from iron implantation into InP and InGaAs as determined by TRIM.
Figure 6.2  Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in nitrogen atmosphere for 1MeV iron implanted n-type InP layers irradiated with $5 \times 10^{14}$ cm$^{-2}$ at 77K, RT, 100°C and 200°C. 88

Figure 6.3  Variation of sheet carrier concentration with post-implant annealing temperature at 77K, RT, 100°C and 200°C. 89

Figure 6.4  RBS channelling spectra of $n^+$ InP samples implanted with 1MeV Fe$^+$ at 77K, RT, 100°C and 200°C using a fluence of $5 \times 10^{14}$ cm$^{-2}$. The RBS simulated spectrum for a depth of 1μm is also plotted. The data for 77K, RT and 100°C implants are identical to the random spectrum. The virgin spectrum is also plotted. 91

Figure 6.5  RBS channelling spectra of $n^+$ InP samples implanted with 1MeV Fe$^+$ at 200°C using a fluence of $5 \times 10^{14}$ cm$^{-2}$ and annealed at 400°C, 650°C and 800°C for 60s in a N$_2$ ambient. Unimplanted sample (virgin) and random spectra are reported for comparison. 92

Figure 6.6  RBS channelling spectra of $n^+$ InP samples implanted with 1MeV Fe$^+$ at RT using a fluence of $5 \times 10^{14}$ cm$^{-2}$ and annealed at 400°C, 650°C and 800°C for 60s in a N$_2$ ambient. Data for 400°C and 650°C is identical to random spectrum. The virgin spectrum is also plotted for comparison. 92

Figure 6.7  SIMS Fe profiles for InP samples implanted at 77K using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. TRIM Fe profile and SIMS Si profile are also plotted on the same graph. 94

Figure 6.8  SIMS Fe profiles for InP samples implanted at RT using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. SIMS Si profile for 800°C annealed is also plotted. 95

Figure 6.9  SIMS Fe profiles for InP samples implanted at 100°C using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. SIMS Si profile for 800°C annealed is also plotted. 95

Figure 6.10 SIMS Fe profiles for InP samples implanted at 200°C using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. SIMS Si profile for 800°C annealed is also plotted. 96
| Figure 6.11 | Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in nitrogen atmosphere for 1MeV iron implanted n-type InGaAs layers irradiated with $5 \times 10^{14}$ cm$^{-2}$ at 77K, RT, 100°C and 200°C. |
| Figure 6.12 | RBS channelling spectra of Fe$^+$ implanted into n$^+$ InGaAs using a fluence of $5 \times 10^{14}$ cm$^{-2}$ at different implantation temperatures namely, 77K, RT, 100°C and 200°C. Unimplanted sample and random spectra are reported for comparison. The RBS spectra for RT and 77K implants are similar to that of the random spectra. |
| Figure 6.13 | RBS channelling spectra of n$^+$ InGaAs samples implanted with Fe$^+$ at 200°C using a fluence of $5 \times 10^{14}$ cm$^{-2}$ at 1MeV and annealed at 550°C and 800°C for 60s in a N$_2$ ambient. Unimplanted sample and random spectra are reported for comparison. |
| Figure 6.14 | RBS channelling spectra of Fe$^+$ implanted into n$^+$ InGaAs at RT and 200°C after annealing at 800°C. Unimplanted sample and random spectra are reported for comparison. |
| Figure 6.15 | SIMS Fe profiles for InGaAs samples implanted at 77K using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. TRIM Fe profile is plotted on the same graph. |
| Figure 6.16 | SIMS Fe profiles for InGaAs samples implanted at RT using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. TRIM Fe profile is plotted on the same graph. |
| Figure 6.17 | SIMS Fe profiles for InGaAs samples implanted at 200°C using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. TRIM Fe profile is plotted on the same graph. |
| Figure 6.18 | Evolution of sheet resistance as a function of dose for 1MeV iron irradiation in n-type InP layers at 77K, RT, and 200°C. |
| Figure 6.19 | RBS channelling spectra for iron implanted n-type InP layers irradiated with different doses at 1MeV at RT. |
| Figure 6.20 | RBS channelling spectra for iron implanted n-type InP layers irradiated with different doses at 1MeV at 77K. Data for a dose of $5 \times 10^{13}$ cm$^{-2}$ is identical to the random spectrum. |
Figure 6.21  RBS channelling spectra for iron implanted n-type InP layers irradiated with different doses at 1 MeV at 200°C.

Figure 6.22  Fractional damage at the near the surface (channel number 190-219) of InP as a function of dose for 77K, RT and 200°C implants.

Figure 6.23  Evolution of sheet resistance as a function of dose for 1 MeV iron irradiation in n-type InGaAs layers at 77K, RT, and 200°C.

Figure 6.24  RBS channelling spectra of Fe+ implanted into n+ InGaAs at RT for different doses namely, 1x10^{12} cm^{-2}, 1x10^{13} cm^{-2} and 5x10^{13} cm^{-2}. Samples implanted above a dose of 5x10^{13} cm^{-2} have channelling spectra similar to that of the random spectrum. Data for 1x10^{12} cm^{-2} is identical to the virgin spectrum.

Figure 6.25  RBS channelling spectra of Fe+ implanted into n+ InGaAs at 77K for different doses namely, 1x10^{12} cm^{-2}, 1x10^{13} cm^{-2} and 5x10^{13} cm^{-2}. Samples implanted at and above a dose of 5x10^{13} cm^{-2} have channelling spectra similar to that of the random spectrum.

Figure 6.26  RBS channelling spectra of Fe+ implanted into n+ InGaAs at 200°C for different doses namely, 1x10^{14} cm^{-2}, 5x10^{14} cm^{-2} and 1x10^{15} cm^{-2}. Samples implanted at a dose lower than 1x10^{14} cm^{-2} have channelling spectra similar to that of the virgin spectrum.

Figure 6.27  Fractional damage at the near surface region (channel 190-219) versus the implanted iron dose for InGaAs implanted at 77K, RT and 200°C.

Figure 6.28  Evolution of sheet resistance with annealing temperature for iron implanted p-type InP layers irradiated with 5x10^{14} cm^{-2} at 1 MeV, as a function of implant temperature.

Figure 6.29  Evolution of sheet resistance with annealing temperature for iron implanted p-type InGaAs layers irradiated with 5x10^{14} cm^{-2} at 1 MeV, as a function of implant temperature.

Figure 6.30  Evolution of sheet resistance as a function of dose for 1 MeV iron irradiation in p-type InGaAs layers at 77K, RT, and 200°C.

Figure 6.31  The damage distribution resulting from 4 MeV nitrogen implantation into InGaAsP as determined by TRIM.
Figure 6.32  Evolution of sheet resistance as a function of dose for 4MeV nitrogen irradiation in n-type InGaAsP layers at 77K and RT.

Figure 6.33  Evolution of sheet resistance as a function of dose for 4MeV nitrogen irradiation in semi-insulating InP at RT and 200°C.

Figure 6.34  Evolution of sheet resistance as a function of dose for 4MeV nitrogen irradiation in n-type InGaAsP and semi-insulating InP at 77K and RT.

Figure 6.35  Evolution of depletion width as a function of dose for n-type InGaAsP irradiated with 4MeV N⁺ at 77K and RT.

Figure 6.36  Si atomic concentration profile for three different initial carrier concentration as determined by TRIM.

Figure 6.37  Sheet resistance of n-InP irradiated at RT with 4MeV N⁺ as a function of dose for three different initial sheet carrier concentrations.

Figure 6.38  Sheet resistance of n-InP irradiated at 77K with 4MeV N⁺ as a function of dose for three different initial sheet carrier concentrations.

Figure 6.39  Initial sheet carrier concentration of n-InP irradiated at RT with 4MeV N⁺ as a function of threshold dose. The solid line represents the best fit.

Figure 7.1  Comparison of sheet resistance curves for electrical isolation of n-type InP and InGaAs at RT and 200°C using 250keV proton at different doses.

Figure 7.2  Comparison of sheet resistance curves for electrical isolation of n-type InP and InGaAs at RT using 600keV helium at different doses.

Figure 7.3  Comparison of sheet resistance curves for electrical isolation of n-type InP and both n and p-type InGaAs at 77K, RT and 200°C using 1MeV iron at different doses.

Figure 7.4  Comparison of sheet resistance curves for electrical isolation of n-type InP and InGaAsP at 77K and RT using 4MeV nitrogen at different doses.
Figure 7.5  Initial sheet carrier concentration of n-InP irradiated at RT with 250keV H⁺, 600keV He⁺ and 4MeV N⁺ as a function of threshold dose. The solid lines represent the best fit.

Figure 7.6  Initial sheet carrier concentration of n-GaAs irradiated at RT with 250keV H⁺, 600keV He⁺, 1.5MeV B⁺ and 2MeV O⁺ as a function of threshold dose. The dotted lines represent the best fit [114].

Figure 7.7  Initial sheet carrier concentration of n-InGaP irradiated at RT with 270keV He⁺ as a function of threshold dose. The solid lines represent the best fit [118].

Figure 7.8  Initial sheet carrier concentration of p-AlGaAs irradiated at RT with 600keV H⁺ as a function of threshold dose. The solid lines represent the best fit [117].

Figure 7.9  Estimated atomic displacement for n-GaAs of four different initial sheet carrier concentrations irradiated at RT with 250keV H⁺, 600keV He⁺, 1.5MeV B⁺ and 2MeV O⁺ as a function of threshold dose [114].

Figure 7.10 Estimated atomic displacement for n-InP of three different initial sheet carrier concentrations irradiated at RT with 4MeV N⁺ as a function of threshold dose.
# LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Table 3.1</td>
<td>Summary of literature review of implant isolation of n-type InP.</td>
<td>29</td>
</tr>
<tr>
<td>Table 3.2</td>
<td>Summary of literature review of implant isolation of p-type InP.</td>
<td>30</td>
</tr>
<tr>
<td>Table 3.3</td>
<td>Summary of literature review of implant isolation of p-type InGaAs.</td>
<td>30</td>
</tr>
<tr>
<td>Table 3.4</td>
<td>Summary of literature review of implant isolation of n-type InGaAs.</td>
<td>31</td>
</tr>
<tr>
<td>Table 3.5</td>
<td>Summary of literature review of implant isolation of n and p-type InGaAsP.</td>
<td>31</td>
</tr>
<tr>
<td>Table 5.1</td>
<td>Initial sheet resistance, effective mobility and sheet carrier concentration of the InP and InGaAs samples before proton implantation.</td>
<td>55</td>
</tr>
<tr>
<td>Table 5.2</td>
<td>Si⁺ implant conditions to create a uniform doping concentration of thickness ~0.6μm into InP.</td>
<td>59</td>
</tr>
<tr>
<td>Table 6.1</td>
<td>Initial sheet resistance, sheet carrier concentration and sheet mobility of both n-type InP and InGaAs before isolation. The projected range and longitudinal straggling of iron into InP and InGaAs as determined by TRIM are also tabulated.</td>
<td>86</td>
</tr>
<tr>
<td>Table 6.2</td>
<td>Initial sheet resistance, sheet carrier concentration and sheet mobility of both p-type InP and InGaAs before isolation. The projected range and longitudinal straggling of 1MeV iron into InP and InGaAs as determined by TRIM are also tabulated.</td>
<td>114</td>
</tr>
<tr>
<td>Table 6.3</td>
<td>Implant conditions to create three different initial carrier concentrations. The projected range and longitudinal straggling of 4MeV nitrogen into InP as determined by TRIM are also tabulated.</td>
<td>126</td>
</tr>
<tr>
<td>Table 6.4</td>
<td>Electrical characteristics of the three wafers after silicon implantation and annealing.</td>
<td>127</td>
</tr>
</tbody>
</table>
Table 6.5  Threshold dose and maximum sheet resistance of n-InP irradiated at 77K and RT with 4MeV N⁺ for three different initial sheet carrier concentrations.
## Glossary of Terms

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_A$</td>
<td>Activation energy</td>
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Chapter One

1. Introduction

1.1 Background

It was only in the late 1940s that any serious work was done in the field of ion implantation. It all began at Bell Laboratories in USA by two research scientists, R. S. Ohl and W. Shockley. In 1954, W. Shockley [1] patented the use of ion beams for producing the buried base layer in the bipolar transistor. Several years later, Rourke et al [2] published the first report on doping a semiconductor using ion implantation. By 1964, several papers on p-n junction formation by ion implantation were published [3,4,5]. The abrupt nature of the ion implanted junction has been used in the fabrication of avalanche photodetectors [6], impatt diodes [7], and a shallow junction having the approximate characteristics of a Schottky diode [8]. Ion implantation has also been used to dope multilayer heterostructure systems such as InGaAs/GaAs, and AlGaAs/GaAs, without appreciable layer mixing [9,10]. For doping of semiconductors using ion implantation, an annealing process is usually required to remove the damage and to carry dopant atoms to the desire lattice sites where they become electrically activated.

Ion implantation can also be used to create high-resistance regions selectively on a wafer that already contains doped layers. This latter application is commonly called implantation induced isolation or isolation by ion irradiation. Production of semi-insulating regions is of considerable technological importance in Integrated Circuit (IC) technology to provide device isolation. The latter is used to restrict the current flow to the active regions of a device without having cross-talk with other areas on the wafer. In other words, the application of a bias voltage to one device should not induce current in a neighbouring device. Implant isolation is a potential alternative to mesa etching, offering simplicity, precise depth control and compatibility with planar technology [11,12]. Semi-insulation is obtained either through defect-induced compensation or through carrier trapping by deep impurities using ion implantation [13]. Ion implantation of inert species is usually used to produce defects, which induce deep levels in the bandgap. This results in the Fermi level moving towards the centre of the
Implant isolation of InP-based materials

Chapter One: Introduction

gap as a result of compensation. Ions that have been used to produce high-resistance regions by defect generation, include helium, and proton in InP [37, 45]. Damage induced isolation is very much dependent on various parameters such as dose, energy, ion mass and implant temperature.

The dose and energy of the ions used depend on the starting doping concentration and thickness of the layer to be compensated. An annealing step is usually required to achieve maximum sheet resistance and to remove hopping conduction. However, defect-induced compensation cannot withstand high temperature processing since the point defects responsible for the high resistivity are annealed out. On the other hand, compensation by deep level impurities requires high temperature processing to activate the implanted impurity.

1.2 Objectives

The objectives of this project are as follows:

(1) To achieve good electrical isolation in InP, InGaAs and InGaAsP materials by implantation of different ions such as proton, helium, iron and nitrogen at different dose, energy and implantation temperature.

(2) To develop a technology to enable implant isolation to be expanded to the fabrication of devices for which no reliable method currently exists.

1.3 Major contributions of the thesis

Ion implantation is an essential technique in InP integrated circuits and microwave devices to produce thermally stable isolated regions. However, a detailed study of implant isolation in InP, InGaAs and InGaAsP has not been done before as compared to GaAs based materials. Few publications can be found on implant isolation of InP, InGaAs and InGaAsP. In the majority of previous studies, implant isolation has been investigated using multiple energy and dose at room temperature and very little work has been done at different dose, implantation temperature and post-implant annealing temperature [35]. Optimisation of the implant isolation process with the right implantation parameters is essential to the fast growth of InP based integration technologies.
In the present work, isolation of n-type InP and InGaAs by implantation of ion species such as proton, helium, nitrogen and iron is studied. Iron implant isolation of p-type InP and InGaAs is also investigated. An introduction to the electrical isolation of n-type InGaAsP using nitrogen is also presented. The effect of ion species, ion mass, dose, energy, substrate temperature during implantation, post-implant annealing cycles and initial carrier concentration of the layer to be isolated on the quality of isolation in terms of optimisation and thermal stability is also examined. The effect of initial carrier concentration of the isolating layer (n-InP) has not been reported anywhere in the literature and is discussed in detail in chapter six. A detailed study of the effect of implantation temperature which has not been reported in the literature, is also discussed in this work.

Semi-insulating InP samples are also bombarded with different ion species and the sheet resistance is measured. From the semi-insulating experiment, a parallel resistor model is formulated to confirm the accuracy and reliability of the measurements of the isolated n and p type doped layer. A linear relationship is obtained between the initial sheet carrier concentration of the n-type InP and the threshold dose which is the minimum dose required for effective electrical isolation.

These results are novel and have applications to the semiconductor industry. They may have some ramifications to the process engineers in choosing the right implant conditions in order to provide effective electrical isolation of In-based devices.

1.4 Structure of the thesis

This thesis is divided into eight main chapters. Chapter one gives a brief introduction, objectives, scope and outline of the thesis. Chapter two describes the fundamental concepts of ion implantation. Chapter three reviews published data on ion implantation for isolation of InP, InGaAs and InGaAsP using different ion species.

In chapter four, a detailed explanation of the experimental process is described including the principles of operation of the ion implantation system. The process flow for making Hall patterns is also discussed in detail. A brief overview of the Accent Hall system, the 8 Lamp Rapid Thermal Annealing system, the Plasma Enhanced Chemical
Chapter One: Introduction

Vapour Deposition system, Secondary Ion Mass Spectroscopy and Rutherford Backscattering Spectrometry are described. The fundamental concepts of the Hall effect measurement are also explained.

Chapter five is devoted to hydrogen and helium implant isolation in InP and InGaAs. The parallel resistor model is also discussed in this chapter. Chapter six presents a detailed discussion of implant isolation of InP and InGaAs using iron. Implant isolation of InGaAsP and InP using nitrogen is also discussed. Chapter seven presents a general summary of the results and the effects of various implantation parameters on the isolation of InP, InGaAs and InGaAsP. Chapter eight presents the main conclusions of the whole work. Suggestions for further work are also outlined in this chapter.
Chapter Two

2. Overview of ion implantation

2.1 Theory of ion implantation

Ion implantation is a process by which ions are accelerated to a target at energies high enough to bury them below the surface. In this process, positively charged ions are extracted from the ion source and are accelerated to a particular energy. Depending on the application, the acceleration energies can range from a few keV to MeV. The ions are then mass analysed to select the required ion species. Finally, the ion beam is focused and scanned over the target. The accumulated charge is monitored as a means of controlling and measuring the dosimetry.

Energetic ions incident on targets have four main effects (see figure 2.1):

(a) Doping/isolation
Ion implantation can lead to the build up of a concentration profile of foreign atoms within a solid, thus altering the electrical properties of the near surface region.

(b) Damage creation
'Hard' nuclear collisions can result in displacement of lattice atoms from their regular sites. A single heavy ion can lead to the displacement of several tens, and even hundreds of lattice atoms within a volume surrounding the ion trajectory. Thus ion bombardment can create considerable structural damage to the material.

(c) Sputtering
In figure 2.1(c), we illustrate that nuclear collisions of a single ion can result in the ejection of one or more substrate atoms from the surface. Following bombardment with many ions, significant erosion (sputtering) of the surface can occur.

(d) Mixing
Figure 2.1(d) shows that the process of atomic mixing in which solid atoms can be transported within the dimensions of the collision cascade at temperatures below those
at which normal diffusion processes would operate. Ion bombardment can produce appreciable intermixing of both the substrate atoms into the film and the constituent atoms of the film into the substrate.

**Figure 2.1:** Effects of ion on targets; (a) doping/isolation, (b) damage creation, (c) sputtering and (d) mixing [120].
2.1.1 Ion stopping and range

When energetic ions enter a material, they start to lose kinetic energy through interaction with the electrons and nuclei of the target atoms and eventually stop. The particles experience a slowing force which is called the stopping power, $S$:

$$S = -\frac{dE}{dx}$$ -----(2.1)

We also define a quantity called the stopping cross section:

$$\sigma = \frac{-dE/dx}{N}$$ -----(2.2)

where $N$ is the atomic density of the material. The stopping cross section measures the contribution of a single atom to the stopping power of the material. The kinetic energy of the ions is mainly transferred to heat while some of it goes to creating damage in the material or detaching atoms from it (sputtering).

Stopping is conventionally divided into two parts, namely the nuclear and electronic stopping:

$$S = S_n + S_e$$ -----(2.3)

where $S_n$ is nuclear stopping and $S_e$ is electronic stopping. Nuclear stopping means the transfer of energy to target atoms via interatomic collisions. The ion can lose most of its energy in a single collision and its direction can change considerably. On the other hand, interaction of the ion with the electrons in the target material is called electronic stopping. The energy transferred in one collision is small and the change in direction is negligible. Sometimes the nuclear stopping is referred to as elastic energy loss and the electronic stopping as inelastic energy loss.
At high energies, electronic stopping dominates whereas at low energies nuclear stopping dominates. As a high-energy ion enters a target, it is slowed down initially by electronic stopping, but as its velocity decreases, nuclear stopping becomes predominant until the ion comes to rest. The contribution of electronic stopping to lattice damage is very small, but nuclear stopping can cause severe damage to crystalline targets and in the extreme, the surface layer may become amorphous.

Figure 2.2 shows a simple picture of an ion slowing down in a solid, which has a random array of atoms. For the high energy ion the path is essentially a straight line in the original direction of motion, since the stopping is electronic, with a small amount of straggle at the end due to nuclear collisions. At lower energies, where the nuclear stopping power and the electronic stopping power are more comparable, the ion path follows a zigzag course with many large angle deflections, the path length between collisions decreasing as the energy falls.
With reference to figure 2.2, we can define the total path length $R$, the projected path length $R_p$ in the original direction of the incident ion and the projected path length $R_\perp$ at right angles to this direction. Each ion that strikes the target will not follow exactly the same path even though its energy is fixed. This is because a beam of ions will have different random impact parameters with the surface atoms so that their subsequent collision sequences will be completely different from each other. Not only will the number of collisions suffered by an individual ion vary but also its total path length. This will naturally result in a distribution of stopping positions which is usually assumed to have a Gaussian (or normal distribution) shape as shown in figure 2.3, where $\overline{R}_p$ now refers to the mean projected range. In calculating ion ranges we must consequently always be concerned with averages of many events and must consider such properties as the average projected range and its standard deviation $\Delta R_p$.

\[
N(x) = \frac{1}{\sqrt{2\pi} \Delta R_p} \cdot \exp \left[ -\frac{(x - \overline{R}_p)^2}{2\Delta R_p^2} \right]
\]

\[\text{Figure 2.3: Gaussian distribution of doping distribution versus range.}\]

Therefore the ion profile distribution is given by [14]:

\[
N(X) = \frac{\Phi}{(2\pi)^{1/2} \Delta R_p} \cdot \exp \left[ -\frac{(X - \overline{R}_p)^2}{2\Delta R_p^2} \right]
\]

where $N(X)$ is the doping concentration at a distance $X$ from the surface
\[
\Phi \quad \text{is the ion dose per unit area}
\]
\[
\overline{R}_p \quad \text{projected range along axis of incidence}
\]
ARP is the statistical fluctuation (standard deviation) in the projected range.

The peak concentration occurs at $R_p$ and is given by

$$N_p = \frac{\Phi}{\sqrt{2\pi}} \frac{1}{\Delta R_p} = \frac{0.4\Phi}{\Delta R_p} \quad (2.5)$$

### 2.1.2 Channelling

In a crystalline material, the atoms form a periodic lattice. Consequently, stopping is not constant because the structure has lattice directions, called channels, where atomic and electron densities are considerably lower than elsewhere in the crystal. Channelling means that ions entering a channel have considerably longer ranges than ions entering the crystal in non-channelling directions [15,16]. Ions in a target material undergo three different types of trajectories (figure 2.4):

(i) Trajectory A: Those ions that are well channelled and penetrate large distances between the atom rows when they are incident along or closely aligned with the channel axis.

(ii) Trajectory B: Those ions which are incident at an angle which is not particularly well aligned with the axis so that nuclear collisions with the atom rows may lead to unstable trajectories and hence dechannelling.

(iii) Trajectory C: Those ions stop as if the target is random. They undergo large angle collisions with target atoms and follow a random path through the lattice.

In order to avoid the channelling effect, the most common approach consists of tilting the wafer by an angle of about $7-10^\circ$ with respect to the incident beam axis. Thus, the beam alignment will not be in the same direction as the major crystal axis. Additionally, the substrate is rotated by about $30-45^\circ$ to further prevent any direct ion path through the crystal. The critical angle of channelling $\theta_c$ is defined as the maximum angle of incidence of implanted ions to a channel between rows of atoms (axial channelling) or planes of atoms (planar channelling) so that the ion remains in a channelled trajectory. Even so, it is extremely difficult to avoid channelling completely, especially with low-
Figure 2.4: Schematic diagram of possible range profiles for ions implanted into a single crystal [120].

dose implants, since some of the ions can be deflected into channelling directions after entering the semiconductor. This residual channelling results in the formation of a tail as the dopant penetrates deeper into the semiconductor. Other approaches for suppressing channelling consist of pre-implanting the semiconductor with a neutral ion such as argon or neon, so as to make it amorphous before the active species is implanted. Implantation through a barrier layer, such as SiO₂, also serves to scatter the ion beam and reduce channelling problems.
2.2 Radiation damage

As an implanted ion enters a solid and slows down, it makes many collisions with the lattice atoms displacing them from their lattice sites. The displaced atoms with sufficient recoil momentum may further undergo several more displacement collisions with the lattice before coming to rest. This is also called a displacement cascade originating from a single primary collision between the implanted projectile and target atom. The net result is the production of a highly disordered region along and around the ion track.

Light ions tend to leave tracks characterized by relatively small amounts of damage. They slow down initially mainly by electronic stopping processes with little displacement damage, until eventually nuclear stopping becomes dominant at the end of their range. There is little lattice damage except near the end of the ion range. Heavy ions, by contrast, may create damage clusters along their path. These ions undergo a relatively higher degree of nuclear stopping than light ions, displacing target atoms right from the surface inward. These recoiling nuclei can also displace other nuclei, leading to considerable lattice damage within a relatively small volume.

At sufficiently high doses of lighter ions or low doses of heavy ions, the implanted surface may become completely amorphous, especially at low implantation temperatures (<RT). Essentially all atoms have been displaced from their lattice position. The models for eventual amorphization can be summarized in two categories:

(i) Heterogeneous model suggests that individual damage clusters are amorphous and complete amorphization occurs as a result of accumulation and merging of individual damage clusters.

(ii) Homogeneous model suggests that when the defect concentration reaches some critical value in a single crystal, the latter becomes unstable and transforms to an amorphous state.

The first model is generally believed to operate for heavy ions whereas the second is suited to light ions. Depending on the ion, the dose and the implant temperature, the implant damage can consist of either amorphous layer or extended defects.
In III-V compound semiconductors, the lattice elements are distinguishable and because they recoil unequally due to their different masses, local perturbations in stoichiometry are created. The lighter element recoils further, leading to an excess of the heavier element near the surface and an excess of the lighter element at greater depths (between $R_p$ and $R_p + \Delta R_p$). Repair of the lattice during subsequent annealing requires displaced atoms to diffuse back to appropriate sites, and in III-V's the diffusion lengths are not great enough to accomplish complete regrowth.

During ion implantation, the simplest defects created are Frenkel pairs, consisting of a vacancy and the displaced atom. More complex defects, such as divacancies, trivacancies can also be created, along with clusters of vacancies or interstitials with impurity atoms [17]. Line defects such as dislocations caused by accumulation of point defects are also common in implanted material. The damage due to ion implantation results in a reduction of the carrier mobility and creation of trap centers which trap the free carriers. Hence the material after implantation but before annealing tends to exhibit high resistance. This technique is commonly used to convert doped III-V compound semiconductors into a semi-insulating form [18,19]. It is also called implantation induced isolation or isolation by ion irradiation.

2.3 Simulation software
The atomic profile and damage distribution are obtained using the Stopping and Range of Ions in Matter (SRIM) program [20]. SRIM is a group of programs which calculate the stopping and range of ions into matter using a quantum mechanical treatment of ion-atom collisions. This program can accept ions of energies from 10eV to 2GeV. The stopping and range tables program gives a rapid calculation of ion ranges, range straggling values, nuclear stopping values and electronic stopping values over a large band of ion energies. The Transport of Ions in Matter (TRIM) program provides the atomic profile and damage distribution inside the target material. It is a Monte-Carlo calculation which follows the ion into the target, making detailed calculations of the energy transferred to every target atom collision. The Monte-Carlo program is based on the Binary Collision Approximation (BCA). The movement of ions in the target material is described by a series of successive binary collisions. The target is considered to be amorphous with atoms at random locations, and thus the directional properties of
the crystal lattice are ignored. The final result is based on the summation of the nuclear and the electronic scattering events occurring in a large number of simulated ion trajectories. TRIM accepts complex targets made of compound materials with up to eight layers, each of different materials. It also calculates both the final distribution of the ions and also all kinetic phenomena associated with the energy loss of the ion, such as target damage, ionisation, and phonon production. All target atom cascades are followed in detail, and the redistribution of these target atoms is determined.
Chapter Three

3. Review of implant isolation

Ion implantation was initially developed as a means of doping the semiconductor elements of integrated circuits. It has several advantages over growth or diffusion techniques such as accurate dose and depth control, good uniformity and reproducibility of doping, low temperature or room temperature implantation, minimal lateral spread of dopants beneath a mask, and several dopants may be added. It is also often possible to introduce dopants, which cannot be introduced by diffusion. Because of the speed, accuracy, cleanliness and controllability of the process, it has become the standard for this type of work.

One of the disadvantages of ion implantation is the introduction of disorder to the crystal during the implantation process. Thus, much of the early research was directed to the problem of determining the optimum implantation conditions and annealing sequence needed to remove the damage, and thus to allow the implant to dope the semiconductor. However, this type of damage can be used to form high resistivity layers in semiconductors. Ion implantation has also become firmly established as a technique for selectively changing the resistivity of semiconductors through the introduction of implantation induced deep levels to trap charge carriers [21]. Isolation by ion irradiation is also commonly known as implant isolation. It provides insulating device isolation for integrated circuits and current blocking layers for heterostructure lasers.

For large scale production of InP-based devices, ion implantation is preferred to mesa etching for device isolation, for several reasons:

(a) Implant isolation maintains a planar structure, which is desirable in device fabrication and wafer processing.

(b) Less intrusion under mask edges as compared to wet etching. The latter is isotropic, so that undercutting at mask edges can be quite severe when a deep mesa must be etched. (Dry-etching can be used to eliminate the undercutting
but the nonplanarity of the surface is still a problem and causes difficulty in subsequent resist application and metal step coverage.

(c) Implant isolation enables the achievement of low parasitic base-collector capacitance leading to high cut-off frequency in Heterojunction Bipolar Transistors (HBTs).

(d) Throughput is greater through implant isolation than mesa etching.

In III-V semiconductors, radiation-induced defects are observed to produce effective compensating centres, stable at room temperature. When light ions such as B$^+$ and N$^+$ are implanted into GaAs at 1MeV, 200 carriers are removed per implanted ion [22]. Thus when implanting dopant ions, even if all the implanted ions become electrically active, one residual defect per implanted ion is sufficient to compensate all of the doping effect. Thus this damage technique is valuable for device-isolation applications. There are two types of mechanism for creating implant-isolated regions in III-V semiconductors:

(1) Damage-induced isolation occurs when neutral ion species are implanted into the target to create damage-related deep levels in the material. Isolation results from the induced lattice damage and is dependent on a variety of parameters such as ion mass, dose, energy and substrate temperature during implantation.

(2) Chemical-induced isolation occurs when ion species are implanted into the material and combine with impurities or dopants already present in the material to create chemically active deep-level state. This type of compensation requires the implanted species to be substitutional, and hence annealing may be required to promote the ion into a substitutional site. An example of this model is the behaviour of iron in InP.

The two methods are somewhat complementary in that damage-induced isolation is effective to a temperature at which the damage anneals out, whereas chemically-induced isolation requires substitutionality of the implanted species through annealing.
High resistivity regions in doped InP can be produced by radiation damage from the implantation of ions such as H\(^+\), He\(^+\), B\(^+\), O\(^+\), N\(^+\) which create “damage” levels and Fe\(^+\), Co\(^+\) and Ti\(^+\) which create chemical related defects. The dose and energy of the ions used depends on the starting doping concentration and depth of the layer to be compensated. Implant isolation has been successfully employed for a number of different device structures, including GaAs metal semiconductor field effect transistors (MESFET) [23,24, 25], InP-based high electron mobility transistors (HEMT) [26,27], optical waveguides [28], heterojunction bipolar transistors (HBT) [29,30,31], PIN photodiodes [32,33], and IMPATT diodes [34]. A brief review of implant isolation in InP will be discussed in the next sub-section.

3.1 Proton and deuterium implant isolation

Hydrogen implantation has been used successfully to reduce electrical conductivity in selective areas of III-V compound semiconductors for device isolation and definition. The mechanism by which proton implantation produces carrier compensation is mainly through the introduction of damage, that is, damage-induced isolation. High proton ion doses are normally used in order to compensate for their low carrier removal rate.

In 1977, Donnelly et al [35] reported high resistivity (10\(^8\) \(\Omega\)cm) in p-type InP by using proton bombardment. But they observed that the bombarded layer converted to n-type material at high dose. For n-type InP, the maximum resistivity with proton bombardment was only ~3x10\(^3\) \(\Omega\)cm. Multiple-energy implantation was used with doses and energies of (3x10\(^{13}\)/cm\(^2\) & 400keV), (1.8x10\(^{13}\)/cm\(^2\) & 300keV), (9x10\(^{12}\)/cm\(^2\) & 200keV), and (6x10\(^{12}\)/cm\(^2\) & 100keV) for p-type InP(5x10\(^{17}\)/cm\(^3\)). They suggested a simple model to explain this behaviour (figure 3.1).

![Figure 3.1: Simple model to explain behaviour of Proton Bombardment on n-type and p-type InP.](image-url)
In n-type InP, the Fermi level moves downward with increasing proton dose, until it is pinned at a position designated as the high-dose Fermi level as shown in figure 3.1. The high-dose Fermi level can only be located within a range of 0.3-0.34eV above the intrinsic Fermi level ($E_i$). At this level, the resistivity is in the order of $10^3 \, \Omega \text{cm}$.

In p-type InP, the Fermi level moves upward with increasing proton dose. When the Fermi level reaches a point near the intrinsic Fermi level the sheet resistance becomes a maximum in the order of $10^8 \, \Omega \text{cm}$. For still higher doses, the Fermi level continues to move towards the conduction band until it is pinned at the high-dose level as shown in figure 3.1 and the bombarded layer becomes weakly n-type. They also investigated the annealing characteristics of proton bombarded InP layers. With increasing dose from $3 \times 10^{13} / \text{cm}^2$ to $1 \times 10^{14} / \text{cm}^2$, the post-annealing temperature at which resistivity was maximum also increased. Underannealing resulted in n-p junctions with high resistance, whereas overannealing resulted in Schottky barriers on p-type InP. This effect was recently verified by Hsieh et al [36]. They fabricated stripe geometry lasers by converting the p-type InP to n-type through bombardment of high flux of proton ions, thereby forming p-n junctions to confine the current.

Boudinov et al [37] studied the effect of proton dose at 600keV into both n and p-type InP at RT. The dose was varied from $1 \times 10^{12} / \text{cm}^2$ to $1 \times 10^{16} / \text{cm}^2$. A maximum sheet resistance of $2 \times 10^6 \, \Omega / \square$ and $2 \times 10^7 \, \Omega / \square$ was obtained for n and p-type InP at a dose of $3 \times 10^{14} / \text{cm}^2$ and $2.8 \times 10^{13} / \text{cm}^2$ respectively. They reported thermally stable isolation up to $200^\circ \text{C}$ and $500^\circ \text{C}$ for n and p-type InP respectively for different doses. They suggested that different traps are formed during proton bombardment into n and p-type InP. They also suggested [38] that the In$_p$ antisite acceptor-like defects are probably responsible for trapping of electrons. They believe that this defect is annealed out in the region of $200^\circ \text{C} - 300^\circ \text{C}$. Hence, a poor thermal stability is observed for proton implant isolation of n-type InP.

Woodhouse et al [39] studied the effect of varying the proton dose from $1 \times 10^{12} / \text{cm}^2$ to $4.4 \times 10^{15} / \text{cm}^2$ at an energy of 100keV on semi-insulating InP:Fe for room temperature implantation. They reported constant sheet resistance of the order of $10^6 \, \Omega / \square$ for ion
doses from $1 \times 10^{12}$ /cm$^2$ to $4.4 \times 10^{14}$ /cm$^2$ and a drop in the sheet resistance to $10^4$ $\Omega$/sq for increasing dose. They attributed the decrease in the sheet resistance at higher dose to the rapid increase in donor defects as compared to acceptor defects. They also suggested the reduction in the effectiveness of the Fe dopant as an acceptor impurity due to the formation of electrically inactive complexes between the Fe atoms and the proton ions or the diffusion of Fe away from the damaged region. They reported a decrease in sheet resistance to $10^3$ $\Omega$/sq at an annealing temperature of 200$^\circ$C for 30min in flowing N$_2$. However, the sheet resistance increased again beyond an annealing temperature of 200$^\circ$C since there was a rapid increase in the donor removal rate to the point that Fe acceptors again dominated the electrical properties of the bombarded layers.

Thompson et al [40] measured resistivity in two different n-type InP structures, namely vertical and lateral geometry after proton bombardment with implantation concentration varying from $1 \times 10^{17}$ to $2 \times 10^{20}$ ions/cm$^3$. Resistivity was higher by two orders of magnitude for vertical geometry than lateral geometry. They ascribed this disparity to the reduction in the resistivity of the Fe-doped InP substrate due to proton implantation, which acted as a shunt for lateral geometry. They showed that for proton doses less than $5 \times 10^{15}$ ions/cm$^2$, the substrate was semi-insulating but for greater doses, the resistivity decreased monotonically with dose. The highest resistivity for the vertical geometry of value $3 \times 10^3$ $\Omega$cm was comparable with the previous work by Donnelly et al [35] who obtained a resistivity of $3 \times 10^3$ to $4 \times 10^3$ $\Omega$cm with multiple energy proton implantation. Annealing was done from 50$^\circ$C to 500$^\circ$C for the vertical geometry and for implantation concentration greater than $2 \times 10^{17}$ ions/cm$^3$, a constant resistivity greater than $10^3$ $\Omega$cm was maintained up to 250$^\circ$C.

Protons do not seem to provide good electrical isolation in either n or p-type InGaAs. Pearton et al [41] reported a sheet resistance of only $10^2$ $\Omega$/sq after multiple low energy proton $[5 \times 10^{13}$/cm$^2$@30keV $+ 7 \times 10^{13}$/cm$^2$@60keV $+ 1 \times 10^{14}$/cm$^2$@320keV] implantation in n$^+$ In$_{0.53}$Ga$_{0.47}$As epilayer of thickness ~0.5$\mu$m. The sheet resistance decreased towards its original value above a post-annealing temperature of 200$^\circ$C. Similar doses and energies were used on p-type In$_{0.53}$Ga$_{0.47}$As. A sheet resistance value of only 400 $\Omega$/sq was obtained. The latter remained quite constant until an annealing
temperature of 500°C. Similar results were obtained by Rao et al [42] for multiple energy proton irradiation on p-type InGaAs of thickness ~1.4 μm. They reported a gradual increase in sheet resistance with dose from 1.4x10¹⁰ /cm² to 1.4x10¹² /cm². An optimal isolation value of 580 Ωcm was reported at a threshold dose of 1.68x10¹² /cm².

Deuterium implants appear to give higher resistivity material over a wider dose range than protons. Focht et al [43] demonstrated high resistivity of the order of 10⁹ Ωcm for deuteron implantation in p-type InP:Zn over a dose range 3x10¹³-1x10¹⁴ /cm² at an energy of 200keV. Best isolation was obtained at a deuteron dose of 3x10¹³ /cm² above which the resistivity fell. A comparison of the compensating behaviour of protons, deuterons and tritons was made by Steeples et al [44]. They concluded that deuterons removed carriers far more efficiently than protons or tritons due to ionisation produced during the irradiation leading to enhanced diffusion of defects and consequent rearrangement of certain damage centres. But the tritons were only slightly more effective than protons at removing carriers.

3.2 Helium implant isolation

Bombardment of InP with deuterons was reported to produce highly resistive regions [43]. Although this approach appears to be controllable and reproducible, there is a health hazard due to neutron generation from the deuteron-deuteron reaction. Thus several authors have investigated the applicability of using ³He⁺, and ⁴He⁺ bombardments to make InP and InGaAs semi-insulating. Focht et al [45] reported that ion bombardment by ³He⁺, and ⁴He⁺ of InP, produced regions of high resistivity; 10⁸ - 10⁹ Ωcm in p-type and 10⁴ Ωcm in n-type InP. The average resistivity due to ³He⁺ bombardment on p-type InP produced a broader peak over a dose range of 10¹³-10¹⁶ /cm², while that due to the ⁴He⁺ implant peaked more sharply at the dose of 10¹⁵ /cm². For all the resistivity curves, the resistivity decreased at highest dose due to the onset of hopping conduction caused by high density of deep states. The use of helium bombardment offers the advantage of reproducibility and no hazardous neutron generation.
Similarly, Sargunas et al [46] obtained a resistivity of \( \sim 10^3 \ \Omega \text{cm} \) in n-type InP (5x10^{17}/cm^3) for both room temperature and 60K He\(^+\) ion implantation using a dose and energy of 3x10^{13} /cm\(^2\) and 55keV respectively. This resistivity was increased up to \( \sim 10^5 \ \Omega \text{cm} \) after suitable annealing for 60s in nitrogen ambient. These highly resistive regions exhibited good stability to heat treatment up to 450\(^0\)C-550\(^0\)C. They also investigated the effect of helium dose for 60K and 300K implants. They reported one order of magnitude decrease in the resistivity (7x10\(^3\) \ \Omega \text{cm}) above a dose of 1x10^{14} /cm\(^2\) for both implantation temperatures and related this effect to the onset of hopping conductivity. For a dose of 10^{16} /cm\(^2\) and an implant temperature of 300K, the sheet resistance increased to a maximum (\( \sim 9x10^6 \ \Omega/\square \)) at 380\(^0\)C and dropped drastically to a low value (\( \sim 10^4 \ \Omega/\square \)) as the damage-related deep levels were annealed. For doses higher than 10^{15} /cm\(^2\), the sheet resistance at 60K implantation was higher than that at room temperature implantation due to amorphisation of the InP layer. Thompson et al [40] also reported resistivity of the order of 10^3 \ \Omega \text{cm} using a dose and energy of 8.4x10^{12} /cm\(^2\) and 160keV respectively.

Quintanilla et al [47] investigated the electrical behaviour in p\(^+\) n InP junctions isolated by He\(^+\) bombardment with an energy and dose of 60keV and 1x10^{14} /cm\(^2\) respectively using Deep Level Transient Spectroscopy (DLTS) measurements. They reported the existence of two deep levels: an electron trap and a hole trap located at 0.19eV below the conduction band and at 0.13eV above the valence band respectively.

Rao et al [42] irradiated a p-type InGaAs epilayer with multiple energy He\(^+\) ions. The layer thickness and doping concentration was \( \sim 1.4\mu\text{m} \) and 5x10^{16} /cm\(^3\) respectively. They reported an as-implanted sheet resistance value of \( \sim 10^5 \ \Omega/\square \) after a helium dose of 5x10^{11} /cm\(^2\). The sheet resistance increased to a maximum value of 3x10^5 \ \Omega/\square \ after annealing at 200\(^0\)C for 10mins and then decreased gradually to \( \sim 10^4 \ \Omega/\square \) above 200\(^0\)C. They also showed that the as implanted sheet resistance value dropped drastically by two order of magnitude above a dose of 5x10^{11} /cm\(^2\). They associated the decrease in the sheet resistance at higher doses to donor defects being created during implantation. A p- to n-type conversion was observed and a net electron concentration was measured for He\(^+\) doses greater than 5x10^{11} /cm\(^2\).
Sargunas et al [48] reported similar p- to n-type conversion after implanting $1 \times 10^{12}$ ions/cm$^2$ into InGaAs. A maximum sheet resistance (~$10^7 \, \Omega/\square$) was obtained at this dose. At higher doses, the sheet resistance decreased to ~$5 \times 10^4 \, \Omega/\square$ and this effect was related to hopping conduction. Samples implanted at a dose of $3 \times 10^{13} \, \Omega/\square$ were annealed from RT to 550$^\circ$C for 60s in nitrogen ambient. A gradual increase in the sheet resistance was observed and maximum sheet resistance (~$10^6 \, \Omega/\square$) was obtained for an annealing temperature in the range of 430$^\circ$C - 480$^\circ$C.

3.3 Boron implant isolation

Boron ion bombardment has also been used in III-V compound semiconductors for isolation. Kamiya et al [49] implanted boron ions at an energy of 30keV with doses in the range of $1 \times 10^{14}$-$5 \times 10^{14} \, \Omega/\square$ into both n$^+$ InP and SI InP. They reported thermally stable isolation up to an annealing temperature of 600$^\circ$C for B$^+$ dose of at least $5 \times 10^{14} \, \Omega/\square$. However, they observed that n-type carriers were generated in the Fe-doped semi-insulating InP for doses lower than $5 \times 10^{14} \, \Omega/\square$. The carrier generation was also observed in undoped InGaAs, but not in SI GaAs. They believed that carrier generation has a close relation to the In atoms in the substrate. Similar results were reported by Favennec et al [50] using a boron concentration of $1 \times 10^{19} \, \Omega/\square$. They observed depletion of carriers in n-type InP and that the carrier depletion disappeared after annealing at 650$^\circ$C.

Nadella et al [51] used high energy from 1 to 5MeV and doses from $10^{11}$ to $10^{16} \, \Omega/\square$ for the creation of buried layers in InP:Sn (~$2 \times 10^{18} \, \Omega/\square$). A maximum resistivity of $2 \times 10^6 \, \Omega/\square$ was obtained using a dose and energy of $1 \times 10^{15} \, \Omega/\square$ and 3MeV respectively and annealed at 500$^\circ$C for 15min. A relatively constant resistivity ($2 \times 10^3 \, \Omega/\square$) was obtained for different implant energy from 1MeV to 5MeV after annealing at 400$^\circ$C for 10mins using a constant dose of $10^{14} \, \Omega/\square$. As the resistivity was a function of the damage concentration, it should remain almost constant with ion energy.

Sargunas et al [52] reported a maximum sheet resistance of $4 \times 10^7 \, \Omega/\square$ in n$^+$ InP after 125keV B$^+$ bombardment at a dose of $1 \times 10^{13} \, \Omega/\square$. The peak of the damage distribution was placed within the doped layer. Above this dose, a decrease in the sheet resistance
was observed due to hopping conduction. Fourre et al [26] investigated implant isolation of InGaAs/InAlAs/InP Modulation Doped Field Effect Transistors using boron ions. The highest sheet resistance ($1 \times 10^6 \, \Omega/\square$) was obtained for a dose of $1 \times 10^{14}/\text{cm}^2$ and energy of 20keV with post-implant annealing at 300°C for 10mins. For higher temperature annealing or longer annealing time, the traps density decreases below the electron density, leading to a decrease in the sheet resistance. They have shown that implant isolation at higher energies does not provide better isolation.

3.4 Oxygen implant isolation

S. J. Pearton [53] reported that the thermal stability of implant isolation achieved with O$^+$ ions exceeded that of protons and thus, the use of heavy O$^+$ ions could be beneficial in increasing device lifetime. Akano et al [54] obtained maximum sheet resistance of approximately $10^5 \, \Omega/\square$ using multiple-energy oxygen implantation in n-type InP(Si doped) and in p-type InP(Zn doped), both of concentration $5 \times 10^{17}/\text{cm}^3$. The multiple-energy implantation schemes (50, 200, 330 and 550keV) with doses in the range of $5 \times 10^{11}/\text{cm}^2$ to $1.2 \times 10^{12}/\text{cm}^2$ were used to produce a uniform defect distribution throughout the InP epilayer thickness. Highest sheet resistance was achieved using the highest doses for both n-type InP and p-type InP when samples were annealed for 30s at a temperature of 400°C and 500°C respectively. A thermally stable isolation up to an annealing temperature of 550°C was reported for both n- and p-type InP for the highest dose. In the case of p-type InP, Hall data showed that type conversion occurred only for the highest dose. They also investigated the effect of O$^+$ into both n- and p-type InGaAsP. Maximum as-implanted sheet resistance of $\sim 10^5 \, \Omega/\square$ was obtained in both n and p-type InGaAsP using the highest dose. Similar type conversion was reported for p-InGaAsP for the highest O$^+$ dose.

Pearton et al [41] also used multiple-energy O$^+$ ion implantation [$7 \times 10^{12}/\text{cm}^2(50\text{keV}+1 \times 10^{13}/\text{cm}^2(150\text{keV})+2 \times 10^{13}/\text{cm}^2(300\text{keV})]$ into an n-type InP (Sn-doped) epilayer. They showed a maximum sheet resistance of $\sim 10^6 \, \Omega/\square$ at an annealing temperature of $\sim 300^\circ\text{C}$ for 30s. When a dose of $\sim 10^{11}/\text{cm}^2$ was used, less thermally stable sheet resistance was obtained. The latter decreased drastically from $\sim 4 \times 10^4 \, \Omega/\square$ to $\sim 10^2 \, \Omega/\square$ with increasing annealing temperature from 100°C to 500°C.
They also implanted O\textsuperscript{+} ion into both n- and p-type InGaAs using multiple-energy and
dose of $1 \times 10^{14}/\text{cm}^2(40\text{keV}) + 2 \times 10^{14}/\text{cm}^2(200\text{keV}) + 3 \times 10^{14}/\text{cm}^2(400\text{keV})$. For the n-
type InGaAs epilayer, they achieved a maximum sheet resistance of $\sim 10^4 \Omega/\square$ at an
annealing temperature of 300$^\circ$C for 30s. There was a drop in sheet resistance value by an
order of magnitude at 400$^\circ$C. However an optimal isolation of $\sim 10^5 \Omega/\square$ was obtained
for p-type InGaAs after annealing at 400$^\circ$C for 30s.

Thompson et al [55] investigated the effect of multiple-energy O\textsuperscript{+} implant into n\textsuperscript{+} InP of
doping concentration $2 \times 10^{17}/\text{cm}^3$. The uniform doping concentration was obtained using
multiple-energy Si\textsuperscript{+} implantation. Maximum sheet resistance of $\sim 2 \times 10^7 \Omega/\square$ was
obtained at a dose of $1 \times 10^{12}/\text{cm}^2$. A gradual decrease in the sheet resistance was
observed with increase in the dose from $10^{12}/\text{cm}^2$ to $10^{15}/\text{cm}^2$. They reported a
classically stable isolation up to 450$^\circ$C and an abrupt decrease in the sheet resistance
between 450$^\circ$C and 500$^\circ$C. Annealing time was 30 mins.

Ridgway et al [56,57] have used single high energy (5MeV) oxygen ion implantation in
p\textsuperscript{+} InP/Semi-insulating InP. Higher sheet resistance as compared to Akano et al [54] of
approximately $5 \times 10^6 \Omega/\square$ was obtained using an annealing temperature of 350$^\circ$C for
10min and a dose of $1 \times 10^{13}/\text{cm}^2$. A dose of $1 \times 10^{15}/\text{cm}^2$ resulted in a decrease of the
sheet resistance by one order of magnitude. They have proved that the maximum sheet
resistance from single MeV energy implantation is of the same order of magnitude as
that from multiple energy implantation. They [58] also reported electrical isolation of
InGaAs/InP p-i-n photodiodes using 0.17 and 0.03MeV O\textsuperscript{+} implantation. Maximum
sheet resistance of $\sim 10^6 \Omega/\square$ was obtained at a dose of $\sim 5 \times 10^{14}/\text{cm}^2$ after annealing at a
temperature of 425$^\circ$C for 5min. They [32] used 0.095, 0.400 and 1MeV O-ion implants
for electrical isolation of p+ InP. The doses were in the ratio 1:1.24:2.57 for energies of
0.095, 0.400 and 1.0MeV respectively to produce a uniform level of disorder over the
extent of the epitaxial layer (thickness of 1.3\mu m). The maximum sheet resistance of
$\sim 3 \times 10^6 \Omega/\square$ was obtained after annealing at 400$^\circ$C for 60s.

Barnard et al [59] studied the effect of dose variation at 100keV into n-type ($3 \times 10^{16}$ cm$^{-3}$)
Ge doped InGaAs. The InGaAs layer was grown by MBE with a thickness and doping
concentration of 1000Å and 3.8x10^{16} /cm^3 respectively. They reported a maximum sheet resistance of 7x10^5 Ω/□ after bombardment at a dose of 1x10^{13} /cm^2 and a post-implant annealing of 200°C for 60s.

3.5 Nitrogen implant isolation

Nitrogen is also a good candidate for implant isolation. Xiong et al [60] reported deep buried layers of resistance ~10^6 Ω/□ in n-type InP after subsequent thermal annealing using a dose range of 5x10^{14}-1x10^{16} /cm^2 at an energy of 5MeV. They also observed better thermal stability for samples implanted at the highest dose (1x10^{16}/cm^2) than the lower dose (1x10^{14}/cm^2). From RBS, an amorphous layer of thickness ~4μm is formed in n-type InP above a dose of 1x10^{15}/cm^2. They finally formed a buried semi-insulating layer in InGaAsP/InP laser by direct nitrogen ion implantation.

Likonen et al [61] studied 30keV N⁺ implantation into both n and p-type InP using a dose of 1x10^{16} /cm^2. They reported higher loss of nitrogen in n-type InP as compared to p-type InP. The n-type InP material had a very dominant tendency for surface nitrogen build-up, whereas the p-type material had a markedly smaller surface peak in the nitrogen distribution. SIMS analyses showed sulphur build-up on the surface during annealing acting partly as a nitrogen diffusion barrier.

Sargunas et al [48] obtained high resistance layers (~5x10^7 Ω/□ for p-InGaAsP and ~5x10^6 Ω/□ for p-InGaAs) following 120keV N⁺ implantation at a dose of 3x10^{11} /cm^2. The initial doping concentration of the p-type layers was ~1x10^{18} /cm^3. For both InGaAsP and InGaAs p- to n-type conductivity conversion occurred. They showed that the thermal stability of the implants increases with ion dose and increased resistivity layers stable to thermal treatment up to 427°C for 60s was obtained.

Comedi et al [62] studied the formation of high resistivity regions in n-type (1x10^{18} cm^{-3}) InGaAsP (0.3μm thick) induced by dual N⁺ bombardment (125keV + 37keV). The dose was varied from 5x10^{11} /cm^2 to 1x10^{16} /cm^2. Maximum sheet resistance (7x10^6 Ω/□) was obtained at a dose of 1x10^{13} /cm^2 followed by a drop in the sheet resistance value above this dose due to hopping conduction. However, they observed that the sheet
resistance increased again at a dose of $2 \times 10^{15} \text{ cm}^{-2}$. This increase in the sheet resistance was correlated to the transition of the InGaAsP layer to an amorphous-like state. Post-implant annealing at the threshold dose showed a gradual increase in the sheet resistance to a maximum value of $2 \times 10^7 \Omega/\square$ at $350^\circ\text{C}$ for 60s.

3.6 Implant isolation using transition metals

It was first shown by Donnelly et al [63] that implantation of iron (Fe) into n-type InP led to the formation of highly resistive material after annealing to promote the Fe into a substitutional site. Wilt et al [64] found that in Fe-implanted InP, chemical doping was the dominant effect, where Fe acted as a simple deep acceptor near the middle of the energy band gap and compensation of free carriers drove the Fermi level to the intrinsic level.

Vellanld et al [65] also reported high energy (0.34-5MeV) implantation of InP:Sn ($1.3 \times 10^{17} \text{ cm}^{-3}$) at RT and $200^\circ\text{C}$. High energy Fe and Co implantation at $200^\circ\text{C}$ was used to obtain thermally stable, buried and high-resistance layers of good crystalline quality in n-type InP and for compensation of the tail of the buried n-type implant. However, due to the low solubility of Fe and Co in InP, the implants of these species were useful only to compensate n-type carriers with concentrations below $10^{17} \text{ cm}^{-3}$. They reported a projected range of 1.19um using SIMS and 1.26um using TRIM 91 data for an energy of 2MeV. At energies greater than 1MeV, the $R_p$ and $\Delta R_p$ values obtained from TRIM 91 were higher than those obtained from SIMS data. The differences were 3%-11% and 1%-5% for $R_p$ and $\Delta R_p$ respectively. Additional peaks around $0.8 R_p$ and $R_p + \Delta R_p$ were observed in the SIMS depth profiles of room-temperature implants after annealing. Schwartz et al [66] showed similar behaviour to Vellanld et al [65] for RT and $200^\circ\text{C}$ implant using a dual Fe implant configuration [(275keV, $1.25 \times 10^{14} \text{ cm}^{-2}$) & (400keV, $1.25 \times 10^{14} \text{ cm}^{-2}$)] from SIMS depth profiles. Surface Fe peaks were observed in the RT samples but not in the $200^\circ\text{C}$ samples. The absence of a double Fe peak in the annealed $200^\circ\text{C}$ implant could be explained by the lack of an amorphous-crystalline interface or by rapid diffusion of interstitial In and P, thereby preventing a build-up of excess P at $R_p + \Delta R_p$. No amorphous region during the $200^\circ\text{C}$ implant and no gross redistribution of Fe after annealing for 1hr at $725^\circ\text{C}$ were observed. Frigeri et al [67]
showed that high energy (2MeV) Fe implantation created a high density of twin defects which were much more difficult to eliminate than at lower beam energies.

Ridgway et al [68] implanted Fe ions in InP:Zn(5x10^17/cm^3) using a dose of 1x10^{15}/cm^2 and reported a maximum sheet resistance of ~5x10^6 Ω/□. The sheet resistance began to decrease towards the unimplanted value at a temperature of 500°C for 30s but the sheet resistance was increased again at an annealing temperature greater than 600°C, potentially the result of Zn out-diffusion. From SIMS measurements, at low temperature ~450°C, Fe diffused towards the sample surface. Annealing at a temperature of 800°C resulted in Fe diffusion to greater depths and the formation of a secondary Fe peak at a depth of ~2μm as observed by Vellanlci et al [65].

Pearson et al [41] implanted multiple-energy Fe⁺ into n⁺InP. The layers of thickness and doping concentration of ~0.5μm and ~10^{18}/cm^2 respectively were grown on semi-insulating (100) InP by MOMBE. For multiple doses [50keV(1x10^{13}/cm^2) + 200keV(2x10^{13}/cm^2) + 400keV(4x10^{13}/cm^2)], the behaviour of the sheet resistance with annealing temperature (30s) was similar to the case of implant damage in GaAs [69]. A peak sheet resistance (~5x10^4 Ω/□) was observed at about 400°C, which decreased to an unimplanted value above 400°C. In the case of higher multiple doses [50keV(7x10^{13}/cm^2) + 200keV(1x10^{14}/cm^2) + 400keV(2x10^{14}/cm^2)], better isolation (~2x10^6 Ω/□) was obtained. Fe⁺ was also implanted into both n⁺ and p⁺-In₀.₅₃Ga₀.₄₇As/InP using a dose and energy of 2x10^{14}/cm²(50keV)+ 3x10^{14}/cm²(200keV) + 2x10^{14}/cm² (400keV). The sheet resistance increased from 10^2 Ω/□ to 7x10^4 Ω/□ as the annealing temperature increased from 23°C to 300°C. Between 300°C and 500°C, a thermally stable region with a sheet resistance (~7x10^4 Ω/□) was obtained.

Fe has also been used to produce semi-insulating regions for isolation in a variety of lightwave devices. Cheng et al [70] have fabricated semiconductor-insulator-semiconductor n⁺ - SI - n⁺ InP structures using Fe implantation into n⁺ InP substrates. This enables current confinement in channelled substrate buried heterostructure (CSBH) lasers.
Implant isolation of InP-based materials

Chapter Three: Review of implant isolation

Gulwadi et al [71] studied multiple-energy Fe, Cr and V implantation into n-type InGaAs. They reported that sheet resistance close to the intrinsic limit (3.5x10^7 Ω/□ [72]) was obtained on annealed samples which had been implanted with Fe. However, they observed a lower resistivity after Cr and V implantation and annealing. They suggested that the introduction of donor levels by either substantial Cr and V atoms or their complexes with some native defects is the cause of the poor isolation in n-InGaAs.

3.7 Implant isolation using other ions

Asahi et al [73] have observed the formation of high sheet resistance region (~10^6 Ω/□) in n-type InP by Ga⁺ implantation at a dose and energy of 7.7x10¹⁴ /cm² and 70keV respectively. The initial doping concentration and thickness of the n-type InP layer was 1x10¹⁸ /cm³ and 700Å respectively. Thermally stable isolation up to 700°C was reported by Asahi et al. Annealing time was 30mins. They suggested that the origin of the formation of the high-resistance region is due to the formation of carrier trapping centers.

Fourre et al [27] investigated implant isolation of InP lattice-matched HEMT layers using Ar⁺. They reported good isolation (~3x10⁷ Ω/□) using a dose of 10¹⁵ /cm² after annealing at a relatively low temperature (300°C) for 10hours. They [26] also published that a sheet resistance of ~1x10⁶ Ω/□ was obtained after bombardment with fluorine at a dose and energy of 1x10¹⁴ /cm² and 35keV respectively. Annealing above 300°C for 10min resulted in a decrease in the sheet resistance.

Almonte et al [74] investigated the electrical properties of Ne⁺ implanted into undoped InGaAs layers. Multiple energy implants (from 50 to 200keV) were used to obtain uniform concentrations over the entire 0.4μm thick InGaAs layer. A thermally stable maximum sheet resistance of 1.25x10⁵ Ω/□ was obtained. Above an annealing temperature of 300°C for 30s, a gradual decrease in the sheet resistance was observed.

3.8 Summary of implant isolation

Tables 3.1, 3.2, 3.3 and 3.4 show a summary of the literature review of implant isolation in both n-type and p-type InP and InGaAs, using different ion species. Table 3.5
summarises the literature review of implant isolation in n and p-type InGaAsP. Most of the implant isolation was done using multiple-energy and dose at RT. Very few investigations of implant isolation at different doses and substrate temperatures were done. We have investigated these two implantation parameters in this work. MeV-like implants instead of multiple low energy implants were done for the formation of high resistivity layers. This area was not fully investigated in the literature. Light ions form less thermally stable isolation in n-type InP as compared to heavy ions. In chapter 5, the effect of substrate temperature and dose for proton and helium implantation into n-type InP and InGaAs is studied in detail. Very poor isolation of n-type InGaAs layers have been demonstrated (see table 3.3) using mostly multiple energy schemes for various ion species. In chapter 6, high sheet resistance (~5x10⁶ Ω/□) in both InP and InGaAs is reported using single energy iron implantation. The effect of initial carrier concentration of the isolating layer has not been investigated in the literature but is discussed in detail in chapter 6.

<table>
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<th>Isolating ion species</th>
<th>Initial doping concentration (cm⁻³)</th>
<th>Dose and energy (cm⁻² &amp; keV)</th>
<th>Implantation temperature (℃)</th>
<th>Post-implant annealing (℃)</th>
<th>Maximum Sheet resistivity (Ω/□)</th>
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<td>Iron</td>
<td>MOCVD grown, Si-doped, 1x10¹⁸</td>
<td>(7x10¹³ &amp; 50)-(1x10¹³ &amp; 200)-(2x10¹³ &amp; 400)</td>
<td>RT</td>
<td>600</td>
<td>2 x 10⁶</td>
<td>41</td>
</tr>
<tr>
<td>Gallium</td>
<td>MBE grown, Si-doped, 1x10¹⁸</td>
<td>(7.7x10¹³ &amp; 70)</td>
<td>RT</td>
<td>none</td>
<td>1 x 10⁶</td>
<td>73</td>
</tr>
</tbody>
</table>

Table 3.1: Summary of literature review of implant isolation of n-type InP.
Chapter Four

4. Experimental procedures

4.1 Process steps for implant isolation in InP, InGaAs and InGaAsP

Czochralski grown wafers of semi-insulating InP of <100> orientation are used in our work. The n-type doped InP layers on semi-insulating InP are obtained by either ion implantation or MBE technique. All the p-type InP, n-type InGaAs, p-type InGaAs and n-type InGaAsP single layers were grown using SSMBE at the EPSRC III-V Central Facility, University of Sheffield. All wafers are cleaned using three stages of cleaning (see section 4.5) before printing the clover-leaf pattern on them.

Formation of n-type doped InP layer by ion implantation

Semi-insulating InP wafers of 2” diameter are implanted with multiple-energy silicon ions to form a uniformly n-type doped layer. All doping implants are performed at room temperature using the Danfysik 1090 Ion Implanter. The normal of the sample is inclined at 7° with respect to the beam to minimize ion channelling. The wafers are then encapsulated with silicon nitride (Si$_3$N$_4$) deposited by plasma-enhanced chemical vapour deposition at a temperature of 300°C. The thickness of Si$_3$N$_4$ on the front and back of all wafers is 500Å and 800Å respectively. Rapid thermal annealing is carried out at 700°C-850°C for 60s-150s in order to electrically activate the implanted dopant. The encapsulant is then stripped off using 10% buffered hydrofluoric acid (HF) and the wafers cleaned in organic solvents.

All the n and p-doped wafers are cleaved to obtain several samples of approximately 1 cm$^2$. The clover-leaf pattern is printed on the samples using an optical lithography technique. It is important to know which side of the wafer has been doped before making the Hall patterns using optical lithography. A detail guide to the optical lithography process is described in section 4.5. The samples are then dipped in 10% buffered hydrofluoric acid to remove the thin layer of oxide on the surface. Ohmic contacts are made at the four corners of the Hall patterns using indium dot for n and p-type InGaAs and n-type InP. Gold/Zinc/Gold was used for ohmic contact on p-type InP. By exposing only the centre of
the Hall pattern, the sample is then implanted with the desired species for the implant isolation process. For 77K and RT implants, the indium (In) contacts mask the region for isolation. For 100°C and 200°C, molybdenum (Mo) foils are used as a mask. Hall measurements are done to obtain the sheet resistance, the sheet carrier concentration and the Hall mobility. Figure 4.1 shows a flowchart of the whole implant isolation process.

**Figure 4.1:** Flowchart of the implant isolation process.
4.2 Basic principles of ion implantation

All low energy implants up to 200keV were implanted using the Danfysik 1090 Ion Implanter and for high energy implants, the 2MV Van de Graaff Ion Implanter was used. The basic features of an ion implantation machine are a variety of ions sources, means of ion extraction, acceleration to high energies, and beam manipulation. Figure 4.2 shows a schematic diagram of the 2MV Van de Graaff ion implanter. All the ion sources, including their power supplies and control elements, are operated by remote means, as are the valves and the high vacuum pumps. For the 2MV Van de Graaff ion implanter, three separate valves and one turbomolecular pump are used to allow individual access to the target chamber, while keeping the rest of the system under high-vacuum conditions at all times. Particular care is taken to operate the target chamber with oil-free pumping systems to avoid hydrocarbon cracking of residual oil vapours.

![Schematic diagram of the 2MV Van de Graaff ion implanter.](image)

**Figure 4.2**: Schematic diagram of the 2MV Van de Graaff ion implanter.

Ion species are produced in an ion source. A gas containing the desired atoms is introduced into the cylindrical anode at a pressure of about $10^{-3}$ torr. Electrons are emitted from the filament in the ionizer and spiral towards the anode in crossed electric and magnetic fields,
Ionising gas atoms along their trajectory to produce plasma. All positive species of ions in the plasma are extracted through an aperture held at a negative DC bias. In the case of iron implantation, a sputter ion source is used. Iron is loaded on the sputter probe which is biased at a few kilovolts negative potential. The discharge is established on a convenient support gas (Argon) and positive ions are produced, which are attracted to the negative potential of the sputter probe, resulting in low energy sputtering of the probe material. The resultant sputtered material is then ionised in the plasma. In both types of ion sources, the ion beams are usually contaminated by atomic and molecular ion species, which are sputtered from the walls and filaments. Purification of the ion beam, to select the desired implant species, is thus an essential requirement. This is carried out by means of a mass analyser, which selects a single desired species. This analysis can be done before or after the beam has been accelerated to the required energy. Analysis before acceleration results in the use of a smaller low-cost machine. However analysis after is preferred when it is required to implant molecular species, which might partially dissociate during the acceleration process. After selection of the desired ion species, electrostatic deflection is used to raster the beam across an earthed aperture plate, which determines the beam area on the sample. This is achieved through parallel vertical and horizontal plates held at different potentials.

Secondary electron emission is limited by the application of a positive bias to a metal plate (suppression plate) located between the sample and aperture plates. Direct observation of the beam current between the sample plate and earth allows the machine conditions to be optimised prior to implantation. Current integration allows the implanted dose to be calculated since

\[ N_D = \frac{Q}{eA} \]

where \( Q \) is the total integrated charge
\( N_D \) is the implanted dose
\( e \) is the electronic charge
\( A \) is the beam area defined by the aperture plate
A pneumatically operated gate valve automatically shuts off the ion beam when a preset charge corresponding to a desired dose is reached by the integrator. Although beam intensity and energy are clearly important, several other parameters must be considered. These include the resolution and shape of the ion beam, as well as its physical separation, or dispersion, from other ion species. The current stability, the duration of continuous operation and the ease with which the dopant species can be changed, are also important.

Two types of specimen holders are used for this work. The first type consists of a carriage upon which a 12” diameter sample plate sits carrying up to fifteen 2” diameter wafers. This plate can be heated up to about 700°C by a resistive heater, with the temperature being monitored by a thermocouple connected directly to the sample plate. This holder is used for high temperature implants. The second type of sample holder is a carousel capable of holding up to eighteen 2” diameter wafers. This holder is used for most of the room temperature InP implants. Small samples are held in position on the sample plate using phosphor-bronze clips.

4.3 Plasma enhanced chemical vapour deposition

After implantation of Si dopants into InP, an annealing temperature of at least 700°C is required to activate the Si⁺ ions and to remove radiation damage. Since this annealing temperature is much higher than the congruent evaporation point of InP, several methods have been developed to prevent the surface of the InP wafer from degrading. These seek either to provide a group V overpressure or to encapsulate the surface. In our work, all doped wafers formed by silicon implantation are encapsulated with a layer of silicon nitride of thickness 500Å to prevent phosphorous loss from the surface. The thin dielectric layer is deposited using Plasmalab 80 Plus Plasma Enhanced Chemical Vapour Deposition (PECVD) (figure 4.3). The main advantage of this reactor is its low deposition temperature, which prevents diffusion of ions within the wafer. The impact of these energetic species also results in excellent adhesion of the film to the substrate. A smooth silicon nitride surface is usually obtained free from voids and pinholes.

Silane and ammonia gases decompose according to the following reaction:

$$3\text{SiH}_4 + 4\text{NH}_3 \rightarrow \text{Si}_3\text{N}_4 + 12\text{H}_2$$  \hspace{1cm} (4.2)
Nitrogen gas is normally used as a dilutant. Film composition and properties are controlled by the ratio of NH$_3$/SiH$_4$ in the gas stream. Deposition is normally conducted at a relatively low temperature (~300°C) because of the high electron temperature associated with the plasma.

Figure 4.3: Schematic diagram of DP80+, rf-powered at 13.56MHz.

4.4 Rapid thermal annealing

The process of ion implantation introduces damage to the lattice, which for high doses of heavy ion species results in a highly disordered material, which in the extreme can become amorphous. This damage can be removed by a post implantation heat treatment in which the damaged layer regrows by solid phase epitaxy onto the underlying single crystal substrate. There exist two types of annealing technique, which are rapid thermal annealing (RTA) and furnace annealing (FA). RTA technique is generally more suitable than conventional furnace annealing. The short annealing time results in much less dopant redistribution, and reduces layer mixing in multi-layer structures. But annealing of III-V semiconductors is complicated by the fact that the group V constituent tends to evaporate incongruently at elevated temperatures [75]. Even for short annealing times, it is necessary to employ a protection scheme to suppress the loss of the group V element. By using a controlled ambient or suitable encapsulant, and keeping processing times short, it is
Implant isolation of InP-based materials

Chapter Four: Experimental procedures

possible to reduce the effects of this problem [76]. In our work, both the front and back of all the wafers are protected with a layer of silicon nitride (Si₃N₄) before RTA process as discussed in section 4.3 above.

RTA can be divided into three classes: adiabatic, thermal flux, and isothermal annealing [77]. In adiabatic annealing, the heating time is so short (less than 10⁻⁷ s) that only a thin surface is affected. A high-energy laser pulse is used to melt the surface to a depth of less than 1μm, and the surface recrystallizes by liquid phase epitaxy with no memory of the previous damage. Dopant diffusion in the liquid state is very fast so the final profile is roughly rectangular. Thermal flux annealing occurs on time scales between 10⁻⁷ and 1s, where heating from one side of the wafer with a laser, electron beam, or flash lamp gives a temperature gradient across the wafer thickness. Almost complete electrical activation is obtained without diffusion but many point defects are formed due to rapid quenching from high temperature. In isothermal annealing, the heating process is longer than 1s. Rapid isothermal annealing uses tungsten-halogen lamps to heat wafers from one or both sides. In our work, rapid isothermal annealing is used since good activation can be obtained.

In general, to recover the lattice order and to enable the dopant atoms to find substitutional sites where they are electrically active, implanted InP needs to be heated to a temperature in the range of 700 to 800°C for relatively short times (say 10-100s). All the wafers are annealed using the 8 lamp rapid thermal annealer as shown in figure 4.4. This RTA was built in-house previously. The sample is placed on the silicon plate, which is inserted into the cylindrical quartz glass and backfilled with nitrogen gas. The annealer is programmed for the desired temperature cycle and runs for the period set. During the cooling process, the lamps are powered off and the sample is allowed to cool in a steady flow of nitrogen gas. The 8 halogen lamps are cooled using a combination of water and fans. The sample is only removed when the temperature reaches about 40°C. The measurement of sample temperature during RTA can have a large error (~15%). Both monitor and control thermocouple with the temperature controller ensure that the desired annealing temperature is reached within a few seconds with little overshoot. The control thermocouple is placed at the centre of the silicon plate as shown in figure 4.4.
The temperature calibration was carried out using a second thermocouple connected to a temperature meter. The second thermocouple was placed inside the chamber and the temperature values from the two thermocouples were recorded for different set temperatures. The temperature difference between the control thermocouple and the calibrated thermocouple is ~1%.

![Figure 4.4: 8 Lamps rapid thermal annealer.](image)

4.5 Photolithography process

Photolithography is one of the most important and most widely used tools in our study. It is used to define the Hall pattern. The technique used is contact printing and is capable of defining geometry near one micron. A positive photoresist is utilised for our purpose, namely, the AZ4330a resist. The first step of the photolithography process is to clean and bake the surface of the samples. A three stage cleaning is used where the samples are dipped consecutively into boiling methanol at 100°C, acetone and isopropanol. After a pre-bake for 30s, photoresist is then applied to the samples by spinning at 4000 rpm for 1 minute to achieve a uniform thickness of approximately 3.5μm. Next, the applied resist is soft-baked on a hotplate at 100°C for 1 minute to dry the resist and improve adhesion. After this, the resist is ready for alignment and exposure. In the case of AZ4330a positive resist, an exposure time of 3.6 seconds is needed. During development, the areas of photoresist exposed to ultraviolet (UV) light are dissolved revealing the desired pattern on
the samples [78]. The AZ400K developer diluted 1:3 with deionised water is used to dissolve those areas which have been exposed. They are then hard-baked for 30 minutes at 100°C before etching. A solution of hydrochloric acid (HCL) and phosphoric acid in the ratio of 1:1 by volume is used to etch the sample until the substrate is reached. The etch rate is approximately 1.4μm/min. For etching InGaAs layers, a solution of sulphuric acid (H\textsubscript{2}SO\textsubscript{4}), hydrogen peroxide (H\textsubscript{2}O\textsubscript{2}) and water (H\textsubscript{2}O) in the ratio of 3:1:1 is used. The latter is selective with respect to InP. For etching InGaAsP layers, a solution of sulphuric acid (H\textsubscript{2}SO\textsubscript{4}), hydrogen peroxide (H\textsubscript{2}O\textsubscript{2}) and water (H\textsubscript{2}O) in the ratio of 1:1:1 is used. The total etch depth is measured using a Rank Taylor Hobson Talystep, the error typically being 5%. The photoresist is then removed in acetone leaving the Hall pattern on the wafer. The photolithography process flow is summarised in figure 4.5.
Implant isolation of InP-based materials

Chapter Four: Experimental procedures

InP, InGaAs & InGaAsP samples

Three stage cleaning
Boil methanol, Acetone & IPA

Pre-bake
Hotplate 100\textdegree{}C, 30s

Spin photoresist (AZ4330a)
at 4000rpm for 1min

Softbake
Hotplate 100\textdegree{}C, 1min

Alignment and Exposure
(AZ4330a = 3.6s)

Development
AZ400K:H\textsubscript{2}O = 1:3

Hardbake
Hotplate 100\textdegree{}C, 30min

Etching of samples

Strip photoresist in acetone

Figure 4.5: Photolithography process flow.
4.6 Hall effect measurement

4.6.1 The Hall effect

The Hall effect has been extensively employed in the electrical studies of ion-implanted layers. The first papers reporting the application of the Hall effect to implant isolated layers appeared in the proceedings of the Grenoble conference on Ion Implantation \[79,80,81\]. When calculating the carrier concentration, \(N\) and mobility, \(\mu\) in thin layers from Hall effect and sheet resistance measurements, it is assumed that the following conditions are fulfilled:

i. The layer is homogenous and continuous over its whole area.

ii. The layer is electrically isolated from any conducting substrate.

iii. The magnetic field is uniform in intensity and normal to the plane of the layer.

iv. The contacts are small and do not disturb the electric field configuration.

Figure 4.6 shows the prototype structure for the Hall-effect experiment. It is assumed that the charge carriers are electrons and that they are all travelling with a velocity \(v\) in the \(x\) direction before application of a magnetic field, \(B\). Upon application of a magnetic field in the \(z\) direction, the electrons experience a force known as the Lorentz force in the \(y\) direction of magnitude \(qvB\), where \(q\) is the electronic charge. The charges build up on the \(+y\) face of the sample until an opposing force \(qE_y\) just balances the Lorentz force.

Thus,

\[
qvB = qE_y \\
E_y = \frac{V_H}{w} = vB
\]

where \(E_y\) is the electric field in the \(-y\) direction (unit is V/m)

\(V_H\) is the Hall voltage (unit is volt)

\(w\) is the width of sample (unit is m)

\(B\) is the magnitude of magnetic field (unit is tesla)
But the total current $I_x$ is

$$I_x = n q v (w.d) \tag{4.5}$$

where $n$ is the free carrier concentration (unit is atoms/m$^3$)

d is the thickness of sample (unit is m)

From equations (4.4) and (4.5)

$$\frac{V_H}{w} = B \left( \frac{I_x}{n q w d} \right) \tag{4.6}$$

$$\frac{R_H}{d} = \frac{V_H}{B I_s} = \frac{1}{n q} \tag{4.7}$$

where $R_H$ is the Hall coefficient

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure46.png}
\caption{Typical configuration for Hall effect measurements.}
\end{figure}
Since electron velocity varies linearly with the electric field in the x direction:

\[ \nu = \mu E_x \]  \hspace{1cm} (4.8)

\[ \mu = \frac{\nu}{E_x} = \frac{I_x}{n q A} \frac{L}{V} = \frac{1}{n q \rho} \]  \hspace{1cm} (4.9)

where \( \mu \) is the electron mobility (unit is m\(^2\)/Vs)
\( E_x \) is the electric field in the x direction (unit is V/m)
\( V \) is the voltage in the x direction (unit is volt)
\( L \) is the length of sample (unit is m)
\( A \) is the cross-sectional area of the sample (unit is m\(^2\))
\( \rho \) is the resistivity of sample (unit is \( \Omega \)m)

There are four different configurations for measuring the Hall effect, which are Hall bar and bridge, Van der Pauw configuration, non-peripheral square four point probe, and Corbino disc. In our work, sheet carrier concentrations, sheet electron mobilities and sheet resistance of the InP, InGaAs and InGaAsP Hall patterns are measured using the standard Van der Pauw technique in a Accent HL5500 system. This method requires that the contacts be at the corners of the sample, that the contacts be small, and that the sample be homogeneous in thickness and not perforated. One must accurately measure the magnetic field intensity, the electrical current, and the voltage.

The Van der Pauw configuration is as shown in figure 4.7. The subscripts A, B, C, and D refer to the four contacts on the Hall pattern. Sheet resistance and sheet Hall coefficient are used because the thickness of the conducting layer is often unknown. The voltage across contact A to C is measured with and without the magnetic field B when a constant current, I is flowing between contact B and D.

The sheet Hall coefficient, \( R_{HS} \) is estimated as follows [82]:

\[ R_{HS} = \frac{(V_{HB} - V_{HO})}{BI} \]  \hspace{1cm} (4.10)
where $I$ is the magnitude of applied current (unit is ampere)

$V_{HB}$ is the Hall voltage with magnetic field (unit is volt)

$V_{HO}$ is the voltage without magnetic field (unit is volt)

Figure 4.7: Electrical Contact Configuration for a sample.

A more accurate analysis of sheet carrier concentration is subsequently obtained by using [82]:

$$n_s = r q R_{HS}$$  \hspace{1cm} (4.11)

where $n_s$ is the sheet carrier concentration (unit is atoms/m$^2$)

$r$ is the Hall scattering factor equal to the ratio of Hall mobility, $\mu_H$ to drift mobility, $\mu_D$ and is normally assumed to be unity

The sheet resistance is given by [82]

$$R_s = \frac{\pi}{\ln 2} \left[ \frac{R_1 + R_2}{2} \right] F$$  \hspace{1cm} (4.12)

where $R_s$ is the sheet resistance of sample (unit is $\Omega/\square$).

$F$ is the correction factor and is normally unity for a symmetrical sample for which $R_1$ is equal to $R_2$. 

45
Implant isolation of InP-based materials

Chapter Four: Experimental procedures

\[
R_1 = \frac{\left| \frac{V_{AB}}{I_{CD}} + \frac{V_{BA}}{I_{DC}} \right|}{2} \tag{4.13}
\]

and

\[
R_2 = \frac{\left| \frac{V_{AD}}{I_{BC}} + \frac{V_{DA}}{I_{CB}} \right|}{2} \tag{4.14}
\]

\(I_{CD}\) is the current entering contact C and leaving contact D
\(V_{AB}\) is \(V_A - V_B\)

Similar naming convention is used for the rest of the parameters in equations 4.13 and 4.14

Thus Hall mobility, \(\mu_e\) is [82]

\[
\mu_e = \frac{1}{qR_s n_s} \tag{4.15}
\]

4.6.2 Resistivity measurement with temperature

The HL5550 liquid nitrogen cryostat (figure 4.8) is an option to the Accent HL5500 system. It enables resistivity and Hall measurements to be extended to temperatures from 90K to 500K. The complete system allows temperatures to be scanned through a predetermined range and results to be collected automatically for a number of steps specified by the user. The comprehensive data acquisition and Windows software allow the data to be collected for fixed linear and/or inverse temperature increments over the specified range. The activation energy \((E_a)\) can then be determined from the Arrhenius plot for sheet resistance using the following equation [83]:

\[
\rho_s = \rho_0 e^{\frac{E_a}{kT}} \tag{4.16}
\]

\[
\ln \rho_s = \left( -\frac{E_a}{1000k} \right) \frac{1000}{T} + \ln \rho_0 \tag{4.17}
\]

where \(\rho_s\) is the sheet resistance of the sample (unit is \(\Omega/\square\))
\[ \rho_0 \] is the sheet resistance of the sample at an infinite temperature (unit is \( \Omega/\square \))

\[ E_A \] is the activation energy (unit is eV)

\( T \) is the temperature of sample holder (unit is K)

\( k \) is the Boltzmann’s constant (unit is eV/K)

A plot of \( \ln (\rho_s) \) versus \( 1000/T \) is a straight line with a slope of \( [E_A/(1000k)] \). The activation energy can then be determined from the slope.

---

**Figure 4.8: Liquid Nitrogen Cryostat with lid removed.**

4.6.3 Sources of errors related to the measurement of Hall effect and sheet resistance

When Hall effect measurements are made a number of errors must be considered such as the Hall scattering factor, uncertainties in the current, magnetic field and voltage measurement [84].

**(a) Magnetic field measurement**

In general, the reproducibility of the magnetic field is good with a maximum error of about 1%. Thus error introduced by magnetic field measurement is very small.
(b) Electrical measurement
The current is supplied by a constant current source, which enables the desired current to be digitally selected. The main source of errors arises from the surface leakage and junction leakage current. Normally, poor surface conditions result in the presence of excessive leakage currents. For greater accuracy the current is reversed and measurements are repeated and averaged. Leakage currents must be small compared to the measurement current.

(c) Hall scattering factor
The uncertainty in the value of the Hall scattering factor can give rise to significant errors in the determination of the carrier concentration. In all measurements, the scattering factor has been assumed to be unity. The actual value of the scattering factor depends on the impurity concentration and the dominant scattering mechanisms present in the sample [85]. According to Stillman et al [86], the error in the assumption that the scattering factor is unity is less than 15% at room temperature and about 5% at 77K. However, the assumption that the scattering factor is unity should not lead to large error when the profiles of similar concentration are compared like the samples used in this study.

4.7 Secondary Ion Mass Spectroscopy (SIMS) technique
SIMS is the mass spectrometry of ionised particles which are emitted when a surface, usually a solid, is bombarded by energetic primary particles. The interaction of a few keV ions with the surface of the target produces monatomic and polyatomic particles of sample material along with electrons and photons. The secondary particles usually carry negative, positive, and neutral charges and they have kinetic energies that range from zero to several hundred eV. It is the secondary ions which are detected and analysed by a mass spectrometer. The primary beam species which are commonly used in SIMS, are Cs⁺, O₂⁺, and O⁻ at energies between 1 and 30keV.

Different information about the sample can be obtained using this technique such as:
(a) Depth profiling (Determination of elemental concentrations of dopant and impurity atoms within a sample as a function of depth).
(b) 2D or 3D imaging of a particular element in the sample.
(c) Mass spectra of the sample (Detection of different atomic and molecular ions present in the sample by scanning a range of mass-to-charge ratios).

(d) Isotope ratio measurements.

All our samples was analysed using the CAMECA IMS 6F at the Institute of Physics Polish Academy of Sciences in Warsaw, Poland. Figure 4.9 shows a schematic diagram of the SIMS system. It has a duoplasmatron source capable of producing O$_2^+$ or O$^-$ ions, and a microbeam source for producing Cs$^+$ ions. Both sources offer premium beam stability, as well as ultra-fine minimum beam size (300nm for O$_2^+$ and 200nm for Cs$^+$). The primary ions pass through the primary beam mass filter (PBMF) to avoid implantation of impurity atoms into the sample. The primary beam line contains lenses and deflection plates for focusing and steering the beam and raster scan plates for scanning it. Secondary ions generated are extracted using an adjustable secondary ion extraction which can be biased from -10kV to +10kV. The electrostatic spherical analyser (ESA) enables the energy filtration of secondary ions and the magnetic analyser allows the selection of different ion masses.

Ultra-high vacuum condition is maintained in the sample chamber to reduce the effect of residual gases. These can cause a dramatic increase in the background counts such that a signal arising from the dopant in the sample cannot be detected to fall to the levels required.

A 12.5keV O$_2^+$ primary beam is rastered scan over a square area of 150µm x 150µm over a period of time to obtain the Fe depth profile in our samples. The analysed area has a diameter of 50µm. The conversion of SIMS depth profile from raw data (count-rates as a function of time) to quantified data (concentrations as a function of depth) involves the use of ion-implanted standards and carter depth measurements. The depth scale is obtained by multiplying the sputter rate of the sample with time. It is assumed that the sample has been sputtered at a uniform rate. To quantify the concentration scale it is necessary to know the relationship between the secondary ion count rate and the dopant concentration.
The relative sensitivity factor (RSF) converts the vertical axis from ion counts into concentration and is defined by the following equation [87]:

$$\frac{I_R}{C_R} = RSF \frac{I_E}{C_E}$$  \hspace{1cm} (4.18)

$$C_E = RSF \frac{I_E C_R}{I_R}$$  \hspace{1cm} (4.19)

where $I_R$ is the secondary ion intensity of reference element, in our case, indium is used as the reference element (unit is ion counts/s)

$I_E$ is the secondary ion intensity of the element (unit is ion counts/s)
RSF_E is the relative sensitivity factor for the element
C_E is the atomic concentration of the element (unit is atoms/cm^3)
C_R is the atomic concentration of the reference element (unit is atoms/cm^3)

4.8 Rutherford Backscattering Spectrometry (RBS) Technique

Rutherford Backscattering Spectrometry involves measuring the number and energy of ions in a beam which backscatter after colliding with atoms in the near-surface region of a sample at which the beam has been targeted. With this information, it is possible to determine the atomic mass and elemental concentrations versus depth below the surface. The combination with channelling effect allows the determination of the lattice damage if the analysis is performed in a single crystal substrate. Thus we can verify whether a sample is crystalline, poly-crystalline or amorphous from RBS channelling experiment. When a sample is channelled, the rows of atoms in the lattice are aligned parallel to the incident ion beam. The bombarding ion will backscatter from the first few monolayers of material at the same rate as a non-aligned sample, but backscattering from buried atoms in the lattice will be drastically reduced since these atoms are shielded from the incident atoms by the atoms in the surface layers. Channelled particles can interact with atoms on non-substitutional lattice sites such as interstitials in regular or random positions and oversized substitutional atoms. Thus each type of defect present in a single crystal is expected to increase the backscattering yield above the aligned yield of a virgin crystal. Most information comes from a comparison of the backscattering spectra from the host with those containing the impurity atoms.

In our work, channelling experiment is done using a 1.5MeV He^+ from a 2MV Van de Graaff accelerator. Tesmer et al [88] have given several reasons for using ^4He^+ in RBS; energy loss data for He^+ are better known than for other ions; silicon surface barrier detectors have a good energy resolution for ^4He^+ at about 12keV. The backscattered ions are detected with a solid-state surface barrier detector located at 145° from the normal beam direction. The detector is position at this angle where the yield is the lowest during channelling measurement. All RBS measurements presented in the work are done with the help of Dr. Chris Jeynes.
Chapter Five

5. Implant isolation using protons and helium

Results and discussions

5.1 Implant isolation of n-type InP and InGaAs using hydrogen ions

This experiment investigates the formation of a high resistance region in n-type InP using hydrogen ions at two different substrate temperatures. The effects of elevated temperature as compared to RT implants are studied in terms of the annealing characteristics of the sheet resistance, sheet carrier concentration and sheet mobility. The n-type InP layer is grown on SI InP substrates with the (100) axis 2° off normal orientation using a Gas Source Molecular Beam Epitaxy (GSMBE) reactor at the University of Sheffield. The InGaAs layer is grown lattice-matched on InP using an indium composition of 0.53. The samples are 1μm thick and doped to a carrier concentration of ~1.0x10^{17} cm^{-3}, using silicon as the dopant. After Hall patterns are formed on the samples, they are irradiated with \( ^1 \text{H}^+ \) at 250keV to doses in the range of \( 1x10^{12} - 4x10^{15} \) cm\(^2\), with an ion current density <0.5μA/cm\(^2\). The samples are divided into two groups for implant isolation at temperatures of 25°C and 200°C using a 2MV HVEE implanter. All the implantations are performed with the sample surface tilted by 7° to the surface normal, representing the beam incidence direction, to minimize the ion channelling effect.

5.1.1 Dose dependence at RT and 200°C

TRIM simulation is performed to calculate range statistics of \( ^1 \text{H}^+ \) ions in both InP and InGaAs. The damage distributions resulting from the proton implant in InP and InGaAs are plotted in figure 5.1. The projected range of protons in both materials is approximately 2μm. An approximately uniform damage concentration is placed in the doped layer (figure 5.1) which is achieved by using a proton energy of 250keV. From figure 5.1, the defect concentration in both InP and InGaAs is about the same magnitude. Using protons at an energy of 250keV, the isolation behaviour is mainly caused by the relatively low concentration of nuclear stopping defects and defects caused via electronic stopping. The end-of-range disorder is buried well in the substrate and removed far from the actual device active region.
Figure 5.1: Damage distribution resulting from 250keV proton implants into InP and InGaAs as determined by TRIM.

Figure 5.2 shows the evolution of the sheet resistance as a function of dose for Si-doped InP material. For low doses below $4 \times 10^{12}$ cm$^{-2}$, the sheet resistance is of the same order of magnitude as the initial sheet resistance (see table 5.1) for the Si-doped InP epilayers before implant isolation. Thus electrical isolation of this n-type ($\sim 10^{17}$ cm$^{-3}$) InP layers is not possible using low dose implants ($< 4 \times 10^{12}$ cm$^{-2}$). As can be seen from figure 5.2, there is a gradual increase in the sheet resistance with dose for both RT and 200°C implants. $R_s$ reaches a maximum value ($\sim 10^6$ $\Omega$/$\square$) at a threshold dose of $4 \times 10^{14}$ cm$^{-2}$ for RT implants. In the case of 200°C implant, a lower maximum sheet resistance ($\sim 5 \times 10^3$ $\Omega$/$\square$) is reached at a threshold dose of $4 \times 10^{13}$ cm$^{-2}$. We infer that this behaviour is due to annealing of antisite defects (In$_p$ or In$_n$, related acceptor-like defects), which are stable up to an annealing temperature of 200°C [37]. Hall measurements reveal that $R_s$ increases due to both mobility degradation and reduction in sheet carrier concentration. Further dose accumulation leads to the formation of a plateau, which is stable up to a dose of $1 \times 10^{15}$ cm$^{-2}$. The plateau ends when the sheet resistance measured might be from the doped layer and the substrate. The latter might become conductive at a high implantation dose of $4 \times 10^{15}$ cm$^{-2}$. 
cm$^{-2}$ as observed by Woodhouse et al [39]. They reported a decrease in the sheet resistance of the SI InP substrate to $\sim 10^5 \Omega/\square$ above a dose of $1 \times 10^{15}$ cm$^{-2}$. The decrease in the sheet resistance above a dose of $1 \times 10^{15}$ cm$^{-2}$ in our case, may be due to the substrate itself, which becomes quite conductive (section 5.3.1).

Figure 5.2: Effect of dose on sheet resistance for 250keV proton irradiation in n-type InP layers at RT and 200°C.

Donnelly et al [35] reported a maximum resistivity of only $\sim 10^3 \Omega cm$ after proton bombardment in n-type InP. On the other hand, a maximum sheet resistance of $\sim 10^5 \Omega/\square$ for 600keV irradiation of H$^+$ at a dose of $3 \times 10^{14}$ cm$^{-2}$ was obtained by Boudinov et al [38]. They also reported a similar thermally stable post-implant annealing window up to 200°C for Si-doped InP using proton implants at a dose of $3 \times 10^{14}$ cm$^{-2}$ at 600keV. Thompson et al [40] and Focht et al [45] reported that the resistivity of n-type InP can only be increased by ion bombardment to a value in the range $10^3 - 10^4 \Omega cm$. 

54
Implant isolation of InP-based materials

Chapter Five: Implant isolation using protons and helium

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sheet resistance, $R_s$ ($\Omega/\square$)</th>
<th>Hall mobility, $\mu_{\text{eff}}$ ($\text{cm}^2\text{V}^{-1}\text{s}^{-1}$)</th>
<th>Sheet carrier concentration, $n_s$ ($\text{cm}^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-type InP</td>
<td>421</td>
<td>3200</td>
<td>5.00x10^{12}</td>
</tr>
<tr>
<td>n-type InGaAs</td>
<td>80.5</td>
<td>7600</td>
<td>1.02x10^{13}</td>
</tr>
</tbody>
</table>

Table 5.1: Initial sheet resistance, Hall mobility and sheet carrier concentration of the InP and InGaAs samples before proton implantation.

Curve 1(a) and 1(b) in figure 5.3 shows the effect of dose on the sheet resistance for 250keV proton irradiation on an n-type InGaAs epilayer at RT and 200°C respectively. For the RT implant, there is a gradual increase in sheet resistance from $\sim 10^2 \ \Omega/\square$ to $1.8x10^4 \ \Omega/\square$ with doses ranging from $10^{12} \ \text{cm}^2$ to $4x10^{15} \ \text{cm}^2$. The increase of sheet resistance occurs as a consequence of a progressive removal of the carriers and mobility degradation. The trend is quite similar to the case of InP. A maximum sheet resistance of $1.8x10^4 \ \Omega/\square$ is obtained at a dose of $4x10^{15} \ \text{cm}^2$ for the RT implant. For the 200°C implant, a maximum sheet resistance of $4x10^3 \ \Omega/\square$ is measured at a threshold dose of $4x10^{14} \ \text{cm}^2$. The value of sheet resistance is very similar for both RT and 200°C implants up to an ion dose of $4x10^{14} \ \text{cm}^2$.

Curves 2(a) and 2(b) in figure 5.3 present the sheet carrier concentration as a function of dose. The decrease of the sheet carrier concentration for both RT and 200°C implants with the accumulation of the irradiation dose (damage) is clear evidence of carrier removal via capture at the trapping centres. The Hall mobility reduces as a consequence of the ion irradiation. It varies from 7470 to 433 cm$^2$/V.s and from 6890 to 675 cm$^2$/V.s for RT and 200°C implant respectively after a dose of $4x10^{15} \ \text{cm}^2$ has been accumulated.
5.1.2 Effect of post-implant annealing temperature

The InP samples, which are implanted at RT, are then annealed at 100°C for 60s. We observed that the sheet resistance curve after annealing the samples is quite similar to that of the as-implanted samples as shown in figure 5.4. When the as-implanted samples are annealed at a post implant temperature of 200°C, the sheet resistance values at different doses decrease and are of the same order of magnitude as that of 200°C implant. Thus electrical isolation is stable up to a temperature of at least 100°C. This behaviour is consistent with the notion that antisite defects are the major cause for the electron trapping for this isolation scheme. For an annealing temperature of 350°C, the sheet resistance decreases further for both RT and 200°C implant for doses above $4\times10^{13}$ cm$^{-2}$. Since the In$_p$ antisite defects are double acceptors, their annealing would result in the release of the captured electrons and hence a decrease in the sheet resistance as observed. We infer that the In$_p$ antisite defects are annealed at temperatures of 200-300°C via recombination with
Implant isolation of InP-based materials
Chapter Five: Implant isolation using protons and helium

Indium vacancies. Similarly, De Souza et al [89] reported GaAs antisite defects as carrier trap centres in the case of GaAs irradiated by protons and helium ions.

The data obtained suggest that the implant temperature is an insensitive parameter for this particular isolation scheme in n-type InP. Antisite defects and/or their related defect complexes created by replacement collisions are suspected to be the major carrier trapping centres due to their low sensitivity to dynamic annealing during hot implants. Most of the defects, which are created during implantation, are annealed out at implant temperature of $200^\circ$C. This is in contrast to the situation with n-type GaAs for which the substrate temperature is established as an important and sensitive parameter that controls the degree of isolation and its stability [90]. Thus unlike GaAs increasing the substrate temperature does not improve the electrical isolation at different doses for n-type InP layers.

![Figure 5.4: Evolution of sheet resistance as a function of dose for 250keV proton irradiation in n-type InP layers at RT and 200°C and annealed at 100°C, 200°C and 350°C.](image)
For doses in the range of $1 \times 10^{12} - 5 \times 10^{13}$ cm$^2$ in n-type InGaAs, the sheet resistance is about the same order of magnitude for both RT and 200°C implants even after an annealing temperature up to 350°C (figure 5.5). After annealing at 100°C, the sheet resistance value follows the same curve as RT implants for all the different doses. However, annealing at 350°C causes the isolation value to drop by one order of magnitude as compared to that measured at RT for doses above $1 \times 10^{14}$ cm$^2$. For a dose in the range of $1 \times 10^{14} - 4 \times 10^{14}$ cm$^2$, a thermally stable annealing window up to 200°C is obtained. Above a dose of $4 \times 10^{14}$ cm$^2$, the electrical isolation is thermally stable up to 100°C.

Steeples et al [121] obtained a maximum sheet resistance of $8 \times 10^4$ Ω/□ in n-type In$_{0.53}$Ga$_{0.47}$As using a post-implant annealing temperature of 200°C. Multiple energy protons of [4$\times 10^{14}$ cm$^2$ (400keV+300keV+200keV+100keV)] were used on Ge-doped (2$\times 10^{17}$ cm$^{-3}$) InGaAs epilayer of thickness 0.87μm. They also reported a thermally stable annealing window up to 200°C. Pearton et al [41] used multiple energy protons on n$^+$ In$_{0.53}$Ga$_{0.47}$As. They obtained an optimum isolation of $\sim 10^2$ Ω/□. Their annealing behaviour was similar to that reported by Steeples et al [121].

![Figure 5.5](image.png)

**Figure 5.5:** Evolution of sheet resistance as a function of dose for 250keV proton irradiation in n-type InGaAs layers at RT and 200°C and annealed at 100°C, 200°C and 350°C.
5.2 Implant isolation of n-type InP and InGaAs using helium ions

In this experiment, the effect of helium dose, substrate temperature, damage accumulation and post-implant annealing temperature in n-type InP and InGaAs are studied. N-type InP samples with clover-leaf pattern are divided into four groups. Group I and II samples are implanted with 55keV and 600keV He⁺ respectively at RT, 100°C and 200°C with an ion dose of $2 \times 10^{14}$ cm⁻². A beam current density of less than 0.12 μA/cm² is used. The post-implant annealing is performed in the range of 100°C-800°C for a time of 60s in a nitrogen atmosphere. Samples which are annealed above 500°C, are capped with Si₃N₄ prior to the anneal. The latter acts as a barrier to out diffusion of either the host atoms or the implanted impurities. Group III and IV samples are irradiated with 55keV and 600keV He⁺ respectively at RT with doses in the range of $5 \times 10^{11} - 1 \times 10^{15}$ cm⁻².

The n-type region is formed by Si implantation in semi-insulating (SI) InP. The implant conditions are given in table 5.2. In our work, TRIM is used to calculate range statistics of Si⁺ and He⁺ ions in InP [20]. Figure 5.6 shows the atomic distributions of the n-type dopant created by multiple low energy implantation. The different doses and energies are chosen so as to obtain a uniform flat doping profile of approximately 0.6μm thick. A post-implant annealing temperature of 700°C for 60s is used to activate the n-type dopants. An activation of ~16% was obtained with an initial sheet carrier concentration of $7 \times 10^{12}$ cm⁻². The initial sheet resistance was 810 Ω/□. As shown in figure 5.6, the energy of the helium beam is chosen to place the damage peak beyond the doped layer so that the defect concentration in the doped layer is approximately uniform with depth.

<table>
<thead>
<tr>
<th>Dose (cm⁻²)</th>
<th>Energy (keV)</th>
<th>Projected range from TRIM calculation (Å)</th>
<th>Longitudinal Straggling from TRIM calculation (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$2.7 \times 10^{12}$</td>
<td>26</td>
<td>330</td>
<td>245</td>
</tr>
<tr>
<td>$1.1 \times 10^{13}$</td>
<td>110</td>
<td>1282</td>
<td>781</td>
</tr>
<tr>
<td>$3.0 \times 10^{13}$</td>
<td>300</td>
<td>3566</td>
<td>1729</td>
</tr>
</tbody>
</table>

Table 5.2: Si⁺ implant conditions to create a uniform doping concentration of thickness ~0.6μm into InP.
Figure 5.6: The relative position of the atomic distributions of the n-type dopant, the helium implant and the damage resulting from helium ions, as determined by TRIM.

5.2.1 Effect of substrate and post-implant annealing temperature

Figure 5.7 shows the sheet resistance as a function of post-implant annealing temperature for a Si-doped InP material after 600keV implantation with helium ions. There are three curves showing the evolution of sheet resistance with annealing temperatures corresponding to the three different substrate temperatures of RT, 100°C and 200°C respectively. The initial sheet resistance of the Si-doped InP layer before implant isolation is as expected 810 \( \Omega/\square \) (on average). The initial Hall mobility and sheet carrier concentration varies from 2150 to 2180 cm²/Vs and from 6.73x10¹² to 7.65x10¹² cm⁻² respectively. After helium implantation, a sheet resistance of 3x10⁶ \( \Omega/\square \) is obtained for RT implant and 7x10⁵ \( \Omega/\square \) for 100°C and 200°C implants, as shown in figure 5.7. Thus there is an increase in the sheet resistance of about four orders of magnitude. Most of the free carriers are trapped at the defect sites. Carrier trapping at defects created by the irradiation and the carrier mobility degradation by the damage cause the change of sheet resistance. Annealing of the RT samples at 400°C results in a maximum sheet resistance of 10⁷ \( \Omega/\square \).
This is the first time such a high sheet resistance value is reported for isolating n-type InP using helium implantation. Sargunas et al [46] reported a maximum sheet resistance of only $\sim 10^6 \, \Omega/\square$ for RT implantation into n-type InP using a dose and energy of $5 \times 10^{14} \, \text{cm}^{-2}$ and 55keV respectively. Akano et al [54] reported an even lower average sheet resistance of $\sim 10^4 \, \Omega/\square$ for $^4\text{He}^+$ using multiple doses and energies of $5 \times 10^{13}$ & $1 \times 10^{14} \, \text{cm}^{-2}$ and 60keV & 200 keV respectively.

![Graph showing the evolution of sheet resistance with different post-implant annealing temperatures](image)

**Figure 5.7:** Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in a nitrogen atmosphere for 600keV helium implanted n-type InP layers irradiated with $2 \times 10^{14} \, \text{cm}^{-2}$ at RT, 100°C and 200°C.

The data obtained here suggest that the implant temperature is not a very sensitive parameter for this particular isolation scheme in n-type InP. This is similar to the proton implantation results from section 5.1. Defects which are created, are thermally stable up to 500°C. We infer that for this particular dose and energy, the isolation resistance is at its threshold. This provides optimum isolation and quite a broad thermally stable region due to
the formation of such defects during implantation, which are stable to higher annealing temperatures. Thus increasing the substrate temperature during implantation does not provide further improvement in electrical isolation under this scheme. Other authors [45,46] have reported optimum isolation ($<10^5 \Omega/\square$) at a similar dose of $\sim10^{14}$ cm$^{-2}$.

As can be seen in figure 5.7, the high sheet resistance ($10^6$–$10^7 \Omega/\square$) is maintained until an annealing temperature of 500$^\circ$C for all three substrate temperatures. Such a wide annealing window is quite useful from the technological point of view. These results for helium implantation are quite different from those obtained using protons (section 5.1.2) which are similar to those of Boudinov. As shown in figure 5.8, higher sheet resistance and better thermal stability is obtained in n-type InP samples implanted with helium compared with protons. As discussed in section 5.1.2, the defects responsible for trapping of carriers in proton implanted material are less thermally stable than those produced using helium ions and hence the poor thermal stability observed. Boudinov et al [37] reported similar smaller annealing window up to 200$^\circ$C for Si-doped InP using proton implants with a dose of $3\times10^{14}$ cm$^{-2}$ at 600keV.

Maximum sheet resistivities of $1\times10^7$, and $9.8\times10^6 \Omega/\square$ are obtained at an annealing temperature of 400$^\circ$C for RT and 500$^\circ$C for both 100$^\circ$C and 200$^\circ$C substrate temperature respectively. With continued annealing, there is a gradual decrease in sheet resistance due to recovery of the carriers as the defects are annealed out. A complete restoration of the initial sheet resistance of about 810 $\Omega/\square$ is obtained after annealing at 750$^\circ$C for all the three cases. This implies that most of the defects are annealed out at temperatures of 600 - 750$^\circ$C which causes an increase in sheet carrier concentration as shown in figure 5.9.
Figure 5.8: Evolution of sheet resistance as a function of post-implant annealing temperature for 250keV proton and 600keV helium implanted n-type InP layers irradiated with 5x10^{14} cm^{-2} and 2x10^{14} cm^{-2} respectively at RT.

Figure 5.9 shows the variation of sheet electron mobility and sheet carrier concentration at different post-implant annealing temperatures. The sheet electron mobility and sheet carrier concentration have values in the range of 200 to 400 cm^{2}/Vs and 2x10^{9} to 5.8x10^{9} cm^{-2} respectively for annealing temperature from 100 to 550°C for all three substrate temperatures. These low values of electron mobility and sheet carrier concentration are due to carriers being trapped at defect sites, leading to a high sheet resistance. For annealing temperatures above 550°C, we see that the mobility and sheet carrier concentration recover until they reach a point where they are comparable to values measured for the pre-isolation implanted substrate. This is due to the fact that the background of isolating defects is very much reduced. All three mobility and sheet concentration curves behave in a similar fashion. Post-implant annealing seems to be an important parameter as compared to elevated temperature implants in this case.
Implant isolation of InP-based materials
Chapter Five: Implant isolation using protons and helium

Figure 5.9: Variation of sheet carrier concentration and sheet electron mobility with annealing temperature for 600keV helium implantation using a dose of $2 \times 10^{14}$ cm$^{-2}$ at RT, 100°C and 200°C.

The post-implant annealing behaviour of a 55keV implant is also investigated at a helium dose $2 \times 10^{14}$ cm$^{-2}$ (Figure 5.10) for the three different substrate temperatures (RT, 100°C and 200°C). The maximum sheet resistance is $\sim 10^7 \Omega/\square$ for all three implantation temperatures. Thus the isolation does not show any improvement for implantation temperatures above RT.

Similar to 600keV isolation schemes, a wide and thermally stable isolation is obtained until a post-implant annealing temperature of 500°C is reached for all three substrate temperatures which represents a wide process window for the device engineer. The sheet resistance is observed to decrease rapidly in both cases above an annealing temperature of 500°C and the initial carriers are recovered at 700°C - 800°C. This implies that most of the defects are annealed out at temperatures of 550°C - 700°C. Figure 5.11 shows the influence of post-implantation annealing on the sheet resistance for 55keV and 600keV implants. It is seen that samples implanted at 55keV and 600keV show comparable sheet resistances for annealing temperatures up to 800°C, although there are some differences in the detail.
Figure 5.10: Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in a nitrogen atmosphere for 55keV helium implanted n-type InP layers irradiated with $2 \times 10^{14}$ cm$^{-2}$ at RT, 100$^\circ$C and 200$^\circ$C.

Figure 5.11: Evolution of sheet resistance with different post-implant annealing temperature for $n^+$ samples implanted with $2 \times 10^{14}$ cm$^{-2}$ helium ions at 55keV and 600keV at RT.
5.2.2 Effect of damage accumulation

An energy of either 55keV or 600keV is used to place the peak of the damage either in the n' doped layer or deep inside the substrate, respectively. The damage profile as calculated by TRIM for the 55keV and 600keV isolation schemes is shown in figure 5.12. The vacancy concentration formed by 55keV helium implantation is about twelve times larger than that formed at 600keV. A constant level of damage within the doped surface region is formed only in the case of 600keV implants. The damage created within the n' doped region in the case of the 55keV isolation scheme has a Gaussian like distribution as shown in figure 5.12.

![Damage distribution profile for 55keV and 600keV He⁺ implants in InP.](image)

Figure 5.12: Damage distribution profile for 55keV and 600keV He⁺ implants in InP.

Figure 5.13 shows the variation of sheet resistance as a function of dose for samples isolated with 55keV and 600keV helium implants at RT. For low doses below $1 \times 10^{12}$ cm$^{-2}$, the sheet resistance is of the same order of magnitude as the initial sheet resistance before implant isolation for both isolation schemes. Thus electrical isolation in n-type ($\sim 10^{17}$ cm$^{-3}$) InP layers is not effective for low doses ($< 1 \times 10^{12}$ cm$^{-2}$). As can be seen from figure 5.13, there is a gradual increase in the sheet resistance with dose for both implant conditions.
The peak sheet resistance of $\sim 1 \times 10^7 \, \Omega/\square$ and $\sim 3 \times 10^6 \, \Omega/\square$ for 55keV and 600keV implants respectively occurs at a threshold dose of $2 \times 10^{14} \, \text{cm}^2$. The sheet resistance for the 55keV implant is about three times higher than that for 600keV implant at the threshold dose. It is also observed that the sheet resistance for 55keV implants is higher than for the 600keV implants for helium doses in the range of $5 \times 10^{11} - 2 \times 10^{14} \, \text{cm}^2$. We believe that this is due to greater damage accumulation inside the doped layer as shown in figure 5.12. Beyond the threshold dose the sheet resistance decreases to $\sim 1 \times 10^6 \, \Omega/\square$ in both cases for a dose of $1 \times 10^{15} \, \text{cm}^2$. A detailed explanation of this behaviour is discussed in section 5.3.

![Sheet resistance of n' InP irradiated with 55keV and 600keV He+ as a function of ion dose at RT.](image)

**Figure 5.13:** Sheet resistance of n' InP irradiated with 55keV and 600keV He+ as a function of ion dose at RT.

Figure 5.14 shows the evolution of sheet electron mobility and sheet carrier concentration at different doses for the two isolation schemes. There is a gradual decrease in the sheet electron mobility and sheet carrier concentration for both schemes with increasing dose. At a dose of $2 \times 10^{14} \, \text{cm}^2$ where maximum sheet resistance is obtained, the sheet carrier concentration is at its minimum, $\sim 3.5 \times 10^9 \, \text{cm}^2$ and $\sim 1 \times 10^{10} \, \text{cm}^2$ for 55keV and 600keV
Implant isolation of InP-based materials
Chapter Five: Implant isolation using protons and helium

Implants respectively. These low values of electron mobility and sheet carrier concentration are due to carriers being trapped at defect sites, leading to a high sheet resistance. Also observed in figure 5.14 is that above the threshold dose, the sheet carrier density increases and this may be due to parallel conduction from the substrate itself. The latter becomes quite conductive at such a high helium dose of $1 \times 10^{15}$ cm$^{-2}$ (section 5.3), and hence the sheet carrier concentration at the highest dose may be from a combination of the doped layer and the substrate (see model developed in section 5.3.2).

![Figure 5.14: Evolution of sheet carrier concentration and sheet electron mobility of n' InP irradiated with 55keV and 600keV He$^+$ as a function of ion dose at RT.](image)

5.2.3 Effect of dose on implant isolation of n-type InGaAs using helium ions
Helium ions were implanted at doses in the range of $1 \times 10^{14}$ - $2 \times 10^{16}$ cm$^{-2}$ into n-type InGaAs epilayers at two substrate temperatures: room temperature (RT) and 200°C. An energy of 600keV was used to create an approximately uniform damage concentration throughout the doped layer and the peak of the damage is buried well in the InP substrate. The Si-doped InGaAs epilayers of thickness 0.4μm were grown on semi-insulating InP substrate of <100> orientation using Metalorganic Chemical Vapour Deposition
Implant isolation of InP-based materials

Chapter Five: Implant isolation using protons and helium

(MOCVD). The InGaAs epilayer was doped to a carrier concentration of 2.0x10^{19} \text{ cm}^{-3}. An undoped InP buffer layer ~0.2\mu m separated the InGaAs from the Si substrate. The initial sheet resistance, sheet carrier concentration and sheet electron mobility are 8.76 \Omega/\square, 2.35x10^{14} \text{ cm}^{-2}, and 3030 \text{ cm}^2/\text{Vs} respectively. The measured sheet carrier concentration is approximately four times smaller than the expected value of 8x10^{14} \text{ cm}^{-2} (2.0x10^{19} \text{ cm}^{-2} \times 0.4\mu m). This discrepancy may be due to a thinner layer being grown instead of 0.4\mu m or the InGaAs layer was doped at a lower carrier concentration. The samples were supplied by University of Manchester Institute of Science and Technology (UMIST).

The variation of the sheet resistance of the helium bombarded n+ InGaAs epilayers with dose for two different substrate temperatures is shown in figure 5.15. After implantation at RT and 200°C for the lowest dose, the samples exhibit an increase in the sheet resistance by two orders of magnitude as compared to the pre-isolated values (8.76 \Omega/\square). As can be seen from figure 5.15, there is a gradual increase in sheet resistance with dose for both RT and 200°C substrate temperature. Hall measurements reveal mobility degradation and a reduction in the sheet carrier concentration. Hence the damage created, traps the carriers at defect sites. For a helium dose in the range of 2x10^{14} - 1x10^{15} \text{ cm}^{-2}, the sheet resistance of the RT implant is higher than that of the 200°C implant by a factor of two. Above a helium dose of 1x10^{15} \text{ cm}^{-2}, the 200°C implant produced a higher sheet resistance than the RT implant. R_s reaches an optimum isolation value of ~7x10^4 \Omega/\square at a threshold dose of 2x10^{15} \text{ cm}^{-2} for the RT implant and ~2x10^5 \Omega/\square at a threshold dose of 5x10^{15} \text{ cm}^{-2} for the 200°C implant. The RT and 200°C implanted samples which were bombarded at a dose of 1x10^{16} \text{ cm}^{-2} and 3x10^{16} \text{ cm}^{-2} respectively, were annealed at 350°C for 60s. The sheet resistance increases to 1.3x10^5 \Omega/\square and 2x10^5 \Omega/\square for the RT and the 200°C implant respectively.

We infer that above a dose of 1x10^{15} \text{ cm}^{-2}, the concentration of defects is much higher than the carrier concentration. Hence there is a high probability of hopping conduction occurring so that the trapped carriers can hop from one defect site to another and no increase in the sheet resistance is observed for RT implants. The data also suggests that parallel conduction from the substrate itself is not responsible for the lower sheet resistance for RT implants (see section 5.3.2). In the case of 200°C implants, the as-implanted sheet
resistance values are at least two times higher than RT implants as there is most probably in-situ dynamic annealing of the defects. Hence during 200°C implants, we believe that most of the excess defects are annealed out.

We infer that the increase in the sheet resistance of the RT implant to the same order of magnitude as that of the 200°C implant after annealing at 350°C is due to further reduction of the excess trap concentration. Thus the choice of the correct implant conditions is vital for device isolation.

Figure 5.15: Evolution of sheet resistance as a function of dose for 600keV helium irradiation in n+ InGaAs layers at RT, and 200°C.

5.3 Helium implantation into semi-insulating InP at RT and 200°C
The effect of helium ion implantation into SI InP at RT and 200°C is studied for doses from 4x10^{12} to 1x10^{16} cm^{-2} at 600keV. The electrical data is then compared with those where similar isolation implants were done in n-type InP and InGaAs. From this
experiment, we are able to check whether the electrical data measured in section 5.2 is correct and reliable. No study of the effect of helium ions on the InP substrate itself has been made before. When the peak of the damage due to helium ions is placed into the SI InP substrate, the operation of the devices employing this material may be affected. The effects exhibited by elevated temperature implants as compared to RT implants are also studied. The post-implant annealing properties of SI InP samples irradiated with helium ions are also investigated.

SI InP wafers were cleaved to obtained samples of size 10mm x 10mm. The samples were cleaned in organic solvents. The clover-leaf pattern was printed on the samples using optical lithography (see section 4.5). The samples were then divided into two groups for implant isolation at temperatures of 25°C, and 200°C using a 2MV High Voltage Engineering Europa (HVEE) implanter. The accuracy in the temperature control was ±3°C. All the implantations were performed with the sample surface normal tilted by 7° with respect to the beam incidence direction to minimize ion channelling.

5.3.1 Effect of dose

Figure 5.16 shows the variation of sheet resistance as a function of helium dose. There are two curves corresponding to the two different substrate temperatures of RT and 200°C respectively. The initial sheet resistance, sheet carrier concentration and sheet mobility of the SI InP substrate before implant isolation are as expected 4x10^8 Ω/□, 1.1x10^7 cm^2 and 1300 cm^2/V.s respectively [91]. After helium implantation, there is a decrease in the sheet resistance by at least an order of magnitude for the RT implant. As the helium dose increases from 1x10^{14} to 1x10^{16} cm^2, the sheet resistance decreases gradually from 1.5x10^7 Ω/□ to 5.5x10^5 Ω/□ respectively. The sheet carrier concentration increases from 1.6x10^9 to 2.2x10^{11} cm^2. Hence the SI InP becomes appreciably conductive at a dose of 1x10^{16} cm^2.

For the 200°C implant, the sheet resistance drops by four times the original value (4x10^8 Ω/□) after helium implantation at different doses.

Minimum sheet resistance of 5x10^7 Ω/□ is obtained at a dose of 1x10^{16} cm^2. The latter is still higher than the maximum sheet resistance for a RT implant at a dose of 1x10^{14} cm^2. All resistance values for 200°C implants are higher than those obtained for RT implants.
Over the dose range between $4 \times 10^{12}$ and $1 \times 10^{14} \text{ cm}^{-2}$ (figure 5.16), $n_s$ and $\mu_s$ ($\sim 720 \text{ cm}^2/\text{Vs}$) are roughly constant for RT implants. As proposed previously by several authors [37,41], both donors and acceptors are produced after helium implantation. Thus, we infer that the acceptor defects are formed at a rate that is comparable to the donor production rate. For higher doses, the observed increase in $n_s$ indicates that donor defects are formed more rapidly than acceptor defects. For a 200°C implant, there is in-situ dynamic annealing due to greater mobility of defects and less donor defects are created at different helium doses. The sheet mobility is almost constant with an average value of 1200 cm$^2$/V.s for different helium doses. Hence we observe less than one order of magnitude decrease of sheet resistance and the sheet carrier concentration increases by only five times its initial value for varying doses from $4 \times 10^{12}$ to $1 \times 10^{16} \text{ cm}^{-2}$.

![Figure 5.16](image_url)

**Figure 5.16:** Evolution of sheet resistance and sheet carrier concentration as a function of dose for 600keV helium irradiation in semi-insulating InP at RT, and 200°C.
The temperature dependence of the sheet resistance and sheet carrier concentration are also studied for RT and 200°C samples implanted with different doses. Figure 5.17 shows a typical log (sheet resistance) versus reciprocal temperature graph obtained for samples implanted with helium ions at RT and at different doses. In the high-temperature range, the log $R_s$ versus 1000/T plots are straight lines, and the activation energies obtained from the slope are also shown in figure 5.17. Details of the calculation of the activation energy are given in section 4.6.2. The activation energy of the SI InP sample before implantation is 0.55eV which is close to the middle of the bandgap as expected [91]. With increasing helium doses, the slope is reduced and the activation energy decreases. The presence of deep and shallow level centers will change the slope of the curve. Samples implanted at a dose of $4 \times 10^{13}$ and $1 \times 10^{14}$/cm$^2$ show similar activation energy of 0.4 eV since similar deep donor levels are formed. At a helium dose of $1 \times 10^{16}$ cm$^{-2}$, an activation energy of 0.23eV is obtained. Thus donor levels at this dose are more dominant leading to an increase in sheet carrier concentration by two orders of magnitude as compared to that at a dose of $4 \times 10^{13}$ cm$^{-2}$ (see figure 5.16).

From figure 5.18, samples implanted at 200°C with doses of $4 \times 10^{12}$, $4 \times 10^{13}$ and $4 \times 10^{14}$ cm$^{-2}$ show a higher activation energy of 0.53, 0.52 and 0.49eV respectively than RT implanted samples at the same dose level. At the highest helium dose, an activation energy of 0.41eV is obtained for 200°C implant. We infer that shallow defects anneal out during hot implantation in SI InP. Deeper donor levels near the mid-gap position are formed compared to RT implants. The difference in the activation energies for both cases also reflects the absence of shallow defects in SI InP for 200°C implants.
Figure 5.17: Sheet resistance as a function of the reciprocal temperature for 600keV helium bombardment in SI InP at RT for different doses.

Figure 5.18: Sheet resistance as a function of the reciprocal temperature for 600keV helium bombardment in SI InP at 200°C for different doses.
5.3.2 The parallel resistor model

The SI InP data is also compared with Si-doped InP and InGaAs data (see section 5.2.2 and 5.2.3) using the same implantation conditions. This comparison provides a check against the sheet resistance measurements of the isolated regions which is an important issue to address when the peak of the damage distribution is placed well inside the semi-insulating InP substrate. The effect of the semi-insulating substrate is only significant when the sheet resistance of the isolated region is equal to that of the substrate and when the sheet carrier concentration of the isolated region is equal or less than that of the substrate. The effect of the depletion width is also important. A depletion layer exists at the air semiconductor interface. A depletion width greater than the doped layer results in a sheet carrier concentration value due to the substrate itself. In the sub-section 5.3.1, we demonstrate that the substrate can become quite conductive at high helium dose (≥1x10^{15} \text{ cm}^{-2}). The device structure used in this work can be represented as three resistors in parallel as shown in figure 5.19. This parallel resistor model works as long as the sheet resistance of the isolated n-type layer is less than that of the semi-insulating substrate and the total depletion width is less than the thickness of doped layer. This model is also valid for samples implanted at different substrate temperatures and also confirms the actual order of magnitude of the sheet resistance created due to ion implantation only.

The parallel resistor model is

\[
\frac{1}{R_T} = \frac{1}{R_1} + \frac{1}{R_2} + \frac{1}{R_3} \hspace{1cm} (5.1)
\]

\[
R_T = \frac{R_2 R_3}{R_2 + R_3} \hspace{1cm} (5.2)
\]

where \( R_T \) is the total sheet resistance obtained using the Van der Pauw technique

\( R_1 \) is the sheet resistance of the surface depletion width

\( R_2 \) is the sheet resistance of the n-type layer

\( R_3 \) is the sheet resistance of the semi-insulating InP substrate
R₁ has a very high sheet resistance (close to infinity) as compared to R₂ and R₃. Hence from equation 5.1, R₁ can be neglected and equation 5.2 includes only R₂ and R₃. However an increase in the width of the depletion region until it is greater than the thickness of the doped layer may cause inaccuracy in the electrical measurements of the doped layer.

The zero bias depletion width is given by [82]

$$ W = \frac{2\varepsilon_0 \varepsilon_r}{qN_B} \left( \frac{V_b}{N_B} - \frac{kT}{q} \right) $$  \hspace{1cm} \text{(5.3)}$$

where $W$ is the depletion width (unit is cm)

$\varepsilon_0$ is the permittivity in vacuum ($8.854 \times 10^{-14}$ F/cm)

$\varepsilon_r$ is the semiconductor permittivity (InP = 12.56, InGaAs = 13.94, InGaAsP = 13.28)

$q$ is the electronic charge ($1.602 \times 10^{-19}$ C)

$N_B$ is the bulk carrier concentration (unit is cm⁻³)

$V_b$ is the built-in potential (unit is V)
Implant isolation of InP-based materials

Chapter Five: Implant isolation using protons and helium

\( k \) is the Boltzmann’s constant \((1.38 \times 10^{-23} \text{ J/K})\)

\( T \) is the temperature during electrical measurement (unit is K)

The surface depletion width at zero bias are given by [92]

\[
W_{ds} = \sqrt{\frac{2\varepsilon_0\varepsilon_r}{qN_B}} \left( V_{bs} - \frac{kT}{q} \right)
\]  \hspace{1cm} (5.4)

\[
V_{bs} = \Phi_b - \frac{kT}{q} \ln \frac{N_e}{N_B}
\]  \hspace{1cm} (5.5)

where \( W_{ds} \) is the surface depletion width (unit is cm)

\( V_{bs} \) is the surface built-in potential (unit is V)

\( \Phi_b \) is the surface barrier height (unit is V)

\( N_e \) is the effective density of states (unit is cm\(^{-3}\))

Figure 5.20 provides a comparison between the 55keV and 600keV helium isolation implant into Si-doped InP and semi-insulating InP at RT. At a dose of \( 1 \times 10^{15} \text{ cm}^{-2} \), the data in figure 5.20 suggests that the sheet resistance value for 600keV implants may be due to the semi-insulating substrate itself and not to the n-type InP layers. We believe that the threshold dose for 600keV implants is not the real one. We infer that the threshold dose for 600keV implants occurs when the defect concentration is the same as that of 55keV implants at the threshold dose value. The parallel conduction due to the substrate is limiting the sheet resistance value for 600keV helium implantation at a dose of \( 1 \times 10^{15} \text{ cm}^{-2} \), that is, the measured sheet resistance is lower than it should be. From figure 5.21, there is an increase in the width of the depletion region with dose for both implants. At a dose of \( 2 \times 10^{14} \text{ cm}^{-2} \), a maximum depletion width of 0.44\( \mu \text{m} \) and 0.30\( \mu \text{m} \) is obtained for 55keV and 600keV implants respectively. Since the thickness of the doped layer is 0.6\( \mu \text{m} \), the data in figure 5.21 suggests that the isolated layer has correctly been measured.

Figure 5.22 provides a comparison between the RT and 200\(^{0}\)C implant isolation for Si-doped InGaAs and semi-insulating InP layers. It is clear that the sheet resistance values are truly coming from the actual n-type region. The sheet resistance of the SI InP is higher by at least one and two orders of magnitude as compared to the n-type doped InGaAs samples.
Implant isolation of InP-based materials

Chapter Five: Implant isolation using protons and helium

for RT and $200^\circ$C implants respectively. The data from figure 5.23 suggests the isolated layer has correctly been measured for both implants since the depletion width is at least four times lower than the thickness of the doped layer (0.4µm) for all doses.

Figure 5.20: Sheet resistance of $n^+$ InP and semi-insulating InP irradiated with 55keV and 600keV He$^+$ as a function of dose for RT implant.
Figure 5.21: Evolution of depletion width as a function of dose for $n^+$ InP irradiated with 55keV and 600keV He$^+$ at RT.

Figure 5.22: Sheet resistance of $n^+$ InGaAs and semi-insulating (SI) InP irradiated with 600keV He$^+$ as a function of dose for RT and $200^0$C implants.
Implant isolation of InP-based materials

Chapter Five: Implant isolation using protons and helium

Figure 5.23: Evolution of depletion width as a function of dose for n' InGaAs irradiated with 600keV He⁺ at RT and 200°C.

5.3.3 Effect of post-implant annealing temperature

Samples implanted at a helium dose of 1x10¹⁴ cm⁻², were annealed in the range of 100°C to 800°C. For samples annealed above 300°C, proximity annealing was done where another SI InP wafer was placed on top of the samples to be annealed. Figures 5.24 and 5.25 show the electrical characteristics of the SI InP after post-implant annealing. There is little change in the sheet mobility for an annealing temperature in the range of 100 – 600°C (figure 5.25). Thus the variation in sheet resistance is mostly due to the variation in sheet carrier concentration. The sheet resistance is almost constant (~1x10⁷ Ω/□) for annealing temperatures between 100°C and 500°C. At 600°C, there is a sharp decrease in the sheet resistance value by three orders of magnitude with the sheet carrier concentration reaching a maximum value of 1x10¹² cm⁻². For an annealing temperature above 650°C, the sheet resistance increases steeply approaching the as implanted value and a corresponding decrease in the sheet carrier concentration is observed.
An explanation of the results obtained after annealing is that the rates of removal of the radiation-induced acceptor and donor defects vary with temperature. The increase in $n_s$ for annealing temperatures in the range of 600°C - 650°C is due to preferential removal of acceptor defects rather than donor defects. Above 650°C, the decrease in $n_s$ is explained by the rapid increase in the donor removal rate. Ridgway et al [93] reported a similar phenomenon after oxygen bombardment of SI InP at a dose and energy of $1 \times 10^{14}$ cm$^{-2}$ and 5MeV respectively. Woodhouse et al [39] observed similar decrease in the sheet resistance by at least three orders of magnitude from the as-implanted value of $\sim 10^6 \, \Omega/\square$ after proton bombardment of SI InP at a dose and energy of $3 \times 10^{14}$ cm$^{-2}$ and 100keV respectively.

**Figure 5.24:** Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in a nitrogen atmosphere for 600keV helium implanted in SI InP at a dose of $1 \times 10^{14}$ cm$^{-2}$ at RT.
Figure 5.25: Sheet carrier concentration and sheet mobility as a function of post-implant annealing temperature for 600keV helium implanted in SI InP irradiated with 1x10^{14} \text{cm}^{-2} at RT.

5.4 Summary

In this chapter, we have looked at the implant isolation of protons and helium ions into both n-type InP and InGaAs. The effect of dose, energy, substrate temperature and post-implant annealing temperature is investigated. A gradual increase in the sheet resistance of both n-type InP and InGaAs up to a maximum value is observed with increase in dose (H^+ or He^+). Helium implant isolation in both InP and InGaAs shows a sheet resistance value of at least one order of magnitude higher than that of protons for the isolation scheme where the peak of the damage is placed outside the doped region. In the case of helium implant isolation, a wide thermally stable region up to 500°C and 350°C is obtained in InP and InGaAs respectively at the threshold dose (section 5.2). The electrical isolation of both n-type InP and InGaAs using protons is thermally stable up to 100°C at the threshold dose (section 5.1.2). Hence helium implant isolation shows better thermal stability as compared to protons.
The effect of placing the peak of the damage inside and outside the InP doped layer has also been investigated using two different helium energies, namely, 55keV and 600keV. The data shows that a similar threshold dose \((2 \times 10^{14} \text{ cm}^{-2})\) is obtained for both schemes but the sheet resistance value for the 55keV isolation scheme is approximately four times higher than that of 600keV scheme. We believe that the threshold dose obtained for 600keV isolation scheme is not the true dose where maximum sheet resistance occurs. A higher sheet resistance should be measured from the doped layer at a higher dose. From the parallel resistor model (see section 5.3.2), it is observed that the substrate becomes conductive above a helium dose of \(1 \times 10^{15} \text{ cm}^{-2}\). Since the peak of the damage is placed in the substrate for 600keV isolation scheme and the substrate becomes quite conductive at such a high dose (section 5.3), the measured sheet resistance is most probably coming from the substrate itself.

Protons are also implanted into both n-type InP and InGaAs at two different substrate temperatures, namely, RT and 200\(^{\circ}\)C. It is observed that 200\(^{\circ}\)C implants do not show better electrical isolation compared to RT implants in both InP and InGaAs. A decrease in the sheet resistance values to the same order of magnitude as those implanted at 200\(^{\circ}\)C, is also observed when RT implanted samples are annealed at 200\(^{\circ}\)C for 60s. The data suggests that these defects responsible for high electrical isolation in both InP and InGaAs for RT implants are annealed out at 200\(^{\circ}\)C. No new thermally stable defects are created during 200\(^{\circ}\)C implant but most of these defects are annealed out at this temperature. For helium implantation into n-type InP at the threshold dose for 55keV and 600keV, the sheet resistance is approximately the same for all three implantation temperatures (RT, 100\(^{\circ}\)C and 200\(^{\circ}\)C). Implantation temperature does not seem to affect the sheet resistance behaviour as a function of post-implant annealing temperature. The sheet resistance values of RT, 100\(^{\circ}\)C and 200\(^{\circ}\)C implants are about the same order of magnitude for post-implant annealing temperature in the range of 100\(^{\circ}\)C – 800\(^{\circ}\)C. We infer that no new defects are formed at 100\(^{\circ}\)C and 200\(^{\circ}\)C for this implant condition. In the case of InGaAs layers implanted with 600keV He\(^{+}\), we observe that 200\(^{\circ}\)C implants show at least two times higher sheet resistance than RT implants above a dose of \(1 \times 10^{15} \text{ cm}^{-2}\). We believe that most of the excess defects in the case of RT implants, are annealed out during implantation at 200\(^{\circ}\)C. Therefore, it can be summarised that 200\(^{\circ}\)C implants do not show better electrical isolation in both n-type InP and InGaAs layers. The ion dose, ion species and
post-implant annealing temperature are very important parameters for choosing the right condition to isolate InP and InGaAs layers.
Chapter Six

6. Implant isolation using iron and nitrogen

Results and discussions

6.1 Implant isolation of n-type InP and InGaAs using iron ions

From chapter five, we have discussed the effect of proton and helium implantation into n-type InP and InGaAs. High and thermally stable sheet resistance values (~$10^7 \Omega/\square$) are demonstrated in n-type InP using helium. However, a maximum sheet resistance of only ~$10^5 \Omega/\square$ is obtained after helium implantation into n-type InGaAs. Proton implantation into both n-type InP and InGaAs results in sheet resistance values of one order of magnitude lower than those obtained after helium implant isolation. In an effort to obtain higher sheet resistivities in both InP and InGaAs, we have investigated the implantation of iron (Fe), an impurity which is known to result in high-resistivity InP when used as a dopant during the growth of bulk crystals [94]. Iron which occupies an indium site [97], creates an acceptor level near mid band gap. This deep acceptor captures free electrons resulting in an increase of the resistivity. In this work, the formation of electrical isolation in both n and p-type InP and InGaAs layers using iron implantation at different doses, post-implant annealing temperatures and substrate temperatures is studied.

Semi-insulating Fe-doped InP wafers of (100) orientation were used as substrates for the growth of n-type InP and InGaAs epilayers, with the (100) axis $2^\circ$ off normal orientation, using a Solid Source Molecular Beam Epitaxy reactor at the University of Sheffield. An undoped InP buffer layer of thickness 1µm was first grown below the n-type layer. Silicon was used to dope the n-type layers with a concentration and thickness of $1\times10^{18}$ cm$^{-3}$ and 1µm respectively. The InGaAs epilayers are lattice-matched to the InP buffer layer using an indium composition of 0.53. The wafers were cleaved to obtain several samples of approximately 1 cm$^2$ for the preparation of the resistors. Full details of the fabrication process of the resistors is given in section 4.1. The initial sheet resistance, sheet carrier concentration and sheet mobility are given in table 6.1. It can be seen that the measured sheet carrier concentration of the InP is approximately two times higher than the expected
value of $1 \times 10^{14}$ cm$^{-2}$ ($1 \times 10^{18}$ cm$^{-3} \times 1$ μm). This discrepancy may be due to a higher concentration of silicon used during the growth of the doped layer. Similarly for InGaAs layer, the lower sheet carrier concentration measured may be due to lower concentration of the silicon used during the growth process.

Table 6.1: Initial sheet resistance, sheet carrier concentration and sheet mobility of both n-type InP and InGaAs before isolation. The projected range and longitudinal straggling of 1MeV iron into InP and InGaAs as determined by TRIM are also tabulated.

<table>
<thead>
<tr>
<th></th>
<th>Projected Range (Å)</th>
<th>Longitudinal straggling (Å)</th>
<th>Initial sheet resistance (Ω/□)</th>
<th>Initial sheet carrier concentration (cm$^{-2}$)</th>
<th>Initial sheet mobility (cm$^2$/Vs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>n$^+$ InP</td>
<td>5950</td>
<td>2083</td>
<td>15.50</td>
<td>$2.28 \times 10^{14}$</td>
<td>1775</td>
</tr>
<tr>
<td>n$^+$ InGaAs</td>
<td>4612</td>
<td>1675</td>
<td>31.60</td>
<td>$6.0 \times 10^{13}$</td>
<td>6777</td>
</tr>
</tbody>
</table>

Figure 6.1 shows damage resulting from the iron implants into both InP and InGaAs, as determined by Transport of Ions in Matter simulation. The projected range of iron ions into InP and InGaAs is approximately 0.6 μm and 0.46 μm respectively. More details of the straggle of iron in these materials are given in table 6.1. The energy of the iron beam was chosen to place most of the iron atoms well inside the doped layer. In this way, the chemical compensation will be more effective for the electrical isolation of the n-type epilayers.

6.1.1 Effect of substrate and post-implant annealing temperature

The samples were divided into four different groups with implant isolation at temperatures of 77K, 25$^0$C, 100$^0$C, and 200$^0$C using a 2MV High Voltage Engineering Europa (HVEE) implanter. The accuracy in the temperature control was ±3$^0$C. During implantation, the samples were tilted about 7$^0$ from the surface normal to minimize channelling. For 77K implants, the samples were mounted on a cold stage, which was cooled using liquid nitrogen. The centre of the Hall pattern for all the samples was irradiated with Fe$^+$ using a dose and energy of $5 \times 10^{14}$ cm$^{-2}$ and 1MeV respectively, with a beam current density < 0.33 μA/cm$^2$. 
Figure 6.1: The damage distribution resulting from iron implantation into InP and InGaAs as determined by TRIM.

Figure 6.2 shows the evolution of sheet resistance as a function of post-implant annealing temperature for layers isolated with 1MeV iron at 77K, RT, 100°C and 200°C, respectively. The initial sheet resistance of the n-type InP layer for all the samples is ~15 Ω/□. After iron implantation, an as-implanted sheet resistance of ~5x10^6 Ω/□ is obtained for substrate temperatures of 77K, RT, and 100°C and that of 200°C is 7x10^5 Ω/□. Thus there is an increase in the sheet resistance by almost five orders of magnitude and a decrease in the sheet carrier concentration by four orders of magnitude (see figure 6.3). Most of the carriers are trapped at defect sites generated during the implantation and cause the large increase in the sheet resistance and a degradation in the mobility. A maximum sheet resistance of ~1x10^7 Ω/□ is obtained for 77K, RT and 100°C implants after a post-implant annealing cycle of 400°C for 60s. After a similar anneal, a sheet resistance of ~2x10^6 Ω/□ is measured for the 200°C implant. Further increase in the post-annealing temperature above 400°C produces a decrease in the sheet resistance by at least one order
of magnitude. However the sheet resistance increases again at 650°C to 2.1x10^6, 1.2x10^6 and 2.5x10^5 Ω/□ for 77K, RT and 100°C implants respectively. This increase is not found for samples implanted at 200°C.

We believe that the isolation for iron implantation into InP is a combination of both damage and chemical compensation. The deep acceptor level of Fe on an indium site is responsible for the chemical compensation [96]. However this Fe deep level acceptor is annealed out at 600°C as reported by Kadoun et al [97]. Hence the decrease in the sheet resistance observed at 600°C is believed to be the annealing of this defect level. The increase in sheet resistance again at 650°C is believed to be due to phosphorus vacancy-iron (V_p - Fe) complex defects which are formed above an annealing temperature of 600°C. Kadoun et al [97] reported similar complex defects formation in InP for annealing temperature between 600°C and 700°C. One possible explanation of not observing this effect for 200°C implants is due to annealing of most of the V_p defects and hence this complex defect responsible for the high sheet resistance is less likely to be formed.

![Figure 6.2: Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in a nitrogen atmosphere for 1MeV iron implanted n-type InP layers irradiated with 5x10^14 cm^-2 at 77K, RT, 100°C and 200°C.](image-url)
Chapter Six: Implant isolation using iron and nitrogen

Figure 6.3: Variation of sheet carrier concentration with post-implant annealing temperature at 77K, RT, 100°C and 200°C.

A high sheet resistance \(10^6 - 10^7 \text{ } \Omega\text{cm}\) is maintained until an annealing temperature of 500°C for all four substrate temperatures (see figure 6.2). This wide thermally stable annealing window is very useful from the technological point of view. Above 650°C, there is a gradual decrease in the sheet resistance for all the substrate temperatures due to recovery of the carriers as the defects are annealed out.

SIMS measurement shows a small amount of iron diffusion towards the epilayer-substrate interface for all four substrate temperatures (see figures 6.7-6.10). Most of the iron atoms are still present in the doped layer even at an annealing temperature of 800°C. At such a high temperature, we believe that most of the iron atoms have moved from active substitutional sites to inactive interstitial sites or have formed inactive complexes with phosphorus or indium atoms. Hence the sheet resistance decreases towards its pre-implanted value above 700°C.

Figure 6.4 shows the RBS channelling spectra of 1MeV Fe\(^+\) implanted n\(^+\) InP at different substrate temperatures. The simulated RBS channelling spectrum for a depth of 1μm is

89
also plotted. The simulated spectrum is based on a simple InP layer of thickness 1µm. It helps to identify the boundaries of the 1µm doped layer for the RBS spectra in figure 6.4.

Hence, the doped layer is between channel number 260 and 111. The lowest curve is a channelled spectrum from the non-irradiated sample, showing a minimum dechannelling yield from the residual defects in the virgin crystal. From figure 6.4, the RBS channelled spectra for 77K, RT and 100°C are the same as the random spectrum. This implies that at these implantation temperatures, an implantation induced buried amorphous layer has been formed from the end of the ion range to the surface. For 200°C implantation, there is a dramatic reduction in the damage produced by iron and no amorphisation of the doped layer is observed. Only a moderate increase of the RBS signal is detected in the 200°C as-implanted samples (due to dechannelling) corresponding to a modest damage production (mostly point defect clusters, as suggested by Wendler et al[100]).

RBS spectra for 200°C implantation temperature following annealing are shown in figure 6.5. The yield for the as-implanted sample is close to that of virgin material indicating very little damage for 200°C implants. The yield for samples annealed from 400°C to 800°C decreases towards the virgin yield, indicating near total restoration of the lattice. For the 800°C annealed sample, in a region corresponding to depth beyond 0.6µm, the yield is higher than that of 650°C annealed samples. This increase is believed to be due to the onset of the production of extended defects. In the case of RT samples, the yield for the as-implanted samples and samples annealed up to 650°C is the same as the random yield indicating total amorphisation of the material (figure 6.6). At 800°C, there is a decrease in the yield but is still higher than for the virgin material. Hence the material is not totally recrystallised even for the highest annealing temperature but contains a high density of extended defects.

This behaviour is typical of III-V compounds if the implant amorphises the material [96,99]. Thus at such a high dose (5x10^{14} cm^{-2}), the whole of 1µm doped InP layer is amorphised for substrate temperature below 200°C. Less damage is produced in the case of 200°C implants due to enhanced dynamic annealing and this is most likely the reason why lower temperature implants (77K, RT and 100°C) produce higher resistivities. Most of the defects created at such an elevated temperature are annealed out during the implantation.
Thus complete recovery of the crystallinity is possible for nonamorphised samples above an annealing temperature of 650°C. Gasparotto et al [100] reported a similar enhanced dynamic annealing phenomenon after Fe ion implantation into Sn doped InP at 200°C. They used Rutherford backscattering spectroscopy (RBS) to show that less damage was created for 200°C implants. RBS was also used by Bahir et al [101] to demonstrate that less damage was formed for 200°C implants of Si into SI InP using a fluence of 3.3x10^{14} cm^{-2} at 180keV.

Figure 6.4: RBS channelling spectra of n⁺ InP samples implanted with 1MeV Fe⁺ at 77K, RT, 100°C and 200°C using a fluence of 5x10^{14} cm⁻². The RBS simulated spectrum for a depth of 1μm is also plotted. The data for 77K, RT and 100°C implants are identical to the random spectrum. The virgin spectrum is also plotted.
Figure 6.5: RBS channelling spectra of $n^+$ InP samples implanted with 1MeV Fe$^+$ at 200$^\circ$C using a fluence of $5 \times 10^{14}$ cm$^{-2}$ and annealed at 400$^\circ$C, 650$^\circ$C and 800$^\circ$C for 60s in a N$_2$ ambient. Unimplanted sample (virgin) and random spectra are reported for comparison.

Figure 6.6: RBS channelling spectra of $n^+$ InP samples implanted with 1MeV Fe$^+$ at RT using a fluence of $5 \times 10^{14}$ cm$^{-2}$ and annealed at 400$^\circ$C, 650$^\circ$C and 800$^\circ$C for 60s in a N$_2$ ambient. Data for 400$^\circ$C and 650$^\circ$C is identical to random spectrum. The virgin spectrum is also plotted for comparison.
Figures 6.7-6.10 show the SIMS Fe depth profiles for 77K, RT, 100°C and 200°C samples which have been annealed from 100°C to 800°C. The detection limit for iron in InP is 2-3x10^{15} \text{ cm}^{-3}. The Fe depth profile of the as-implanted samples for the four different substrate temperatures fits quite well the TRIM profile, except for 100°C and 200°C implants where broadening of the SIMS profile is observed at the tail. One possible explanation of this broadening of the profile at the tail is the high mobility of the iron during elevated temperature implants as a result of radiation enhanced diffusion. During 100°C and 200°C implantation, the dynamic interaction of defects with the injected iron by irradiation may be the reason behind the tail broadening. The Fe profile for samples annealed up to 650°C is similar to the as-implanted profile for all substrate temperatures. Even for annealing temperatures as high as 800°C, only a limited amount of Fe atoms diffuses from the deeper side of the Fe peak towards the doped layer-substrate interface for 77K, RT, 100°C implants. No strong iron diffusions or accumulation peaks are observed at these implantation temperatures. For samples implanted at 200°C, SIMS measurements show that the iron profile broadens during annealing at 700°C and 800°C. The tail of the broadened profile extends up to 1 \mu m inside the substrate. The origin of this tail is not clear but probably is related to the extended defects as reported from our RBS results for 200°C. These extended defects can act as Fe gettering and precipitation centres [96].

These results are novel from the device engineering point of view. High sheet resistance is obtained after annealing at 400°C for 77K, RT and 100°C implants and no diffusion of iron is observed within this annealing temperature range. The immobility of iron is a great advantage since the effect of Fe on the device performance is insignificant.

The silicon signal is also monitored during the SIMS measurements, showing the expected flat doping profile without any particular feature that could be related to those observed for the Fe signal (figures 6.7-6.10). An abrupt interface between the silicon doped layer and the substrate is observed for all samples from SIMS measurements. We believe that total amorphisation of the doped layer and the formation of the amorphous/crystalline interface far from the peak Fe concentration have an effect on the immobility of iron at an annealing temperature of 650°C. Carnera et al [102] have preamorphised the InP samples with phosphorus to create an amorphous/crystalline interface at depth of ~1.8 \mu m which is outside the iron implanted profile (range = 0.6 \mu m). They reported no iron diffusion at an
Implant isolation of InP-based materials

Chapter Six: Implant isolation using iron and nitrogen

An annealing temperature of 400°C for 90 mins. Partial iron diffusion towards the amorphous/crystalline interface was observed at an annealing temperature of 650°C for 90 mins. Gettering of iron is observed only when the iron implanted profile is in proximity to the amorphous/crystalline interface. Another possible reason for the immobility of iron at such a high annealing temperature is the Si retarding effect on Fe diffusion. The Fe diffusion mechanism in InP is not completely understood, although it is believed that Fe diffuses with an interstitial – substitutional mechanism [103-106]. This diffusion mechanism involves the migration of iron atoms from an interstitial position to a neighbouring substitutional one. Hence the presence of Si atoms on substitutional sites will reduce the vacant sites available for diffusion of Fe. Van Gurp et al [107] have observed a similar retarding effect for zinc diffusion in silicon doped InP. They reported a decrease in the diffusion of zinc with an increase in the silicon doping concentration.

![Graph showing SIMS Fe profiles for InP samples implanted at 77K using a dose of 5x10^{14} \text{ cm}^{-2} for different annealing temperatures. TRIM Fe profile and SIMS Si profile are also plotted on the same graph.]

**Figure 6.7:** SIMS Fe profiles for InP samples implanted at 77K using a dose of 5x10^{14} \text{ cm}^{-2} for different annealing temperatures. TRIM Fe profile and SIMS Si profile are also plotted on the same graph.
Figure 6.8: SIMS Fe profiles for InP samples implanted at RT using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. SIMS Si profile for 800°C annealed is also plotted.

Figure 6.9: SIMS Fe profiles for InP samples implanted at 100°C using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. SIMS Si profile for 800°C annealed is also plotted.
Article 6.10: SIMS Fe profiles for InP samples implanted at 200°C using a dose of 5x10^{14} cm^{−2} for different annealing temperatures. SIMS Si profile for 800°C annealed is also plotted.

A similar implant isolation experiment was carried out on n^+ InGaAs epilayers grown on SI InP substrate. Figure 6.11 shows the evolution in the sheet resistance as a function of post-implant annealing temperature for Si-doped InGaAs. A similar behaviour to InP in the sheet resistance variation as a function of annealing temperature is observed in n^+ InGaAs bombarded with Fe. A maximum sheet resistance of 1x10^7 and 2.3x10^6 Ω/□ is obtained for 77K and RT implants respectively after annealing at 650°C for 60s. In the case of 100°C and 200°C implants, the maximum sheet resistance is only ~1x10^5 Ω/□ after annealing at 550°C. As shown in figure 6.11, the sheet resistance for 77K implants is only two times higher than that of RT implants for samples annealed in the range of 100°C – 500°C. Since all samples implanted at 77K are measured at RT, the relaxation mechanism during cold implantation and measurement at RT may affect the isolation behaviour of the samples. We infer that most of the defects formed at 77K are annealed out as the samples are...
Implant isolation of InP-based materials

Chapter Six: Implant isolation using iron and nitrogen

warmed up to room temperature. Similar to n⁺ InP, 200°C implants result in sheet resistivities at least two times lower as compared to 77K and RT implants.

A thermally stable region (~7 × 10⁴ Ω/□) up to an annealing temperature of 550°C is obtained for substrate temperatures of 100°C and 200°C. For 77K and RT implants, the thermally stable region from 100°C to 650°C has an average sheet resistance of 5 × 10⁶ Ω/□. A decrease in the sheet resistance by at least four times is observed for 77K and RT implants at an annealing temperature of 600°C for 60s (figure 6.11). The sheet resistance increases again by one order of magnitude at an annealing temperature of 650°C for both implants. Beyond the post-implant annealing temperature where maximum sheet resistance is obtained for all substrate temperatures, the sheet resistance decreases gradually towards its original value (~32 Ω/□). This implies that most of the defects are annealed out and that out-diffusion of Fe at temperatures between 700°C – 800°C may have an effect on the observed decrease in the sheet resistance. Our SIMS measurements show that most of the iron atoms diffuse towards the surface and the epilayer-substrate interface above an annealing temperature of 650°C. Hence the presence of iron within the doped epilayer is an important factor for isolation of n-type InGaAs materials.

We believe that no new thermally stable defects are created during elevated temperature implants (100°C and 200°C) for this isolation scheme. Most of the defects created during elevated temperature implants are annealed out. We reported similar behaviour for proton implant isolation of n-type InP and InGaAs (section 5.1.2). The increase in the sheet resistance at 650°C for 77K and RT implants is most probably related to the presence of similar Fe complex defects which are responsible for similar increase in the sheet resistance for InP. One possible explanation for not observing this increase in the sheet resistance at 650°C for 100°C and 200°C implants is that these complex defects are annealed out at these implantation temperatures in the case of InGaAs. We are not very sure about the exact Fe-related defects which are responsible for the high sheet resistivity at 650°C.
Figure 6.11: Evolution of sheet resistance with different post-implant annealing temperature after rapid thermal annealing for 60s in nitrogen atmosphere for 1MeV iron implanted n-type InGaAs layers irradiated with $5 \times 10^{14}$ cm$^{-2}$ at 77K, RT, 100°C and 200°C.

To evaluate the crystal lattice perfection qualitatively, RBS channelling measurements are performed on these samples. Figure 6.12 shows the RBS channelling spectra of 1MeV Fe$^+$ implanted n$^+$ InGaAs at different substrate temperatures. The simulated RBS channelling spectrum of the 1μm InGaAs layer only is also shown on the same plot. The peak at the low channel number (90) for all the RBS spectra is the interface between the doped epilayer and the substrate. This peak is due to the contributions from the deep-lying indium and the damage in the GaAs sublattice. Channel numbers 239 and 257 correspond respectively to the surface peak positions for the GaAs and In. The lowest curve is a channelled spectrum from the non-irradiated sample, showing a minimum dechannelling yield from the residual defects in the virgin crystal. The RBS channelled spectra for 77K...
and RT implants are the same as the random spectrum. At this implantation temperature, an implantation induced amorphous layer has been formed throughout the whole 1μm doped layer as shown in figure 6.12. For 100°C and 200°C implants, there is a reduction in the damage produced by iron and no amorphisation of the doped layer is observed. It is clear therefore that under the present implant conditions, the material is resistant to amorphisation for implantation temperatures above 100°C, whereas in the case of InP, implantation temperatures above 200°C are required to prevent amorphisation. Since the measured damage is always a result of the competition between defect production and dynamic defect annealing, the reduced damage accumulation rate measured above 100°C in the case of InGaAs and 200°C in the case of InP indicates that a strong dynamic annealing of the radiation-produced defects is operational at these implantation temperatures and largely suppresses growth of lattice disorder.

Figure 6.13 shows the RBS channelling spectra for 200°C implants at different annealing temperatures. In the channelling spectra, the yield from the as-implanted sample is similar to that of the virgin sample at the near-surface (0.1μm) and is higher deep inside. With increasing annealing temperatures, the yield decreases towards the virgin yield. This indicates that the implantation damage concentration is low due to substantial dynamic annealing of the damage which occurs at high annealing temperatures. In the case of RT implant where the as-implanted sample is amorphous, even after annealing at 800°C, only a minor decrease in the yield at the near surface is observed (figure 6.14). Hence partial recrystallisation only occurs at the surface and the doped layer deep inside is still amorphous. Once the material becomes amorphous, total removal of lattice damage is unattainable even at a high annealing temperature of 800°C as observed in the case of InP.
Figure 6.12: RBS channelling spectra of Fe⁺ implanted into n⁺ InGaAs using a fluence of $5 \times 10^{14}$ cm$^{-2}$ at different implantation temperatures namely, 77K, RT, 100°C and 200°C. Unimplanted sample and random spectra are reported for comparison. The RBS spectra for RT and 77K implants are similar to that of the random spectra.

Figure 6.13: RBS channelling spectra of n⁺ InGaAs samples implanted with Fe⁺ at 200°C using a fluence of $5 \times 10^{14}$ cm$^{-2}$ at 1 MeV and annealed at 550°C and 800°C for 60s in a N$_2$ ambient. Unimplanted sample and random spectra are reported for comparison.
Implant isolation of InP-based materials
Chapter Six: Implant isolation using iron and nitrogen

Figure 6.14: RBS channelling spectra of Fe⁺ implanted into n⁺ InGaAs at RT and 200°C after annealing at 800°C. Unimplanted sample and random spectra are reported for comparison.

The SIMS Fe depth profiles on as-implanted and annealed InGaAs samples are shown in figures 6.15 - 6.17 for 77K, RT and 200°C implants respectively. The Ga SIMS signal is also monitored for all measurements to locate the interface between the InGaAs epilayer and the InP substrate and is not quantified. As shown in figure 6.15, the InGaAs epilayer thickness of 1μm was correctly grown on the InP substrate. The TRIM profile fits quite well the RT as-implanted profile whereas the SIMS profile for 200°C as-implanted sample shows a broader profile. A similar effect is observed in the case of Fe-implanted InP and is most probably due to radiation-enhanced diffusion. For 77K implants, above an annealing temperature of 550°C, the iron atoms redistribute towards the surface and the interface between the InGaAs epilayer and the InP substrate. However, the SIMS profile of the 800°C Fe implanted sample indicates larger surface pileup, which is followed by a sharp dip in the profile at 0.67μm. This sharp dip, which is not observed in the case of 550°C, is due to more Fe diffusing out to the surface and into the bulk. Similar Fe redistribution phenomenon is observed for the RT and 200°C implants (see figures 6.16 and 6.17).
In the case of the RT sample, which has been annealed at 650°C, SIMS measurement shows two distinct peaks after the iron projected range (figure 6.16). The first peak at the interface is also observed in 77K and 200°C annealed samples. However, the second peak is only observed in RT sample, which has been annealed at 650°C. One possible explanation for the peak at a depth of 0.84μm is the segregation of the iron to the end-of-range defects which are formed after the amorphous/crystalline interface.

Hence the presence of Fe in the doped layer is believed to be an important parameter for the high sheet resistance obtained in the case of 77K and RT implants. Annealing at a high temperature of 800°C results in the rapid out-diffusion of Fe towards the surface and interface, which is evident from the SIMS profiles (figure 6.15-6.17). These results are consistent with the decrease in the sheet resistance observed from figure 6.11. The exact diffusion mechanism of iron in InGaAs is not clear and is a topic of research by itself. No one has reported a suitable mechanism to explain the diffusion of iron in InGaAs. Koumetz et al [108] reported interstitial – substitutional diffusion mechanism for beryllium dopant in InGaAs. We believe that similar diffusion mechanism is operational.

![Figure 6.15: SIMS Fe profiles for InGaAs samples implanted at 77K using a dose of 5x10^{14} \text{ cm}^{-2} for different annealing temperatures. TRIM Fe profile is plotted on the same graph.](image-url)
Figure 6.16: SIMS Fe profiles for InGaAs samples implanted at RT using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. TRIM Fe profile is plotted on the same graph.

Figure 6.17: SIMS Fe profiles for InGaAs samples implanted at 200°C using a dose of $5 \times 10^{14}$ cm$^{-2}$ for different annealing temperatures. TRIM Fe profile is plotted on the same graph.
6.1.2 Effect of dose at different substrate temperatures

In this experiment, the effect of iron damage accumulation is investigated. Similar n-type InP and InGaAs samples described in section 6.1 are used. The samples are divided into three different groups with implantation temperatures of 77K, RT and 200°C for this experiment. The samples are irradiated with iron at 1MeV to doses in the range of $1 \times 10^{12} - 1 \times 10^{15}$ cm$^{-2}$.

Figure 6.18 shows the evolution of sheet resistance with the variable implanted doses for n$^+$ InP isolated with 1MeV iron at 77K, RT and 200°C, respectively. The initial sheet resistance before implant isolation is 15.5 Ω/□. After implantation with $1 \times 10^{12}$ cm$^{-2}$ Fe$^+$ ions, an increase in the sheet resistance by at least one order of magnitude as compared to the pre-implanted value (15.5 Ω/□) is observed for all the three implantation temperatures. An optimum isolation of $4 \times 10^6$ Ω/□ and $6 \times 10^6$ Ω/□ is obtained at a dose of $5 \times 10^{13}$ cm$^{-2}$ for InP implanted at 77K and RT, respectively. Further dose accumulation up to $5 \times 10^{14}$ cm$^{-2}$ results in a plateau for both 77K and RT implants.

For 200°C implants, a relatively low isolation value of $\sim1 \times 10^6$ Ω/□ is obtained at a threshold dose of $1 \times 10^{13}$ cm$^{-2}$. Implantation beyond the threshold dose results in a plateau until an iron dose of $1 \times 10^{15}$ cm$^{-2}$. We infer that no thermally stable defects are created during the 200°C implant, thus enhanced dynamic annealing is present during the high temperature implantation. This is consistent with the RBS results in figure 6.21, which show significantly lower backscattering yields for samples irradiated with iron at 200°C relative to the layers bombarded at lower temperatures. Most of the defects responsible for the higher sheet resistance at 77K and RT implants are annealed out during 200°C implants.
Figure 6.18: Evolution of sheet resistance as a function of dose for 1MeV iron irradiation in n-type InP layers at 77K, RT, and 200°C.

The RBS spectra for 1MeV iron implant of n⁺ InP at RT are shown in figure 6.19 for different doses. The lowest curve is a channelled spectrum from the non-irradiated sample, showing a minimum dechannelling yield. The doped layer is between channel numbers 260 and 111 as simulated in figure 6.4. There is a gradual increase in damage both at the surface and in the doped layer as the iron dose increases. The spectra from the as-implanted RT samples for doses of 5×10¹⁴ cm⁻² and 1×10¹⁵ cm⁻² are not shown in figure 6.19, as they look similar to the random spectrum. This implies that at these doses, an implantation induced buried amorphous layer has been formed from the doped layer/substrate interface to the surface. The RBS spectra for samples implanted at 77K are shown in figure 6.20. Similar to RT implants, an increase in damage both at the surface and in the doped layer is observed with increasing iron doses. However for an iron dose of 1×10¹³ cm⁻², the yield is significantly lower near the surface but similar to the random yield deep inside the doped layer. This indicates that the surface is still crystalline but very highly damaged inside the doped layer. The RBS yield of RT implants at this dose is lower by 20% with respect to the random yield. For the lowest dose, the region near the surface
of the samples is crystalline and the damage inside the substrate is reduced by 80% for both 77K and RT implants. Hence less damage for the lowest dose results in less generation of defects and most probably related to the lower sheet resistance obtained.

Figure 6.21 shows the RBS spectra for 1MeV iron implant of n⁺ InP at 200°C. For doses lower than $1 \times 10^{14}$ cm$^{-2}$, the RBS spectra are similar to that of the unimplanted sample indicating no crystalline damage for these samples. The yield for the region near the surface is close to that of the virgin spectrum for all the doses. However there is a small increase in the RBS yield in the region below 0.5μm from the surface with increasing doses from $1 \times 10^{14}$ cm$^{-2}$ to $1 \times 10^{15}$ cm$^{-2}$. This is consistent with the presence of point defects and the dechannelling effect. Therefore only small differences in crystalline quality between the unimplanted sample and those implanted at 200°C with doses between $1 \times 10^{14}$ cm$^{-2}$ and $1 \times 10^{15}$ cm$^{-2}$ are observed. It can be deduced that significant annealing or recombination of defects is activated during bombardment at 200°C. Thus a lower density of dislocations is present in those samples irradiated at 200°C.

Figure 6.22 shows the damage fraction at the near surface region (channel number 190-219) as a function of dose for InP samples implanted at 77K, RT and 200°C. The fraction damage is the ratio of the aligned damaged spectrum to the random yield at the defined channel range. For 77K and RT implants, the fraction damage increases with dose until it saturates at a dose of $\sim 5 \times 10^{13}$ cm$^{-2}$ where full amorphisation occurs. In contrast with 77K and RT implants, the damage buildup in samples implanted at 200°C remains very small for doses up to $1 \times 10^{15}$ cm$^{-2}$. The accumulated damage (0.23) is still very much below the amorphous level for doses up to $1 \times 10^{15}$ cm$^{-2}$ during implantation at 200°C. From figure 6.22, less damage is created when samples are implanted at 200°C than 77K and RT implants for all the different doses. The dose required for complete amorphisation of the InP implanted at 77K and RT is $5 \times 10^{13}$ cm$^{-2}$ and $1 \times 10^{14}$ cm$^{-2}$ respectively. However no amorphisation of InP is obtained for 200°C even after a high dose of $1 \times 10^{15}$ cm$^{-2}$. 


Figure 6.19: RBS channelling spectra for iron implanted n-type InP layers irradiated with different doses at 1 MeV at RT.

Figure 6.20: RBS channelling spectra for iron implanted n-type InP layers irradiated with different doses at 1 MeV at 77K. Data for a dose of $5 \times 10^{13}$ cm$^{-2}$ is identical to the random spectrum.
Chapter Six: Implant isolation using iron and nitrogen

Figure 6.21: RBS channelling spectra for iron implanted n-type InP layers irradiated with different doses at 1MeV at 200°C.

Figure 6.22: Fractional damage at the near the surface(channel number 190-219) of InP as a function of dose for 77K, RT and 200°C implants.
The dose dependence experiment for iron implantation into $n^+$ InGaAs was also investigated. Figure 6.23 shows the sheet resistance of InGaAs layers implanted at 77K, RT and 200°C by iron as a function of dose. The initial sheet resistance of the InGaAs layer before implant isolation is $\sim 35 \ \Omega/\square$. It can be seen that similar to what is observed in the case of InP, the lowest dose irradiation results in about two orders of magnitude increase in the sheet resistance for all three implantation temperatures. There is a gradual increase in the sheet resistance with dose for all three substrate temperatures. A maximum sheet resistance of $\sim 5 \times 10^6 \ \Omega/\square$ and $8 \times 10^4 \ \Omega/\square$ is obtained at a dose of $5 \times 10^{14} \ \text{cm}^2$ for 77K and RT implants respectively. In the case of 200°C implant, the maximum sheet resistance is $5.25 \times 10^4 \ \Omega/\square$ after an iron dose of $1 \times 10^{13} \ \text{cm}^2$. The increase in the sheet resistance for RT and 200°C is only about one order of magnitude from the lowest dose to the highest dose. Hence, the sheet resistance data in figure 6.23 show that better isolation is obtained for samples implanted at 77K as compared to those implanted at 200°C above a dose of $5 \times 10^{13} \ \text{cm}^2$. We believe that the defects responsible for the high sheet resistance in 77K implanted samples are annealed out during implantation at 200°C. From RBS results in figure 6.26, significantly lower backscattering yields are obtained for samples irradiated with iron at 200°C relative to the layers bombarded at 77K. The RBS results are quite consistent with the electrical results shown in figure 6.23.

Figure 6.23: Evolution of sheet resistance as a function of dose for 1MeV iron irradiation in $n$-type InGaAs layers at 77K, RT, and 200°C.
Figures 6.24-6.26 show the channelling spectra obtained for InGaAs samples implanted with iron at different substrate temperatures with fluences from $1 \times 10^{12}$ to $1 \times 10^{15}$ cm$^{-2}$. Backscattering spectra obtained for unimplanted material in channelling and random orientations are also shown for comparison purposes. In figure 6.24, the peak at channel 257 in the damage spectrum corresponds to the peak In damage position. The peak at channel 239 corresponds to the peak damage in the GaAs sublattice in addition to contributions from deep-lying In. The InGaAs doped layer is between channel 257 and 93. The lowest dose RBS channelling spectrum is similar to the unimplanted spectrum for RT implants (figure 6.24). It can be seen that there is a gradual increase in damage both at the surface and in the doped layer as the iron dose increases. Thus the height of the damage spectrum increases until it coincides with the random spectrum, indicating the formation of a continuous amorphous layer ~1.0μm thick. The spectra from the as-implanted RT samples for doses of $1 \times 10^{14}$ cm$^{-2}$, $5 \times 10^{14}$ cm$^{-2}$ and $1 \times 10^{15}$ cm$^{-2}$ are not shown in figure 6.24, as they look similar to the random spectrum. At a dose of $5 \times 10^{13}$ cm$^{-2}$, the RBS spectrum for RT implant shows an amorphous layer deep inside the doped layer but crystalline layer near the surface region. The RBS spectra for samples implanted at 77K are shown in figure 6.25. Similar to RT implants, an increase in damage both at the surface and in the doped layer is observed with increasing iron doses. However, the $5 \times 10^{13}$ cm$^{-2}$ dose produces a continuous amorphous layer up to the surface for the 77K implant but for a RT implant, a buried amorphous layer is formed.

RBS spectra for 200°C implants are also shown in figure 6.26. In contrast with 77K and RT implants, the damage build-up in samples implanted at 200°C remains quite small for doses up to $1 \times 10^{15}$ cm$^{-2}$ and no amorphous layer is formed. This is due to enhanced dynamic annealing during implantation. The RBS spectra for doses lower than $1 \times 10^{14}$ cm$^{-2}$ are not shown in figure 6.26 as they are similar to that of the unimplanted sample indicating no crystalline damage for these samples. Similar to InP, the yield for the region near the surface is close to that of the virgin spectrum for all the doses. But there is an increase in the RBS yield in the region below 0.5μm from the surface with increase in doses from $1 \times 10^{14}$ cm$^{-2}$ to $1 \times 10^{15}$ cm$^{-2}$. This is due to the introduction of damage which increases the dechannelling yield. As the He$^+$ ions penetrate deeper inside, they suffer more and more de-channelling in the irradiated sample.
Figure 6.27 shows the variation of implant-induced disorder at the near surface region (channel 219-190) with iron dose for InGaAs samples implanted at 77K, RT and 200°C. The fractional damage is the ratio of the aligned damaged spectrum to the random yield at the defined channel range. The figure shows that for the 77K implants, the damage builds up with increasing dose until it saturates as full amorphisation is reached at a dose of $5 \times 10^{13}$ cm$^{-2}$. It is clear, therefore, that under the present implant conditions the threshold dose for amorphisation of InGaAs implanted at 77K and RT is $5 \times 10^{13}$ cm$^{-2}$ and $1 \times 10^{14}$ cm$^{-2}$ respectively, while the material is resistant to amorphisation for doses up to $1 \times 10^{15}$ cm$^{-2}$ during implantation at 200°C. Since the measured damage is always a result of the competition between the defect production and dynamic defect annealing, the reduced damage accumulation rate measured at 200°C indicates that a strong dynamic annealing of the radiation-produced defects is operational at 200°C and largely suppresses production of lattice disorder.

Figure 6.24: RBS channelling spectra of Fe$^+$ implanted into n$^+$ InGaAs at RT for different doses namely, $1 \times 10^{12}$ cm$^{-2}$, $1 \times 10^{13}$ cm$^{-2}$ and $5 \times 10^{13}$ cm$^{-2}$. Samples implanted above a dose of $5 \times 10^{13}$ cm$^{-2}$ have channelling spectra similar to that of the random spectrum. Data for $1 \times 10^{12}$ cm$^{-2}$ is identical to the virgin spectrum.
Implant isolation of InP-based materials
Chapter Six: Implant isolation using iron and nitrogen

Figure 6.25: RBS channelling spectra of Fe⁺ implanted into n⁺ InGaAs at 77K for different doses namely, $1 \times 10^{12} \text{ cm}^{-2}$, $1 \times 10^{13} \text{ cm}^{-2}$ and $5 \times 10^{13} \text{ cm}^{-2}$. Samples implanted at and above a dose of $5 \times 10^{13} \text{ cm}^{-2}$ have channelling spectra similar to that of the random spectrum.

Figure 6.26: RBS channelling spectra of Fe⁺ implanted into n⁺ InGaAs at 200°C for different doses namely, $1 \times 10^{14} \text{ cm}^{-2}$, $5 \times 10^{14} \text{ cm}^{-2}$ and $1 \times 10^{15} \text{ cm}^{-2}$. Samples implanted at a dose lower than $1 \times 10^{14} \text{ cm}^{-2}$ have channelling spectra similar to that of the virgin spectrum.
Implant isolation of InP-based materials

Chapter Six: Implant isolation using iron and nitrogen

Figure 6.27: Fractional damage at the near surface region (channel 190-219) versus the implanted iron dose for InGaAs implanted at 77K, RT and 200°C.

6.2 Implant isolation of p-type InP and InGaAs using iron ions

Many InP-based Heterojunction Bipolar Transistors and lasers have also p-type doped regions. Thus information is needed on the production of high resistivity in these materials. Fewer investigations have been published on the electrical isolation of p-type InP and InGaAs using the ion implantation technique [39,42]. In this section, the electrical characterisation of both p-type InP and InGaAs after iron implantation is reported. The effect of post-implant annealing temperature, implantation temperature and dose are also studied.

Semi-insulating Fe-doped InP wafers were used as substrates for the growth of both p-type InGaAs and InP epilayers using SSMBE at the National Centre for III-V Technologies (University of Sheffield). An undoped InP buffer layer of thickness 0.1µm was first grown below the 1.0µm p-type layers. Zinc was used as the dopant with a concentration $1 \times 10^{18}$ cm$^{-3}$. The wafers were cleaved to obtain several samples of approximately 1 cm$^2$ for the
preparation of the resistors. The initial sheet resistance, sheet carrier concentration and sheet mobility are given in table 6.2. The measured initial sheet carrier concentration is close to the expected value of 1x10^{14} \text{ cm}^{-2} (1x10^{18} \text{ cm}^{-3} \times 1 \mu\text{m}).

<table>
<thead>
<tr>
<th></th>
<th>Projected Range (Å)</th>
<th>Longitudinal straggling (Å)</th>
<th>Initial sheet resistance (Ω/☐)</th>
<th>Initial sheet carrier concentration (cm(^2))</th>
<th>Initial sheet mobility (cm(^2)/Vs)</th>
</tr>
</thead>
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<tr>
<td>p(^+) InP</td>
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<td>2083</td>
<td>871.5</td>
<td>7.7x10^{13}</td>
<td>93.22</td>
</tr>
<tr>
<td>p(^+) InGaAs</td>
<td>4612</td>
<td>1675</td>
<td>683.6</td>
<td>7.0x10^{13}</td>
<td>135</td>
</tr>
</tbody>
</table>

**Table 6.2:** Initial sheet resistance, sheet carrier concentration and sheet mobility of both p-type InP and InGaAs before isolation. The projected range and longitudinal straggling of 1MeV iron into InP and InGaAs as determined by TRIM are also tabulated.

6.2.1 Effect of substrate and post-implant annealing temperature
The p-type samples were divided into four different groups with implant isolation at temperatures of 77K, 25°C, 100°C and 200°C. The centre of the Hall pattern for all the samples was irradiated with Fe\(^+\) at a dose and energy of 5x10^{14} \text{ cm}^{-2} and 1MeV respectively, using a 2MV High Voltage Engineering Europa (HVEE) implanter.

The evolution of sheet resistance as a function of post-implant annealing temperature for p-type InP at 77K, RT, 100°C and 200°C is shown in figure 6.28. The initial sheet resistance of the Zn-doped InP layer before implant isolation is 872 Ω/☐. After iron implantation, it is interesting to note that the sheet resistance for 77K and RT implants is \~6x10^6 Ω/☐ and that for 100°C and 200°C implants is \~10^6 Ω/☐. Thus there is an increase in R\(_s\) by at least three orders of magnitude for all samples. It is also observed that the initially p-type layers convert to n-type conductivity for all as-implanted samples. Maximum sheet resistivities of 2.7x10^7 Ω/☐ and 1.5x10^7 Ω/☐ are obtained after a post-implant annealing temperature of 500°C for 77K and RT implants respectively.
This type conversion is also observed by other authors [35,37] after proton bombardment in p-type InP. They have used the bombardment created deep levels model to explain this behaviour. They have suggested that in p-type InP, the Fermi level moves close towards the middle of the bandgap with increasing proton dose as both donor- and acceptor-like defects are created. When it reaches a point near and above the middle of the bandgap where the defect related free electron concentration just exceeds the hole concentration, the resistivity becomes maximum and p-type bombarded layers convert to n-type. This agrees with the interpretation of the sheet resistance data presented in this work. The same model can be used to explain the sheet resistance results. We believe that the iron dose is close to the threshold value which occurs when p-type material converts to n-type. This is the reason why as implanted samples produce high sheet resistance ($\sim 10^6 \Omega/\square$).

Hot implants show one order of magnitude lower sheet resistance as compared to 77K and RT implants until a post-implant annealing temperature of 500°C. Less damage is produced for 100°C and 200°C implants due to enhanced dynamic annealing and this is most likely the reason why lower temperature implants produce higher resistivities. From RBS, we have demonstrated that less damage is formed for hot implants of iron into n-type InP using a fluence of $5 \times 10^{14}$ cm$^{-2}$ at 1MeV. The RBS channelling spectrum of the as-implanted 200°C sample is close to that of the virgin non-implanted sample. We believe that most of the defects responsible for the high sheet resistance during 77K and RT implants are annealed out during 100°C and 200°C implants. From figure 6.28, 77K and RT implants show similar post-implant annealing behaviour and the changing sheet resistance may therefore be due to the same isolation mechanism in both cases.

A broad thermally stable region up to a post-implant annealing temperature of 500°C is obtained for all four substrate temperatures. Such a wide annealing window is quite useful from the technological point of view. With continued annealing above 600°C, the carriers are converted back to p-type as defects responsible for trapping of mobile carriers are gradually annealed out and the sheet resistance recovers towards its initial unimplanted value. Similar behaviour is observed for all implant temperatures. A gradual recovery of the sheet carrier concentration above an annealing temperature of 600°C is obtained.
Figure 6.28: Evolution of sheet resistance with annealing temperature for iron implanted p-type InP layers irradiated with $5 \times 10^{14}$ cm$^{-2}$ at 1MeV, as a function of implant temperature.

The implant isolation of p-type InGaAs using 1MeV iron is also studied. The sheet resistance versus post-implant annealing temperature for Fe$^+$ implanted p-type InGaAs samples at 77K, RT, 100$^0$C and 200$^0$C is shown in figure 6.29. Conductivity type conversion from p to n is also observed for all as-implanted samples. The post-implant annealing behaviour is quite similar to that of p-type InP. However only samples implanted at 77K show better and thermally stable isolation as compared to those implanted at RT, 100$^0$C and 200$^0$C.

One possible explanation of this result is that the type of defects formed during 77K implants is different from RT implants in the case of InGaAs and this may be the explanation for the two orders of magnitude difference in the sheet resistance between 77K and RT as-implanted samples. For 77K implants, a thermally stable isolation is maintained up to 600$^0$C. A maximum sheet resistance of $\sim 2.2 \times 10^7$ $\Omega/\square$ is achieved for 77K implanted
samples which have been annealed at 500°C for 60s. For higher annealing cycles, the sheet resistance decreases sharply and the initial p-type conductivity is restored by 700°C even for the other three implantation temperatures.

The implant and anneal behaviour of p' InGaAs samples resemble that of p' InP samples. Depending on the ion species and initial doping concentration, there is a threshold dose at which the p-type InGaAs converts to n-type conductivity and the Fermi level is pinned in the upper half of the bandgap. The lower isolation values obtained for RT, 100°C and 200°C implants are most probably due to the effect of enhanced dynamic annealing as seen in the case of p' InP.

Figure 6.29: Evolution of sheet resistance with annealing temperature for iron implanted p-type InGaAs layers irradiated with 5x10^{14} cm^{-2} at 1 MeV, as a function of implant temperature.
6.2.2 Effect of dose on implant isolation of p-type InGaAs

In this experiment, the effect of iron damage accumulation in p-type InGaAs is investigated. The samples are divided into three different groups with implantation temperatures of 77K, RT and 200°C for this experiment. The samples are irradiated with iron at 1MeV to doses in the range of $1 \times 10^{12} - 5 \times 10^{14}$ cm$^{-2}$.

Figure 6.30 shows the evolution of sheet resistance with doses for 1 MeV iron implantation into p$^+$ InGaAs at 77K, RT and 200°C, respectively. The initial sheet resistance value of the p$^+$ InGaAs epilayer is 685 Ω/$\square$. It is increased by two orders of magnitude after implantation with $1 \times 10^{12}$ cm$^{-2}$ Fe$^+$ ions for all the three implantation temperatures. It is also observed that the initially p-type layers convert to n-type conductivity for all three substrate temperatures after the lowest implantation dose used ($1 \times 10^{12}$ cm$^{-2}$). The sheet resistance remains essentially unchanged with increasing dose for RT and 200°C implants. However in the case of 77K implant, there is a gradual increase in the sheet resistance with increasing dose. A maximum sheet resistance of $\sim 6.8 \times 10^6$ Ω/$\square$ is obtained at a dose of $5 \times 10^{14}$ cm$^{-2}$ for samples implanted at 77K. For all doses and substrate temperatures, p to n-type conversion of the epilayer is observed.

The sheet resistance data in figure 6.30 show that better isolation is obtained for samples implanted at 77K as compared to those implanted at RT and 200°C above a dose of $1 \times 10^{13}$ cm$^{-2}$. Similar to n-type InGaAs, we believe that defects responsible for the high sheet resistance in 77K implanted samples are annealed out during RT and 200°C implantation.
6.3 Implant isolation of n-type InGaAsP using nitrogen ions

With the increasing use of InGaAsP for the fabrication of lasers and other optoelectronic devices [109], there is a need to establish the effect of ion bombardment on the behaviour of this material, specifically on the formation of high resistivity regions for current confinement and device isolation. This work may also have applications to integrate laser and modulator structures and indeed to integrate opto-electronics in general. Little is known about how to electrically isolate quaternaries compared with implant isolation studies in GaAs and InP.

In this work, we are reporting an introduction to implant isolation of InGaAsP. The effect of dose and substrate temperature on the implant isolation of n-type InGaAsP is studied. Semi-insulating iron-doped InP wafers of (100) orientation were used as substrates for the growth of n-type InGaAsP epilayers, with the (100) axis 2° off normal orientation, using a Metal Organic Vapour Phase Epitaxy reactor. An undoped InP buffer layer of thickness 0.7μm was first grown between the epitaxial layer and the substrate. Silicon was used to
dope the n-type layers with a concentration and thickness of $1 \times 10^{18}$ cm$^{-3}$ and 1$\mu$m respectively. The InGaAsP epilayers are lattice-matched to the InP buffer layer using an indium and arsenic composition of approximately 0.75 and 0.54. The wafers were cleaved to obtain several samples of approximately 1 cm$^2$ for the preparation of the resistors. A full detail of the fabrication process of the resistors is given in section 4.1. The initial sheet resistance, sheet carrier concentration and sheet mobility are 15.40 $\Omega$/$\square$, $8.75 \times 10^{13}$ cm$^2$ and 4695 cm$^2$/V.s respectively. The measured sheet carrier concentration agrees quite well with the expected value of $1 \times 10^{14}$ cm$^{-2}$.

Figure 6.31 shows damage resulting from the nitrogen implants into InGaAsP, as determined by TRIM simulation. The projected range and longitudinal straggle of nitrogen ions into InGaAsP is approximately 2.72$\mu$m and 0.29$\mu$m respectively. The energy of the nitrogen beam was chosen to place the damage peak beyond the doped layer. In this way the defect concentration in the doped layer is approximately uniform with depth. The end-of-range disorder is buried well in the substrate and most of the nitrogen ions are buried deep inside the substrate as shown in figure 6.31.

Figure 6.31: The damage distribution resulting from 4MeV nitrogen implantation into InGaAsP as determined by TRIM.
6.3.1 Effect of dose at RT and 77K

The samples were divided into two different groups with implant isolation at temperatures of 77K and 25°C, using a 2MV High Voltage Engineering Europa (HVEE) implanter. The accuracy in the temperature control was ±3°C. During implantation, the samples were tilted about 7° to the surface normal to minimize channelling. The samples were irradiated with 4MeV N⁺ at doses ranging from \(5 \times 10^{11}\) cm\(^{-2}\) to \(1 \times 10^{14}\) cm\(^{-2}\).

Figure 6.32 shows the sheet resistance of InGaAsP layers implanted at 77K and RT as a function of the ion dose. It can be seen that similar to what is observed in proton and helium implantation into InP (section 5.1.1 and 5.2.2), the lowest dose irradiation results in a sheet resistance value (20.7 \(
\Omega/\square\)) which is of the same order of magnitude as that before nitrogen implantation.

There is gradual increase in the sheet resistance with dose for both implants. A maximum sheet resistance of \(~7 \times 10^4\ \Omega/\square\) is reached at a dose of \(1 \times 10^{14}\) cm\(^{-2}\). Xiong et al [60] reported high sheet resistance(\(~10^7\ \Omega/\square\)) in n-type InP for nitrogen doses above \(5 \times 10^{14}\) cm\(^{-2}\). Thus we believe that higher doses are required to achieve better isolation in InGaAsP. Cold implant (77K) does not result in better electrical isolation. We believe that all defects produced at 77K and responsible for trapping of carriers are presumably frozen-in during implantation. When samples are brought to air at RT, some of the defects would be annealed out through the process of diffusion and recombination. Hence the sheet resistance values for samples implanted at RT and 77K are of the same order of magnitude.
6.3.2 Reliability of the sheet resistance measurements

The effect of nitrogen ion implantation into SI InP at RT and 200°C is also studied for doses from $3 \times 10^{11}$ to $1 \times 10^{14}$ cm$^{-2}$ at 4MeV. This experiment checks the reliability of the electrical data measured in sections 6.3.1 and 6.4 using the parallel resistor model discussed in section 5.3.2. When the peak of the damage due to nitrogen ions is placed into the SI InP substrate, the operation of the devices employing this material may be affected. Hence it is important to check the effect of nitrogen ions on the semi-insulating InP substrate. No study of this effect has been reported in the literature before. The electrical data can be compared with those where similar isolation implants were done on n-type InP and InGaAsP layers grown on SI InP. The effect exhibited by elevated temperature implants as compared to RT implants is also studied.

SI InP wafers were cleaved to obtained samples of size 10mm x 10mm and the clover-leaf pattern was printed on the samples using optical lithography (see section 4.5). The samples were then divided into two groups for implant isolation at temperatures of $25 ^\circ$C, and $200 ^\circ$C using a 2MV High Voltage Engineering Europa (HVEE) implanter. The initial sheet
resistance, sheet carrier concentration and mobility of the SI InP are $6.5 \times 10^8 \ \Omega/\square$, $7.42 \times 10^6 \ \text{cm}^2$, and $1280 \ \text{cm}^2/V.s$ respectively.

Figure 6.33 shows the variation of sheet resistance and sheet carrier concentration of SI InP after nitrogen implantation as a function of dose. After nitrogen implantation at the lowest dose, the sheet resistance is decreased to $\sim 1 \times 10^8 \ \Omega/\square$ and the sheet carrier concentration is increased to $\sim 4 \times 10^7 \ \text{cm}^2$ for both substrate temperatures. There is a decrease in the sheet resistance by approximately six times. As the nitrogen dose increases from $3 \times 10^{11}$ to $1 \times 10^{14} \ \text{cm}^2$, the sheet resistance is more or less constant with values of about $1 \times 10^8 \ \Omega/\square$ for both RT and $200^\circ \text{C}$. Similarly the sheet carrier concentration does not change much at the highest dose. Hence the material should not be conductive. This trend is quite similar to that of helium implantation into SI InP (section 5.3). Higher doses ($>1 \times 10^{14} \ \text{cm}^2$) may be required to make the substrate conductive as reported in the case of helium (section 5.3). Thus the semi-insulating InP does not become appreciably conductive after nitrogen implantation at a dose in the range of $3 \times 10^{11}$ to $1 \times 10^{14} \ \text{cm}^2$ for RT and $200^\circ \text{C}$ implants. Therefore the material still remains semi-insulating.

![Figure 6.33: Evolution of sheet resistance as a function of dose for 4MeV nitrogen irradiation in semi-insulating InP at RT and 200°C.](image-url)
By comparing the data presented in figure 6.33 with those obtained using similar implant isolation conditions in Si-doped InGaAsP samples (section 6.3.1), it is clear that the sheet resistance values are coming from the actual Si-doped layer and the effect of the semi-insulating substrate is not significant. The sheet resistance of the SI InP is higher by at least three orders of magnitude as compared to the Si-doped InGaAsP samples for doses in the range of $5 \times 10^{11}$ to $1 \times 10^{14}$ cm$^{-2}$ (figure 6.34). From figure 6.35, the maximum depletion width (~0.16μm) is still at least five times smaller than the total thickness of the doped layer (1μm) for both RT and 77K implants. Hence we are truly measuring the sheet resistance value from the doped layer unless the as-implanted resistance of the n-type isolated region is equal to that of the semi-insulating substrate and the depletion width is equal or larger than the doped layer. This comparison provides control over the sheet resistance measurements of the regions to be isolated which is an important issue to address when the peak of the damage distribution is placed well inside the SI InP substrate.

![Figure 6.34: Evolution of sheet resistance as a function of dose for 4MeV nitrogen irradiation in n-type InGaAsP and semi-insulating InP at 77K and RT.](image-url)
Implant isolation of InP-based materials

Chapter Six: Implant isolation using iron and nitrogen

6.4 Implant isolation of n-type InP using nitrogen ions

In this experiment, n-type InP samples of different initial carrier concentrations are implanted with various doses of nitrogen ions at two different implantation temperatures, namely 77K and RT. An energy of 4MeV is used to create an approximately uniform damage within the doped layer and the peak of the damage is placed well inside the substrate.

Three semi-insulating InP wafers were implanted with silicon using multiple dose and energy to create three different initial carrier concentrations and a uniform flat doping profile of approximately 0.75μm. All silicon implantations were done at RT. The implantation conditions to create the three groups of different initial sheet carrier concentrations from $1.51 \times 10^{13}$ to $2.2 \times 10^{14}$cm$^{-2}$ are shown in table 6.3. The projected range and straggle as determined by TRIM are shown in table 6.3. Figure 6.36 shows the uniform silicon atomic concentration profile as determined by TRIM. The doses and energies (see table 6.3) are chosen to obtain the desired uniform concentration values.

Figure 6.35: Evolution of depletion width as a function of dose for n-type InGaAsP irradiated with 4MeV $N^+$ at 77K and RT.
A Si₃N₄ layer was deposited on both the front and back side of the three wafers before annealing. A post-implant annealing temperature of 850°C for 210s was used to activate the n-type dopants. The wafers were then cleaved into samples of size 10mm x 10mm. The clover-leaf pattern was printed on the samples using optical lithography (see section 4.5). The initial sheet resistance, sheet carrier concentration, Hall mobility and percentage activation of the three wafers are given in table 6.4. These samples from group I, II and III are then implanted with 4MeV N⁺ ions at temperatures of 77K and RT using a 2MV High Voltage Engineering Europa (HVEE) implanter.

<table>
<thead>
<tr>
<th>Group no</th>
<th>Dose (cm⁻²)</th>
<th>Energy (keV)</th>
<th>Projected Range (Å)</th>
<th>Longitudinal straggling (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>2.2x10¹²</td>
<td>23</td>
<td>286</td>
<td>216</td>
</tr>
<tr>
<td></td>
<td>8.0x10¹²</td>
<td>125</td>
<td>1352</td>
<td>797</td>
</tr>
<tr>
<td></td>
<td>1.8x10¹³</td>
<td>360</td>
<td>3888</td>
<td>1748</td>
</tr>
<tr>
<td>II</td>
<td>1.48x10¹³</td>
<td>23</td>
<td>286</td>
<td>216</td>
</tr>
<tr>
<td></td>
<td>6.0x10¹³</td>
<td>125</td>
<td>1352</td>
<td>797</td>
</tr>
<tr>
<td></td>
<td>1.8x10¹⁴</td>
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<td>3888</td>
<td>1748</td>
</tr>
<tr>
<td>III</td>
<td>4.8x10¹³</td>
<td>23</td>
<td>286</td>
<td>216</td>
</tr>
<tr>
<td></td>
<td>2.0x10¹⁴</td>
<td>125</td>
<td>1352</td>
<td>797</td>
</tr>
<tr>
<td></td>
<td>4.0x10¹⁴</td>
<td>360</td>
<td>3888</td>
<td>1748</td>
</tr>
</tbody>
</table>

Table 6.3: Implant conditions to create three different initial carrier concentrations. The projected range and longitudinal straggling of 4MeV nitrogen into InP as determined by TRIM are also tabulated.
Table 6.4: Electrical characteristics of the three wafers after silicon implantation and annealing.

<table>
<thead>
<tr>
<th>Group no</th>
<th>Sheet resistance (Ω/□)</th>
<th>Sheet carrier concentration, n_s (cm⁻²)</th>
<th>Hall mobility (cm²/V.s)</th>
<th>Total ion dose n_D (cm⁻²)</th>
<th>% activation = [(n_s/n_D) x 100]</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>161.60</td>
<td>1.51 x 10¹³</td>
<td>2573</td>
<td>2.82 x 10¹³</td>
<td>53.5</td>
</tr>
<tr>
<td>II</td>
<td>46.47</td>
<td>9.83 x 10¹²</td>
<td>1366</td>
<td>2.55 x 10¹⁴</td>
<td>38.5</td>
</tr>
<tr>
<td>III</td>
<td>30.48</td>
<td>2.20 x 10¹⁴</td>
<td>930</td>
<td>6.48 x 10¹⁴</td>
<td>34</td>
</tr>
</tbody>
</table>

Figure 6.36: Si atomic concentration profile for three different initial carrier concentrations as determined by TRIM.
The samples from group I to III were bombarded with 4MeV N⁺ ions to doses in the range of 5x10¹¹ to 3x10¹⁴ cm⁻². Figure 6.37 shows the sheet resistance variation as a function of dose for RT implanted samples from group I to III. The three curves exhibit similar dose dependence except that the ion dose required to achieve the maximum sheet resistance is shifted to the higher doses with the increasing original free sheet carrier concentration. As the dose is increased the sheet resistance is increased from its pre-implanted values of 161.6, 46.47, and 30.48 Ω/□ to a maximum of 5x10⁶, 1x10⁶ and 7.9x10⁵ Ω/□ for samples from group I, II and III respectively. Such an increase in the sheet resistance is caused by the trapping of carriers at defects created by ion irradiation. The threshold doses for maximum sheet resistance are 1x10¹³, 5x10¹³ and 1x10¹⁴ cm⁻² for RT implanted samples of initial sheet carrier concentration of 1.51x10¹³, 9.83 x10¹³ and 2.20x10¹⁴ cm⁻², respectively. The threshold dose is observed to shift towards higher values with the increase of the initial free electron concentration. Further dose accumulation beyond the threshold doses results in a plateau as shown in figure 6.37.

Similar curves are presented for 77K implants in figure 6.38. The sheet resistance increases with accumulation of dose. The sheet resistance is of the same order of magnitude as that of RT implants for samples of different initial sheet carrier concentration at various doses. Hence the threshold doses of 77K implant is similar to RT implant for samples from group I, II and III. Similar trend is observed for nitrogen implantation into n-type InGaAsP. We believe that similar diffusion and recombination of defects occur when samples are brought to air at RT after implantation at 77K. We believe that most of these defects produced at 77K, which are responsible for trapping of carriers, are annealed out when samples are warmed up to RT. Hence, the sheet resistance for samples implanted at RT and 77K are of the same order of magnitude. Table 6.5 shows a summary of the threshold doses and maximum sheet resistivities for 77K and RT implants.

The threshold dose values obtained from the data in figure 6.37 are plotted versus the initial sheet carrier concentration in figure 6.39. A unique linear relationship is found that fits closely the data points obtained from the n-type InP layers. A straight line with a slope of +1 indicates that the initial carrier concentration is directly proportional to the required minimum damage concentration for maximum sheet resistance. This relationship is
discussed in more detail in section 7.3. Knowing the threshold dose at a particular initial sheet carrier concentration, one can easily determine the threshold dose for a different initial carrier concentration from figure 6.39. Similarly for 77K implants, a straight line with a slope of +1 is obtained since the same threshold dose is observed for samples from group I to III (figure 6.39).

<table>
<thead>
<tr>
<th>Group no</th>
<th>77K</th>
<th>RT</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Threshold dose (cm$^2$)</td>
<td>Maximum $R_s$ (Ω/□)</td>
</tr>
<tr>
<td>I</td>
<td>$1\times10^{13}$</td>
<td>$5.2\times10^6$</td>
</tr>
<tr>
<td>II</td>
<td>$5\times10^{13}$</td>
<td>$1.7\times10^6$</td>
</tr>
<tr>
<td>III</td>
<td>$1\times10^{14}$</td>
<td>$1.7\times10^5$</td>
</tr>
</tbody>
</table>

Table 6.5: Threshold dose and maximum sheet resistance of n-InP irradiated at 77K and RT with 4MeV N$^+$ for three different initial sheet carrier concentrations.

Figure 6.37: Sheet resistance of n-InP irradiated at RT with 4MeV N$^+$ as a function of dose for three different initial sheet carrier concentrations.
Figure 6.38: Sheet resistance of n-InP irradiated at 77K with 4MeV N⁺ as a function of dose for three different initial sheet carrier concentrations.

Figure 6.39: Initial sheet carrier concentration of n-InP irradiated at 77K and RT with 4MeV N⁺ as a function of threshold dose. The solid line represents the best fit.
6.5 Summary
In this chapter, we have investigated the effect of dose, initial sheet carrier concentration, substrate temperature and post-implant annealing temperature for iron and nitrogen implantation into InP and InGaAs. An introduction to implant isolation of InGaAsP using nitrogen has also been presented.

High and thermally stable sheet resistance up to $500^\circ$C ($5 \times 10^6 \, \Omega/\square$) has been obtained in both n and p-type InP and InGaAs using 1MeV iron implantation. No diffusion of iron in both InP and InGaAs layers has been observed from SIMS measurements below a post-implant annealing temperature of $550^\circ$C. This ensures its applicability for device fabrication which usually involves high temperature processing. These results are novel since devices consisting of n and/or p-type InP and/or InGaAs layers can use iron implantation for electrical isolation. Also, the immobility of iron below $550^\circ$C in InP and InGaAs layers will cause insignificant effect on the device performance.

The experiments have also revealed substantial changes in electrical and structural properties with increasing iron dose and implantation temperature both in InP and InGaAs. There is an increase in damage for both InP and InGaAs with increasing iron dose. An amorphous layer is formed in InP above a dose of $5 \times 10^{13} \, \text{cm}^2$ for RT and 77K implants. However small damage buildup and no amorphisation of the InP layer are observed up to a dose of $1 \times 10^{15} \, \text{cm}^2$ for $200^\circ$C implants. Similarly, no amorphous layer is formed in InGaAs at a dose of $1 \times 10^{15} \, \text{cm}^2$ for $200^\circ$C implants. The InGaAs layer becomes amorphous above a dose of $5 \times 10^{13} \, \text{cm}^2$ and $1 \times 10^{14} \, \text{cm}^2$ for 77K and RT implants respectively. In-situ dynamic annealing occurs during $200^\circ$C implants so that less lattice dis ordering is observed in both InP and InGaAs. We also observed an increase in the sheet resistance with increasing iron dose in both materials. This is most probably due to higher defects accumulation with increasing iron dose. However $200^\circ$C implants show at least one order of magnitude lower sheet resistance compared to 77K implants for both InP and InGaAs.

A gradual increase in the sheet resistance of n-type InP is observed with increasing nitrogen dose. 77K and RT implants show similar isolation behaviour with increasing dose.
A linear relationship has been obtained between the initial sheet carrier concentration of the doped InP layer and the threshold dose. A higher dose is required to isolate effectively and efficiently a doped InP layer of higher initial sheet carrier concentration. Both 77K and RT implants show similar linear relationship between the initial sheet resistance and the threshold dose.

In the case of n-type InGaAsP, a gradual increase in the sheet resistance with dose is observed for nitrogen implantation at 77K and RT. 77K implants show similar isolation behaviour as a function of dose to RT implants. From the parallel resistor model, we have confirmed that the sheet resistance has accurately been measured.
Chapter Seven

7. Comparative discussions of implant isolation using protons, helium, iron and nitrogen

The previous two chapters presented a systematic study of implant isolation of InP, InGaAs and InGaAsP using hydrogen, helium, iron and nitrogen ions. In the following sections, these results are summarised and compared with the literature. The effect of various implantation parameters is also discussed in detail.

7.1 General summary of the results

Usually In-based HBTs and lasers are made up of several layers of different materials such as InP, InGaAs and InGaAsP. In order to isolate these devices, high sheet resistance is required for all the layers which are usually of different materials. For example, electrical isolation of InP/InGaAs HBTs requires high sheet resistance \((\sim 10^6 \Omega/\square)\) for both InP and InGaAs layers.

Figures 7.1, 7.2 and 7.3 show the evolution of sheet resistance with dose for 250keV protons, 600keV helium and 1MeV iron implantation into n-type InP and both n and p-type InGaAs at different substrate temperatures. For all implantations, we observed a gradual increase in the sheet resistance with dose. From figure 7.1, better electrical isolation is obtained in n-type InP as compared to n-type InGaAs after 250keV proton implantation at RT. However the isolation is thermally stable up to 200\(^\circ\)C for both cases (section 5.1). Boudinov et al [38] reported a similar thermally stable post-implant annealing window up to 200\(^\circ\)C for Si-doped InP using proton implants at a dose of \(3 \times 10^{14} \text{ cm}^{-2}\) at 600keV. They infer that the isolation is due to antisite defects \((\text{In}_{p}\) or \(\text{In}_{p}\) related acceptor like defects), which are stable up to an annealing temperature of 200\(^\circ\)C. Our InP data suggest a similar isolation mechanism as reported by Boudinov et al [38]. We also notice that 200\(^\circ\)C implants do not provide better isolation when compared with RT implants. We believe that most of the antisite defects responsible for trapping of electrons are annealed out during 200\(^\circ\)C implantation via recombination with indium vacancies. In the case of InGaAs, the
Implant isolation of InP-based materials
Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

increase in the sheet resistance with proton dose is also most probably due to radiation-induced crystal defects. But the exact nature of the defects responsible for the high sheet resistance in InGaAs is not known yet and is a topic of research by itself.

In the case of 600keV helium implantation into n-type InP and InGaAs, a maximum sheet resistance of $3 \times 10^6 \, \Omega/\square$ and $1.7 \times 10^5 \, \Omega/\square$ is obtained respectively as shown in figure 7.2. Annealing of the InP samples at 400°C results in a sheet resistance value of $\sim 10^7 \, \Omega/\square$ (section 5.2.1). Higher doses are required to isolate InGaAs as compared to InP material. A thermally stable isolation up to 500°C and 350°C is obtained for InP and InGaAs at the dose where maximum sheet resistance is obtained (section 5.2.1 and 5.2.3). 200°C implantation does not result in higher sheet resistance for both n-type InP and InGaAs. Hence at least four times higher sheet resistance and better thermal stability are obtained in both InP and InGaAs materials implanted with helium as compared to proton.

Figure 7.3 shows the evolution of sheet resistance as a function of 1MeV iron dose for n-type InP and both n- and p-type InGaAs samples implanted 77K, RT and 200°C. A sheet resistance comparison is made among these materials as a function of dose. A high sheet resistance value of $\sim 5 \times 10^6 \, \Omega/\square$ is obtained in all three different materials after 1MeV iron implantation using a dose of $5 \times 10^{14} \, \text{cm}^{-2}$ at 77K. This is the first time, such a high sheet resistance is reported in both n- and p-type InP and InGaAs using iron. A sheet resistance of $10^7 \, \Omega/\square$ can be obtained in both n- and p-type materials after annealing at 400°C - 650°C for 60s. A wide and thermally stable region up to 500°C is obtained for both n- and p-type InP and InGaAs samples implanted at a dose of $5 \times 10^{14} \, \text{cm}^{-2}$ (section 6.1.1 and 6.2.1). These results are novel and reported for the first time to the best of our knowledge. Pearton et al [41] reported a sheet resistance of only $7 \times 10^4 \, \Omega/\square$ in both n- and p-type InGaAs after iron implantation at multiple energy and dose and annealed at 300-400°C for 5mins. The removal of carriers may be due to a combination of both damage and chemical induced compensation. From SIMS measurement, no diffusion of iron is observed within the annealing temperature range where maximum sheet resistance is obtained. Hence the immobility of iron in these materials is a great advantage for device engineers. Iron will not have an effect on the device performance.
Implant isolation of InP-based materials

Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

Hence iron is suitable for isolation of InP/InGaAs HBTs or any devices having InP and InGaAs layers since high sheet resistance ($10^6 \ \Omega/\square$) and a thermally stable isolated region up to an annealing temperature of 500°C can be obtained in both InP and InGaAs.

The isolation behaviour of initially n-type InP and InGaAsP resulting from 4MeV nitrogen implantation at various doses is shown in figure 7.4. A gradual increase in the sheet resistance with dose is observed in both materials. A maximum sheet resistance of $\sim 1 \times 10^6 \ \Omega/\square$ and $\sim 6 \times 10^4 \ \Omega/\square$ is obtained in InP and InGaAsP respectively, at a dose of $1 \times 10^{14}$ cm$^{-2}$. We believe that higher doses ($>1 \times 10^{14}$ cm$^{-2}$) are required to achieve higher electrical isolation ($>10^6 \ \Omega/\square$) in InGaAsP. Xiong et al [60] reported high sheet resistance ($\sim 10^7 \ \Omega/\square$) in n-type InP for nitrogen doses above $5 \times 10^{14}$ cm$^{-2}$.

77K and RT implants for both InP and InGaAsP show similar isolating behaviour. As discussed in chapter 6, we believe that most of the defects created during 77K implantation are annealed out when samples are brought to atmosphere at RT. Hence, the sheet resistance for samples implanted at RT and 77K is of the same order of magnitude. Comedi et al [62] reported similar behaviour after 33keV He$^+$ implantation into n-type InGaAsP. They reported similar sheet resistance values for both RT and 80K implants until a dose of $1 \times 10^{14}$ cm$^{-2}$.

Finally a comment should be made on the nature of defects responsible for electrical isolation in InP and InGaAs. For proton implantation, the irradiation temperature dependency revealed in this study suggests that the centres responsible for isolation are simple point defects such as antisite defects. We observe recovery of the sheet resistance above 200°C which is the transition temperature for annealing the simple point defects as reported by Boudinov et al [38]. For helium and nitrogen implantation, we believe that the centres responsible for the isolation are not simple point defects but the product of defect migration and interaction processes leading to more complex defects. Simple point defects are expected to anneal out using much smaller thermal budgets than those necessary to recover the sheet resistance in InP and InGaAs ($>350^0$C). These defects responsible for trapping free carriers in the case of helium and nitrogen implantation are believed to be complexes of defects. Simpson et al [110] suggest that implanted helium into InP generates
Implant isolation of InP-based materials

Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

Implantation defects which are converted to a more stable trap during annealing. The nature of the traps responsible for the high and thermally stable isolation is not known so far. Xiong et al [60] reported that the formation of complex defects with nitrogen is responsible for the resistivity behaviour in InP. In the case of iron implantation into InP and InGaAs, we believe that carrier removal in these layers is a combination of defects created by irradiation damage and chemical compensation. Carnera et al [96] suggested similar compensation to be responsible for their high sheet resistance values after iron implantation into n-type InP. A. Gaarder et al [111] reported that the presence of a deep Fe acceptor level is responsible for the high sheet resistance in semi-insulating InP. One possible explanation of the high sheet resistance observed in InP and InGaAs may be due to the chemical compensation from the iron substituting the indium sites and the damage compensation created by the iron irradiation itself. However, we are not sure of the exact types of defects which are responsible for isolation in InP and InGaAs after iron irradiation.

Figure 7.1: Comparison of sheet resistance curves for electrical isolation of n-type InP and InGaAs at RT and 200°C using 250keV proton at different doses.
Implant isolation of InP-based materials

Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

Figure 7.2: Comparison of sheet resistance curves for electrical isolation of n-type InP and InGaAs at RT using 600keV helium at different doses.

Figure 7.3: Comparison of sheet resistance curves for electrical isolation of n-type InP and both n and p-type InGaAs at 77K, RT and 200°C using 1MeV iron at different doses.
Implant isolation of InP-based materials

Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

7.2 Influence of implantation parameters

In this section, a summary of the effect of different implantation parameters such as ion species, energy, dose, initial sheet carrier concentration and implantation temperature on implant isolation in InP and InGaAs is presented based on the results reported in chapters 5 and 6.

Ion species

Based on data reported in chapters 5 and 6, we observe that better and thermally stable electrical isolation is obtained with heavier ion species (Fe\(^+\)) as compared to lighter ones (H\(^+\), He\(^+\) and N\(^+\)). Heavier ions are expected to create more damage per ion than lighter ions. Hence the mass of the ion specie has an effect on the electrical isolation of InP and InGaAs. An inverse linear relationship exists between the ion mass and the dose required for effective isolation of InP. This relationship is discussed in more detail in section 7.3. A similar relationship is observed in other materials such as GaAs and GaN [112-114].
Implant isolation of InP-based materials

Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

Energy

Energy is another important factor which has to be considered when choosing the right implant conditions for effective electrical isolation. By changing the energy, the position of the damage profile can be changed. Hence, the peak of the damage profile can be placed inside or outside the layer to be isolated by varying the energy. Most of the work presented in this report is based on the energies which are chosen to create a uniform damage concentration in the layer to be isolated with the peak of the damage profile deep inside the substrate. An energy of 250keV for protons and 600keV for helium ions produces a similar constant damage level in the InP and InGaAs layers to be isolated as does 4MeV energy for nitrogen. At such energies, the profile of the damage distribution is essentially uniform throughout the doped region. This scheme has the advantage of compensating near surface and bulk devices at the same time. Implantation using multiple energy to create an approximately uniform damage distribution is another scheme, which is mostly reported in the literature (see chapter 3). This scheme involves more time and money since it requires multiple implantations.

We have also investigated the effect of placing the peak of the damage distribution inside and outside the InP doped region (section 5.2.2). The sheet resistance is three times higher when the peak of the damage is placed inside the doped layer than outside for different helium doses. We believe that the dose required for effective isolation is lower when the damage is placed inside than outside the doped layer. Our results show similar effective dose ($1 \times 10^{14} \text{ cm}^{-2}$) for both cases since the measurement of the sheet resistance for samples having damage outside the doped layer is inaccurate. From the parallel resistor model, the substrate becomes conductive above a helium dose of $1 \times 10^{15} \text{ cm}^{-2}$. Since the peak of the damage is placed in the substrate which becomes quite conductive at such a high dose (section 5.3), the measured sheet resistance is most probably coming from the substrate itself.

In the case of iron implant isolation, an energy of 1MeV is chosen to place most of the iron ions within the doped region. In bulk materials, highly resistive behaviour is usually obtained by doping the crystal with iron during the growth [94]. These iron atoms occupy indium sites substitutionally and act as deep acceptor centres for free electrons [95].
Hence an iron energy of 1MeV is used to take advantage of both damage and chemical compensation to some extent.

**Dose**

Dose is a very important parameter in isolation of InP and InGaAs. It is related to the concentration of damage created within the doped region. The sheet resistance of the doped region is initially low compared to the substrate for low doses (section 7.1). A gradual increase in the sheet resistance is observed with increase in the dose for all the ion species ($H^+$, $He^+$, $N^+$ and $Fe^+$) in both InP and InGaAs. This increase in the sheet resistance is believed to be the result of the carrier removal process which traps the free carriers. A threshold dose, which is defined as the dose when maximum sheet resistance is reached, is obtained in both InP and InGaAs samples. At this dose, most of the free electrons from the dopants are trapped at the defect sites. This threshold dose has a different value for different ion species implanted into InP and InGaAs (section 7.1).

**Initial sheet carrier concentration**

The effect of initial sheet carrier concentration of the doped region is also investigated. Since InP and InGaAs doped layers are usually of different initial free carrier concentration, it is important to investigate its effect on the isolation behaviour in these materials. It is found that higher doses are required for effective isolation of InP doped regions of higher initial free carrier concentrations. A linear relationship is found between the initial sheet carrier concentration and the threshold dose for nitrogen implantation in n-type InP (section 6.4). A similar linear relationship is expected to exist in the case of InGaAs. Knowing the initial sheet carrier concentration of the doped layer, the dose required for effective isolation of the layer can be determined from this linear relationship (section 7.3).

**Implantation temperature**

An investigation of the effect of substrate temperature during implantation has been done using different ion species implanted into InP and InGaAs (chapters 5 and 6). Such experiments are done in order to achieve better electrical isolation as observed in GaAs where 200-350°C implants show thermally stable and higher sheet resistance than RT.
Implant isolation of InP-based materials

Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

Ahmed et al [90,112] have claimed that different trap structures responsible for higher and thermally stable sheet resistance in GaAs are generated during 200°C implants compared with RT implants. It is also worth noting that most previous studies on the implant isolation of InP and InGaAs have, to the best of our knowledge been performed at room temperature (section 3.8).

In the case of 250keV proton implantation into n-type InP and InGaAs, the sheet resistance for a 200°C implant is an order of magnitude lower than that for a RT implant. When RT implanted samples are annealed at 200°C, the sheet resistance decreases to the same order of magnitude as that of 200°C implants. Hence, the data suggests that these defects responsible for high electrical isolation in both InP and InGaAs are annealed out at 200°C. For 55keV and 600keV helium implantation into InP at the threshold dose (2x10^14 cm^-2), the implantation temperature is an insensitive parameter for electrical isolation. The as-implanted sheet resistance value is approximately the same for the three different substrate temperatures (RT, 100°C and 200°C). We infer that no new thermally stable defects are formed during 100°C and 200°C implants.

For implant isolation of InP and InGaAs using iron, the data shows that 200°C implants do not result in better electrical isolation than RT or 77K implants. We believe that most of the defects responsible for the high electrical isolation are annealed out during 200°C and no new thermally stable defects are created at this temperature. These data are in apparent contrast with the lower damage accumulation (as shown by RBS) that occurs by virtue of in situ dynamic annealing during hot implants. RBS on InP and InGaAs samples implanted with 1MeV iron shows that substantial in situ dynamic annealing occurs during implantation at 200°C. For 100°C implants, InGaAs shows much lower damage as compared to InP. In situ dynamic annealing occurs readily in InGaAs at this temperature whereas as-implanted InP samples still remain highly damaged.

In simplest form, the lattice damage processes involved in ion implantation can be classified into two main categories: (1) defect generation by collision cascades, and (2) defect migration and recombination due to associated thermal effects. The final state of implantation is just the result of competition between these two processes. In low
temperature implantation (<RT), the first process may dominate the second one, and then amorphisation can take place; alternatively in high temperature implantation, the latter process may overcome the first one. Lattice damage at a particular temperature is also material dependent. Hence, in the case of 200°C implants for InGaAs and InP and 100°C implants for InGaAs, strong in situ annealing is present and sufficient lattice disorder cannot be sustained to form an amorphous zone in these samples as a result of ion implantation.

7.3 Isolation matrix

In this section, the model proposed by Ahmed [112] is applied to InP. From sections 5.1, 5.2 and 6.4, the data suggest similar electrical isolation behaviour at RT as a function of ion mass, original carrier concentration and ion dose like GaAs, AlGaAs, InGaP and GaN [112,113,114,117,118]. Based on the data presented in chapter 5 and 6, the following conclusions can be deducted:

(a) The threshold dose increases with increase in original free carrier concentration of the layer to be isolated for a particular ion specie.

(b) Threshold dose, D_{th} (the dose required to achieve maximum sheet resistance) is linearly proportional to the original free carrier concentration of the layer to be isolated.

The following relationship between dose and the initial sheet carrier concentration is deduced:

\[ n_{s0} \propto D \]  

\[ n_{s0} = k \cdot D \]

where \( n_{s0} \) is the initial sheet carrier concentration before implant isolation (unit is \( \text{e}^-/\text{cm}^2 \))

\( D \) is the dose required to isolate the structure (unit is ion/\( \text{cm}^2 \))

Rearranging equation 7.1,

\[ n_{s0} = k \cdot D \]

\( k \) is a constant of proportionality with a slope of +1 (section 6.4)
Implant isolation of InP-based materials

Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

In figure 7.5, the initial sheet carrier concentration values are plotted versus the threshold doses for nitrogen, helium and proton implantation into n-type InP. Although, the complete electrical isolation data of protons and helium as a function of initial carrier concentration is not available, we believe that the data should follow the same relationship as proved by other people in the case of GaAs, InGaP and AlGaAs (figures 7.6, 7.7 and 7.8). Ahmed [119] has demonstrated similar linear relationship between the initial sheet carrier concentration and the threshold dose for different ion species implanted into n-type GaAs (figure 7.6). We have shown that the electrical isolation data of nitrogen fits quite well the straight line. For the first time this relationship is shown in InP. We believe that the electrical isolation data for helium and proton will follow the same trend as nitrogen as shown in figure 7.5.

Figure 7.9 shows the data reported by Ahmed [112] on the estimated atomic displacement (calculated using TRIM) produced by hydrogen, helium, boron and oxygen implantation into four different doped GaAs layers versus the threshold doses. Figure 7.9 reveals that the threshold dose decreases with the increasing atomic displacements. The latter is determined by ion mass and energy. For samples of similar initial sheet carrier concentrations, a lower dose is required for effective electrical isolation with increasing ion mass. Ions of heavier mass create more atomic displacements than those of lighter mass. A straight line fit of slope -1 is reported by Ahmed [112]. This means that the number of atomic displacements is inversely proportional to the threshold dose. For identical samples, an increase in the atomic displacements along the depth of the conductive layer results in a decrease in the threshold dose to isolate samples of similar carrier concentration. For samples of similar initial sheet carrier concentrations, a lower dose is required for effective electrical isolation with increasing ion mass. Ions of heavier mass create more atomic displacements than those of lighter mass. Boudinov et al [114] reported similar relationship between the number of atomic displacements and the threshold dose for n-type GaN. In figure 7.10, our experimental nitrogen data could be fitted to a straight line of slope -1. Although the proton, helium and oxygen data are not available to confirm the inverse relationship between the threshold dose and the number of atomic displacements, we believe that this relationship should also hold for n-type InP.
Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

**Figure 7.5:** Initial sheet carrier concentration of n-InP irradiated at RT with 250keV H⁺, 600keV He⁺ and 4MeV N⁺ as a function of threshold dose. The solid lines represent the best fit.

**Figure 7.6:** Initial sheet carrier concentration of n-GaAs irradiated at RT with 250keV H⁺, 600keV He⁺, 1.5MeV B⁺ and 2MeV O⁺ as a function of threshold dose. The dotted lines represent the best fit [114].
Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

**Figure 7.7:** Initial sheet carrier concentration of n-InGaP irradiated at RT with 270keV He$^+$ as a function of threshold dose. The solid lines represent the best fit [118].

**Figure 7.8:** Initial sheet carrier concentration of p-AlGaAs irradiated at RT with 600keV H$^+$ as a function of threshold dose. The solid lines represent the best fit [117].
Figure 7.9: Estimated atomic displacement for n-GaAs of four different initial sheet carrier concentrations irradiated at RT with 250keV H⁺, 600keV He⁺, 1.5MeV B⁺ and 2MeV O⁺ as a function of threshold dose [114].

Figure 7.10: Estimated atomic displacement for n-InP of three different initial sheet carrier concentrations irradiated at RT with 4MeV N⁺ as a function of threshold dose.
Implant isolation of InP-based materials

Chapter Seven: Comparative discussions of implant isolation using protons, helium, iron and nitrogen

In summary, we have discussed an overall picture of the results obtained in chapters 5 and 6. A comparison has been made between implant isolation of InP, InGaAs and InGaAsP layers which are commonly present in InP based devices. The effect of ion species, energy, dose, initial sheet carrier concentration and implantation temperature based on our results in chapters 5 and 6 for implant isolation of InP and InGaAs has been discussed. A similar isolation matrix as found in GaAs, GaN, AlGaAs and InGaP is also valid in the case of n-type InP. These results may be very useful in the production of implant conditions required to produce high resistance material in a range of III-V compounds.
Chapter Eight

8. Conclusion and further work

8.1 Conclusion

This thesis focuses on the electrical isolation of both n- and p-type InP and InGaAs by implantation of ion species such as protons, helium, nitrogen and iron. A brief introduction to implant isolation of n-type InGaAsP using nitrogen is also presented. Both n- and p-type InP, InGaAs and InGaAsP layers were grown by SSMBE using silicon and zinc dopants respectively. Some of the n-type InP samples were doped with silicon using multiple-energy implantation in order to create a uniform dopant distribution throughout the n-type region. The electrical method of characterisation of the isolating layers is discussed in detail (chapter 4).

Thus the ultimate goal of this work was to investigate the effect of ion species, ion mass, dose, energy, substrate temperature during implantation, post-implant annealing cycles and initial carrier concentration of the layer to be isolated on the quality of isolation in terms of optimisation and thermal stability. Semi-insulating InP samples were also bombarded with different ion species and the sheet resistance was measured. From experiments using the semi-insulating InP, a parallel resistor model has been formulated to confirm the accuracy and reliability of the measurements of the isolated n- and p-type doped layer. A major part of this work is dedicated to the investigation of the effect of implantation temperature on the electrical characteristics of the isolated n- and p-type doped layers. The effect of variable doses on the other implantation conditions is also examined in detail.

Rutherford backscattering spectrometry was used on iron implanted samples to detect and quantify the effect of implant parameters on defect concentration. Secondary ion mass spectroscopy was also used to measure atomic profiles of implanted ions in a selection of samples providing key information related to two aspects. Firstly, to check that the desired range and doping concentration profile have been successfully attained and secondly to
investigate the effects of iron diffusion for samples annealed at different temperatures with particular emphasis on raised substrate temperature implantation. No diffusion of iron is observed below a post-implant annealing temperature of 550\(^\circ\) in both n-type InP and InGaAs layers.

These results are very useful to choose the right implant conditions in order to provide effective electrical isolation of In-based devices such as HEMTs, HBTs and lasers. They are novel and have applications to the semiconductor industry.

We can conclude from this project the following:

- RT and 77K implants provide a more effective and thermally stable isolation as compared to 200\(^\circ\)C. We believe that the defects responsible for the high sheet resistance during RT and 77K implants are annealed out during 200\(^\circ\)C implant.
- Proton implantation results in poor and less thermally stable isolation in both n-type InP and InGaAs of initial carrier concentration, 1\times10^{17} \text{ cm}^{-3}. RT implants show two orders of magnitude higher sheet resistance as compared to 200\(^\circ\)C implants at doses above 5\times10^{13} \text{ cm}^{-2} and 5\times10^{14} \text{ cm}^{-2} for InP and InGaAs respectively.
- Helium implantation shows good and thermally stable isolation in n-type InP as compared to n-type InGaAs of initial carrier concentration of \sim1\times10^{17} \text{ cm}^{-3}. Higher damage accumulation within the doped layer results in higher sheet resistance as discussed in section 5.2.2.
- The effect of helium implantation into semi-insulating InP is discussed in section 5.3. The substrate becomes appreciably conductive above a dose of 1\times10^{16} \text{ cm}^{-2} for RT implants. However high sheet resistance of the semi-insulating InP is still maintained at a dose of 1\times10^{16} \text{ cm}^{-2} for 200\(^\circ\)C implants. A parallel resistor model is then formulated to check the accuracy of the sheet resistance measurements.
- A heavier ion species such as iron gives high sheet resistance values in both n and p-type InP and InGaAs. The effect of dose, implantation temperature and post-implant annealing temperature is discussed in detail in chapter 6. It is found that the choice of the right implant conditions is important in order to obtain an effective and reproducible isolation.
A brief introduction to implant isolation of n-type InGaAsP using 4MeV nitrogen is presented in section 6.3. A maximum sheet resistance of $7 \times 10^4 \, \Omega/\square$ is obtained at RT using a dose of $1 \times 10^{14} \, \text{cm}^{-2}$. Higher doses ($>1 \times 10^{14} \, \text{cm}^{-2}$) may be required to obtain better isolation ($>10^6 \, \Omega/\square$) in this material. Implantation at 77K does not show better isolation as compared to RT implants.

Nitrogen implantation also shows good electrical isolation in InP. RT and 77K implants show same order of magnitude in the sheet resistance as a function of nitrogen dose. The effect of initial sheet carrier concentration is also investigated. It is found that the ion dose required to achieve the maximum sheet resistance shifts to the higher doses with increasing initial sheet carrier concentration.

Based on the obtained data, it is found that the ion dose required for effective and efficient isolation of n-type InP is linearly dependent on the initial sheet carrier concentration and reciprocally dependent on the number of atomic displacements produced in the InP layer to be isolated.

8.2 Suggestions for further work
The main aim of this work is to develop recipes for isolation of both n- and p-type InP and InGaAs and n-type InGaAsP. In spite of the encouraging results obtained, further studies are important especially in the following:

- Further implant isolation studies need to be done on both n- and p-type InGaAsP to obtain high and thermally stable electrical isolation in this material. This will help to isolate effectively devices having InGaAsP layers.

- The exact nature of the defects responsible for the high sheet resistance in both n- and p-type InP, InGaAs and InGaAsP is not known yet. Some spectroscopic techniques such as Deep Level Transient Spectroscopy (DLTS), Photoconductive Frequency Resolved Spectroscopy (PCFRS), Positron Annihilation Spectroscopy (PAS) and Photoluminescence (PL) can be used to identify the nature of defects responsible for the isolation in these materials. This will help in the defect engineering of devices made from these materials.

- The isolation matrix experiment can be done using other ion species such as hydrogen, boron and oxygen. This will confirm the validity of the predictive model.
This isolation matrix experiment can also be done on InGaAs. This will have useful applicability to devices such as HEMTs and HBTs. This will help the device engineers to choose the right implant conditions for the isolation of their devices.

This work can be extended to three-dimensional integration of devices which requires the creation of an additional buried high resistivity layer within the epilayers. Such a process can be achieved by creating defects which are thermally stable at the end of ion range as compared to those created along the ion tracks. The appropriate annealing window must then be found which removes all defects except those at the end of the ion range.
Implant isolation of InP-based materials

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Implant isolation of InP-based materials

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