Characterization and Control of Crystallographic Defects in Thin film SIMOX Materials

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Abstract

Crystallographic defects present in the silicon overlayer of thin (< 1000 Å) SIMOX material have been characterized using a newly developed etchant and by transmission electron microscopy. Rutherford backscattering spectroscopy has also been used to determine the chemical composition and thickness of the synthesised layers. The thin SIMOX layers were produced by two different methods, namely (i) sacrificial oxidation of the silicon overlayer of thick (2000 Å) SIMOX films and (ii) low energy O⁺ implantation.

The main crystallographic defects present in materials prepared by sacrificial oxidation are threading dislocations and oxidation induced stacking faults (OISF) whose density and size depends mainly upon the oxidation conditions (temperature, time) and also on the number of nucleation sites present before oxidation. The nucleation of these OISF has been investigated and it has been observed that stacking fault (SF) complexes are the main nucleation sites. The density of threading dislocations within the Si overlayer does not increase during oxidation. The lowest density of OISF (5.0x10² cm⁻²) was observed in layers thinned by dry oxidation although some layers contained up to 5.0x10⁵ cm⁻². This difference in OISF densities was attributed to differences in the densities of the SF complexes before oxidation.

Thin film SIMOX structures formed by low energy implantations also contain two principal defect types namely, threading dislocations and stacking fault complexes, where the densities depend upon the implantation and annealing conditions. A low density of threading dislocations is only obtained when parameters, such as dose and implantation temperature are optimised. Furthermore, it is also observed that thermal and mechanical stresses produced in the silicon overlayer during implantation or annealing, need to be minimised in order to obtain low defect density material. It has been shown by whole wafer defect mapping of six inch SIMOX wafers implanted under optimised conditions, that a uniform distribution of defects can be achieved, having an average defect density as low as 1.0x10⁴ cm⁻².

Additionally, the effects of implantation damage on the formation of secondary defects have also been investigated. The results have shown that the coalescence of point defects generated during implantation produce a high density of dislocation loops that, depending upon the annealing treatment, develop into threading dislocations or OISF.

These experiments confirm that careful optimisation of the processing conditions, such as implantation temperature and dose uniformity, can significantly reduce defect densities thus enhancing the prospect of thin film SIMOX as a suitable substrate for fully depleted MOS devices.
Dedicated to my parents
Barbara and Antonio,
to my wife Danielle,
to my brothers & sisters,
Antonio, Manolo, Carla, Luciana
and to Guillermo Antunez de Mayolo.
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Abbreviations

AES - Auger Electron Spectroscopy
BOX - Buried Oxide
BF - Bright Field
CMOS - Complementary Metal Oxide Semiconductor
CVD - Chemical Vapour Deposition
DF - Dark Field
DL - Dislocation loop
ELO - Epitaxial Lateral Overgrowth
FIPOS - Full Insulation by Porous Oxidised Silicon
FWHM - Full Width Half Maximum
HTA - High Thermal Annealing
IIB - Ion Implanted Boundary
IR - Infra Red
LSI - Large Scale Integration
MBE - Molecular Beam Epitaxy
MOS - Metal Oxide Semiconductor
NSF - Narrow Stacking Fault
OISF - Oxidation Induced Stacking Fault
OM - Optical Microscopy
PVTEM - Plan View Transmission Electron Microscopy
RBS - Rutherford Backscattering Spectroscopy
RED - Radiation Enhanced Diffusion
SDL - Semi Dislocation Loop
SEM - Scanning Electron Microscopy
SF - Stacking Fault
SFP - Stacking Fault Pyramidal
SFT - Stacking Fault Tetrahedron
SIA - Sequential Implants and Anneals
SIMOX - Separation by Implanted Oxygen
SIMS - Secondary Ion Mass Spectroscopy
SOI - Silicon On Insulator
SOS - Silicon On Sapphire
SPE - Solid Phase Epitaxy
TD - Threading Dislocation
TEM - Transmission Electron Microscopy
VLSI - Very Large Scale Integration
WB - Weak Beam
XRD - X-Ray Diffraction
XTEM - Cross Section Transmission Electron Microscopy
ZMR - Zone Melt and Regrowth
Contents

Abstract

Acknowledgements

Abbreviations

Chapter One: Introduction

1. Introduction 1

Chapter Two: Literature Survey

2.1 Introduction 7

2.2 Evolution of SIMOX technology 8

Chapter Three: Background

3.1 Introduction 22

3.2 Ion beam synthesis of SiO₂

3.2.1 Formation of a buried oxide layer 24

3.3 Crystallographic defects in oxygen implanted silicon

3.3.1 Radiation damage as a source of secondary defects 34

3.3.2 Internal oxidation of silicon as a source of defects 37

Chapter Four: Experimental details

4.1 Sample description

4.1.1 Oxygen implantations at the University of Surrey 40

4.1.2 Oxygen implantations at Eaton Corporation 46

4.1.3 Standard SIMOX substrates 48

4.1.4 BF₂⁺, Ge⁺ and Si⁺ implantations 49

4.1.5 Sacrificial oxidation 52

4.2 Characterization methods

4.2.1 Chemical defect etching 54

4.2.1.1 Etching experiment details 55

4.2.2 TEM 57

4.2.3 RBS 57

4.2.4 Other methods 58
Chapter Five: New etchant

5.1 Introduction 59
5.2 Development of an optimum defect etchant for thin Si layer 60
  5.2.1 Dilute silicon etchants 60
  5.2.2 New etchant 72
5.3 Distribution of defects in thick SIMOX 81
  5.3.1 Lateral distribution of defect 81
  5.3.2 Defect distribution-depth 84

Chapter Six: Results I - Sacrificially oxidised SIMOX

6.1 Introduction 92
6.2 Defects in sacrificially oxidised SIMOX 93
  6.2.1 Crystallographic defects in standard SIMOX 93
  6.2.2 Crystallographic defects in sacrificially oxidised SIMOX 93
6.3 Sacrificial oxidation of ion implanted SIMOX materials 110

Chapter Seven: Results II - Low energy oxygen implants

7.1 Introduction 126
7.2 Wafers prepared by Eaton Corporation 127
  7.2.1 General description 127
  7.2.2 Microstructure 128
  7.2.3 Defect distribution-depth 132
  7.2.4 Defect distribution-lateral 134
  7.2.5 Crystallographic defects in the silicon substrate 144
  7.2.6 Defects associated with edge effects 145
7.3 Wafers prepared at the University of Surrey 147
  7.3.1 General description 147
  7.3.2 SIMOX structures 148
  7.3.3 Defect distribution-lateral 150
  7.3.4 Evolution of threading dislocations during annealing 157
  7.3.5 Defect density- dependence upon implant temperature 158
  7.3.6 Edge effects-stress related 160
  7.3.7 Defect densities in the silicon overlayer 165
  7.3.8 Crystallographic defects in the Si substrate 167
## Chapter Eight: Discussion

8.1 Introduction 170
8.2 Crystallographic defects in sacrificially oxidised SIMOX 171
  8.2.1 Nucleation process of OISF in SIMOX 171
  8.2.2 Evolution of OISF during oxidation 179
8.3 Crystallographic defects in low energy and low dose SIMOX 187
  8.3.1 Effects of thermal stresses on the nucleation of defects in SIMOX 189
  8.3.2 Effects of contamination on the formation of crystallographic defects 191

## Chapter Nine: Conclusions and Suggestions for future work

9.1 Crystallographic defects in SIMOX thinned by sacrificial oxidation 194
9.2 Crystallographic defect in low energy, low dose SIMOX 195
  9.2.1 SIMOX wafers produced at the University of Surrey 195
  9.2.2 SIMOX wafers produced at Eaton Corporation 196
9.3 Suggestions for future work 196

## References

198

## Appendix A - SOI Technologies

208

## Appendix B - Chemical defect etching

211

## Appendix C - Dislocations and plastic flow in the diamond structure

221

## Appendix D - Preparation of TEM samples

232

## Appendix E - List of sacrificial oxidised samples

234

## Publications

243
Chapter One: Introduction

Complementary metal oxide semiconductor (CMOS) technology is the most common device architecture used for very large scale integration (VLSI) microelectronics and although bulk silicon is still used as its main substrate, it is expected that for the next generation devices (say < 0.25 μm), silicon on insulator (SOI) substrates could replace bulk silicon in order to reduce parasitic effects between neighbouring devices and to increase speed [1]. Indeed, the improved performance of devices formed on SOI substrates results from reduced parasitic capacitance, latch-up suppression due to the dielectric insulation and inherent radiation hardness[2]. In addition it is possible that SOI substrates will be used, in the near future, for low power circuits for "hand held" equipments [3].

During the last thirty years, a great deal of work has been dedicated to improve the quality of SOI substrates. Indeed, during this time several SOI technologies were developed and among the most important are Silicon on Sapphire[4](SOS), Fully
Insulated Porous Oxidized Silicon (FIPOS), Wafer Bonding, Zone Melt Regrowth (ZMR) and Separation by Implanted Oxygen (SIMOX). However, in spite of all the technological advances made during the 1980s these materials are only now just beginning to reach a desirable quality (defect density, lateral uniformity) to compete with bulk Si technology (appendix A). Control of crystal quality, reduction of wafer cost and designing of new compatible architectures are some of the main problems currently being addressed (see appendix A).

Among all of the SOI technologies, SIMOX is arguably the most promising material for high complexity CMOS circuits and even bipolar applications. It has been successfully used as the substrate for 1 Mbit SRAM memories and many others. Furthermore, fully depleted MOS devices have been successfully fabricated in thin film SIMOX (< 1000 Å) and it has been shown that higher drive currents and frequencies of operation can be achieved and that deleterious effects, arising from parasitic capacitances and short channel effects, can be reduced leading to further improvement to circuit performance.

SIMOX substrates are fabricated by a two step process which involves the implantation of high doses of energetic oxygen ions into (100) silicon wafers followed by a post-implantation thermal annealing used to annihilate defects and to redistribute the implanted oxygen. Standard SIMOX substrates having a silicon film thickness of about 3000 Å and an oxide thickness of about 4000 Å are normally produced by implanting a dose of $1.8 \times 10^{18}$ O$^+$ cm$^{-2}$ at energies of 180 keV to 200 keV and annealing at 1300°C for typically six hours in an argon plus oxygen ambient. Thin film SIMOX can be obtained either by sacrificial thermal oxidation of the overlayer of thick SIMOX or by implanting with lower energies and doses.

Introduction
As with other SOI technologies, SIMOX has still to overcome some obstacles before becoming the preferred SOI substrate for mainstream CMOS technology. The main problems facing SIMOX technology are the presence of residual defects (usually $>10^4 \text{cm}^2$) in the silicon overlayer, the quality of the buried oxide layer and the wafer cost [13]. Although the quality of the silicon overlayer has improved dramatically from the early days (from $10^9 \text{cm}^2$ to $10^4 \text{cm}^2$ and lower) it still needs to be reduced to values typical of bulk silicon before it will be acceptable for bipolar applications. In order to achieve this goal (defect free SIMOX) it is important to identify the mechanisms of formation of crystallographic defects in SIMOX.

Paradoxically, the success in producing relatively low defect density materials ($10^4 \text{cm}^2$) created new problems which impeded studies of the mechanisms of formation of crystallographic defects due to the lack or inadequacies of available characterization methods to delineate such low defect densities.

Among the direct characterization methods available to delineate crystallographic defects in silicon, the most versatile are plan view transmission electron microscopy (PVTEM), cross-section transmission electron microscopy (XTEM) and X-ray topography. Arguably, XTEM is the best technique, however it is unfortunate that the high magnification which is necessary to resolve the defects gives poor statistics for low defect densities ($<10^7 \text{cm}^2$) and, due to the complexity of sample preparation, the throughput of samples is generally low. The spatial resolution of plan view TEM means that it is impractical to use the technique to determine the areal density and the defect population in samples containing defect densities lower than $10^4 \text{cm}^2$.

Introduction
X-ray topography[14] is another direct method extensively used to characterize defects in bulk silicon. Like TEM, the contrast observed originates from regions that have a non-periodical arrangement of atoms and therefore, do not obey the Bragg law[15]. The main difference between X-ray and electron diffraction is the radiation penetration depth, which in the case of X-rays is almost two orders of magnitude greater (100 μm) than electron penetration [16]. This feature makes the use of X-ray diffraction topography extremely difficult for the analysis of thin films since the defect contrast from the thin films (< 1.0 μm) is extremely weak compared with the contrast of defects present in the bulk of the material. Therefore, although very attractive, since large areas can be analysed at the same time, X-ray topography is generally not practical for the analysis of defects in the silicon overlayer of SIMOX substrates.

Indirect methods such as RBS/channeling [17], photo luminescence[18], device leakage currents[19] and chemical defect etching[20] could be used as alternative methods to TEM analyses since these methods are sensitive to the presence of crystallographic defects. However, only chemical defect etching can be used to accurately estimate the density of crystallographic defects. The method is based upon the formation of etch pits at the intersection of defects with the surface. Its advantage is that it is a simple technique and only requires SEM or even optical microscopy to visualise the etch pits and, therefore, can be used to analyse samples with a wide range of defect densities. Indeed, chemical etching may be used to analyse samples containing a very low density (say 10 cm⁻²) up to a density of 10⁹ cm⁻² (see Fig.1.1).
Fig. 1.1 - Schematic showing the range of defect densities for the different characterization methods.

However, standard bulk silicon defect etchants can not be applied directly to the study of thin film SOI substrates because they require at least 1 μm of Si to be removed in order to form resolvable etch pits (by optical microscopy). If less silicon is removed the technique is found to be unreliable. Indeed, the reliability of the etchant will depend upon its effectiveness to nucleate an etch pit at each crystallographic defect and this will depend upon the actual thickness of the silicon film since the probability to nucleate all defects will increase with etching time, that is to say with thickness of silicon removed.

In this project a systematic analysis of different silicon etchant systems has been carried out in order to identify and optimise an etchant suitable for thin silicon films (≤ 1000 Å). This etchant has subsequently been used to identify crystallographic defects and to facilitate a study of the evolution and annihilation of extended defects.

The overall aim of this project was to characterize the extended defects in thin film SIMOX material and determine methods to control the nucleation and growth of these defects. For this purpose a new defect etchant suitable for ultra thin silicon films has been developed. The development of a novel characterization technique which is at the same
time simple, quick and reliable opens the way not only to assess the quality of materials produced in different ways but also enables the processing conditions to be optimised in order to control crystallographic defect formation and, therefore, to obtain materials with lower defect densities.

Furthermore, the availability of this new characterization technique can be used, in the future, not only in SOI technology but also in other group IV materials such as Si/SiGe hetero-epitaxy structures or ion beam synthesised Si/silicide structures which also call for analysis of thin films of silicon.

This thesis contains nine chapters including this introductory one. In chapter 2, we present a literature survey of publications relevant to this work. A background to the basic physical aspects of ion beam synthesis of SiO₂ and to the main sources of defects in SIMOX are presented in chapter 3 whilst in chapter 4, we describe the experimental conditions and procedures used to prepare and analyse the thin films. In chapters 5, 6 and 7 we present the experimental results. In chapter 5, we investigate and optimise a new etchant suitable for thin silicon overlayers and in chapter 6, we apply it to the analysis of thin film SIMOX prepared by sacrificial oxidation. In chapter 7 we report further use of the new etchant together with TEM analysis of thin film SIMOX produced by low energy and low dose oxygen implantation. In chapter 8, we discuss the results presented in chapters 6 and 7. Finally, in chapter 9 we present our conclusion and suggestions for future work.
Chapter Two: Literature Survey

2.1 Introduction.

In this chapter we present a literature survey of the processing and materials characterization of SIMOX structures. We first review the advances made in SIMOX technology from the early idea of using ion implantation as a method to produce a buried layer of SiO\textsubscript{2} up to todays ultra thin SIMOX structures. We also report on the characterization methods that have been used to delineate crystallographic defects in the SIMOX materials.
The first report on ion implantation as a method to introduce impurities in a controllable way into a substrate was made by Smith et al. [21] in 1956. However, it was eleven years later (1967) that Watanabe and Tooi [22] reported for the first time, the successful synthesis of \( \text{SiO}_2 \) by \( \text{O}^+ \) implantation into bulk silicon. They used a relatively low energy (40 keV) and high dose (2.0x10^{18} \text{ O}^+ \text{cm}^{-2}) and successfully formed a surface oxide which was identified by infra-red (IR) spectroscopy and ellipsometry measurements. They speculated on the use of ion beam synthesis as an alternative to thermal oxidation for the formation of thin oxide layers. In the same year, Pavlov and Shitova [23] analyzed the properties of the synthesised oxide using infra-red spectroscopy and electron diffraction and concluded that the oxide layer consisted of a mixture of \( \text{SiO}_2 \) and sub oxides (\( \text{SiO}_x \), where \( x < 2 \)).

At this time, the implantation energies were low, typically 40 keV to 60 keV, because the main objective was to produce a surface oxide. During the early 1970s, the quality of the synthesised oxides was further assessed and the consensus was that they were inferior to thermal oxides [24]. However, IR spectroscopy and I-V measurements [25] showed that at least some of their properties (IR and passivation properties) were comparable to a thermal oxide.

In 1977, the use of a synthesised buried oxide layer to isolate semiconductor devices was proposed by Badawi and Anand [26]. They suggested that high energy and a medium dose would enable a device worthy buried oxide layer to be formed. In the following year, Izumi et al. [27] reported the successful synthesis of a buried \( \text{SiO}_2 \) layer formed by...
implanting a high dose ($\phi = 2.0 \times 10^{18}$ O$^+$ cm$^{-2}$) of energetic oxygen ions (E= 150 keV) into (100) silicon followed by an anneal at 1150°C for 2 hours. Afterwards, they grew an epitaxial layer on top of the silicon overlayer in which CMOS devices, having operation speeds twice as faster as devices on bulk silicon, were successfully fabricated. They proposed the acronym SIMOX (Separation by IMplanted OXygen) for this new SOI technology.

Subsequently, several groups[28,29] were involved in the formation and analysis of SIMOX structures. The University of Surrey in collaboration with Middlesex Polytechnic were the first groups to use Rutherford Backscattering (RBS) of 1.5MeV He$^+$ to characterize both the silicon overlayer and buried oxide layer of SIMOX structures. This analysis technique enabled the oxygen depth profiles to be determined and Gill [30] investigated the evolution of the buried oxide layer with increasing oxygen dose using RBS. He observed that by increasing the oxygen dose a Gaussian-like depth distribution of oxygen evolved into a flat topped distribution. Gill and Wilson [31] suggested that after the oxygen reached the stoichiometric value of oxygen in SiO$_2$ (4.48x10$^{22}$ O$^+$ cm$^{-3}$) the excess oxygen (at the peak of concentration depth-profile) diffused to the wings of the distribution enabling the growth of the buried oxide layer.

Meanwhile in Japan, Hayashi et al.[32] were the first to use transmission electron microscopy (TEM) to determine the microstructure of SIMOX materials. They analyzed samples implanted at 150 keV with a wide range of doses (0.3x10$^{18}$ to 2.4x10$^{18}$ O$^+$ cm$^{-2}$) and annealed at 1150°C for 2 hours. They observed that the quality of the silicon overlayer in samples implanted with the highest doses was very poor, with the presence of microtwins and polycrystalline silicon. Hayashi et al. [33] used Auger Electron Spectroscopy (AES) to determine the concentration-depth profiles of silicon and oxygen.

\textit{Literature Survey}
in the buried oxide layers in samples implanted with $1.8 \times 10^{14} \text{ O}^+ \text{ cm}^{-2}$ and found that the concentrations saturated at the values of stoichiometric SiO$_2$, namely, 33% and 67% respectively. In addition, they found that the interface between the implanted buried oxide layer and the silicon overlayer was abrupt, suggesting that no intermediate regions were present at these interfaces.

The application of cross-sectional TEM (XTEM) by Booker and co-authors[34] in 1980 gave more information about the microstructure of the buried oxide layers and the quality of the silicon overlayers. Subsequently, the application of Secondary Ion Mass Spectroscopy (SIMS) [35,36,37] enabled the evolution of the oxygen distributions to be followed with increasing dose and anneal conditions. Studies carried out by Hemment et al [38] showed that oxygen diffusion was preferentially driven to the upper Si/SiO$_2$ interface. They attributed this behaviour to a radiation enhanced diffusion (RED) process[39] due to a higher population of point defects in the vicinity of the upper Si/SiO$_2$ interface (see Fig. 2.1).

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*Fig. 2.1 - Schematic of the evolution of the SiO$_2$ layer with increasing oxygen dose.[38]*
In spite of this worldwide research effort, the relatively poor quality of the silicon overlayer impeded the further development of SIMOX technology. Analysis by XTEM [40] showed that the main defects present in the silicon overlayer were SiO$_2$ precipitates and dislocations which in the early 1980s had a density, typically of $10^{11}$ cm$^{-2}$. It was observed that two different types of dislocations were present namely, type I, which were dislocations that thread from the buried oxide layer to the surface. These dislocations were named "threading dislocations" and were generally observed in pairs separated by a distance of less than 0.1 μm. Type II dislocations, started from a SiO$_2$ precipitate and ended at a neighbouring precipitate remaining parallel to the surface. The threading dislocations were later characterized by Krause et al [41] as being edge dislocations having $1/2 <110>$ Burgers vectors. Although the mechanisms of formation of the threading dislocations were not understood, it was clear at that time that the use of higher implantation temperatures would lead to lower residual defect densities due to the enhanced self annealing during implantation. In 1984, Holland et al [42] were the first to introduce background heating to increase and control the implantation temperature. By so doing, they successfully reduced the level of dislocations in the silicon overlayer by three orders of magnitude (from $10^{11}$ to $10^8$ cm$^{-2}$). However, the introduction of higher implantation temperatures did not produce a reduction in the number of SiO$_2$ precipitates in the silicon overlayer[43].

The big breakthrough in SIMOX technology occurred in 1986, when the mechanisms of coalescence and dissolution of SiO$_2$ precipitates became better understood. Stoemenos et al. [44] proposed that by using higher temperature annealings (1300°C) the SiO$_2$ precipitates present in the silicon overlayer could be completely eliminated. He
demonstrated that at these high temperatures only the dissolution of the SiO₂ precipitates occurred whilst the buried oxide layer remained thermally stable (see chapter 3).

The improvements in the quality of the silicon overlayer, both by the elimination of SiO₂ precipitates and reduction of dislocations, suddenly, placed SIMOX in the position of the most promising SOI technology. Moreover, in the spring of 1986, the commissioning of the first 100 mA high-current oxygen implanter (NV-200), where the engineering development was funded by Nippon Telephone and Telegraphs (NTT) [45] and the machine was built by the Eaton Corporation, put SIMOX a step closer to implementation as the preferred SOI substrate for radiation hard and small geometry CMOS circuits. This change in the status of SIMOX technology was reflected by an increase in the number of papers presented at the IEEE conferences during the late 1980s[46] (see Fig 2.2).

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**Fig. 2.2 - Number of SIMOX papers presented at the IEEE SOS/SOI technology workshops over the period 1983 to 1993 [46].**
During 1987 to 1992, several groups [47,48,49] were involved in the materials characterization of SIMOX with the primary objective of determining the mechanisms responsible for the formation of threading dislocations. Although several mechanisms were proposed none of them was experimentally verified, however, all of the researchers agreed that the origin of threading dislocations was either due (i) to the evolution of defects present in the as-implanted material (before annealing) or (ii) to the creation of defects during annealing.

Jaussaud et al [47] proposed that dislocations could be created by precipitate-induced stresses during thermal ramping and annealing. Nevertheless, they concluded that under optimum conditions (to give a stress free silicon overlayer) defect creation during annealing was not the dominant mechanism but that the principal mechanism was due to the evolution of defects present in the as-implanted state. This idea was shared by other researchers and indeed, Stoemenos et al [48] proposed that the formation of paired threading dislocations was due to the growth of surface defects (semi dislocation loops) during annealing. They attributed the formation of these defects to the inability of the surface to accommodate the flux of self interstitials, arriving at the wafer surface and leading to a non ideal internal epitaxial growth. The defects were thus viewed as "growth defects". On the other hand, Visitserngtrakul et al. [49] proposed another mechanism in which intrinsic stacking faults (SF) located near the surface grow towards the buried oxide layer during annealing. They suggested that threading dislocations were formed by the unfaulting of the SF during the expansion. De Veirman et al [50] also found near surface defects on {111} habit planes and suggested that these defects were the precursors of threading dislocations. Finally, Venables et al [51] suggested that implantation-induced stresses in the silicon overlayer played a role in dislocation
formation. Based on X-ray rocking curve determinations of the strain within the overlayer, they showed that the upper portion of the silicon overlayer was in a stress state of biaxial tension, probably due to the formation of vacancy-oxygen clusters. The stress was believed to be of sufficient magnitude to provide a climb force for the expansion of prismatic extrinsic half loops. These half loops could, then, expand over the whole silicon overlayer and form paired dislocations.

As a result of these detailed studies a better understanding of the mechanism of defect formation was achieved leading to the successful optimisation of the implantation conditions and consequently to lower dislocation densities (< 10^5 cm^-2). The introduction of multiple implants and anneals by Hill et al [52] enabled the formation of SIMOX structures with dislocation densities as low as 10^4 cm^-2 to be achieved. The reason for using multiple O⁺ implants instead of the more conventional single O⁺ implants, was to avoid exceeding an experimentally established critical dose for crystallographic defect formation[53].

Other methods to produce low defect density SIMOX appeared later, for example, El Ghor et al. [54] demonstrated that implanting at high current densities together with high implantation temperatures (600-675 °C) enabled the formation of cavities along the path of the incident ions. These cavities act as sinks for point defects and reduce the stress state of the silicon overlayer resulting in a low defect density.

Another method proposed by Van Ommen [55] is the implantation of oxygen ions in channelled directions using low current beam densities. In this way, a cubic superlattice of SiO₂ precipitates was formed. The ordering of the precipitates resulted in a decrease in
the stress state of the silicon overlayer due to easier accommodation of the strain and, as a consequence, the defect density was lower, being typically less than $10^5 \text{ cm}^{-2}$.

At the end of the 1980s, when the first commercial SIMOX wafers appeared in the market a recipe for good quality device worthy substrates was already well known (see table 2.1).[56]

<table>
<thead>
<tr>
<th>Implantation</th>
<th>Dose ($\text{O}^+ \text{ cm}^2$)</th>
<th>Energy (keV)</th>
<th>Implantation temperature ($^\circ\text{C}$)</th>
<th>Annealing temperature ($^\circ\text{C}$)</th>
<th>Annealing time (hrs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single</td>
<td>$1.8\times10^{18}$</td>
<td>190</td>
<td>700</td>
<td>1300</td>
<td>6</td>
</tr>
<tr>
<td>Multiple</td>
<td>$3\times(6\times10^{17})$</td>
<td>190</td>
<td>700</td>
<td>1300</td>
<td>$3\times2$</td>
</tr>
</tbody>
</table>

*Table 2.1 - Implantation and annealing conditions to obtain good quality SIMOX*

**Thin film SIMOX**

At the beginning of the 1990s SIMOX was already well established as the most promising SOI materials technology for high performance CMOS devices. However, the advent of fully depleted CMOS devices[11] necessitated the fabrication of thin film ($<1000 \text{ Å}$) SIMOX substrates. Additionally, the desirability of forming thin buried oxide (BOX) layers to lower the dose and, hence, cost of the substrates shifted the attention of researchers and substrate vendors to low dose and low energy implantations.

In 1990, Nakashima and Izumi [57] reported the formation of a continuous thin buried oxide layer in samples implanted with low O$^+$ doses. They implanted $^{16}\text{O}^+$ into 100 mm (100) silicon wafers at an energy of 180 keV over a large range of doses ($0.1$ to $2.4\times10^{18}$ O$^+ \text{ cm}^{-2}$) and observed extremely low defect densities ($10^3 \text{ cm}^{-2}$) in substrates implanted with doses $\phi < 0.7\times10^{18} \text{ O}^+ \text{ cm}^{-2}$ (see Fig. 2.3).
Subsequently, several groups\cite{58,59} in different continents (Europe and USA) used low dose O$^+$ implantations to form thin buried oxide layers. The SPIRE Corporation\cite{58} was a pioneer in the use of low dose and low energy O$^+$ implantations to produce thin SIMOX structures. The low doses enabled thin buried oxide layers to be formed whilst the use of low energies ensured that the silicon overlayer was thin also. Although they succeeded in producing continuous buried oxide layers, no reports on the quality of the silicon overlayer appeared in the open literature.

During the period of 1990 to 1993 the University of Surrey in collaboration with three others laboratories (Imperial College, Oxford University and Liverpool University) characterized thin SIMOX structures \cite{59} prepared by low dose and low energy O$^+$ implantations. In 1991, Li et al. \cite{59} reported, for the first time, the production of good
quality thin silicon overlayers (1000 Å) on top of continuous thin buried oxide layers. They produced these device worthy structures by implanting low oxygen doses (\( \Phi < 1.0 \times 10^{11} \text{ O}^- \text{ cm}^{-2} \)) using a wide range of energies (40 to 90 keV) and annealed at 1300°C for 6 hours. Furthermore, they proposed a semi-empirical model to determine the minimum oxygen dose (\( \Phi_c \)) needed to form a continuous buried oxide as a function of the implantation energy. They suggested that the necessary condition to obtain a single oxide layer was that the separation between the peak of maximum damage (\( R_d \)) and the peak of concentration (\( R_p \)) had to be smaller or equal to the estimated thickness (\( T_{\text{SiO}_2} \)) of the synthesised layer of oxide, given by:

\[
T_{\text{SiO}_2} = \frac{\Phi}{N_{\text{SiO}_2}^{\text{O}}} 
\]

(2.1)

Where \( \Phi \) is the oxygen dose and \( N_{\text{SiO}_2}^{\text{O}} \) is the oxygen concentration in stoichiometric SiO\(_2\) (\( N_{\text{SiO}_2}^{\text{SiO}_2} = 4.48 \times 10^{22} \text{ cm}^{-3} \)).

Therefore, according to Li et al. \( \Phi_c \) is:

\[
\Phi_c = \alpha R_p N_{\text{SiO}_2}^{\text{O}} 
\]

(2.2)

where \( \alpha \) is:

\[
\alpha = \frac{R_p - R_d}{R_p} 
\]

(2.3)

From eq. (2.2) it is possible to obtain the critical dose for the formation of a continuous buried oxide layer. Li et al found a good agreement between the experimental values and those obtained from eq. (2.2) (see Table. 2.2).
<table>
<thead>
<tr>
<th>Energy ((1\text{keV}))</th>
<th>(\Phi_0^c (\times 10^{15}/\text{cm}^2))</th>
<th>(\Phi_0^c (\times 10^{17}/\text{cm}^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>2.34</td>
<td>2.3</td>
</tr>
<tr>
<td>50</td>
<td></td>
<td>5.4</td>
</tr>
<tr>
<td>70</td>
<td>7</td>
<td>6.7</td>
</tr>
<tr>
<td>140</td>
<td>9.9</td>
<td>3</td>
</tr>
<tr>
<td>150</td>
<td>10.2</td>
<td>4</td>
</tr>
<tr>
<td>200</td>
<td>12-13</td>
<td>5.6</td>
</tr>
</tbody>
</table>

**Table 2.2 - Estimated and experimental values for \(\Phi_0^c\) according to Li et al [59].**

An alternative method to produce ultra thin silicon overlayers (\(t_{\text{SiO}_2} < 1000\ \text{Å}\)) is to employ sacrificial oxidation of the overlayer (\(t_{\text{SiO}_2} = 3000\ \text{Å}\)) of conventional (200 keV) SIMOX material. The method consists of thermal oxidation of a thick film (3000 Å) structure and subsequent removal of the oxide by a HF etch. First reports [60, 61] suggested that the quality of the silicon overlayer was not degraded during the extra oxidation step.

**Low defect density studies**

Until 1988, the main method used to characterise crystallographic defects in SIMOX materials was TEM in planar and cross section views. However, with the improvements in the quality of the silicon overlayer and much reduced defect densities, TEM rapidly became inefficient for the analysis of samples containing low defect densities (< 10^5 cm^-2) because direct observation methods which involve very high magnifications were not compatible with the low areal density of defects [62].
Conventional characterization methods used in bulk silicon technology such as X-ray topography\cite{14}, electrochemical etching\cite{63} and chemical defect etching\cite{20} were investigated as alternatives to TEM analyses since the spatial resolution limits of these methods facilitated the analysis of samples containing extremely low defect densities.

A recent report on the use of X-ray topography to the analysis of crystallographic defects in SOI materials (prepared by Wafer Bonding) was given by Theunissen et al \cite{64}. They succeeded in separating the images of the silicon overlayer from the substrate due to a misorientation between the layers. They intentionally introduced this misorientation by bonding two wafers of different orientations, namely (001) and (111). However, they did not succeed in analysing SIMOX since for these materials, the silicon overlayer and the substrate have the same orientation and, therefore, a distinction between the image of defects from the silicon overlayer and from the substrates was not possible.

The application of chemical defect etching to the analysis of SIMOX substrates is also very difficult and there have been only relatively few reports on the successful use of that method to delineate defects in SIMOX. Probably, the first report was by Homma et al \cite{53} in 1987. They grew a thick epitaxial silicon layer (0.6 µ- 1.0 µm) on top of the original silicon overlayer using molecular beam epitaxy (MBE) prior to the etching process. The etch pits formed in these relatively thick layers (≈1 µm) were large enough to be observed by scanning electron microscopy (SEM). However, it was not possible, at that time, to determine a one to one correlation between the etch pits observed in the epitaxial layer and the "threading dislocations" in the silicon overlayer since no cross-correlations with TEM analysis were carried out. In 1990, Liaw et al \cite{65} reported extensive analyses by TEM of epitaxial SIMOX samples and determined that not all the "threading dislocations" (TD) propagated into the epitaxial layer. Indeed, they found that
the dislocation density measured at the top surface decreased as a function of the epitaxial silicon thickness due to the "looping" of threading dislocations during the epitaxial growth.

In 1990 Fechner et al. [66] introduced a second step in the etching process which enabled the etch pits to be observed by optical microscopy without growing an epitaxial layer. The second etching step consisted of a dip in a HF(40%) solution which dissolved the SiO₂ layer below the etch pit. Using this second etch step, large cavities (1-2 μm) were formed underneath the etch pits (see Fig. 2.4). The "decorated" etch pits could then be easily observed by optical microscopy. Margail et al [62] used a similar method to delineate defects in commercial SIMOX and reported that the double etch method was only reliable for samples having a silicon overlayer thickness greater than 1000 Å.

In 1993, Giles et al. [67] reported a new etchant capable of reliably delineating crystallographic defects (dislocations and SF) in silicon overlayers as thin as 500 Å. (see chapter 5).
Fig. 2.4 - Cross section schematic of the "decorated" etch pits.
Chapter Three: Background

3.1 Introduction

An understanding of some physical aspects, such as diffusivity and solid solubility of oxygen in silicon is fundamental to a better appreciation of the mechanisms of the formation of the buried oxide layer in SIMOX materials. In this chapter, we present basics aspects related to the synthesis of a continuous buried oxide layer by O⁺ ion implantation. In addition, we also present some basic aspects related to the formation of crystallographic defects in oxygen implanted silicon.
3.2 Ion beam synthesis of SiO₂

Ion implantation is a non-equilibrium process in which highly energetic ions are injected into a target in order to change the mechanical, optical or electrical properties of the target material [68,69]. In some cases (for extremely high doses), the volume concentration of the ions (impurities) introduced by the implantation process can exceed the solid solubility limits and the formation of new chemical phases is likely to occur. The use of ion implantation as a method to synthesise new phases has been widely used in the past, to form compounds like silicon dioxide, silicon nitrides[70] and more recently, to form silicides such as iron silicides [71], cobalt silicides[72] and many others [73]. However, the complex nature of the structures created by ion beam synthesis makes theoretical descriptions of the evolution of the new phase and identification of the dominant processes and prediction of the final structure rather difficult.

The fundamental processes that occur during the nucleation and segregation of a second phase depend on the processing conditions but the general scheme of the evolution of an incipient buried layer can be considered as consisting of four overlapping stages[74]. These stages are namely, supersaturation of implanted atoms (ions), nucleation of precipitates, precipitate growth and, finally, the coalescence of precipitates into a planar continuous layer with abrupt interfaces. In the next sub-section we briefly describe the physical processes which control the four stages of the growth of the BOX layer.
3.2.1 Formation of a buried oxide layer.

A. Diffusivity of oxygen in silicon.

Below the solubility limits, oxygen in single crystal silicon is known\[^{75}\] to be a covalently bonded interstitial impurity, incorporated in slightly off-centred interstitial positions between two neighbouring silicon atoms (see Fig. 3.1).

\[
\begin{align*}
\text{Fig. 3.1- Configuration of interstitial oxygen in silicon} \ [75].
\end{align*}
\]

The diffusion of interstitial oxygen is an activated process with a diffusion coefficient \( D \) given by \[^{76}\]:

\[
D(T) = D_0 \exp(-E^*/k_B T) \tag{3.1}
\]

Where \( T \) is the absolute temperature, \( k_B \) is the Boltzmann constant, \( D_0 = 0.13 \text{ cm}^2/\text{s} \) and \( E^* = 2.53 \text{ eV} \). Fig 3.2 shows the diffusion coefficient of interstitial oxygen in silicon as a function of temperature.
In some special cases, such as for example in ion implanted silicon, anomalous fast diffusing oxygen has been reported[77,78]. It has been suggested that this anomalous diffusion is due to the diffusion of molecular oxygen (O_2) formed during heat treatments at moderate temperatures.

**B. Solid solubility of oxygen in silicon**

The solid solubility of oxygen in silicon is given by[79]:

\[ C_{\text{sol}}(T) = C_{\text{sol}}^0 \exp \left( -\frac{\Delta E}{k_B T} \right) \] (3.2)

Where \( C_{\text{sol}}^0 = 9 \times 10^{22} \text{ cm}^{-3} \) and \( \Delta E = 1.52 \text{ eV} \).

Fig. 3.3 shows the dependence of the solubility upon temperature [79].
In oxygen implanted silicon, the oxygen concentration, \( C_O(z) \), varies significantly with depth, having its maximum value near the projected range \( (R_p) \) and decreasing progressively towards the surface and at greater depths into the substrate. To a first approximation, neglecting effects such as crystal orientation, \( C_O(z) \) is given approximately by a Gaussian distribution:

\[
C_O = C_{\text{peak}} \exp \left( \frac{(z - R_p)^2}{2\delta^2} \right)
\]  

(3.3)

where \( \delta \) is the standard deviation (straggles) and \( C_{\text{peak}} = \frac{b}{\sqrt{2\pi}\delta} \).

For \( C_O(z) > C_{O_{\text{sol}}} \), segregation of the implanted oxygen into a SiO\(_2\) phase will occur through the nucleation and growth of SiO\(_2\) precipitates (see 3.2.1 C). Mantl[80] has shown that this phase transformation is driven by the decrease in the free energy of the system. The change of Gibbs free energy, \( \Delta G \), when a new phase of volume, \( V \), and interface area, \( A \), is formed is given by[80]:

\[
\Delta G = V(\Delta G_V + \Delta G_{\text{interface}}) + A\sigma
\]

(3.4)
where $\Delta G_v$ is the change in free energy per unit volume associated with the formation of the SiO$_2$ phase, $\Delta G_e$ is the increase in elastic energy per unit volume and $\sigma$ is the specific interface energy. Since the molar volume of the SiO$_2$ is approximately twice as large as the molar volume of Si[81], the formation of the SiO$_2$ phase will produce internal strains, and therefore will tend to increase the elastic energy of the crystal. The typical dependence of the change of Gibbs free energy $\Delta G$ upon the radius of a precipitate is presented in Fig. 3.4[82]. Precipitates with radii smaller than a critical radius $r_c$ (shown in the figure as $r_{c1}$ at a temperature $T_1$) are unstable and tend to dissolve in the matrix.

![Fig. 3.4 - Dependence of $\Delta G$ upon precipitate radius [80]](image_url)
In oxygen implanted silicon, several mechanisms of SiO$_2$ precipitation can occur simultaneously depending upon the processing conditions (oxygen dose, implanted temperature, and current density). We can basically separate these mechanism into three major processes, that are namely, homogeneous[83], heterogeneous[84] and 'etherogeneous' precipitation.

Homogeneous precipitation [86,87] is the dominant process when the oxygen concentration exceeds a minimum value for which spontaneous precipitation occurs independently of the presence of nucleation sites. Experimental evidence of homogeneous precipitation in high dose oxygen implanted silicon (\( \phi = 1.8 \times 10^{14} \) O$^+$ cm$^{-2}$) has been extensively reported by Hemment et al[86] and Marsh et al[87].

On the other hand, Cerofolini et al [88] have suggested that heterogeneous precipitation is the dominant process for lower oxygen concentrations (low dose regimes) and high implantation temperatures. For these conditions the oxygen supersaturation inside the matrix is not high enough to enable homogeneous precipitation to occur, and therefore the formation of the SiO$_2$ precipitates will depend upon the existence of preferencial nucleation sites. Several authors [89,90] have identified vacancy clusters as being the preferential nucleation sites for SiO$_2$ precipitates. Preferential nucleation at vacancy clusters occurs because the elastic energy associated with the formation of the SiO$_2$ phase is considerably reduced in these localized areas [91]. In oxygen implanted silicon, vacancy clusters are predominantly located near the peak of damage (\( R_p \)) and, therefore, heterogeneous precipitation will occur preferentially near that region. Experimental
evidence that heterogeneous precipitation occurs near the peak of damage has been recently shown by, Li et al [92] and Aspar et al [93], for low dose oxygen implanted silicon submitted to high temperature annealings. These authors showed that in these cases two bands of SiO$_2$ precipitates are formed. One located near the peak of the damage and the other close to the peak of the oxygen concentration-depth profile. The formation of the first band of precipitates is explained by the radiation enhanced diffusion[39] of the implanted oxygen to the region of higher concentration of vacancy clusters where the heterogeneous precipitation process takes place.

More recently, Cerofolini et al [94] have proposed a new precipitation mechanism named 'etherogeneous' precipitation. This new mechanism differs from the conventional heterogeneous precipitation because it takes into account the effects of the implantation process. In their model, they propose that for certain conditions (low implantation temperature and high beam currents) SiO$_2$ precipitates can be heterogeneously nucleated inside highly disordered regions formed by the implantation process. The driving force for this precipitation process is the local heating created by the collisional events and according to their model only projectiles that release an energy higher than a minimum threshold energy are able to induce the formation of SiO$_2$ precipitates [95].

**D. Growth and dissolution process of SiO$_2$ precipitates.**

The evolution of the SiO$_2$ precipitates during thermal processing is initiated by second phase nucleation, and then controlled by a process in which only precipitates having a radius larger than $r_c$ (see Fig. 3.4) will remain stable while smaller ones will dissolve[96]. Vanhellemont and Claey[97] have derived an expression for the critical
radius $r_c$ which takes into account the influence of oxygen, vacancy and self-interstitial concentrations in the Si matrix. This expression is given by [98]:

$$r_c = \frac{2\sigma}{K T \ln \left( \frac{C_o}{C_i} \left( \frac{C_i}{C_j} \right)^{\alpha} \left( \frac{C_j}{C_i} \right)^{\beta} - E_{str} \right)}$$

(3.5)

with $\sigma$ is the interface energy per unit area, $E_{str}$ the strain energy and $K$ a constant associated with the silicon oxide polymorph (SiO$_x$). The parameters $C_o$, $C_v$ and $C_i$ are respectively the oxygen, vacancy and self-interstitial concentrations in the silicon matrix. Their thermal equilibrium values are given by $C_0^*$, $C_v^*$ and $C_i^*$. In addition $\alpha$ is the number of vacancies absorbed from the matrix and $\beta$ the number of self-interstitials injected into the matrix. For high temperature anneals (say $> 1250^\circ$C) the recombination rate of vacancies and interstitials is very high [99] and therefore, $C_v$ and $C_i$ rapidly approach the thermal equilibrium values $C_v^*$ and $C_i^*$. Also the interstitial oxygen concentration rapidly approaches its equilibrium value due to the combined effect of oxygen out-diffusion (at these high temperatures the diffusion length of oxygen in silicon is larger than the silicon overlayer thickness) and precipitate growth. This makes the denominator of eq. 3.5 very small provided that $E_{str}$ is also small. A quantitative value for $E_{str}$ is not well known, however, at these high temperatures several strain relief mechanisms are active [100] and therefore $E_{str}$ is expected to be small. The decrease of the denominator of eq. 3.5, means that the critical radius increases at these high temperatures (1250$^\circ$C). Therefore, as we have already suggested, precipitates having a radius smaller than $r_c$ will become unstable and will dissolve. The oxygen atoms released by the dissolving precipitates can out-diffuse or diffuse towards the larger precipitates.
The oxygen diffusion from the dissolving precipitates towards the large precipitates can be described in terms of the Gibbs-Thomson equation, given by [101]:

\[
C_s(r) = C_\infty \left(1 + \frac{2\sigma V_a}{r k_B T}\right)
\]  

(3.6)

where \(C_s\) is the solute concentration at the surface precipitate of radius \(r\) and \(C_\infty\) denotes the solute concentration at a planar interface \((r = \infty)\), \(V_a\) the atomic volume, \(\sigma\) is the specific interface energy and \(k_B\) the Boltzman constant. From equation 3.6 it is observed that \(C_s(r_2) > C_s(r_1)\) for \(r_2 > r_1\) (see Fig. 3.5).

Fig. 3.5 - Solute concentration around precipitates having radius \(r > r_c\) and \(r < r_c\) [80].
Therefore, the oxygen released by the dissolving precipitate \((r_1 < r_c)\) will diffuse towards the growing precipitate \((r_2 > r_c)\). The competitive growth of precipitates, in which larger ones grow at the expense of the smaller ones, is known as Ostwald ripening [102]. The growth of precipitates larger than the critical radius is, then, a diffusion limited process in which the limiting factor is the diffusion of oxygen to the periphery of the precipitate. Wada et al. [103] have expressed the volume increase of a precipitate with time above the critical radius as:

\[
I = \frac{C_0^I - C_0^E}{C_0^T - C_0^P} D_0 t^{1/2}
\]

where \(r\) is the radius of the precipitate, \(C_0^I\) is the initial oxygen concentration expressed in atoms per cubic centimeter, \(C_0^E\) is the equilibrium oxygen concentration at the annealing temperature and \(C_0^P\) is the oxygen concentration in the precipitate.

**E. Coalescence of SiO₂ precipitates.**

A further "coarsening" mode observed in oxygen implanted silicon is the coalescence of SiO₂ precipitates. If the density of these large precipitates is high enough, coalescence of adjacent precipitates may occur as the two phase system will change in order to reduce the surface energy [104]. The coalescence process is mainly controlled by solvent diffusion from the matrix to the 'neck' region at the point of contact of the precipitates (see Fig. 3.6). Brown [105] calculated the solute concentration at the neck \(C_s(\rho)\) as being:

\[
C_s = C_\infty \left(1 - \frac{aV_a}{\rho k_B T}\right)
\]

where \(\rho\) is the radius of the neck, as depicted in Fig. 3.6.
The equation (3.8) resembles the Gibbs-Thomson equation (eq. 3.6), but with the difference that the solute concentration at the neck is smaller than the concentration in the matrix, in contrast to the concentration at the surface of a small spherical particle. This is a consequence of the 'negative curvature' at the neck of the two touching precipitates (see Fig. 3.6).

Mantl [80] showed that the solute flux towards the neck is approximately:

$$j \sim \frac{\sigma C_{\infty} D V_0}{\rho^2 K_B T}$$

A consequence of this solute flux towards the neck is that the radius of the neck will increase leading to a localized planarization of the resulting precipitate. Thus, precipitate coalescence can promote planar layer formation provided the degree of overlapping of the large precipitates is high enough to enable their coalescence into a single continuous layer.
3.3 Crystallographic defects in oxygen implanted silicon.

In a comprehensive review of defects in SIMOX materials, Jaussaud et al [46] proposed that crystallographic defects in oxygen implanted silicon are mainly formed by two mechanisms, namely (i) condensation of point defects produced both by the implantation and by internal oxidation and (ii) plastic deformation of the silicon structure caused by the large volume mismatch between atomic Si and the SiO₂ molecule. Therefore, we can see that the two main sources of defects are the radiation damage itself and the internal oxidation of silicon and in the following sub-sections we describe the consequential formation of crystallographic defects in the implanted layer.

3.3.1 Radiation damage as a source of secondary defects

The loss of kinetic energy by the implanted ions occurs through electronic (inelastic) and nuclear (elastic) interactions with the atoms of the target[69]. In silicon, elastic collisions are mainly responsible for the primary implantation damage. Collisions with energy transfers above a threshold, of about 15 eV, will displace Si atoms from their lattice sites [106]. The displaced atoms may in turn displace further atoms resulting in highly disordered regions. The displacement of an atom from its regular lattice position to an interstitial one forms an interstitial/vacancy pair known as a Frenkel pair[107]. Therefore, the highly disordered regions produced by the implantation process consist predominantly of a non equilibrium concentration of vacancies and interstitials. Since the internal energy of isolated point defects is high, interstitials and vacancies will tend to recombine with each other or condensate into aggregates in order to decrease the total energy.
In diamond structures, the condensation of vacancies (interstitials) occurs preferentially along \{111\} planes, since they are the lowest surface energy planes in these structures\[108\] (see appendix C). The growth of aggregates occurs because there is a net reduction in energy when an additional vacancy (interstitial) is captured by the aggregate. However, Thomas et al \[109\] have proposed that the continuous growth of the vacancy (interstitial) aggregate is limited by the possible collapse into a dislocation loop (DL) with a lower energy configuration. Since the stacking fault energy in silicon is relatively low (\(\gamma \approx 50 \text{ erg cm}^2\)) \[110\] both faulted (Frank sessile DL) and perfect (prismatic DL) dislocation loops can be formed from the collapse of those aggregates. Moreover, it has also been proposed \[109\] that a vacancy aggregate can collapse into a stacking fault tetrahedron (SFT)\[111\] when this configuration is a lower energy state.

Further, Thomas et al \[109\] have shown that the relative energies of intrinsic (vacancy type) faulted DL, SFT and perfect DL are given approximately by:

\[
E_F = \frac{G a^2 l}{4\pi(1-\nu)} \ln \left( \frac{l}{r_0} \right) + \frac{\sqrt{3}}{4} l^2 \gamma \tag{3.10 a}
\]

\[
E_T = \frac{G a^2 l}{12\pi(1-\nu)} \ln \left( \frac{l}{r_0} \right) + \sqrt{3} l^2 \gamma \tag{3.10 b}
\]

\[
E_p = \frac{G a^2 l}{2.4(1-\nu)} \ln \left( \frac{2l\sqrt{2}}{a} \right) \tag{3.10 c}
\]

where \(E_F\), \(E_T\) and \(E_p\) are the energies of a triangular faulted DL, a SF tetrahedron and a perfect loop, respectively, each having side \(l\). Additionally, \(G\) is the shear modulus, \(a\) is the unit cell dimension, \(r_0\) is the dislocation core radius, \(\nu\) is Poisson's ratio and \(\gamma\) is the stacking fault energy.

It is observed from eq. (3.10 a-c) that the relative energies of these three different types of defects depend on the size \(l\) of the defect. For illustrative purposes we show in Fig. 3.7 the dependence on the energy of the defect upon the size \(l\) \[109\].
Fig. 3.7 - Energy dependence with size $l$ of faulted DL, SFT and perfect loop in Ag [109].

It is observed that SFT are more stable (have lowest energy) below a critical size, $l_o$, while perfect loops are always favoured above an upper critical size (330 Å). Table 3.1 lists the critical value, $l_o$, for which the SF tetrahedron (SFT) is the most stable defect in different metals [109].

<table>
<thead>
<tr>
<th>Material</th>
<th>$\gamma$ (erg cm$^{-2}$)</th>
<th>$l_o$ (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-Zn</td>
<td>20</td>
<td>1.6x10$^4$</td>
</tr>
<tr>
<td>Au</td>
<td>33</td>
<td>430</td>
</tr>
<tr>
<td>Cu</td>
<td>40</td>
<td>515</td>
</tr>
<tr>
<td>Ag</td>
<td>40</td>
<td>330</td>
</tr>
</tbody>
</table>

Table 3.1 - Calculated values of $l_o$ below which SFT are the most stable defect (after Thomas et al [109])

Transformation of one type of defect to another can occur if the energy associated with the defect (see eq. 3.10) is decreased by the transformation and also if the activation energy for this process is low [112]. The transformation of a faulted DL (Frank type, b
to a perfect DL ($b = \frac{1}{2} <110>$) was first considered by Saada[113] who showed that it basically involves the nucleation of a Shockley partial ($b = \frac{1}{6} <211>$) in the plane of the fault. The unfaulting mechanism can be written, then, as:

$$\frac{1}{3} <111> + \frac{1}{6} <211> \rightarrow \frac{1}{2} <110>$$  \hspace{1cm} (3.11)

Frank Shockley perfect

Similarly, SFT can arise from a multiple dissociation mechanism of an intrinsic Frank DL. The dissociation mechanism that enables the formation of the SFT from an intrinsic Frank DL is described in appendix C.

3.3.2 Internal oxidation of silicon as a source of defects.

The molar volume of the SiO$_2$ molecule is approximately 2.25 larger than that of Si ($V_{si} = 2.23 \times 10^{23} \text{ cm}^3$)[81] and therefore, the formation of the SiO$_2$ phase will produce large internal stresses unless an efficient stress relief mechanism occurs. The extra volume can be accommodated either by (a) emission of self-interstitials away from the Si-SiO$_2$ interface or by (b) plastic deformation of the silicon matrix near the SiO$_2$ precipitate.

A. Emission of Si interstitials

The large volume change associated with SiO$_2$ formation requires that silicon atoms will be displaced from the vicinity of the SiO$_2$/Si interface. This may be accomplished by generation of silicon interstitials, $Si_i$, at the SiO$_2$/Si interface. Self interstitials are created in this region because the crystal elastic energy associated with the stress generated by the SiO$_2$ precipitate generally exceeds the formation energy of the $Si_i$ [114]. Therefore,
the internal oxidation of silicon involves the formation of $\text{Si}_i$ according to the following reaction[115]:

$$ (r + 1) \text{Si} + \text{O}_2 \rightarrow \text{SiO}_2 + r \text{Si}_i $$

(3.12)

where $r = 1.25$.

From eq. 3.12, it is observed that a supersaturation of $\text{Si}_i$ will be created near the $\text{SiO}_2$/Si interface and, consequently, condensation of interstitials into aggregates is likely to occur giving rise to the formation of crystallographic defects through the mechanisms described in section 3.2.1.

**B. Plastic deformation**

Plastic deformation of the silicon matrix can arise if the compressive stresses around the $\text{SiO}_2$ precipitate are not completely relieved. The silicon matrix will deform plastically through the nucleation of prismatic dislocation loops. This process was first described by Seitz et al [116] as "prismatic punching". They pointed out that a pair of prismatic loops of opposite nature, one interstitial and one vacancy type, can be generated under the influence of a misfit stress field. This is accomplished by expansion of "generator" loops around a glide prism.

When the precipitate is compressed by the matrix, the interstitial loop is punched out along the glide directions, $<110>$, while the vacancy loop moves onto the precipitate surface where it is annihilated, thereby reducing the misfit strain. This mechanism can be repeated several times and can produce a family of loops "punched" out along crystallographic directions which defines the axis of the glide surface. In some cases, pinning of the prismatic dislocation loop to the $\text{SiO}_2$ precipitate can occur and the expansion of the dislocation is only possible by a climb process [117].
Chapter Four: Experimental Details

In this chapter we describe the experimental conditions in which samples were prepared and we give details on the different methods used to characterize the SIMOX materials.

4.1 Sample description.

Two different sets of samples have been used in this project. The first group of samples consisted of (100) CZ, Si wafers implanted with oxygen using a wide range of energies (30 keV-200 keV) and a wide range of doses (0.2-1.0 x10^{18} O^+ cm^{-2}). The samples were implanted either at the University of Surrey or at Eaton Corporation. The wafers prepared at the University of Surrey were produced as part of a SERC funded programme (IED 1777) to produce and characterize low energy and low dose SIMOX materials whilst the wafers prepared at Eaton Corporation were obtained to support a collaborative activity involving that company and the University of Surrey. Annealing of the Surrey implanted wafers were carried out at Southampton University. Further details of the implants and anneals are included in section 4.1.1 and 4.1.2.

A second group of specimens consisted of SIMOX samples thinned by sacrificial oxidation. The original thick SIMOX substrates were supplied by LETI [118] and IBIS.
Corporation [119]. Details of the implantation and annealing conditions are given in section 4.1.3 whilst the details of the sacrificial oxidation are given in section 4.1.5. In addition, some SIMOX samples were implanted with Si⁺, Ge⁺ or BF⁺₂, to amorphise the silicon overlayer in the vicinity of the Si/BOX interface prior to oxidation (see section 4.1.4). Details of all the samples used in this project are listed in tables 4.1 to 4.8 and in appendix E.

The SIMOX wafers were then cleaved into small samples (typically 5x5 mm²) and cleaned using acetone, isopropyl alcohol, and trichloroethylene prior to a dip in an ultrasonic bath in order to remove particles from the surface. The samples were then ready to be analyzed using the different analytical methods described in section 4.2. Furthermore, selected SIMOX samples were etched using a KOH solution (1 molar) at 70 °C in order to selectively remove the silicon overlayer. Then, the buried oxide layer was removed using a HF(40%) solution. This two-fold etch treatment enabled both the silicon overlayer and BOX layer to be removed, thus exposing the silicon substrate for further analysis.

4.1.1 Oxygen implantations at the University of Surrey.

Ion implantation was carried out on the 500 kV implanter at the University of Surrey. This machine has an open air terminal from which the ions are accelerated to their full velocity, (maximum voltage of 500 kV) prior to analysis at the earth potential. The ion beam is electrostatically scanned in the x and y directions over an area of maximum size 2"x2". A schematic of the implanter showing the main components is presented in Fig. 4.1. From Fig. 4.1 we can see that the implanter has six major components; namely: (i)
ion source, (ii) focussing element, (iii) acceleration tube, (iv) analysing magnet, (v) electrostatic scan and (vi) the target chamber.

Fig. 4.1 - Schematic of the 500kV implanter.

The sample holder was specially designed in order to provide background heating of the wafer during implantation using a bank of quartz halogen lamps mounted behind the wafer. A thermocouple was inserted between the wafer and the lamps to control the
implantation temperature. Furthermore, the sample holder was water cooled in order to
dissipate the excess heat generated by the lamps. A schematic of the sample holder is
shown in Fig. 4.2.

Fig. 4.2 - Schematic of the halogen lamp heater sample holder.

The 3 inch (100) Si wafer was placed inside the sample holder facing the beam but with
the wafer normal at an angle of 7° in order to reduce the occurrence of channelling.
Additionally, another silicon wafer was also incorporated into the sample holder to be
used as a 'dummy'. This enabled the beam parameters to be set up before the beam was
directed onto the wafer. A silicon aperture was used to define the implant area which is
smaller than the wafer's diameter. In these experiments the implanted area was
approximately 6.5 cm². Fig. 4.3 shows a schematic of the 3 inch wafer and the implanted
area.

42  Experimental details
Both the beam defining aperture and suppressor electrode, biased to -300 V to suppress secondary electrons, were faced with silicon to minimise metallic contamination due to sputtering. The target chamber was pumped by a conventional diffusion and rotary pump system to a pressure of less than $10^{-6}$ Torr before commencing the implantation. The chamber included a liquid $N_2$ cold finger to condense hydrocarbons.

The ion source used was a r-f source which was capable of producing total beam currents of up to 100 to 150 $\mu$A. After scanning the total current incident upon the sample was 50 $\mu$A to 70 $\mu$A with a current density of typically 3-10 $\mu$A/cm$^2$. Molecular singly charged oxygen ($O^+$) were implanted at energies of 140, 180 and 400 keV. Doses in the range of $3\times10^{17}$ $O^+$/cm$^2$ to $7\times10^{17}$ $O^+$/cm$^2$ were selected to be close to the critical dose for the formation of a continuous buried oxide layer [59], although some wafers were also implanted with higher doses ($1.0\times10^{18}$ $O^+$/cm$^2$) to form a thicker oxide layer.
After implantation, all wafers were annealed at 1300 °C or 1320° C for 2 or 6 hours under argon (Ar + 1/2% O₂) or nitrogen (N₂ + 1/2% O₂) ambients. The anneals were carried out either at Southampton University or Imperial College. Prior to annealing, the samples were capped with a low temperature CVD oxide of thickness 5000 Å in order to protect the surface from contamination, unintentional oxidation and thermal etching. Implantation and annealing details of the samples prepared at the University of Surrey are listed in table 4.1.
### Experimental details

<table>
<thead>
<tr>
<th>SIMOX wafer</th>
<th>Energy (keV)</th>
<th>Oxygen dose, x10¹⁷ (O² cm⁻²)</th>
<th>Current density [µA/cm²]</th>
<th>Implantation temperature (°C)</th>
<th>Area Implanted (cm²)</th>
<th>Annealing ambient</th>
<th>Annealing temperature (°C)</th>
<th>Annealing time (hrs.)</th>
</tr>
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<tbody>
<tr>
<td>G1/G2/G4 G5/G6</td>
<td>70</td>
<td>3.3</td>
<td>3.1</td>
<td>500/550/600/650/700</td>
<td>6.45</td>
<td>Ar + 0.5% O₂</td>
<td>1300</td>
<td>6</td>
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<tr>
<td>G3</td>
<td>70</td>
<td>3.3</td>
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<td>700</td>
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<td>&quot;</td>
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<td>G7/G8/G9/G10/G11</td>
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<td>5</td>
<td>&quot;</td>
<td>500/550/600/650/700</td>
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<td>6</td>
</tr>
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<tr>
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<td>10</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
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<td>200</td>
<td>4</td>
<td>7.75</td>
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<td>&quot;</td>
<td>N₂ + 0.5% O₂</td>
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<td>&quot;</td>
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<td>&quot;</td>
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<td>&quot;</td>
<td>&quot;</td>
</tr>
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<td>7.75</td>
<td>&quot;</td>
<td>&quot;</td>
<td>Ar + 0.5% O₂</td>
<td>1300</td>
<td>&quot;</td>
</tr>
<tr>
<td>G17</td>
<td>&quot;</td>
<td>6</td>
<td>7.75</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
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<td>7</td>
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<td>5</td>
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</table>

*Table 4.1 - SIMOX wafers prepared at the University of Surrey.*
Six inch SIMOX wafers were prepared at Eaton Corporation using a prototype commercial implanter, namely a modified Eaton NV-10-80 high current batch implanter [120]. This machine was used to implant batches of 6" (100) Cz wafers using mechanical scanning to achieve lateral dose uniformity. The silicon wafers were implanted using either atomic or molecular singly charged ($O^+_2$ or $O^+$) ions at energies of 60 keV($O^+_2$ implantations), 30 keV or 40 keV with a range of doses which varied from $2.2 \times 10^{17}$ $O^+/cm^2$ to $3.0 \times 10^{17}$ $O^+/cm^2$. This range of doses was selected in order to form a continuous buried oxide layer[59]. Background heating of the wafers was obtained using quartz halogen lamps which enabled the wafers to be pre-heated at 500°C before commencing the implantations. The average wafer temperature was measured using an optical fibre thermometer placed 3mm in front of the wafers. The complete 6 inch wafer was exposed to the ion beam except for small areas at the periphery beneath the wafer clips (see fig. 4.4).

High temperature annealing (HTA) was performed subsequently to implantation at a temperature of 1300°C for six hours in a nitrogen ambient mixed with 0.25% oxygen. Table 4.2 shows the implantation and annealing details of the complete batch of wafers prepared at Eaton Corporation.
Fig. 4.4 - Schematic of the implanted areas of the 6" wafers supplied by Eaton Corporation.

![Diagram showing implanted areas of wafers.]

<table>
<thead>
<tr>
<th>SIMOX</th>
<th>oxygen mass implanted</th>
<th>Dose x10¹⁷ O² cm⁻²</th>
<th>Energy keV</th>
<th>Implantation temperature (°C)</th>
<th>Annealing temperature (°C)</th>
<th>Annealing ambient</th>
</tr>
</thead>
<tbody>
<tr>
<td>E1</td>
<td>32</td>
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<td>60</td>
<td>530</td>
<td>1300</td>
<td>N₂ +0.25% O₂</td>
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<tr>
<td>E2</td>
<td>32</td>
<td>2.2</td>
<td>60</td>
<td>530</td>
<td>1300</td>
<td>&quot;</td>
</tr>
<tr>
<td>E3</td>
<td>16</td>
<td>2.2</td>
<td>30</td>
<td>530</td>
<td>1300</td>
<td>&quot;</td>
</tr>
<tr>
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<td>2.2</td>
<td>40</td>
<td>530</td>
<td>1300</td>
<td>&quot;</td>
</tr>
<tr>
<td>E5</td>
<td>16</td>
<td>2.4</td>
<td>40</td>
<td>550</td>
<td>1300</td>
<td>&quot;</td>
</tr>
<tr>
<td>E6</td>
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<td>2.4</td>
<td>40</td>
<td>600</td>
<td>1300</td>
<td>&quot;</td>
</tr>
<tr>
<td>E7</td>
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<td>3.0</td>
<td>40</td>
<td>510</td>
<td>1300</td>
<td>&quot;</td>
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</table>

Table 4.2 - Implantation details of wafers produced by Eaton Corporation using the modified NV-10-80 implanter. All wafers were annealed for six hours.
4.1.3 Standard SIMOX Substrates.

Device grade 4" SIMOX substrates were supplied by two different vendors (IBIS [118], LETI [119]) who prepared the substrates using commercial Eaton NV-200 implanters either by single or multiple step oxygen implantations (SIA SIMOX). In these samples the complete wafer was implanted. The implantations were carried out at high temperatures, typically 700°C, by pre-heating the wafers using halogen lamps. The implanted wafers were subsequently annealed for six hours at 1300°C in an Ar + 1/2% O₂ atmosphere. Details of the implantation conditions are listed in table 4.3.

<table>
<thead>
<tr>
<th>SIMOX Batch</th>
<th>Source</th>
<th>Implant method</th>
<th>Nominal dose (cm²)</th>
<th>Energy (keV)</th>
<th>Silicon overlayer thickness (Å)</th>
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<td>A1</td>
<td>IBIS</td>
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<td>2000</td>
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<td></td>
<td>single</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A3</td>
<td></td>
<td>single</td>
<td></td>
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<td>2000</td>
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<td>A4</td>
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<td></td>
<td></td>
<td>1800</td>
</tr>
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<td>LETI</td>
<td>multiple</td>
<td></td>
<td></td>
<td>2100</td>
</tr>
<tr>
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<td></td>
<td>single</td>
<td></td>
<td>200</td>
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<tr>
<td>B3</td>
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<td>single</td>
<td></td>
<td></td>
<td>2500</td>
</tr>
<tr>
<td>B4</td>
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<td>190</td>
<td>2100</td>
</tr>
<tr>
<td>B5</td>
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<td>2100</td>
</tr>
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<td>B6</td>
<td></td>
<td>single</td>
<td></td>
<td></td>
<td>500</td>
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</table>

Table 4.3- Implantation details of standard SIMOX substrates supplied by IBIS and LETI.
It is observed from Table 4.3, that all samples had a relatively thick silicon overlayer (typically > 2000 Å) except wafer B6, which was sacrificially oxidised by the vendor (LETI) in order to reduce the silicon overlayer thickness. We now designate these thick SIMOX substrates as "standard" SIMOX.

4.1.4 BF$^+$, Ge$^+$ and Si$^+$ implantations.

Selected samples cut from standard SIMOX wafers (namely wafers B1, B3(2) and B4(2)) were subsequently implanted with BF$^+_2$, Ge$^+$ and Si$^+$ ions at the University of Surrey. The purpose of the implantations was to amorphise the silicon overlayer in the vicinity of the BOX layer in these standard SIMOX wafers. The implantations were carried out at room temperature using a carousel sample holder, in which a maximum of 16 samples (approximately 1 cm$^2$) could be loaded. The samples were lightly clamped with aluminium clips and the total area implanted was 1 cm$^2$. The BF$^+_2$ implantations were carried out at energies of 270 keV with doses which varied from 8.0 x 10$^{13}$ cm$^{-2}$ to 2.0 x 10$^{14}$BF$^+_2$ cm$^{-2}$ (see table 4.4). For the Si$^+$ implantations the energy used was 190 keV and the doses varied from 1.0 x 10$^{13}$ cm$^{-2}$ to 5.0 x 10$^{15}$ cm$^{-2}$ (see table 4.5). Finally, the Ge$^+$ implantations were carried out at energies of 330 keV with doses which varied from 1 x 10$^{13}$ cm$^{-2}$ to 3.0 x 10$^{14}$ cm$^{-2}$ (see table 4.6). The energies and doses were selected using the LUPIN [121] simulation programme in order to place the peak of the damage profile near the Si/SiO$_2$ interface, and therefore selectively amorphise that region.
<table>
<thead>
<tr>
<th>sample ID</th>
<th>SIMOX wafer</th>
<th>Ion implanted</th>
<th>Energy (keV)</th>
<th>Dose ($x10^{13}$ cm$^{-2}$)</th>
</tr>
</thead>
<tbody>
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<td>B1/BF$_{+}$/1</td>
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<td>BF$_{+}$</td>
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<td>0.8</td>
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</tr>
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<td>B1/BF$_{+}$/3</td>
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<td>&quot;</td>
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<td>2</td>
</tr>
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<td>B1/BF$_{+}$/4</td>
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<td>&quot;</td>
<td>&quot;</td>
<td>5</td>
</tr>
<tr>
<td>B1/BF$_{+}$/5</td>
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<td>&quot;</td>
<td>&quot;</td>
<td>8</td>
</tr>
<tr>
<td>B1/BF$_{+}$/6</td>
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<td>&quot;</td>
<td>&quot;</td>
<td>10</td>
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<td>0.8</td>
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<td>&quot;</td>
<td>&quot;</td>
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</tr>
<tr>
<td>B3/BF$_{+}$/3</td>
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<td>&quot;</td>
<td>&quot;</td>
<td>2</td>
</tr>
<tr>
<td>B3/BF$_{+}$/4</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>5</td>
</tr>
<tr>
<td>B3/BF$_{+}$/5</td>
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<td>&quot;</td>
<td>&quot;</td>
<td>8</td>
</tr>
<tr>
<td>B3/BF$_{+}$/6</td>
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<td>&quot;</td>
<td>&quot;</td>
<td>10</td>
</tr>
<tr>
<td>B4/BF$_{+}$/1</td>
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Table 4.4 - Details of samples implanted with BF$_{+}$ ions.

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<th>Ion implanted</th>
<th>Energy (keV)</th>
<th>Dose ($x10^{13}$ cm$^{-2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B4/Si$^+$/1</td>
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<td>Si$^+$</td>
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<td>&quot;</td>
<td>&quot;</td>
<td>7.5</td>
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<td>B4/Si$^+$/5</td>
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</tr>
<tr>
<td>B4/Si$^+$/6</td>
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<td>&quot;</td>
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Table 4.5 - Details of samples implanted with Si$^+$ ions.
<table>
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<th>Sample ID</th>
<th>SIMOX wafer</th>
<th>I on implanted</th>
<th>Energy (keV)</th>
<th>Dose (x10¹³ cm⁻²)</th>
</tr>
</thead>
<tbody>
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<td>B3(2)</td>
<td>Ge⁺</td>
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</tr>
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<td>10</td>
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<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>30</td>
</tr>
</tbody>
</table>

Table 4.6 - Details of samples implanted with Ge⁺ ions
4.1.5 Sacrificial oxidation

Standard SIMOX as well as BF$_2$, Ge$^+$ and Si$^+$ implanted SIMOX samples were thermally oxidised at the University of Surrey using a dedicated dry oxidation furnace. The furnace consisted of a quartz semi-open tube (3 inch in diameter) which enabled that a flux of oxygen to be flown continuously during the oxidation treatment. The temperature inside the furnace was monitored using a Pt-Pt (13%)/Rh thermocouple and the maximum temperature variation along the central zone of the furnace (along a zone of length 10 cm) was estimated to be ± 2 °C. A flow of 1.5 litre/min of pure oxygen gas was bled into the tube, approximately 10 min. before loading the samples and then reduced to 1.0 litre/min a couple of minutes before the oxidation in order to perform the thermal treatment under standard dry oxidation conditions [122]. The samples were, then, horizontally loaded into a quartz sample holder (5 cm long) which was placed inside the tube near its extremity. The quartz sample holder was maintained at that position until the air inside the tube was blown away (typically for 3 min.). Then, the sample holder was pushed into the centre of the furnace using a quartz rod. The loading time was kept short, typically one minute, in order to minimise the thermal oxidation during the loading procedure. The range of oxidation temperatures used varied from 800°C to 1100°C and the oxidation times from 30 min. to 74 hours. After the thermal oxidation, the samples were immersed in a HF(40%) solution for 2 min. to strip off the thermal oxide. The thickness of the thermal oxide was estimated measuring the height of an oxide window using a Rank Taylor Talystep stylus instrument. The oxide window was formed by protecting a selected area of the sample with a HF(40%) resistant mask. The final thickness of the silicon overlayer was determined by 1.5 MeV RBS analyses. Table 4.7 shows a summarised list of the samples oxidised in this project. A complete list of all
sacrificially oxidised samples is presented in Appendix E.

<table>
<thead>
<tr>
<th>Sample</th>
<th>SIMOX wafer</th>
<th>Oxidation temperature (°C)</th>
<th>Oxidation time (hours)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B1/1 → B1/20</td>
<td>B1</td>
<td>850/900/950 1000/1050/1100</td>
<td>0.5 → 60</td>
</tr>
<tr>
<td>B2/1 → B2/22</td>
<td>B2</td>
<td>900/1000/1050 1100</td>
<td>0.6 → 74</td>
</tr>
<tr>
<td>B3/1 → B3/23</td>
<td>B3</td>
<td>900/950/1000 1050/1100</td>
<td>0.5 → 74</td>
</tr>
<tr>
<td>B4/1 → B4/23</td>
<td>B4</td>
<td>850/900/950 1000/1100</td>
<td>0.5 → 60</td>
</tr>
<tr>
<td>B5/1 → B5/22</td>
<td>B5</td>
<td>900/950/1050 1100</td>
<td>0.5 → 60</td>
</tr>
<tr>
<td>B1/\text{BF}_2^-/1/1 → B1/\text{BF}_2^-/6/1</td>
<td>B1</td>
<td>1100</td>
<td>4</td>
</tr>
<tr>
<td>B3/\text{BF}_2^-/1/1 → B3/\text{BF}_2^-/6/1</td>
<td>B3(2)</td>
<td>1100</td>
<td>4</td>
</tr>
<tr>
<td>B4/\text{BF}_2^-/1/1 → B4/\text{BF}_2^-/7/1</td>
<td>B4(2)</td>
<td>1100</td>
<td>4</td>
</tr>
<tr>
<td>B4/\text{Si}^-/1/1 → B4/\text{Si}^-/14/1</td>
<td>B4(2)</td>
<td>1100</td>
<td>4</td>
</tr>
<tr>
<td>B4/\text{Ge}^-/1/1 → B4/\text{Ge}^-/11/1</td>
<td>B4(2)</td>
<td>1100</td>
<td>4</td>
</tr>
<tr>
<td>B3/\text{Ge}^-/1/1 → B3/\text{Ge}^-/11/1</td>
<td>B3(2)</td>
<td>1100</td>
<td>4</td>
</tr>
</tbody>
</table>

Table 4.7 - Sacrificially oxidised SIMOX samples.
4.2 Characterization methods.

Crystallographic defects in the SIMOX samples were characterized using chemical defect etching, plan view transmission electron microscopy (PVTEM), cross-section transmission electron microscopy (XTEM). Rutherford backscattering (RBS), double crystal X-ray diffraction (XRD) analysis and Secondary Ion Mass Spectroscopy (SIMS) were also used to analyze a limited number of samples.

4.2.1 Chemical defect etching.

Chemical defect etching was used to delineate crystallographic defects in the silicon overlayer and in the silicon substrate just beneath the BOX. A number of standard bulk Si etchants were used in this project (see Table 4.8). However, for the analysis of thin silicon overlayers (<1000 Å) it was found necessary to develop a new defect etchant capable of reliably deliniating crystallographic defects in these thin layers. Experimental results regarding the chemical composition and the main characteristics of this new etchant are presented in chapter 5.

<table>
<thead>
<tr>
<th>Silicone Etchant</th>
<th>Formula</th>
<th>Etch pit morphology</th>
</tr>
</thead>
<tbody>
<tr>
<td>Secco[123]</td>
<td>2 HF(49%): 1 K$_2$Cr$_2$O$_7$ (0.15 M)</td>
<td>isotropic</td>
</tr>
<tr>
<td>Schimmel[124]</td>
<td>2 HF(49%): 1 CrO$_3$ (1 M)</td>
<td>isotropic</td>
</tr>
<tr>
<td>Yang[125]</td>
<td>1 HF(49%): 1 CrO$_3$ (1.5 M)</td>
<td>Anisotropic</td>
</tr>
<tr>
<td>Wright[126]</td>
<td>60 ml HF(49%): 30 ml HNO$_3$: 30 ml CrO$_3$ (1 M): 60 HAc: 60 H$_2$O (1 gr. CuNO$_3$)</td>
<td>Anisotropic.</td>
</tr>
</tbody>
</table>

Table 4.8 - Bulk silicon etchants used in this project.
4.2.1.1 Etching experiment details

A. Etching temperature

All the etching experiments were carried out in a climate controlled room (20°C ± 1°C) at the University of Surrey. Additionally, the etchant temperature was further controlled by placing the beaker containing the etchant in a temperature controlled bath, for a couple of hours, before the commencement of the etching experiment. In this way, the etchant temperature was controlled over the range from 21 ± 1°C to 35 ± 1°C.

B. Etch rates

The etch rates of the chemical solutions were estimated by etching a partially masked silicon sample for a predetermined time. After removing the mask (HF resistant tape), the step height between the etched and non etched region was determined using a Rank Taylor Tallystep stylus instrument. Furthermore, the etch rates were double checked by estimating the etching time needed to completely remove the silicon overlayer of a standard SIMOX sample of known thickness (Tsi= 2000 Å). By so doing, the uncertainty was estimated to be less than ± 50 Å/min.

C. Ageing of the chemical solution.

In order to avoid problems with the deterioration of the etchant during storage, fresh solutions were mixed for each set of analyses. Additionally, the etch rates of the fresh solutions were always measured before beginning a new set of experiments.
**D. Determination of the defect etch pit densities.**

All the SIMOX samples submitted to chemical etching were, additionally, immersed in a HF(40%) solution for a minimum of 30 sec. and a maximum of 5 min. in order to form cavities underneath the etch pits (see Fig. 2.6). The double etched samples were, then, observed either by optical microscopy (OM) using an Olympus microscope or by scanning electron microscopy (SEM) using a Cambridge S250 Stereoscan electron microscope. The defect densities were estimated by counting the etch pits in a minimum of ten micrographs (optical microscopy or SEM) taken from the area of interest. The mean etch pit density and the standard deviation could then be estimated for every region of interest. The lateral distribution of defects in the SIMOX wafers was determined by measuring the etch pit densities in different areas of samples cut along the [110] diameter of the SIMOX wafers. In some cases, defect etch pit maps of whole SIMOX wafers were obtained. Furthermore, the depth distribution of defects was also obtained by determining the etch pit density as a function of the thickness of silicon removed (e.g. etching time). For this purpose, small samples cut from adjacent areas were etched for different times and the thickness removed was estimated measuring the step height at the edge of a non-etched region. Whenever possible, the etch pit results were correlated with plan view TEM or even XTEM analysis in order to determine the nature and geometry of the crystallographic defects.
4.2.2 Transmission electron microscopy

Characterization of crystallographic defects was carried out by transmission electron microscopy. Samples were observed under plan view and cross section views using the Jeol 200 CX and 2000 FX high resolution microscopes in the Microstructural Studies Unit (MSSU) at the University of Surrey. The preparation of the samples for TEM analyses is described in appendix D. After preparation the samples were loaded in a double tilt sample holder in order to accurately obtain the two beam condition[127] and also to scan through different zone axes. Determination of the Burgers vectors of the crystallographic defects was not carried out in this work. The areas analyzed by plan view TEM were larger than 1000 \( \mu m^2 \) enabling the determination of defect densities as low as \( 1.0 \times 10^5 \text{ cm}^{-2} \).

4.2.3 Rutherford Backscattering (RBS)

Rutherford backscattering (RBS) and channelling ([100] direction) analyses were used to determine the composition and crystalline quality of selected SIMOX samples. In this project, we have used the 2.0 MV Van de Graaff accelerator at the University of Surrey, to produce a 1.5 MeV or 1.0 MeV \( \text{He}^+ \) beam. The backscattered \( \text{He}^+ \) ions were collected using a silicon surface barrier detector, having an energy resolution of 16 keV. This detector was located inside the target chamber at an approximate angle of 32° to the incident beam axis. The energy spectrum of the backscattered \( \text{He}^+ \) ions was used to determine the mass and depth distribution of the component elements of the target. Furthermore, simulations of the RBS spectra were carried out, using the XRBSSIM[128]
simulation programme, in order to accurately determine both the thickness of the silicon overlayer and the buried oxide layer.

4.2.4 Other methods.

Double crystal X-ray diffraction (XRD) analysis was also used to determine the stress near the implanted/ non implanted area of a typical 'as-implanted' SIMOX wafer implanted with a dose of $3.0 \times 10^{17}$ O$^+$ cm$^{-2}$ and energies of 70 keV. The XRD experiments were performed by Dr. M. Lourenco using the 50 kV X-ray generator at the University of Surrey. The rocking curves were obtained by scanning through the (004) diffracted beam ($\theta_B = 34.58^\circ$) using the characteristic Cu $K_\alpha$ line (1.5432 Å).

Furthermore, Secondary Ion Mass Spectroscopy (SIMS) was also used to quantify the Cu concentration present in the (100) Cz-Si wafers implanted at the University of Surrey and at Eaton Corporation. Dr. A. Nejim is acknowledged for arranging for the SIMS analyses to be carried out.
Chapter Five: New etchant for thin SOI.

5.1 Introduction

In this chapter we present the experimental results associated with the development and optimisation of the new defect etchant, which was subsequently used in the study of crystallographic defects in thin silicon films (Chapters 6 and 7). TEM analyses have been used to validate the interpretation of the data obtained by chemical defect etching. Furthermore, the lateral and depth distributions of crystallographic defects in thick film SIMOX have been determined to investigate the uniformity of device grade wafers and also to show the improved selectivity of the new etchant.

This chapter contains two sections. In the first one (5.2), we present the results of the new defect etchant developed to delineate defects in ultra thin film silicon layers, while in the second section (5.3), we apply this new etchant together with TEM analysis to study the lateral and depth distribution of crystallographic defects present in thick SIMOX.
5.2 Development of an optimum defect etchant for thin silicon layers

5.2.1 Dilute silicon etchants

In chapter two, we have seen that although different etching systems have been developed for the study of bulk silicon materials, so far, no defect etchant has been specially developed for ultra thin silicon layers (< 1000 Å). Bulk silicon etchants, such as the Wright[123], Secco[124], Yang [125 ] and Schimmel [126], can be used to analyse thin silicon layers, however these solutions have etch rates of about 1.0 μm/min. or faster (see Fig. 5.1-a) and therefore, the removal of thin silicon overlayers is difficult to control, specially if the layer is as thin as 500 Å.

A simple way of decreasing the etching rate is to dilute the chemical solution by increasing the H₂O content. In figure 5.1 b) we plot the etch rates of different silicon etchants upon dilution. From Fig. 5.1 b) it is observed that the etch rates of these etchants decrease considerably as a function of the dilution. Indeed, the etch rate of the ten fold dilute Secco solution is decreased to approximately 200 Å/min, which is slow enough to allow good control of the removal of the silicon overlayer. However, a defect etchant can only be considered as reliable if all the crystallographic defects intersecting the surface are delineated. Therefore, it is also important to determine the effectiveness of the dilute solutions to delineate defects.
Fig. 5.1 - a) Thickness removed upon etching time and b) etch rate upon the number of times the solution has been diluted for the Wright, Yang, Schimmel and Secco etchants.
In order to assess the effects of the dilution on the formation of etch pits, two standard SIMOX wafers produced commercially by the IBIS Corporation, namely wafers A1 and A2 (silicon overlayer thickness of 2000 Å, see table 4.3), were etched, following the procedure described in section 4.2.1, and observed by optical and scanning electron microscopy (SEM). Selected samples cut from each of the wafers were analysed by plan view TEM and the threading dislocation density in these samples were estimated to be 

\[(1.0 \pm 0.5) \times 10^6/\text{cm}^2\] for wafer A1 and \[(9.0 \pm 1) \times 10^6/\text{cm}^2\] for wafer A2.

Figure 5.2 shows SEM micrographs of samples prepared from adjacent regions of wafer A1, etched with a two-fold dilute Wright solution for different etching times (15 sec, 30 sec and 35 sec). It is seen (see fig. 5.2 a to c) that the etch pit density increases with etching time and only reaches a value which corresponds to the crystallographic defect density determined from the TEM analyses when 1900 Å of silicon has been removed (e.g. for 35 sec etching time).
Figure 5.2- SEM micrographs of samples from wafer A1 etched with the 2 fold Wright etch for a) 15 sec. (850 Å) b) 30 sec (1650 Å) c) 35 sec. (1900 Å)

Similar experiments have been carried out for the other etchants and in Figure 5.3 we plot the etch pit density upon the thickness of silicon removed for the Schimmel, Yang, Wright and Secco dilute etchants using as standard material, samples prepared from adjacent regions of wafer A1.
Fig. 5.3 a- b (continued)
Figure 5.3 - Etch pit density against thickness of silicon removed for a) Wright, b) Yang, c) Schimmel and d) Secco dilute etchants (data collected from wafer Al:1.0x10^6 cm^-2; etchant temperature: 20 ±1°C).
From Fig. 5.3 (a-d) it is observed that for each of the etchants the etch pit density only reached a saturation value that corresponds to the defect density determined by plan view TEM (e.g. 1.0x10⁶/cm) when a certain minimum thickness of silicon had been removed. The minimum thickness (T_m) of silicon that needs to be removed to obtain reliable results depends upon the etchant and also upon the dilution of the solution. For example, for the 5-fold diluted Secco etch (Fig. 5.3 d), it is necessary to remove about 1100 Å in order to delineate all of the defects (density : 1.0x10⁶ cm⁻²).

![Figure 5.4- Etch rate against T_m for the Wright, Yang, Schimmel and Secco based solutions.](image)

Fig. 5.4 summarises the results of the different etchants in terms of etch rate against T_m. It is evident from Fig. 5.4 that silicon etchants such as the Wright and Yang etchants, give poor results since most of the silicon overlayer has to be removed (T_m > 1500 Å) to obtain a one to one correlation between etch pits and defects imaged in the TEM.
result is not unexpected since both the Yang and Wright etchants form well defined etch pits due to preferential etching of silicon in the {111} planes near the dislocation core and consequently, the formation of etch pits using these systems is very slow. Upon dilution the capability to form etch pits is further decreased and fewer than 1% of the crystallographic defects (see Fig. 5.3a) are delineated, even after removing almost all the silicon overlayer.

Preferential etchants that form isotropic etch pits (Schimmel and Secco) give better results. However, it is observed that Schimmel based solutions are inferior to the Secco based ones because more material has to be removed to obtain reliable results (see Fig. 5.4). $T_m$ for the standard Schimmel etchant is 1200 Å and increases rapidly for the dilute solutions. The best results were obtained for solutions having potassium dichromate ($K_2Cr_2O_7$) as the oxidising agent (e.g. Secco solutions). Indeed, the minimum thickness for the standard Secco is 700 Å and increases only to 1250 Å for the 10 fold dilute solution.

Considering that the initial thickness of the silicon overlayer was 2000 Å, these results mean, in practical terms, that all the dilute CrO$_3$ based solutions analysed in this work (e.g. Yang, Wright and Schimmel etchants) are not suitable for the study of ultra thin film SIMOX ($< 1000$ Å) since the formation of etch pits is not fast enough to allow the delineation of all crystallographic defects in the thin films. It is important to point out that the main difference between Secco and Schimmel based solutions is the presence of the alkali ions ($K^+$) in the former. We believe that the presence of $K^+$ ions in the solution increases the selectivity of the etchant. This assumption is backed by the early work from Gilman et al[129] which showed that the selectivity of chemical defect etchants was improved by the introduction of metal ions. They showed that the metals ions are

\[ \text{New etchant} \]
Etch rate (Å/min.) generally adsorbed on the active sites of the surface and inhibit the retreat of monomolecular steps across the surface, reducing their velocity \((V_s)\) without modifying the etch rate \((V_e)\) at the crystallographic defect (see appendix B).

The etching temperature is another parameter that influences the kinetics of the reaction. For example, Figure 5.5 shows the dependence of the etch rate upon temperature for the dilute Secco solutions. It is seen, that the etch rate increases linearly with temperature for the three Secco solutions investigated.

Figure 5.5 - Etch rate dependence of dilute Secco solutions upon temperature.

Figure 5.6 (a to d) show the temperature dependence of the etch pit density upon the thickness of silicon removed for samples prepared from adjacent areas of wafer A2 (defect density: \(9.0 \times 10^6 \text{ cm}^{-2}\)). All four sets of data, which were recorded using samples similarly etched at different temperatures, namely 21°C, 26°C, 31°C and 35°C, respectively show the same trend. The etch pit density reaches a saturation value between \(5.0 \times 10^6 \text{ cm}^{-2}\) and \(8.0 \times 10^6 \text{ cm}^{-2}\) after a particular thickness of silicon \(T_m\) has been removed.
Fig. 5.6 (a-b) continued
Figure 5.6 - Etch pit density versus thickness of silicon removed for samples prepared from wafer A2 at a) 21° C, b) 26° C, c) 31° C and d) 35° C.
It is also observed that the value of the minimum thickness $T_m$ is insensitive to temperature and depends only on the dilution of the solution, as previously observed (see fig. 5.4). This result suggests that the temperature of the solution does not accelerate the nucleation of etch pits.

Figure 5.7 is a typical plan view TEM micrograph from a sample prepared from wafer A2 etched for 2 min. (1000 Å removed) using the 5 fold dilute Secco solution. Three well delineated etch pits are observed while no defect contrast was seen in other regions indicating that all of the crystallographic defects were delineated.

Figure 5.7 - Typical plan view TEM micrograph of sample A2 etched for 2 min. using the 5 fold dilute Secco.

It is concluded that crystallographic defects can be reliably delineated using the 5 fold dilute Secco solution providing the silicon overlayer is greater than 1000 Å. However, for silicon layers thinner than 1000 Å, a new etchant is required.
5.2.2 New etchant

In section 5.2.1 we have shown that the effectiveness of the defect etchants was controllably reduced upon dilution of the solution (see Fig. 5.4). We believe that this decrease in the effectiveness of the dilute solutions is due to a decrease in the concentration of the oxidising agent reducing its etching capabilities (see appendix B). On the other hand, we have also showed, that the preferential etching characteristic of the solution will be enhanced by the presence of alkali ions (K⁺) in the solution. From these findings, we concluded that to obtain a highly preferential etchant we need to develop a new system with a strong oxidising power (i.e. with a high concentration of the oxidising agent) [125] and at the same time having a high concentration of alkali (K⁺) and metal (Cu⁺⁺) ions. The introduction of the metal ions being aimed to further inhibit the retreat of monomolecular steps and by so doing reduce the planar etch rate of the solution [129].

The new etchant that we developed in this project is based on the HF:HNO₃:H₂O:K₂Cr₂O₇ : Cu(NO₂)₃ system. The oxidising power of this system, compared to the HF:HNO₃:H₂O, is increased by the presence of Cr⁺⁺ ions (see appendix B) and at the same time the introduction of K⁺ and Cu⁺⁺ ions in the solution will inhibit the retreat of monomolecular steps. An optimum formula was obtained by changing the relative quantities of the etchant components in order to obtain the highest etching anisotropy with the slowest planar etch rate. The optimum formula for this new system is 50 ml HF(40%):70 ml HNO₃(70%):170 ml H₂O (1.0 gr. K₂Cr₂O₇ + 4.0 gr. Cu(NO₂)₃):H₂O, which was established by carrying out a large number of etching experiments.
In Figs. 5.8 and 5.9 we present calibration data for the new chemical solution. Figure 5.8 (a) and (b) show the etch rate and dependence of the etch rate with temperature, respectively.

The new silicon etchant is found to be well behaved, as the removal of silicon varies linearly with increasing time and also the etch rate has a linear dependence upon temperature. The etch rate of the new solution at room temperature (21°C) is 450 Å/min, which ensures good control of the dissolution of silicon from the thinnest films (≤ 500 Å).

Fig 5.9 (a) and (b) show the dependence of the etch pit density in samples cut from wafers A1 and A2 upon the thickness of silicon removed. For comparison we have also plotted data obtained using dilute Secco solutions. It is clear from Fig. 5.9 (a) and (b) that the minimum thickness $T_m$ for the new solution is about 500 Å for samples from both wafers. The increase in the effectiveness of the new etchant confirms our supposition that the introduction of Cu$^{++}$ and K$^+$ ions inhibits the retreat of monomolecular steps across the surface, reducing the dissolution rate of the surface without modifying the etch rate at the crystallographic defect and, therefore, enabling the formation of large etch pits even when the thickness of silicon removed is small.

The evolution of etch pits using this new etchant is shown in Figure 5.10 (a-d) which includes plan-view TEM micrographs of samples prepared from wafer A2 after different etching times. For an etching time of 7 sec. (Fig. 5.10 a) we observe that no preferential etching of dislocations has occurred. The small amount of material removed (about 50 Å) is insufficient to show nucleation of etch pits at the site of the dislocations. The onset of nucleation (Fig. 5.10 b) appears to occur after the removal of 100 Å (14 sec.) of material.
It is noted that the growth of the etch pits appears not to be homogeneous during the initial stage and that initially not all the dislocations have resolvable etch pits.

Fig. 5.8 a) Thickness removed upon etching time and b) dependence of the etch rate upon temperature for the new etchant.
Fig. 5.9 - Etch pit density upon thickness of silicon removed for samples prepared from wafers a) A1 and b) A2.
Fig. 5.10 (continued)
Fig. 5.10 - Plan view TEM micrographs of samples prepared from adjacent samples from wafer A2 etched for a) 7 sec., b) 14 sec., c) 24 sec., d) 60 sec., and e) 120 sec. using the new etchant.
After the removal of 150 Å of material (Fig 5.10 c) etch pits are visible at the sites of all resolvable dislocations, however the diffraction contrast indicates that the etch pits have not penetrated completely through the silicon overlayer. When 450 Å of the silicon overlayer has been removed (Fig. 5.10 d), it is clear that well defined etch pits have formed, with all the silicon around all of the dislocations being etched away. These observations agree closely with the data shown in Fig. 5.9. Upon a further increase in the amount of material removed (900 Å, Fig. 5.10 e) the etch pit size increases considerably but the etch pit density is unchanged, since there are no further dislocations to nucleate additional etch pits in this material. Analysis by plan view TEM of samples etched using the 5 fold dilute Secco solution showed similar trends, although the amount of material needed to be removed for the onset of nucleation was considerably greater (250 Å) and the etch pits only penetrated through the top silicon layer when about 900 Å of silicon had been removed. These results give confirmation that the formation of etch pits is indeed a nucleation-dependent process. From the nucleation theory of etch pits (Appendix B) it is known that the probability that an etch pit will be nucleated at a dislocation is directly proportional to the undersaturation of the medium (Si surface/etchant) and to the square of the Burgers vector of the defect. In terms of this theory the different etching times for the etchants (new etchant and dilute Secco etchant) to nucleate etch pits can be explained by the presence of inhibitors that decrease the free enthalpy required for nucleation[20].

Fig. 5.11 shows the diameter of the etch pits as a function of the thickness of silicon removed, where the data has been collected from plan view TEM micrographs. It is seen that the new chemical solution forms etch pits that are larger by a factor of two than those formed by the five-fold dilute Secco. For 900 Å of silicon removed the diameter of
the etch pit produced by the new chemical etch is 0.2 µm, whilst it is less than 0.1 µm for the 5 fold dilute Secco. This feature illustrates, once more, the increased effectiveness of the new solution over the 5 fold dilute Secco.

\[
\begin{align*}
\text{Diameter of the etch pit (1e-2 µm)}
\end{align*}
\]

\[
\begin{align*}
\text{Thickness of Si removed (Å)}
\end{align*}
\]

**Fig. 5.11 - Etch pit diameter against thickness of silicon removed.**

The use of this new etchant is further reported in section 5.3 of this chapter and in chapters 6 and 7. It is shown that threading dislocations are successfully delineated in thin silicon layers by using the new etchant. Stacking faults (SF) in thin layers (Sample B6, silicon overlayer thickness: 500 Å) are also delineated using this etchant (see fig. 5.12). Identification of stacking faults has been made possible by the faceted nature of the etch pits and their orientation along <110> directions. Fig. 5.13 shows SF present in another sample which has been thermally oxidised. It is observed that SF can be distinguished from a pair of dislocations if the projected length of the fault is larger than 0.5µm.

New etchant
**Fig 5.12** - SEM micrograph of SF observed in thin SIMOX ($Ti = 500 \text{ Å}$).

**Fig 5.13** - SEM micrograph of threading dislocations and SF observed in sacrificially oxidised SIMOX. It is observed that SF can be distinguished from a pair of dislocations if the projected length of the SF is greater than $0.5 \text{ μm}$. 
5.3 Distribution of defects in thick SIMOX.

In this section we use the new chemical defect etchant to determine the distribution of defects present in the silicon overlayer of standard SIMOX wafers produced commercially by the IBIS and LETI Corporations.

5.3.1 Lateral distribution of defects.

Figure 5.14 a) shows the lateral distribution of defects from the centre to the edge of wafer A2. It is observed that although small variations are evident (from $8.0 \times 10^6 \text{ cm}^2$ to $6.0 \times 10^6 \text{ cm}^2$), the overall defect distribution is relatively constant suggesting that the vendors have good control of the processing parameters during implantation and annealing. Lateral uniformity could also be evaluated from the uniform interference colour (green) observed over the whole wafer and was found qualitatively to be good.

This result is contrasted with the one presented in Fig. 5.14 b) obtained from another SIMOX wafer (A3) which had a large colour difference between the centre (green) and the edge of the wafer (pink). A difference in the defect density of one order of magnitude is observed from the centre ($6.0 \times 10^9 \text{ cm}^2$) to the edge ($6.0 \times 10^7 \text{ cm}^2$) of this wafer (Fig. 5.14 b).
Fig. 5.14 - Etch pit density distribution from the centre to the edge of a) wafer A2 and b) wafer A3. The colour (by eye) of the layers is indicated at the top of the figure.
RBS analysis of different areas of the wafer A3 (see Fig. 5.15) confirmed that the silicon overlayer and buried oxide layer thicknesses were significantly different in the centre and the edge of the wafer. With the aid of an RBS computer simulation programme it was possible to determine that the thickness of the silicon overlayer near the edge of the wafer was 2100 ± 60 Å whilst it was only 1900 ± 60 Å close to the centre. On the other hand, the BOX thickness near the edge was thicker (4100 ± 50 Å) than in the centre (3900 ± 50 Å). These results, associated with the different interference colours (green to pink), indicate that lateral dose inhomogeneities occurred during implantation. The increased defect density in the centre of the wafer is attributed to the higher implanted dose which will increase the number of point defects created during implantation and lead to greater lattice disorder.
5.3.2 Defect distribution- depth

The depth distribution of defects in the silicon overlayer was also determined. Fig. 5.16 shows the etch pit density upon the thickness of silicon removed for two multiple implanted SIMOX substrates, namely wafers A4 and B1 (see table 3.2). It is observed that the etch pit density (threading dislocations) for both materials is around $1.0 \times 10^3$ cm$^{-2}$. However, an increase in the etch pit density for thickness higher than 1000 Å is observed for both wafers which saturates at a value of about $3.0 \times 10^6$ cm$^{-2}$. We speculate that this increase in the etch pit density is caused either by the presence of localized defects or by irregularities found near the Si/SiO$_2$ interface.

Plan view TEM analysis of a sample prepared from wafer A4, confirmed that the
threading dislocation density is below the resolution limit for this technique (e.g. < 1.0x10^7 cm^-2) and also revealed the existence of small extended defects in the silicon overlayer. Fig. 5.17 shows a plan view TEM micrograph of a sample prepared from wafer A4 observed under weak beam conditions. It is possible to observe alpha fringes inside two opposite {111} faulted planes. This contrast is characteristic of a stacking fault pyramid: (SFP)[130]. The SFP density measured was 1.0x10^7 cm^-2, explaining the increase in the etch pit density observed in Fig. 5.16. From Fig. 5.16 and 5.17, we conclude that the increase in the etch pit density is due to the presence of SFP localized near the back of the Si overlayer. Although the etch pit density is approximately 3 times lower than the actual SFP density estimated by plan view TEM analyses, we speculate that this difference is due to the different magnifications used to estimate the defect densities.

5.17 Plan view TEM micrograph of sample A4 under weak beam conditions. An extended defect is observed. Fringes inside two faulted planes are clearly visible.
The presence of non threading defects was also observed in other SIMOX wafers. Fig. 5.18 (a-b) include plan view TEM micrographs of a sample prepared from wafer B4. In Fig. 5.18 a) two paired threading dislocations can be observed. The threading dislocation density estimated by plan view TEM analysis of this particular sample was $5.0 \times 10^5 \text{ cm}^2$.

In Fig. 5.18 b) an extended defect is observed which we conclude is an elongated SFP since the edges of the projected defect are orientated along $\langle 110 \rangle$ directions, and contrast from the (111) and ($\bar{1}$11) planes are visible under $(g=220)$ and invisible under $(g=\bar{2}20)$.

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Fig. 5.18- Plan view TEM of a sample prepared from wafer B4. a) A pair of threading dislocations and b) an elongated SFP are observed.
Fig. 5.19 (a-b) are XTEM micrographs of sample prepared from wafer B4. Fig. 5.19 a) and b) are respectively a bright field and dark field micrographs of the silicon/BOX interface of a sample prepared from wafer B4. From Fig. 5.19, it is observed that a small stacking fault complex is localized in a region close to the Si/SiO$_2$ interface. Due to poor statistics a meaningful estimation of the small SF complex density was not possible, however the height of this particular defect was estimated to be of 100 Å. Furthermore, XTEM analyses have also shown the presence of these defects in the silicon substrate below the BOX layer (see Fig. 5.20). The presence of small SF complexes far from the substrate/BOX interface suggests that some of them are stacking fault tetrahedra (SFT).

PV TEM analyses of a similar sample prepared from wafer B4, enabled us to determine the density of these defects (SFP/SFT) as $2.0 \times 10^7 \text{ cm}^{-2}$. A distribution of sizes is apparent with a mean length of 0.1 μm (see Fig. 5.21).
Fig. 5. 19- XTEM micrographs from a sample prepared from wafer B2 under a) bright field and b) dark field.
Figure 5.20: Bright field XTEM micrograph from a sample prepared from wafer B4 showing the presence of a SF complex below the BOX layer.
Table 5.1 summarises the crystallographic defect densities observed in standard SIMOX wafers produced by IBIS and LETI during the period 1989 to 1992. The dislocation densities were estimated using the new chemical etchant whilst the SFP/SFT densities were determined by PVTEM analyses. In general we find that in single implanted materials the threading dislocation density is lower for LETI than for IBIS samples being ~1.0x10^5 cm^2 and 1.0x10^6 cm^2 respectively. On the other hand, IBIS material contains the lower SFP/SFT densities.
<table>
<thead>
<tr>
<th>SIMOX wafer</th>
<th>Supplier</th>
<th>Implantation method</th>
<th>Dislocation density (cm⁻²)</th>
<th>SFP/SFT density (cm⁻²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>IBIS</td>
<td>single</td>
<td>1.0x10⁶</td>
<td>1.0x10⁶</td>
</tr>
<tr>
<td>A2</td>
<td>&quot;</td>
<td>&quot;</td>
<td>8.0x10⁶</td>
<td>5.0x10⁵</td>
</tr>
<tr>
<td>A3</td>
<td>&quot;</td>
<td>&quot;</td>
<td>50.0 /6.0 x10⁵</td>
<td>**</td>
</tr>
<tr>
<td>A4</td>
<td>&quot;</td>
<td>multiple</td>
<td>1.0x10³</td>
<td>1.0x10⁷</td>
</tr>
<tr>
<td>B1</td>
<td>LETI</td>
<td>multiple</td>
<td>1.0x10³</td>
<td>&lt; 1.0x10⁴</td>
</tr>
<tr>
<td>B2</td>
<td>&quot;</td>
<td>single</td>
<td>5.0x10⁴</td>
<td>1.0x10⁶</td>
</tr>
<tr>
<td>B3</td>
<td>&quot;</td>
<td>&quot;</td>
<td>5.0x10⁴</td>
<td>1.0 x10⁷</td>
</tr>
<tr>
<td>B3(2)</td>
<td>&quot;</td>
<td>&quot;</td>
<td>5.0x10⁴</td>
<td>1.0x10⁷</td>
</tr>
<tr>
<td>B4</td>
<td>&quot;</td>
<td>&quot;</td>
<td>5.0x10⁵</td>
<td>2.0x10⁷</td>
</tr>
<tr>
<td>B4(2)</td>
<td>&quot;</td>
<td>&quot;</td>
<td>5.0x10⁵</td>
<td>2.0X10⁷</td>
</tr>
<tr>
<td>B5</td>
<td>&quot;</td>
<td>&quot;</td>
<td>8.0x10⁵</td>
<td>3.0x10⁷</td>
</tr>
<tr>
<td>B6&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>1.0x10³</td>
<td>**</td>
</tr>
</tbody>
</table>

* Table 5.1 - Threading dislocation and SFP/SFT densities in standard SIMOX

*: The silicon overlayer thickness of wafer B6 was 500 ±50 Å.
**: SFP/SFT density not measured.
Chapter Six: Results I

Sacrificially oxidised SIMOX

6.1 Introduction

In this chapter we report the use of the new etchant to study the nature and density of defects present in thin SIMOX materials produced by sacrificial oxidation. Furthermore, data from plan view TEM and RBS analyses are also reported to provide complementary characterizations.

This chapter is divided in two sections. In the first one (6.2) we characterize the thin silicon overlayer in terms of the nature and density of crystallographic defects produced during sacrificial oxidation of thick SIMOX materials whilst in section 6.3 we describe an experiment to control the density of OISF by the implantation of $BF_2^+$, Ge$^-$ or Si$^+$ ions prior to the oxidation. The aim of this latter experiment is to annihilate the nucleation sites for OISF by selectively amorphising the region near the Si/SiO$_2$ interface, leaving the near surface region relatively free of damage in order for it to be used as a seed for a solid phase epitaxial (SPE) regrowth during a subsequent thermal treatment.
6.2 Defects in sacrificially oxidised SIMOX.

6.2.1 Crystallographic defects in standard SIMOX.

In section 5.3 we have shown that the main defects present in the silicon overlayer of thick SIMOX substrates are threading dislocations and small SF complexes (SFP/SFT). We have also shown that SIMOX wafers showing a uniform light interference colour also showed a uniform distribution of defects. Furthermore, we observed that although the nominal implantation and annealing conditions were the same, samples implanted in different batches contained different defect densities. Indeed, the range of defect densities observed in wafers from different batches varied from $10^3$ cm$^2$ to $8 \times 10^6$ cm$^2$ for threading dislocations and from $10^5$ cm$^2$ to $3.0 \times 10^7$ cm$^2$ for SF complexes (see table 5.1).

6.2.2 Crystallographic defects in sacrificially oxidised SIMOX.

Selected samples (1 cm$^2$) cut from different SIMOX wafers (namely B2, B3, B4 and B5, see table 5.1) were sacrificially oxidised following the procedure described in section 4.1.5. The thicknesses of the silicon overlayer of the sacrificially oxidised SIMOX samples were measured by RBS analyses. Fig. 6.1 shows 1.5 MeV He$^+$ RBS spectra from samples (cut from wafer B2) submitted to dry oxidations at 1100°C for different times. The RBS analyses were carried out after removing the sacrificial oxide using a HF(40%) solution. From Fig. 6.1, it is observed that the thickness of the silicon overlayer decreases according to the oxidation time. The estimation of the thicknesses of the silicon overlayers was carried out by simulation using the XRBSSIM programme[128]. The simulated data
Counts showed that the thicknesses of the silicon overlayer (T=2500 Å) were reduced to 1000 Å and 500 Å after oxidation times of 4 hrs. and 8 hrs., respectively. The thicknesses of the sacrificial oxide layers were previously estimated by measuring the step height of an oxide window using a Rank Taylor Tallystep stylus instrument (see section 4.1.5). The RBS data together with the Tallystep trace analysis showed that the ratio between the thickness of the oxide and the consumed silicon was approximately 2.3 which is consistent with published values[60].

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Fig. 6.1- 1.5 MeV He⁺ RBS spectra from wafer B2 and samples sacrificially oxidised at 1100 °C for 4 hrs. and 8 hrs.
Fig. 6.2 shows the dependence of the oxide thickness upon oxidation time for SIMOX samples oxidised in dry oxygen at different temperatures. For comparison we have also plotted the theoretical values obtained from the Deal-Grove model [131]. It is observed that the oxidation rate of the silicon overlayer in our SIMOX materials is similar to that predicted for bulk silicon, and thus, suggesting that the existence of the buried oxide layer (BOX) does not affect the oxidation rate.

![Graph showing oxide thickness vs. oxidation time for SIMOX samples](image)

**Fig. 6.2- Dependence of the oxide thickness upon time for SIMOX samples oxidised in dry O$_2$ at different temperatures.**

Fig. 6.3 shows the dependence of the etch pit density upon thickness of silicon removed for a sample cut from wafer B2 and oxidised at 900$^\circ$C under dry oxidation conditions for 2 hours. The thickness of silicon consumed was 200 Å. As a reference, data from a non oxidised sample cut from wafer B2 is also included. The data presented in Fig. 6.3...
was collected by estimating the etch pit density in a minimum of ten (SEM) micrographs for each point (see section 4.2.1) and shows the depth distribution of the crystallographic defects in the oxidised and non-oxidised material. From Fig. 6.3 two different regions are observed. Up to 1500 Å of silicon removed, the etch pit density in both oxidised and non-oxidised samples is found to be the same (5.0 ± 1x10⁴ cm⁻²). However, the etch pit density for the oxidised sample increases by a factor of six close to the back of the silicon overlayer (at an estimated depth of 1700 Å), suggesting an increase in the defect density at approximately 800 Å from the top Si/SiO₂ interface.

![Graph showing etch pit density vs. thickness of silicon removed](image)

**Fig. 6.3-** Etch pit density upon thickness of silicon removed from a SIMOX substrate (B2) oxidised at 900°C for 2 hrs. Data from wafer B2 before oxidation is included as a reference.

Fig. 6.4 a-b-c) show plan view TEM micrographs of samples cut from wafer B2 oxidised at 900°C for 2, 10 and 24 hours respectively. In Fig. 6.4 a) we can observe a well defined oxidation induced stacking fault (OISF) bounded by a dislocation loop (arrowed). The
density and mean size of the OISF have been determined from a set of micrographs recorded using the two beam condition \((g = 220)\) at a magnification of 30 thousand times. The OISF density estimated in this sample is \(5.0 \times 10^5 \text{ cm}^{-2}\) and the mean size of the OISF is 0.2 \(\mu\text{m}\). Furthermore, detailed analysis by plan view TEM has shown that the density of threading dislocations is not modified during a 2-hour dry oxidation at 900°C. This result together with data presented in Fig. 6.3 suggests that for short oxidation times (< 2 hours), the OISF are formed in the silicon overlayer close to the Si overlayer/BOX interface. The micrographs shown in Fig. 6.4 b and c) show that the OISF grow larger during longer oxidation times at this temperature. In Fig. 6.4 b, (10 hours), it is observed that the OISF grow and eventually reach the silicon surface (arrowed) which we propose is by capturing interstitials emitted during oxidation [132]. Upon a further increase in the oxidation time (24 hours) the OISF grows laterally, being bounded by two partial dislocations to a length of approximately 2 \(\mu\text{m}\).(see fig. 6.4 c).
Fig. 6.4 - Plan view TEM of samples prepared from wafer B2 after dry oxidation at 900°C for a) 2 hrs. b) 10 hrs and c) 24 hrs

Fig. 6.5 (a-b) are SEM micrographs of OISF in samples prepared from wafer B2 and B3 submitted to 900°C (10 hrs) and 1100°C (4 hrs.) respectively. The OISF have been delineated by etching the samples for 1 min. using the new etchant.
Fig. 6.5- SEM micrographs of OISF which are delineated by the new etchant. a) wafer B2 after dry oxidations at 900 °C for 10 hours and b) wafer B3 after oxidation at 1100°C for 4 hours.

It is observed that the mean OISF length is strongly dependent upon the oxidation temperature having an average length of 1.0 μm (900 °C, 10 hrs) and 35 μm (1100°C, 4
hrs) in these two representative samples. Fig. 6.6 shows the dependence of the mean length of the OISF upon the oxide thickness (i.e. oxidation time, see Fig 6.2) for samples prepared from wafer B2. It is observed that the OISF length increases linearly with increasing oxide thickness for different temperatures. Similar results were obtained from samples prepared from other wafers. A complete list showing the length and densities of OISF of all the sacrificial oxidised SIMOX samples is presented in Appendix E.

Fig. 6.6 Dependence of the mean length of OISF upon oxide thickness (wafer B2).

Fig. 6.7 shows the dependence of the OISF density upon the thermal oxide thickness for wafers B1, B2 and B3. From Fig. 6.7, it is observed that before the oxidation treatment,
the OISF density measured was below the sensitivity limit of the optical microscope (typically less than $10^2 \text{ cm}^2$). However, after growing a thin oxide (say 500 Å) all samples cut from the same wafer, presented a constant OISF density. Furthermore, it is also observed that the OISF density is insensitive to temperature and to oxide thickness (i.e. oxidation time).

The areal density of OISF along the [110] diameter of wafer B3 was also quantified. Fig. 6.8 shows the OISF distribution in a sample cut along the diameter of wafer B3 and oxidised at 1000°C for 16 hours. It is observed that the OISF density is constant within the mean standard deviation which is typically ± 50%, except at a distance of less than 5 mm from the [110] flat edge. The data points do appear to show a periodic variation which may be reflecting a lateral non uniformity. However, as the variation is within the estimated uncertainty further measurements would be required to establish if this variation is real or an experimental artifact. For comparison we have also plotted the distribution of OISF along the diameter of a wafer sacrificially oxidised in the clean room facility at the LETI laboratory (wafer B6). It is observed that the OISF density is very similar to the values $(1.5 \pm 0.5 \times 10^4 \text{ cm}^2)$ determined from the oxidised sample cut from wafer B3 (see Fig. 6.8), which we present as evidence for the growth of the OISF not being a feature of the particular equipment or laboratory.
Fig. 6.7 Dependence of OISF density upon oxide thickness for different SIMOX materials (wafers B1, B2 and B3) oxidised at 900°C, 950°C, 1000°C and 1100°C.
Fig. 6.8 - Density of OISF along a diameter of wafer B3. Data from wafer B6 is also presented for comparison.
We conclude from the results presented in Fig. 6.7 and 6.8 that the nucleation of OISF in our sacrificially oxidised SIMOX materials is controlled by the presence of nucleation sites prior to oxidation. It is important to emphasise that all of the SIMOX substrates chosen for the oxidation experiments were produced in the same laboratory (LETI) and that the main difference between the substrates was the density of crystallographic defects. These results, then, suggest that crystallographic defects (SF complexes, threading dislocations) present in the silicon overlayer before oxidation are the main nucleation sites for the OISF. However, we conclude that the nucleation sites are not threading dislocations since specimens B3/22 and B5/23 (see table 6.1) had different threading dislocation densities, yet, the density of the OISF is very similar. In table 6.1 we also list the SFP/SFT densities observed in different samples before oxidation. Although the experimental data is limited, there is a trend for the density of the OISF to be lower in samples with a higher density of SFT/SFP before oxidation. This behaviour seems to indicate that the small SF complexes (SFP/SFT) act as effective sinks for silicon interstitials that are emitted during the thermal oxidation[133]. However, it is important to point out that this trend was not observed for a multiple implanted SIMOX sample (B1/19, see table 6.1) in which no SFP/SFT were resolved under plan view TEM (e.g. < 1.0x10^4 cm^-2) before oxidation and the OISF density was very low (5.0x10^2 cm^-2). Therefore, it is not clear if the SF complexes act as sinks for self interstitials or are the effective nucleation sites for OISF. In order to test whether the SFP (or SFT) act as effective sinks for point defects, samples cut from wafer B2 were annealed at high temperatures (1150°C) for a prolonged time (24 hours) in a N_2 ambient. By so doing, vacancies generated during the nitridation process[133] could promote the growth or shrinkage of the SF complexes.

Results I
<table>
<thead>
<tr>
<th>SIMOX sample</th>
<th>Implantation method</th>
<th>Threading Dislocation density before oxidation (cm$^{-2}$)</th>
<th>SFT/SFP density before oxidation (cm$^{-2}$)</th>
<th>OISF density (cm$^{-2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B2/22</td>
<td>single</td>
<td>5.0x10$^4$</td>
<td>1.0x10$^6$</td>
<td>3.0x10$^5$</td>
</tr>
<tr>
<td>B3/22</td>
<td>&quot;</td>
<td>5.0x10$^4$</td>
<td>1.0x10$^7$</td>
<td>2.0x10$^4$</td>
</tr>
<tr>
<td>B4/24</td>
<td>&quot;</td>
<td>5.0x10$^5$</td>
<td>2.0x10$^7$</td>
<td>1.0x10$^4$</td>
</tr>
<tr>
<td>B5/23</td>
<td>&quot;</td>
<td>8.0x10$^5$</td>
<td>3.0x10$^7$</td>
<td>8.0x10$^3$</td>
</tr>
<tr>
<td>B1/19</td>
<td>multiple (SIA)</td>
<td>1.0x10$^3$</td>
<td>&lt;1.0x10$^3$</td>
<td>5.0x10$^2$</td>
</tr>
</tbody>
</table>

Table 6.1 - OISF densities observed in different samples, oxidised at 1100°C for 4 hours in dry oxidation conditions.
Fig. 6.9 shows a plan view TEM micrograph of an extended defect observed in a sample cut from wafer B2 and submitted to a thermal annealing at 1150°C for 24 hours in a N₂ ambient. The contrast of this defect is very similar to that observed in Fig. 5.18 b, suggesting that it is an elongated SFP (l = 0.5 μm). Fig. 6.10 (a-b) are plan view TEM micrographs in a) dark and b) bright field contrast of another region of the same sample. In Fig. 6 a) and b) an extended defect (l = 1.0 μm) is clearly observed. The extended defect was again identified as being an SFP since the two elongated faulted planes are orientated along the [110] direction and the contrast from the two small planes are clearly observed under dark field contrast (see Fig. 6.10 b).

**Fig. 6.9- Plan view TEM micrograph of a SIMOX sample submitted to a thermal annealing in a N₂ ambient (1150°C)**
Fig. 6.10- Plan view TEM micrographs in  a) bright and b) dark field contrast of a long SFP observed in a sample (B3) after a prolonged annealing at 1150°C for 24 hours in N₂ ambient.
The increase in size of the SFP from 0.1 μm (before the N₂ annealing, see Fig. 5.18 b) to 1.0 μm (after the N₂ annealing) suggests that SFP can grow by capturing vacancies and therefore, can act as effective sinks for point defects. However, it is important to point out that not all the SFP have grown to sizes as large as 1.0 μm. In fact, the density of long SFP (1 = 1.0 μm) was estimated to be $10^5$ cm$^{-2}$. This result could suggest that not all the SFP in SIMOX are intrinsic and therefore, it is probable that some of them can also be eliminated during a thermal anneal in a nitrogen ambient.

To further investigate if single extrinsic SF can also be eliminated by the capture of vacancies, we annealed (1150°C, 24 hours in N₂) a sample which was previously oxidised at 900°C for 24 hours (cut from wafer B2), resulting in an OISF density of $3.0 \times 10^5$ cm$^{-2}$. Fig. 6.11 shows the lateral variation of the OISF density in the silicon overlayer along a 30 mm wide sample (cut from wafer B2 and oxidised at 900°C for 24 hours), before and after the annealing in the N₂ ambient. The data was obtained by etching (SEM) adjacent areas of the same sample before and after the high temperature annealing in the N₂ ambient. From Fig. 6.11, it is observed that after the annealing, the OISF density is reduced from $3.0 \pm 1 \times 10^5$ cm$^{-2}$ to $1.0 \pm 0.3 \times 10^4$ cm$^{-2}$. 

108 Results I
Fig. 6.11 - OISF densities in a sample (cut from wafer B2) which was thermally oxidised at 900°C for 24 hours and then further annealed in a nitrogen ambient at 1150°C for 24 hours. The OISF density before annealing is also plotted for comparison.
6.3 Sacrificial oxidation of ion implanted SIMOX materials.

In section 6.2, we have shown that the OISF density in sacrificially oxidised SIMOX materials depends upon the presence of SFP/SFT at the back of the silicon overlayer. A method to determine the influence of these defects on the formation of OISF is to annihilate the SFP/SFT by selectively amorphising the silicon in the vicinity of the Si/SiO₂ interface by ion implantation. Subsequently, the amorphous region can be (thermally) epitaxially regrown using the top of the silicon overlayer as a seed.

Fig. 6.12 (a - c) show 1.5 MeV He⁺ RBS/channelling spectra from SIMOX samples implanted with different doses of a) BF₂⁺, b) Ge⁺ and Si⁺. The samples implanted with BF₂⁺ were cut from wafer B1 while samples implanted with Ge⁺ and Si⁺ were cut from wafer B3(2) (see table 4.3). From Fig. 6.12 (a-c) it is observed that for doses of 8.0x10¹⁴ BF⁺/cm², 1.7x10¹⁴ Ge⁺/cm² and 1.0x10¹⁵ Si⁺/cm², the channelled yield between ch. 220-260 is the same as the non channelled (random) spectra. Therefore, we conclude that the threshold dose for the amorphisation is approximately 8.0x10¹⁴ BF⁺/cm², 1.7x10¹⁴ Ge⁺/cm² and 1.0x10¹⁵ Si⁺/cm² for the BF₂⁺, Ge⁺ and Si⁺ implantations respectively. We also conclude that the amorphous region is confined to the back of the silicon overlayer and that the top silicon layer remains relatively free of damage.
*Fig. 6.12 (a-b) continued.*
Fig. 6.12- 1.5 MeV He⁺ RBS/channelling spectra from SIMOX samples implanted with different doses of a) BF⁺ b) Si⁺ and c) Ge⁺ ions

Fig. 6.13 (a-b) are XTEM micrographs of samples (cut from wafer B3(2)) implanted with $\phi = 1.0 \times 10^{15}$ Si⁺ cm⁻² under a) bright and b) dark field contrast. From the dark field micrograph (Fig. 6.13 b) we can see that an amorphous region has been formed near the back of the silicon overlayer confirming the results obtained by RBS analyses (see Fig. 6.12 b). It is also observed that the silicon surface, although defective, remains crystalline.
Fig. 6.13 - XTEM micrographs of a sample (cut from wafer B3(2)) implanted with $\phi = 1.0 \times 10^{15}$ Si cm$^{-2}$ (190 keV) under a) bright and b) dark field contrast.
The silicon overlayer was regrown during thermal treatments in Ar (700°C, 30 min.) and in dry oxygen (1100°C, 10 min.) ambients. Fig. 6.14 is a XTEM micrograph of a sample (wafer B3(2)) implanted with a dose of $\phi=5.0 \times 10^{14}$ BF$_2$cm$^{-2}$ and thermally treated at 1100°C for 10 min. It is observed that the silicon overlayer is completely regrown after the thermal treatment. Similar results were obtained for the thermal treatments in Ar ambients (700°C, 30 min.).

Fig. 6.14- XTEM micrograph of SIMOX B3(2) implanted with $5.0 \times 10^{14}$ BF$_2$cm$^{-2}$ and submitted to a thermal treatment (1100°C, 10 min., dry O$_2$)
Analysis by plan view TEM of samples implanted with BF$_2^+$, Si$^+$ and Ge$^+$ ions and thermally treated at 1100 °C for 10 min. in dry O$_2$ were carried out in order to determine the quality of the regrown silicon overlayer. Fig. 6.15 is a plan view TEM micrograph of a sample (wafer B3(2)) implanted with $\phi = 1.0 \times 10^{15}$ BF$_2^+$ cm$^{-2}$ and thermally treated at 1100°C for 10 min. in dry O$_2$. It is observed that a high density of dislocation loops is formed (2.0x10$^5$ cm$^{-2}$) during the regrowth process. Similar results were obtained for samples implanted with Si$^+$ with the threshold dose for amorphisation ($\phi = 1.0 \times 10^{15}$ Si$^+$ cm$^{-2}$) and thermal treated under the same conditions (see Fig. 6.16).

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**Fig. 6.15** - Plan view TEM micrograph from a sample (wafer B3(2)) implanted with $1.0 \times 10^{15}$ BF$_2^+$ cm$^{-2}$ and thermally treated at 1100°C for 10 min. in dry O$_2$. The DL density estimated from PV TEM observations was 2.0x10$^5$ cm$^{-2}$. 

*Results 1*
On the other hand, no dislocation loops nor SF complexes were observed by plan view TEM ($< 10^5 \text{ cm}^2$) of samples implanted with Ge$^+$ ions with the threshold dose for amorphisation ($\phi = 1.7 \times 10^{14} \text{ Ge}^+ \text{ cm}^2$) and submitted to a thermal treatment at 1100°C for 10 min in dry $O_2$. However, upon further increasing the Ge$^+$ dose ($\phi = 2.5 \times 10^{14} \text{ Ge}^+ \text{ cm}^2$), the dislocation loop density increases to a value of $7.0 \times 10^6 \text{ cm}^2$ (see Fig. 6.17).
Fig. 6.17 - a) bright and b) dark field plan view TEM micrographs of a sample (cut from wafer B3(2)) implanted with 2.5x10^{14} Ge^+/cm^2 (1100°C, 10 min. dry O). A DL density of 7.0x10^6 cm^{-2} was observed.
Fig. 6.18 is a SEM micrograph of a sample (cut from wafer B3(2)) implanted with Ge⁺ (\( \phi = 1.7 \times 10^{14} \text{ Ge}^+ \text{ cm}^{-2} \)) and oxidised at 1100°C for 4 hours in dry O₂ after defect etching. The mean OISF density estimated from a set of SEM micrographs was 5.0 \times 10^2 \text{ cm}^{-2}.

On the other hand, samples implanted with the threshold dose for amorphisation with BF₂ ions showed a high density of OISF after an oxidation at 1100°C for 4 hours in dry O₂ (see Fig. 6.19). The OISF density estimated by plan view TEM analysis was 1.0 \times 10^7 \text{ cm}^{-2}. Chemical etching plus SEM analysis of an adjacent area of the same sample showed that a network of SF existed, which we suggest are formed due to the interaction of OISF lying in different \{111\} planes (see Fig. 6.20).
Fig. 6.19 - Plan view TEM micrograph of a sample (prepared from wafer B3(2)) implanted with $\phi = 1.0 \times 10^{15}$ BF$_2$ cm$^{-2}$ and oxidised at 1100°C, 4 hours in a dry oxygen ambient. OISF are clearly identified by the characteristic fringe contrast in the plane of the fault and also by the partial dislocations that bound the fault.

Fig. 6.20- SEM micrograph of a sample (prepared from wafer B3(2)) implanted with $\phi = 1.0 \times 10^{15}$ BF$_2$ cm$^{-2}$ and oxidised at 1100°C for 4 hours in dry oxygen. The sample was etched using the new etchant for 1 min.
Fig. 6.21 (a-c) show the dependence of the OISF density upon the ion dose after oxidising the samples at 1100°C for 4 hours in dry oxygen. From Fig. 6.21 a) it is observed that for doses close to the threshold dose for Ge⁺ the OISF density decreases to approximately 5.0x10³ cm⁻² indicating that a very low density of nucleation sites are present in the silicon overlayer before the oxidation. However, for higher doses (ϕ = 2.5x10¹⁴ Ge⁺ cm⁻²) the OISF density increases significantly due to the increase in dislocation loop density during the regrowth process (see Fig. 6.17). We conclude that the effective annihilation of SF complexes gives rise to a low OISF density, thus, suggesting that they act as effective nucleation sites for OISF.

In Fig. 6.21 b) we observe that although a decrease in the OISF density is not obtained, an interesting trend is apparent. In the high dose regimes (Φ > 5.0x10¹⁴ BF⁺₂ cm⁻²) the OISF density is strongly dependent upon the BF⁺₂ dose. This is due to a high density of dislocation loops (2.0x10⁸ cm⁻²) that are formed during the regrowth process (see Fig. 6.14). We believe that the dislocation loops act as the main nucleation sites for OISF. For intermediate doses (1.0x10¹⁴ BF⁺₂ cm⁻² < Φ < 5.0x10¹⁴ BF⁺₂ cm⁻²), the nucleation process of OISF is more complex since the SFT/SFP are not completely removed and therefore, they coexist with the dislocation loops produced during the regrowth process. We, therefore, conclude that in this case, the OISF density is primarily dependent on the SFP/SFT density found in the original SIMOX (see table 5.1) and only partially dependent upon the implanted dose (see Fig. 6.21b). For lower doses (ϕ ≤ 10¹⁴ BF⁺₂ cm⁻²) no significant increase in the OISF density has been observed indicating that crystallographic defects present before the amorphisation process are the main nucleation sites for OISF.

In Fig. 6.21 c) it is observed that for sub-threshold doses (Φ < 1.0x10¹³ Si⁺ cm⁻²) the OISF density is independent of the Si⁺ dose.
Fig. 6.21 a) OISF density against Ge⁺ dose after oxidation at 1100°C for 24 hours in dry oxygen.
Fig. 6.21 b) OISF density against $BF_2^-$ dose after oxidation at 1100°C for 4 hours in dry oxygen.
**Fig. 6.21 c**) - OISF density against Si$^+$ dose after oxidation at 1100°C for 4 hours in dry oxygen.
However, for doses equal or higher than $1.0 \times 10^{15}$ Si$^+$ cm$^{-2}$, an increase in the OISF density is observed. This increase, as in the case of Ge$^+$ and BF$_2^+$ implantations, is due to the nucleation of the dislocation loops during the solid phase epitaxial regrowth process (see Fig. 6.15).

In order to determine the effects of the regrowth ambient on the formation of secondary defects, short thermal annealings (1100°C, 10 min.) under N$_2$ ambients were used to regrow the amorphous regions of the BF$_2^+$, Si$^+$ and Ge$^+$ implanted SIMOX samples. Once more, low DL densities were determined for samples implanted with $\phi = 1.7 \times 10^{14}$ Ge$^+$ cm$^{-2}$ ($\rho < 10^5$ cm$^{-2}$) while a considerably higher density of dislocation loops ($\sim 10^8$ cm$^{-2}$) were observed for the BF$_2^+$ implanted samples, indicating that, for our processing conditions, the formation of DL seems not to depend upon the regrowth ambient. The reason why the amorphisation with Ge$^+$ ions gives better results is not well known, however recent work by Laanab et al [134] have shown that the density of secondary defects (known as 'end of range' defects [135]) formed during the solid phase epitaxial (SPE) regrowth of amorphous silicon depends upon the number of excess interstitials at the amorphous/crystalline interface. Moreover, Laanab[136] have shown that Ge$^+$ implants (compared to Si$^+$ implants) give rise to a lower number of excess interstitials, and perhaps, explains the low DL densities observed in our Ge$^+$ Implanted samples.

Finally, an interesting result observed for samples implanted with Si$^+$ ions and annealed at 1100°C for 10 min. in a N$_2$ ambient is the evolution of the dislocation loops into paired threading dislocations (arrowed in Fig.6.22 a-b). We presume that this process occurs by the climb of vacancy type perfect dislocation loops due to the capture of vacancies that are found in super saturation under those conditions[133]. This result is interesting since it confirms the assumption, made separately by Stoemenos [48] and Venables [51], that
the formation of threading dislocations in SIMOX occurs through a climb process of perfect dislocation loops.

Fig. 6.22 - Plan view TEM micrographs of samples implanted with Si a) $\phi = 1.0 \times 10^{15} \text{ Si}^+ \text{ cm}^{-2}$ and b) $\phi = 2.5 \times 10^{15} \text{ Si}^+ \text{ cm}^{-2}$ annealed at $1100^\circ \text{C}$ for 10 min. in a $N_2$ ambient.
Chapter Seven: Results II
Low energy oxygen implants

7.1 Introduction.

In this chapter, we report experimental studies of crystallographic defects in thin film SIMOX substrates produced by low energy and low dose O⁺ implantation. The new defect etchant (chapter 5) was used to delineate defects and complementary data is obtained by RBS, XTEM and PV TEM methods. Additionally, SIMS and double crystal X-ray diffraction analyses were also used to further characterize a selected number of samples.

This chapter contains two sections. In Section 7.2 we report experiments to characterize thin film SIMOX wafers produced by Eaton Corporation while in Section 7.3 we characterize crystallographic defects in thin film SIMOX substrates produced at the University of Surrey.
7.2 Wafers prepared by Eaton Corporation.

7.2.1 General description.

The wafers prepared by Eaton Corporation were implanted with low energies (30 keV or 40 keV) and low doses (typically $2.2 \times 10^{17}$ O$^+$ cm$^2$) with the specific purpose of producing a thin overlayer above a thin continuous buried oxide (BOX). The doses were selected to be close to the critical dose $\Phi_c$ for the formation of a continuous BOX layer according to the semi-empirical model proposed by Li et al.[59] (see section 2.1). Table 7.1 shows the main analytical methods used in this project to determine the microstructure and the crystalline quality of the wafers prepared by Eaton Corporation.

<table>
<thead>
<tr>
<th>Wafer</th>
<th>Ion implanted</th>
<th>Energy (keV)</th>
<th>Dose $x 10^{17}$ (O$^+$ cm$^2$)</th>
<th>Analytical Methods</th>
</tr>
</thead>
<tbody>
<tr>
<td>E1</td>
<td>$^{32}$O$^+$</td>
<td>60</td>
<td>2.2</td>
<td>RBS/XTEM/PVTEM</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Chemical etching + SEM/Optical microscopy</td>
</tr>
<tr>
<td>E2</td>
<td>$^{32}$O$^+$</td>
<td>60</td>
<td>2.2</td>
<td>RBS/XTEM/PV TEM</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Chemical etching + SEM/Optical microscopy</td>
</tr>
<tr>
<td>E3</td>
<td>$^{16}$O$^+$</td>
<td>30</td>
<td>2.2</td>
<td>RBS/PV TEM</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Chemical etching + SEM/Optical microscopy</td>
</tr>
<tr>
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<td>$^{16}$O$^+$</td>
<td>40</td>
<td>2.2</td>
<td>RBS/PV TEM</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Chemical etching + SEM/Optical microscopy</td>
</tr>
<tr>
<td>E5</td>
<td>$^{16}$O$^+$</td>
<td>40</td>
<td>2.4</td>
<td>RBS/PV TEM</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Chemical etching + SEM/Optical microscopy</td>
</tr>
<tr>
<td>E6</td>
<td>$^{16}$O$^+$</td>
<td>40</td>
<td>2.4</td>
<td>RBS/PV TEM</td>
</tr>
<tr>
<td></td>
<td></td>
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<td></td>
<td>Chemical etching + SEM/Optical microscopy</td>
</tr>
<tr>
<td>E7</td>
<td>$^{16}$O$^+$</td>
<td>40</td>
<td>3</td>
<td>RBS/PV TEM</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Chemical etching + SEM/Optical microscopy</td>
</tr>
</tbody>
</table>

Table 7.1 - Description of the analytical methods to determine the microstructure and crystalline quality of the wafers prepared by Eaton Corporation.
The microstructure of the SIMOX wafers was determined by RBS and XTEM analyses while the nature and density of crystallographic defects in the silicon overlayer and in the substrate below the BOX were determined by chemical defect etching (SEM/optical microscopy) and PVTEM analyses.

7.2.2 Microstructure

Fig. 7.1 shows a typical 1.0 MeV He\(^+\) RBS spectrum from wafer E1. The two main features of this spectrum are marked by the letters A and B. In region A, the reduced backscattered yield of the He\(^+\) ions is due to the reduced density of silicon atoms in the buried oxide layer whilst the increase in the backscattered yield, in region B, is due to the overlapping of the oxygen and silicon signals. From Fig. 7.1, due to the abruptness of the features A and B, we conclude that wafer E1 has a well defined buried oxide layer. In order to determine if the signal from region A corresponds to stoichiometric SiO\(_2\), we have carried out simulations using the XRBSSIM programme[128]. The accurate fitting to the experimental data of the simulated spectrum for a test structure containing a BOX layer of 500 Å confirms that a stoichiometric SiO\(_2\) layer exists in the Eaton wafer. Furthermore, the thicknesses of the silicon overlayer and buried oxide layer were also determined by matching the experimental spectrum with the simulated one. RBS analyses of other Eaton wafers were carried out and table 7.2 summarises the determined thicknesses of the silicon overlayers and buried oxide layers.
Table 7.2 - Si overlayer and BOX layer thicknesses estimated by using computer simulation of the RBS spectra.
Fig. 7.2 (a-b) are typical XTEM micrographs taken from a representative area of wafer E1 showing respectively, bright and dark field images. From these and other micrographs, it was found that the structure of wafer E1 consists of a single crystal silicon overlayer of thickness $500 \pm 20 \text{ Å}$, beneath which a thin continuous buried oxide layer exists of thickness $500 \pm 20 \text{ Å}$. All of these thicknesses determinations agreed consistently with the RBS data (table 7.2). No silicon islands [137] were found inside the BOX layer after extensive observations by XTEM suggesting that their density is below the practical sensitivity limit for XTEM analysis, which is typically $10^7 \text{ cm}^2$. Additionally, crystallographic defects were not observed either in the silicon overlayer or in the silicon substrate confirming that the crystallographic defect density in these layers also is below the sensitivity limit for XTEM analysis.
Fig. 7.2 - XTEM micrographs of a typical region of wafer E1 in a) bright and b) dark field.
Chemical defect etching (optical microscopy and SEM) and PVTEM analyses have been used to quantify the density and depth distribution of crystallographic defects in the thin silicon overlayers of wafers prepared by Eaton.

Fig. 7.3 shows the dependence of the etch pit density against thickness of silicon removed from a representative sample (No 6, Fig. 7.6) cleaved from wafer E1. It is observed that for half of the thickness removed the etch pit density reaches a saturation value of $4.0 \pm 2 \times 10^4$ cm$^{-2}$. However, upon etching to greater depths ($> 350$ Å) the etch pit density increases to approximately $1.0 \times 10^7$ cm$^{-2}$. From Fig. 7.3 and results presented in section 5.3 (see for example Fig. 5.16), we conclude that although the density of threading dislocations is low ($4.0 \times 10^4$ cm$^{-2}$), a high density of extended but non threading defects ($\sim 1.0 \times 10^7$ cm$^{-2}$) is present at the back of the silicon overlayer. Similar results were obtained from other wafers implanted by Eaton.

![Graph showing the dependence of etch pit density against thickness of silicon removed](image)

**Fig. 7.3 - Dependence of the etch pit density upon thickness of silicon removed from a representative area of wafer E1.**
Analyses by plan view TEM of samples prepared from wafer E1 indicates the presence of small SFP (arrowed) at the back of the silicon overlayer (see Fig. 7.4). The maximum size of these defects was measured to be 300 Å and the SFP density was $\approx 8 \times 10^6$ cm$^{-2}$. Furthermore, no threading dislocations were observed by plan view TEM analysis confirming that the threading dislocation density in wafer E1 is lower than the sensitivity limit of plan view TEM (typically less than $10^4$ cm$^{-2}$). The plan view TEM analyses together with the results presented in Fig. 7.3 suggest that a high density of SFP are located close to the silicon /BOX interface. It is important to point out that the presence of SF complexes at the back of the silicon overlayer is not simply related to the energy and dose of the oxygen implantation since these defects were also observed in samples implanted with high energy and high doses (see section 5.3). The formation of SF complexes in SIMOX is discussed in chapter 8.

**Fig. 7.4- Plan view TEM micrographs of a sample prepared from wafer E1 showing two small SFP.**
7.2.4 Defect distribution - lateral

The lateral distribution of threading dislocations across the diameter of wafer E1 is shown in Fig. 7.5. while in Fig. 7.6 we show a map of the density of etch pits for the same wafer (the schematic showing the 48 areas analyzed by chemical etching is also shown).

From Fig. 7.5 and 7.6, it is observed that the average defect density near the centre of the wafer is $1.0 \times 10^4 \text{cm}^2$. However, small variations from $3.0 \times 10^3 \text{cm}^2$ to $2.0 \times 10^4 \text{cm}^2$, which are greater than the experimental uncertainty, were observed. It is speculated that this non uniformity is associated with temperature inhomogeneities during implantation or during the subsequent thermal annealing.
Fig. 7.5 - Lateral variation of the etch pit density along the [110] diameter of wafer E1.
Fig. 7.6: Whole wafer defect etch pit density map (wafer E). A schematic of the areas analyzed is also shown.
Defect densities higher than $10^7$ cm$^{-2}$ were observed near the borders of the areas masked from the oxygen ion beam (see fig. 7.6). These highly defective areas extended several millimetres inside the implanted area and are symmetrically distributed around the wafer. Fig. 7.7 (a-c) show plan view TEM micrographs of samples prepared from the highly defective areas. From Fig. 7.7 b) it is observed that a high density of SF complexes are present in these regions. The density of these defects is very high and has been estimated to be $5.0 \times 10^9$ cm$^{-2}$. From Fig. 7.7 c) we can clearly see that the size of these defects is not uniform, and they consist mainly of SFP (labelled X) and SFT (labelled Y). The distinction between the SFP and SFT was carried out by determining the direction of projection of the faulted planes. The SF complexes having the projected edges orientated along <110> directions were identified as being SFP whilst defects having the projected edges orientated along <100> directions were identified as being SFT[130]. The origin and growth of the SFP/SFT in SIMOX are discussed in chapter 8.
Fig. 7.7 - a and b (continued)
The existence of high defect densities in areas near the non-implanted regions were observed in other Eaton wafers. Fig. 7.8 (a-b) show etch pit density maps close to the non-implanted area for wafers E2 and E3. It is observed that a high defect density (\( > 10^7 \) cm\(^2\)) is always observed close to the edge of the non-implanted area. The data shown in Fig. 7.8 suggests that the increased defect density is related to processing problems during implantation or annealing of the 6 inch wafers. A further discussion of the reasons for this anomalous behaviour is given in chapter 8.
Fig. 7.8 Defect maps of regions close to the non-implanted border for wafers a) E2 and b) E3.
At a distance of approximately > 1cm from the implanted/non implanted boundary, the
defect density is considerably lower for all of the Eaton wafers. Fig 7.9 (a-b) show
typical SEM micrographs of defect pits in wafer E7 etched using the new etchant for 35
sec. It is observed that although a relatively small amount of silicon has been removed
(250 Å), well defined paired etch pits can be clearly seen, identified by the arrows in Fig
7.9 b.

Fig. 7.9  a) and b) SEM micrographs of an area close to the centre of wafer E7
etched using the new etchant for 35 sec.
The etch pit density distribution along a 35 mm long sample cut from a region close to the centre of wafer E7 is shown in Fig 7.10. It is observed that the defect density has a mean value of $2.0 \times 10^6$ cm$^{-2}$.

![Lateral etch pit density distribution along a 35 mm sample cut from a region near the centre of wafer E7.](image)

The mean threading dislocation densities measured for all Eaton wafers are listed in table 7.3 where it be can seen that the lowest defect densities were observed in wafers implanted with the lowest doses and energies. Another interesting result is that the defect density of wafers implanted with molecular oxygen ($^{16}$O$_2^-$) (E1 and E2) were lower (by more than one order of magnitude) than wafers implanted with atomic oxygen ($^{16}$O$^+$).
This result, although not conclusive (since further analysis of other wafers are needed to confirm the trend), suggests at least that the defect density is not increased due to the use of molecular oxygen and, therefore, opens a way to achieve low cost thin film SIMOX structures. Moreover, the results presented in table 7.3 suggest that good quality thin film SIMOX structures can be obtained using existing equipment (NV-10-80 implanter) and hence development costs for a new generation of low voltage machines will be avoided, providing a route to achieve thin film SIMOX substrates without incurring a cost penalty.

<table>
<thead>
<tr>
<th>SIMOX wafer</th>
<th>Ion implanted</th>
<th>Energy (keV)</th>
<th>Dose (O^+ \text{cm}^{-2})</th>
<th>Implantation temperature (^\circ\text{C})</th>
<th>Si overlayer thickness (Å)</th>
<th>Dislocation density</th>
</tr>
</thead>
<tbody>
<tr>
<td>E1</td>
<td>$^{32}\text{O}^+$</td>
<td>60</td>
<td>2.2x10$^{17}$</td>
<td>530</td>
<td>500 ± 50</td>
<td>1.0x10$^4$</td>
</tr>
<tr>
<td>E2</td>
<td>&quot;</td>
<td>60</td>
<td>2.2x10$^{17}$</td>
<td>&quot;</td>
<td>530 ± 50</td>
<td>1.0x10$^4$</td>
</tr>
<tr>
<td>E3</td>
<td>$^{16}\text{O}^+$</td>
<td>30</td>
<td>2.2x10$^{17}$</td>
<td>&quot;</td>
<td>550 ± 50</td>
<td>5.0x10$^5$</td>
</tr>
<tr>
<td>E4</td>
<td>&quot;</td>
<td>40</td>
<td>2.2x10$^{17}$</td>
<td>&quot;</td>
<td>630 ± 50</td>
<td>5.0x10$^5$</td>
</tr>
<tr>
<td>E5</td>
<td>&quot;</td>
<td>&quot;</td>
<td>2.4x10$^{17}$</td>
<td>550</td>
<td>650 ± 50</td>
<td>2.0x10$^5$</td>
</tr>
<tr>
<td>E6</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>600</td>
<td>600 ± 50</td>
<td>5.0x10$^6$</td>
</tr>
<tr>
<td>E7</td>
<td>&quot;</td>
<td>&quot;</td>
<td>3.0x10$^{17}$</td>
<td>510</td>
<td>700 ± 50</td>
<td>2.0x10$^6$</td>
</tr>
</tbody>
</table>

*Table 7.3 - Mean threading dislocation densities found in Eaton thin film SIMOX wafers.*
7.2.5 *Crystallographic defects in the silicon substrate.*

The crystalline quality of the silicon substrate below the BOX layer was also assessed using the new defect etchant. Before the defect etchant was applied the silicon overlayer of the SIMOX sample was stripped off using a KOH etch and the buried oxide layer was removed by HF(40%) etching (see section 4.1). It is important to point out that the exposed surface was featureless and was free of etch pits prior to the chemical defect etching.

Fig. 7.11 shows a typical SEM micrograph of the silicon substrate below the BOX layer of wafer E1 after defect etching. The main defects observed in this and other micrographs were stacking faults (SF) having a mean density of $5.0 \times 10^{4}$ cm$^{-2}$ (labelled X). Analysis of the silicon substrate below the BOX of other Eaton wafers have also shown the presence of SF with a similar density ($5.0 \times 10^{4}$ cm$^{-2}$).

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*Fig. 7.11 - SEM micrograph of the substrate below the BOX layer of wafer E1 after chemical etching.*
7.2.6 Defects associated with edge effects.

The crystalline quality of the non-implanted area of the Eaton wafers was also assessed. Analysis by SEM and optical microscopy (after chemical defect etching) of the non-implanted areas of wafers E1, E2 and E3 show the presence of stacking faults with a mean density of $5.0 \times 10^4$ cm$^{-2}$. The overall distribution of SF in the non-implanted area is uniform, although at a distance of approximately 20 µm away from the visible ion implanted boundary (IIB), SF are distributed in bands separated by a denuded zone 20 µm wide (see Fig. 7.12 a-b). Analysis of other areas of wafers E1, E2 and E3 have shown that the SF are essentially parallel to the visible IIB. We believe that these bands appear due to a stress related process caused by the lateral pinning of the silicon overlayer. It is important to point out that no SF were observed inside the implanted area. A discussion about the stress related edge effects is given in section 8.3.1.

Fig. 7.12 a- (continued).
Fig. 7.12 a) and b) SEM micrographs showing the SF band close to the ion implanted boundary of wafer E1 observed after defect etching.
7.3 Wafers prepared at the University of Surrey.

7.3.1 General description.

The wafers prepared at the University of Surrey were implanted with doses equal or greater than the critical dose for the formation of a continuous buried oxide layer at energies of 70 keV, 90 keV and 200 keV [59]. Table 7.4 shows the main analytical methods used to determine the microstructure and characterize the crystallographic defects.

<table>
<thead>
<tr>
<th>Wafer</th>
<th>Energy (keV)</th>
<th>Dose (O'cm² x10¹⁷)</th>
<th>Implantation temperature (°C)</th>
<th>Analytical methods</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1/G2/G3 G5/G6</td>
<td>70</td>
<td>3.3</td>
<td>650/550/700 700/600/500</td>
<td>RBS/channeling Chemical etching (SEM/Optical microscopy) PVTEM</td>
</tr>
<tr>
<td>G4</td>
<td>70</td>
<td>3.3</td>
<td>700</td>
<td>RBS/channeling Chemical etching (SEM/Optical microscopy) PVTEM Double X ray diffraction</td>
</tr>
<tr>
<td>G14/G15 G16/G17 G18/G19 G20/G21</td>
<td>200</td>
<td>4/5 5/6 6/7 7/5</td>
<td>700</td>
<td>RBS/channelling Chemical etching (SEM/Optical microscopy) PVTEM</td>
</tr>
<tr>
<td>(100) Cz Si Wacker</td>
<td>**</td>
<td>**</td>
<td>**</td>
<td>SIMS Chemical etching</td>
</tr>
</tbody>
</table>

**: sample non implanted. 

Table 7.4 - Description of the analytic methods used to determine the crystalline quality of the SIMOX wafers prepared at the University of Surrey.
7.3.2 SIMOX Structures

The structure of the wafers prepared at the University of Surrey were determined using RBS/channeling analyses. Fig. 7.13 shows typical 1.5 MeV He⁺ RBS spectra from wafers G12, G10, G13 implanted at energies of 90 keV and doses of $3 \times 10^{17}$ O⁺ cm², $5 \times 10^{17}$ O⁺ cm² and $1 \times 10^{18}$ O⁺ cm², respectively. From Fig. 7.13, it is observed that the thickness of the BOX layer increases with increasing ion dose. The thicknesses of the Si overlayer and BOX layer have been determined by curve fitting of computed spectra, generated using XRBSSIM [128], to the experimental data. The thicknesses estimated by RBS for the wafers prepared at the University of Surrey are summarised in table 7.5.

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**Fig. 7.13—1.5 MeV RBS spectra of samples G12, G10 and G13 implanted at 90 keV with doses of $3 \times 10^{17}$ cm², $5 \times 10^{17}$ cm² and $1 \times 10^{18}$ cm², respectively.**

---

Results II
<table>
<thead>
<tr>
<th>Sample</th>
<th>Energy (keV)</th>
<th>Dose(x10^17) O'/cm²</th>
<th>Current density (µA/cm²)</th>
<th>Implant temperature (°C)</th>
<th>Anneal ambient</th>
<th>Anneal time (hrs.)</th>
<th>Silicon thickness (Å) ± 60 Å</th>
<th>BOX layer thickness (Å) ± 60 Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>70</td>
<td>3.3</td>
<td>3.1</td>
<td>650</td>
<td>Ar + 0.5%O₂</td>
<td>6</td>
<td>1200</td>
<td>800</td>
</tr>
<tr>
<td>G2</td>
<td>70</td>
<td>3.3</td>
<td>*</td>
<td>550</td>
<td>&quot;</td>
<td>6</td>
<td>1200</td>
<td>800</td>
</tr>
<tr>
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<td>70</td>
<td>3.3</td>
<td>*</td>
<td>700</td>
<td>&quot;</td>
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<td>*</td>
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<td>3.3</td>
<td>&quot;</td>
<td>700</td>
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<td>6</td>
<td>1300</td>
<td>800</td>
</tr>
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<td>G5</td>
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<td>3.3</td>
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<td>700</td>
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<td>1500</td>
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<td>&quot;</td>
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<td>6</td>
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<td>1500</td>
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Table- 7.5 Thickness of the Si overlayer and SiO₂ layer.
It is found that the silicon overlayer and BOX thicknesses of the samples implanted at 70 keV with the critical dose for the formation of a continuous buried oxide layer ($\phi = 3.3 \times 10^{17} \text{ O}^+ \text{ cm}^{-2}$) are $1200 \pm 60 \text{ Å}$ and $800 \pm 60 \text{ Å}$, respectively. Similar silicon overlayer and BOX thicknesses were obtained for samples implanted under similar conditions but with different implantation temperatures showing a good reproducibility in the samples produced at the University of Surrey. It is also observed that the thinnest film ($600 \text{ Å}$) were formed in samples implanted with a dose of $\phi = 1.0 \times 10^{18} \text{ O}^+ \text{ cm}^{-2}$ and energy of 90 keV (wafer G13, see Fig. 7.13). On the other hand, the thickest silicon overlayers ($4300 \text{ Å}$) were observed in low dose samples implanted with 200 keV.

7.3.3 Defect distribution - lateral.

Chemical defect etching associated with SEM observations were used to determine the lateral distribution of the areal density of crystallographic defects within a 30 mm central implanted region. Fig. 7.14 shows a schematic of the central implanted area of the 3" wafers used to determine the lateral variation of the crystallographic defects.

7.14- Schematic showing the central area of the 3 inch SIMOX used to determine the lateral distribution of the areal density.
Fig. 7.15 (a-b) show SEM micrographs from two different areas of wafer G1 (E= 70 keV, 3.3x10^17 cm^2, Ti = 650 °C), namely areas A and B (see Fig. 7.14), etched using the new etchant for 1 min. It is observed that the etch pit density is considerably different in distinct areas of wafer G1. Indeed, a difference of more than one order of magnitude was observed from one side (region A: 5.0x10^6 cm^2) to the other of the implanted area (region B: 1.0x10^4 cm^2) of wafer G1.
Fig. 7.16 shows the distribution of the etch pit density across the implanted area of wafer G1 and G2. It is seen that for specimen G1 (Ti = 650 °C) a low etch pit density is only observed on one side of the implanted area where the density is ≈ 5.0x10^6 cm⁻². Furthermore, it is observed that for wafer G2, the etch pit density is relatively uniform across the implanted area. It is important to point out that both wafers G1 and G2 were implanted under the same conditions except that the implantation temperature for wafer G1 was 650 °C whilst it was only 550 °C for wafer G2.

Plan view TEM analyses were carried out in order to determine the nature of the defects present in wafer G1. Fig. 7.17 is a typical plan view TEM micrograph of the silicon overlayer of a sample taken from the highly defective area of wafer G1. It is observed that
the most numerous crystallographic defects present in the silicon overlayer are threading dislocations (labelled X), with a density of $2.0 \times 10^4 \text{ cm}^2$. Identification of the defects as threading dislocations was possible due to the classical stress contrast associated with inclined dislocations [127].

Fig. 7.17 - Plan view TEM micrograph from a sample cut from area A of wafer G1.

Fig. 7.18 shows the lateral distribution of the areal density of defects obtained by chemical defect etching (SEM observations) across the implanted area of wafer G18 ($\phi = 6.0 \times 10^{17} \text{ O}^- \text{ cm}^2$, $E= 200 \text{ keV}$, $T_i = 700^\circ\text{C}$) confirming that a high lateral non uniformity is typical of the SIMOX substrates prepared at Surrey at high implantation temperatures.
From the results presented in Fig. 7.16 and 7.18, it is evident that the non uniformity of the defect densities is more accentuated for the wafers implanted at temperatures greater or equal to 650°C. From Figure 7.18, it is also apparent that the presence of areas, in the same wafer, containing different defect densities is independent of the oxygen dose and energy. We, therefore, speculate that this behaviour is due to thermal gradients across the wafer during implantation which cause large thermal stresses. These thermal inhomogeneities are accentuated because only a 6.45 cm$^2$ area is implanted causing a large gradient of temperature across the wafer. Indeed, an approximate temperature difference of 70°C between the centre and edge of the wafer was determined using a pyrometer for samples implanted at 700°C[138,139]

In order to better understand the reasons for the non uniform nucleation of crystalloigraphic defects inside the implanted area, we annealed for shorter times (2
hours, 1300 °C) a wafer implanted under the same conditions as wafer G1, namely wafer G3. Fig. 7.19 shows the lateral variation of the defects inside the implanted central area of wafer G3 delineated by chemical etching plus SEM observations. Again a similar pattern is evident, namely non uniformity of the areal defect density confirming that the effect is independent of the post implantation annealing process.

![Graph](image)

**Fig. 7.19 - Lateral etch pit density distribution across the implanted area of wafer G3. The data was obtained after etching the sample for 1 min. using the new etchant.**

Fig. 7.20 a) and b) are SEM micrographs from two different regions of wafer G3. It is observed that in the region of high defect density, etch pits are distributed in larger clusters (7.20 a), whilst in the region of lower defect density the etch pits are single or paired (7.20 b)
Fig. 7.20 - SEM micrographs of samples from the a) high defect density region and b) low defect density region of wafer G3 (after 1 min. etching).

From Fig. 7.20, it is concluded that the presence of regions containing the high defect densities occurs due to a higher degree of overlapping of large defect clusters. We propose that these clusters of dislocations are nucleated in the regions of highest stress (discussed further in section 8.3.1).
7.3.4 Evolution of threading dislocations during annealing.

The evolution of threading dislocations during the high temperature thermal annealing process was assessed by plan view TEM analysis of a sample implanted with $\phi = 6.0 \times 10^{17} \text{ O}^+ \text{ cm}^{-2}$ (200 keV) and annealed for 2 hours at 1300°C, namely wafer G21. Fig. 7.21 is a plan view TEM micrograph of the silicon overlayer of a sample taken from the implanted area of wafer G21. It is observed that a high density of dislocations (labelled D) are pinned to large SiO$_2$ precipitates (labelled P). Some of the dislocations, however, are only partially pinned (labelled T) to the SiO$_2$ precipitates and thread through the silicon overlayer. The observation of dislocations totally and partially pinned to large SiO$_2$ precipitates suggests that the former are the precursors of the threading dislocations in low dose SIMOX samples. We propose that during the annealing these dislocations evolve into threading dislocations by a non conservative climb process during the anneal[117]. This assumption is backed by recent proposals made by Venables et al [51] that the evolution of threading dislocations in high dose SIMOX materials occur through a stress assisted climb process (see section 2.2.).
Fig. 7.21 - Plan view TEM micrograph from a sample cut from wafer G21. Dislocations pined (labelled D) and partially pinned (labelled T) to SiO₂ precipitates (labelled P) are observed.

7.3.5 Defect density - dependence upon implant temperature (T)

Figure 7.22 a) shows the etch pit density dependence upon temperature for samples implanted at 70 keV with a dose of 3.3x10¹⁷ O·cm² and Fig. 7.22 b) for samples implanted at 90 keV with doses of 5.0x10¹⁷ O·cm². From the figure it is observed that the defect density decreases considerably for temperatures equal or higher than 650°C. This data agrees closely with recent results presented by Nakashima and Izumi[140] which showed a large decrease of the defect density with temperature. However, it is important to point out that, in the samples implanted at the University of Surrey, this decrease does not occur uniformly across the implanted area, which we propose is due to machine specific temperature gradients.
Fig. 7.22 - Dependence of the defect density upon temperature for a) samples implanted at 70 keV, \(3.3 \times 10^{17} \text{O}^+ \text{cm}^{-2}\) and b) at 90 keV, \(5.0 \times 10^{17} \text{O}^+ \text{cm}^{-2}\).

*: The defect densities plotted in Fig. 7.22 are from the centre of a particular wafer.
7.3.6 Edge effects - stress related.

Fig. 7.23 is a typical SEM micrograph from the non implanted area of wafer G1 (previously etched for 1 min) at a distance of about 100 μm from the ion implanted boundary (IIB, see schematic). It is noted that bands of etch pits (A) exist separated by zones (B) which are relatively free of defects.

Fig. 7.23 - SEM micrograph of etch pit bands observed in the non implanted area from wafer G1. A schematic showing the region of analyses is included.

Fig. 7.24 (a-b) shows low magnification (100 x and 1000 x) optical micrographs of the non implanted area of the same sample (after defect etching). Bands of etch pits are found to follow the implanted/non-implanted boundary as can be seen in the lower magnification optical micrograph (fig. 7.24 a). Furthermore, it is observed that the separation between the etch pit bands increases as a function of the distance from the IIB until they completely disappear at a distance of about 200 μm from the IIB. The defects responsible for the bands of etch pits were identified as faulted dislocation loops[109], since it was possible to grow them into large OISF (l= 20 μm) during a
subsequent dry oxidation treatment[122] (2 hours at 1100 °C). The OISF were identified by SEM observations after being defect etched.

Fig. 7.24 (a-b) Optical micrographs of the etch pits bands following the ion implanted boundary (IIB).
The existence of defect bands parallel to the IIB suggests that large stresses are present in this region due to the pinning of the silicon overlayer (see section 7.2.6).

In Fig. 7.25 (a-b) we show SEM micrographs (after defect etching) from the implanted area of wafer G14 which was implanted at a higher current density than normal, namely at 7.75 µA/cm². From these micrographs, it is observed that bands of defects, which are orientated along <110> directions, exist inside the implanted area. We postulate that this effect is due to slip within the silicon overlayer. Similar results were observed in other wafers implanted with current densities of 6.0 µA/cm² and 7.75 µA/cm², namely wafers G15, G16 and G17. The occurrence of slip is assumed to be related to the higher thermal gradients[141] in wafers implanted at the higher current densities (greater power loading).

In order to confirm that large stress fields can exist in these wafers, double crystal X-ray diffraction analyses have been carried out. Fig. 7.26 a) shows a X-ray diffraction rocking curve (Cu Kα) from the non implanted area at a distance of 0.5 cm from the visible IIB border of an as-implanted wafer (ϕ = 3.0x10¹⁷ O⁺ cm⁻², E = 70 kV). Fig. 7.26 b) shows the rocking curve of the non implanted area of the same wafer, far removed (3cm) from the IIB. We observe that a broadening of the peak occurs with the FWHM increasing from 14.15° to 25.27° close to the IIB. This broadening of the FWHM confirms an increase in the stress state of that region [142]. A discussion on the effects of large stress on the nucleation of crystallographic defects is given in section 8.3.1.
Fig. 7.25 (a-b) SEM micrograph of defect bands in the implanted area of wafer G14. The defect bands are orientated along <110> directions.
Fig. 7.26 - X-rays diffraction rocking curves from the non implanted region a) 0.5 cm and b) 3 cm away from the visible IIB. \( \Phi = 3.3 \times 10^{17} \, O \, \text{cm}^{-2} \) at \( E = 70 \, \text{keV} \) (as-implanted).
7.3.7 - Defect densities in the silicon overlaver

The defect densities in all wafers implanted at the University of Surrey are summarised in table 7.6. It is observed that optimisation of the implantation parameters such as dose, current beam density and temperature can lead to the formation of thin SIMOX structures with a relative low defect density ($\approx 10^5 \text{ cm}^2$). Indeed, for the low energy SIMOX samples the lowest defect density ($3.0 \times 10^5 \text{ cm}^2$) was observed in wafer G4 which was implanted at an energy of 70 keV and a dose of $\phi = 3.3 \times 10^{17} \text{ O}^+ \text{ cm}^{-2}$ ($T_i = 700^\circ\text{C}$). On the other hand, the highest defect density was observed in wafer G13 which was implanted at an energy of 90 keV and dose of $\phi = 1.0 \times 10^{18} \text{ O}^- \text{ cm}^{-2}$ ($T_i = 700^\circ\text{C}$). This high defect density is attributed to the non optimised dose, which leads to an extremely high defect density due to the high concentration of point defects produced by the implantation process and by the internal oxidation of silicon (see section 3.3). We conclude that, the best results are obtained for samples implanted with doses close to the threshold dose [59] for the formation of the buried oxide layer, which are, for the 70 keV and 90 keV implantations, $3.3 \times 10^{17} \text{ O}^+ \text{ cm}^{-2}$ and $5.0 \times 10^{17} \text{ O}^- \text{ cm}^{-2}$ respectively.

For the 200 keV SIMOX samples, the lowest defect density ($1.0 \times 10^4 \text{ cm}^2$) was observed in wafer G14 which was implanted with a dose of $\phi = 4.0 \times 10^{17} \text{ O}^- \text{ cm}^{-2}$ at $T_i = 700^\circ\text{C}$. However, it is important to point out that this wafer was implanted with a high current beam density (7.75 $\mu$A/cm$^2$) and that slip was observed in part of the silicon overlayer (see section 7.3.6).
<table>
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Table 7.6 - Dislocation densities in wafers implanted at the University of Surrey
7.3.8 Crystallographic defects in the Si substrate

The crystalline quality of the silicon substrate below the BOX was also assessed using chemical defect etching. Before applying the defect etchant the silicon overlayer was removed using a KOH etch and the BOX layer was removed using a HF(40%) solution (section 4.1). Fig. 7.27 (a-b) are typical SEM micrographs of the silicon substrate of wafer G7 after being defect etched for 1 min. The etch pits characteristic of stacking faults are clearly observed, and are labelled SF in the figures.

Fig. 7.27 (a-b) Typical SEM micrographs of the silicon substrate of wafer G7 after being etched for 1 min.
The SF density measured in the silicon substrate was \((5.0 \pm 2.0) \times 10^5 \text{ cm}^{-2}\). Furthermore, from Fig. 7.27 b) it is observed that the SF are decorated since the etch pits spread also along \(<100>\) directions. Plan view TEM analyses of the silicon substrate of a similar sample, carried out at the University of Oxford [143], have confirmed that the SF are decorated by Cu colonies orientated along \(<100>\) directions. SF were also observed in the non implanted area of the SIMOX wafers as described for the Eaton substrates in Section 7.2.6. It is important to point out that the Si wafers used both at the University of Surrey and at Eaton Corporation were supplied by the same vendor (Wacker) and are expected to have similar concentrations of contaminants and growth defects. In order to determine if the nucleation of SF in these wafers was related to the implantation process or the high thermal treatment, a non-implanted bulk silicon wafer was annealed at 1300°C for 6 hours and examined in the SEM after chemical defect etching. The annealed bulk silicon wafer showed that SF (density: \(1.0 \times 10^6 \text{ cm}^{-2}\)) were formed during the annealing process. This result suggests that the nucleation sites for the SF are not introduced during the \(O^+\) but pre-exist in the silicon wafer prior to the \(O^+\) implantation.

SIMS analysis (see fig. 7.28 a-b) of Wacker silicon wafers have shown an increase in the Cu concentration in the near surface region from \(3 \times 10^{15} / \text{cm}^3\) to more than \(2 \times 10^{18} / \text{cm}^3\) during high temperature annealing (1300°C, 6 hours). In the absence of other experimental data we assume that this increase is due to diffusion and segregation of Cu from the bulk of the Si wafer. This increase in the metal contamination near the surface could explain the formation of the decorated SF observed in the non implanted area of the SIMOX wafers.
Fig. 7.28 - SIMS copper concentration-depth profiles of (100) Si wafer (supplied by Wacker) a) non annealed and b) annealed at 1300°C for 6 hours.
Chapter Eight: Discussion.

8.1 Introduction.

In this chapter, we discuss the main results obtained in chapter 6 and 7 and give some interpretations on the possible mechanisms of formation of crystallographic defects in thin film SIMOX.

This chapter has two sections. In the first (8.2), we discuss the formation of crystallographic defects in sacrificially oxidised SIMOX and in the second (8.3) we discuss the origin of defects in low energy, low dose SIMOX.
8.2 Crystallographic defects in sacrificially oxidised SIMOX

In chapter 6, we have shown that during the thermal oxidation of SIMOX materials, oxidation induced stacking faults (OISF) are formed. We have also shown that the OISF density, which varies from $5.0 \times 10^2$ cm$^{-2}$ and $5.0 \times 10^3$ cm$^{-2}$ in the particular samples under investigation, is strongly dependent upon the density of SF complexes present in the silicon overlayer before oxidation. In the next sub-section, the nucleation and evolution of OISF are discussed in more detail.

8.2.1 Nucleation process of OISF in SIMOX

The conversion of Si to SiO$_2$ involves a 2.2 fold increase in molar volume and therefore, unless there is an efficient mechanism to account for this extra volume, high stress fields will be formed at the Si/SiO$_2$ interface. Although several stress relief mechanisms may be active during the thermal oxidation of silicon[144,145,146] the most widely accepted one, involves the emission of silicon interstitials or absorption of vacancies[147]. This model was first proposed by Dunham et al [147] and they suggested that most of the interstitials ejected are immediately captured by the superficial oxide with only a few, perhaps less than one in $10^4$, diffusing into the bulk silicon. Therefore, we can see that the thermal oxidation of bulk silicon creates a perturbation in the point defect equilibrium concentration.
In the case of SIMOX, a super-saturation of silicon interstitials in the silicon overlayer is expected since the diffusivity of Si through the buried oxide (BOX) layer is extremely low \( D = 1.0 \times 10^{-20} \text{ cm}^2 \text{ s}^{-1} \) at 1100°C [148] and, therefore, the BOX layer acts as an effective barrier for the diffusion of the interstitials. Indeed, several authors [149, 150] have suggested that the formation of \{113\} defects below the buried oxide layer of SIMOX is due to a super-saturation of Si interstitials that are unable to diffuse in the opposite direction to the surface through the buried oxide layer. Recently, Guillemot et al [151] have proposed that Si could diffuse through the buried oxide layer as SiO since the diffusivity of SiO in SiO\(_2\) (at 1000 °C) is four orders of magnitude \( (1.6 \times 10^{-16} \text{ cm}^2 \text{ s}^{-1}) \) higher than that of Si in SiO\(_2\) and therefore, the supersaturation of silicon interstitials could be reduced through the formation of SiO molecules. However, this mechanism is unlikely to occur at the temperatures used in our oxidation studies (800°C-1100°C) since the activation energy for the dissociation of SiO\(_2\) into SiO is very high (4.4eV) [151]. We conclude that the BOX layer is an effective barrier for the downwards diffusion (towards the bulk) of Si interstitials, giving rise to an interstitial super-saturation in the silicon overlayer.

In crystalline materials the free energy associated with a point defect is high [152] and therefore, recombination with other point defects or annihilation at sinks is generally energetically favourable. In SIMOX the surface and the Si/SiO\(_2\) interface are the natural sinks for interstitials, since point defects can be easily incorporated in surface 'kinks' [153].

Other sinks for interstitials are the crystallographic defects present in the silicon overlayer. It is nowadays widely accepted [154] that crystallographic defects can act as effective sinks for point defects. Indeed, proposed annealing models for point defect in metals...
[155] generally take into account the presence of defects such as SFT, dislocation loops and others.

In section 5.3, we have shown that threading dislocations and SF complexes are the main crystallographic defects present in the silicon overlayer of SIMOX materials. However, we have also shown (see chapter 6) that threading dislocations do not influence the formation of OISF. In order to understand why threading dislocations do not act as effective sinks for interstitials it is important to know the origin and nature of the threading dislocations. Stoemenos et al [48] have proposed that threading dislocations originate from the evolution of perfect semi dislocation loops (SDL) \((b = 1/2<110>)\) that expand by a climb mechanism through the capture of point defects. Once the SDL reaches the surface it opens up into a paired dislocation by the escape of the segment parallel to the surface. However, it has been recently suggested by Lee et al [156], that after reaching the surface the perfect dislocation can dissociate into two Shockley partials through a mechanism given by [157]:

\[
\frac{1}{2}[110] \rightarrow \frac{1}{6}[211] + \frac{1}{6}[2\overline{1}] 
\]

perfect Shockley Shockley

This dissociation reaction can occur when a segment of the originally perfect dislocation lies along the intersection of the \(\{110\}\) habit plane of the half loop [157] and a \(\{111\}\) slip plane. This process forms a narrow stacking fault (NSF) in a \(\{111\}\) plane bounded by the two partials. However, it is unlikely that the NSF can act as an efficient sink for interstitials since the Shockley partials that bound the fault can not efficiently capture point defects[158]. Therefore, we conclude that the threading dislocations will not influence directly the formation of OISF.
On the other hand, in chapter 6, we have also presented extensive evidence to show that SF complexes act as efficient sinks for interstitials and influence the nucleation process of OISF in SIMOX. Although the nature and origin of these defects in SIMOX is still uncertain some models for their formation have been presented recently by Stoemenos[159] and Venables [160] and these are discussed below.

A. Mechanisms for the formation of SF complexes (SFT/SFP).

1. Stacking fault tetrahedra (SFT)

Stacking fault tetrahedra (SFT) were first reported in quenched gold (polycrystalline Au, 99.998%) by Silcox and Hirsch in 1959[111]. They attributed the formation to the dissociation of intrinsic Frank dislocation loops through a mechanism described in Appendix C. Another mechanism for the formation of SFT in metals (Al, Cu) was later proposed by Jong and Koehler[161] who suggested that SFT could be formed by the aggregation of tetravacancies into a SFT by the capture of a divacancy (where the six-vacancy cluster is the smallest tetrahedron). Although these two models differ substantially in nature, they agree in the fact that SFT arise directly or indirectly due to the condensation of vacancies.

The nature and origin of SFT in SIMOX have still not been determined. However, several authors [159,162] have suggested that they are formed due to the condensation of vacancies produced during the dissolution of the SiO₂ precipitates during the latest stages of the annealing process. This proposal is backed by the fact that intrinsic SFT have been observed in P⁺ implanted Si [163].
2. Stacking Fault Pyramids (SFP)

As in the case of SFT, the nature of SFP in SIMOX has still not been determined, however, several mechanisms for the formation of SFP have been proposed recently.

A recent proposal made by Stoemenos et al [159], is that SFP are "as-grown" defects formed during the high temperature anneal as a result of the considerable silicon redistribution near the Si/SiO$_2$ interface that occurs during the dissolution of large SiO$_2$ precipitates in the vicinity of the interface. Stoemenos also suggested that the sequence of faults inside the SFP has to be intrinsic/extrinsic/intrinsic/extrinsic in order to satisfy conditions imposed by the stair rod partial dislocations that bounds the fault[ 163].

A second mechanism recently proposed by Venables et al [160] is that the formation of SFP occurs due to the interaction between two or more narrow stacking faults (NSF) that are pinned by a SiO$_2$ precipitate. In this way the NSF can interact with each other forming a SF complex. Upon the dissolution of the SiO$_2$ precipitate the interacting NSF can further interact and develop into a complete SFP.

Here, it is our supposition, that the formation of SFP occurs due to the interaction of two adjacent Frank dislocation loops pinned by a SiO$_2$ precipitate through a mechanism similar to that proposed by Venables (see Fig. 8.1 ). We believe that the Frank dislocation loops can be nucleated either (i) by the condensation of vacancies produced by the dissolution of the SiO$_2$ precipitates or (ii) by a prismatic punching process [116] (see section 3.3.2) in which an intrinsic and an extrinsic dislocation loop are formed near the SiO$_2$ precipitate. This supposition is substantiated by experimental evidence shown in chapter 6, that suggests that the SFP are effective sinks for vacancies (see Fig.6.11 ). Although the model proposed by Venables can account for the formation of square like
SFP, it cannot explain the presence of elongated SFP (see Fig. 6.9), since the Shockley partials are not efficient sinks for point defects [158].

8.1- Schematic showing a proposed mechanism for the formation of SFP during the dissolution of a SiO₂ precipitate.
B. SF complexes as nucleation sites for OISF

1. Stacking fault tetrahedra (SFT).

Koehler and Lund [165] have reported that the activation energy for the annealing rate of SFT in gold is 2.2 eV. Furthermore, Cotterill and Jones [166] have observed that SFT present in quenched gold can be completely eliminated when submitted to α particle-irradiation. They suggested that the SFT are removed by the capture of the excess interstitials generated by the irradiation. Moreover, Jong and Koehler [161] have proposed that the incorporation of point defects in the SFT occurs through the capture of point defects by the stair-rod dislocations that bound the fault. During the incorporation of point defects the SFT can grow or shrink depending upon the nature of the point defects. Indeed, Silcox and Hirsch [111] have suggested that under certain conditions, the SFT can collapse into a Frank sessile dislocation loop on any of the four {111} planes associated with the SFT.

Although the nature of SFT in SIMOX is unknown it is our supposition that they also arise from the condensation of vacancies and therefore, they will also act as effective sinks for point defects. However, according to the annealing model proposed by Jong and Koehler [161] the capture of interstitials by the SFT will cause the defect to shrink until it is completely eliminated. We conclude that, if the nature of SFT in SIMOX is intrinsic, they will not act as nucleation sites for OISF.

On the other hand, if the SFT in SIMOX are extrinsic, the capture of interstitials by the stair rod dislocations will lead to the growth of the defect until its size reaches a critical value ($l_c$) when it will become unstable and will collapse into an extrinsic Frank loop, according to the model by Silcox and Hirsch [111]. In this case the SFT will act as direct

Discussion
nucleation sites for OISF, since the extrinsic Frank loop can further capture interstitials and expand into a large OISF.

2. *Stacking fault pyramidal*

Although the nature of the SFP have not yet been determined, the different mechanisms of formation of these defects suggest that they can be either intrinsic, extrinsic or mixed. If the nature of a SFP is extrinsic then, the incorporation of interstitials will lead to the growth of the defect until it collapses into extrinsic Frank loops in a similar manner to the SFT. The Frank loops can then grow by capturing further interstitials and evolve into OISF. In the case of an intrinsic/extrinsic SFP, the two opposite planes will grow through the capture of interstitials while the other two intrinsic planes will shrink and probably disappear. Once again, upon further growth, the defect can collapse and give rise to the formation of OISF.

In the case of a pure intrinsic SFP, the capture of interstitials will lead to a reduction in the size of the defect and, therefore, although they will act as efficient sinks for interstitials they will not nucleate OISF.
8.2.2 Evolution of OISF during oxidation.

Once an embryonic OISF is nucleated, it will evolve through the capture of self interstitials by the Frank partial that bounds the fault. It is important to remember that the Frank dislocation (b=1/3 <111>) loop that bounds the fault is not glissible and therefore it can only evolve through a non-conservative motion. This can only be a climb process involving the capture of interstitials. Fig. 8.2 is a schematic showing different stages in the evolution of the OISF in SIMOX. In the first stage of evolution an embryonic OISF is formed near the Si/SiO₂ interface probably through the collapse of a SF complex which will form one Frank dislocation loop (See Fig. 8.2 a). Upon further oxidation, the Frank partial that bounds the fault will grow by a climb process through the capture of interstitials (see Fig. 8.2 b ). Once the partial loop reaches the surface it opens up into two Frank partial dislocations in a similar way to that observed for threading dislocations. After splitting of the partial, the fault is only able to grow laterally by the incorporation of interstitials by the two partials (See Fig. 8.2 )
8.2 - Schematic of the evolution of an OISF during the oxidation process.
A. Activation energy for the growth of OISF in SIMOX

In chapter six we have shown (see for example Fig. 6.6) that the OISF length was strongly dependent upon temperature. Using data presented in Fig. 6.6 and in appendix E, we have estimated the activation energy for the growth of the OISF in SIMOX. The results are displayed in Fig. 8.3, showing $\ln R$ (rate of growth) as a function of $T^{-1}$.

Performing a linear regression on the data, it was possible to estimate an activation energy $E_a = 1.5 \pm 0.5$ eV. This value is comparable to the values for bulk silicon (2.3 eV) [167] when the rather large experimental uncertainty is taken into account.

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**Fig. 8.3 -** $\ln R$ (rate of growth of OISF) as a function of $T^{-1}$. 

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B. Estimation of the number of self interstitials emitted during dry oxidation.

Assuming that the number of interstitials captured by the OISF is proportional to the total number of interstitials emitted during oxidation, we can make an estimation of the interstitial super-saturation at different oxidation temperatures under dry oxidation conditions. This can be done by estimating the total number of interstitials inside the OISF ($N_{\text{Si, OISF}}$) and correlating this with the total number of silicon atoms consumed by the thermal oxidation ($N_{\text{OX}}$). The total surface area of the OISF ($S_{\text{OISF}}$) is expressed by [151]:

$$S_{\text{OISF}} = D_{\text{OISF}} L_{\text{OISF}} (h / \sin 55\degree)$$  \hspace{1cm} (8.1)

where $D_{\text{OISF}}$ is the OISF density, $L_{\text{OISF}}$ is the OISF mean length and $h$ is the thickness of the silicon overlayer and $55\degree$ is the angle that $\{111\}$ planes form with the (001) surface (see Fig. 8.4). Since the number of atoms per cm$^2$ in the $\{111\}$ extra plane is $N_0 = 1.6 \times 10^{15}$ atoms/cm$^2$, $N_{\text{Si, OISF}}$ is given by: $N_{\text{Si, OISF}} = N_0 S_{\text{OISF}}$. The total number of silicon atoms consumed ($N_{\text{OX}}$) can be estimated by knowing the thickness of the silicon consumed and the molar density of silicon ($\rho = 12.1$ cm$^3$/mol.).

![Fig. 8.4 - Schematic showing an OISF inside the silicon overlayer.](image)
Fig. 8.5 (a-d) shows the dependence of the $N^\text{Si}_{\text{OISF}}$ as a function of $N_{\text{ox}}$. From Fig. 8.5 it is observed that the $N^\text{Si}_{\text{OISF}}$ increases according to the temperature for a given SIMOX substrate. This result is not unexpected since for high temperatures the oxidation rate increases considerably and therefore the incorporation of the ejected interstitials by the oxide interface is more difficult and as a consequence more interstitials will be able to diffuse into the silicon overlayer[165]. The other feature observed from Fig. 8.5 is that the number of silicon interstitials captured by the OISF depends upon the particular choice of the SIMOX wafer. This result indicates that other sinks for interstitials exist, and as we have already suggested we believe that this difference is due to the presence of SFT in the silicon overlayer.

![Graph](image_url)

*Fig. 8.5 - a (continued) [the lines are drawn only to guide the eye].*
Discussion

$N_{Si^{\text{OISF}}}^{Si} \ (x1e10 \ \text{Si/cm}^2)$

$N_{Si^{\text{OISF}}}^{Si} \ (x1e10 \ \text{Si/cm}^2)$

8.5 b-c (continued)
Using the data presented in Fig. 8.5, an estimation of the number of interstitials emitted per silicon atom consumed during oxidation ($N_{\text{Si}}^{\text{OSF}}/N_{\text{ox}}$) can be obtained. In Fig. 8.6, we plot the $N_{\text{Si}}^{\text{OSF}}/N_{\text{ox}}$ (for $N_{\text{ox}} = 4.0 \times 10^{17}$ cm$^{-2}$) as a function of temperature for wafer B2. It is observed that in the particular case of wafer B2 the value for $N_{\text{Si}}^{\text{OSF}}/N_{\text{ox}}$ at 1100°C is $5.0 \times 10^5$, which is in close agreement to the value theoretically obtained by Dunham et al. [147].
Fig. 8.6 - $\frac{N_{Si\ OSF}}{N_{ox}}$ as a function of temperature for wafer B2.
8.3 Crystallographic defects in low energy and low dose SIMOX.

The results presented in chapter 7 have shown that the main defects observed in the silicon overlayer of low energy and low dose SIMOX samples were "threading" dislocations and in some cases, SF complexes (see Fig. 7.7). We have also shown that implantation parameters such as dose, energy and implantation temperature, have a great effect on the final defect density. In fact, a wide range of defect densities (from 10^4 cm^2 to 10^9 cm^2) was observed depending upon the implantation conditions.

In order to understand the reasons why the implantation parameters affect so dramatically the formation of crystallographic defects it is important to refer back to chapter 3. In chapter 3 we have shown that the main sources of defects in oxygen implanted silicon were basically the radiation damage and the internal oxidation of silicon. We have also indicated that the last factor contributed twice as a source of defects since it was responsible for the generation of self interstitials and also for the plastic deformation of the silicon matrix around the SiO₂ precipitate. In this way, we would expect that low dose O⁺ implantations (close to Φ₂⁺) at high temperatures (700°C) would minimise the effects of both the radiation damage and the internal oxidation of silicon, and therefore, would produce low defect densities. Results by other groups[57] (see Fig. 2.3) have suggested that extremely low defect densities (10^3 cm^2) are formed for low dose and high implantation temperatures. However, by scaling down the implantation energy in order to form thin silicon overlays, a new problem is introduced since, in doing so, we bring the peak of damage (R_n) closer to the surface and therefore, dislocations that are pinned to
large SiO₂ precipitates (see Fig. 7.21) can escape more easily to the surface during the annealing process.

In spite of that problem, our results have shown that low defect densities can be obtained in thin film SIMOX produced by low energy and low dose O⁺ implantations using high implantation temperatures (Ti > 500°C) (see Fig. 8.7).

![Defect density vs. Energy](image)

Fig. 8.7 - Defect density upon implantation energy for doses close to \( \Phi_c \) [59]. The line is to guide the eye.

From Fig. 8.7 we can see that, interestingly, the best results (1.0x10⁴ cm⁻²) were obtained for the lowest energy (30 keV). However, it is important to point out that in Fig. 8.7, we have plotted the results obtained from samples implanted at Eaton and Surrey without making any distinction between them. Obviously, clear differences in the way that these
samples have been prepared (see chapter 4) may have influenced the final crystallographic defect density but what emerges from Fig. 8.7 is that the defect density does not necessarily increase with decreasing ion energy, indeed in these samples the density decreases. These results indicate that the positioning of the peak of damage $R_d$ closer to the surface (by using lower energies) does not increase significantly the defect density as might be initially expected[62]. Meda et al [169] have proposed that the gradual merging of the damage and atomic concentration-depth profiles with decreasing ion energy will facilitate the formation of a continuous BOX layer. The results presented in Fig. 8.7 may be a consequence of the improved planarity of the final structures with the proximity to the free surface having a significantly smaller effect.

Comparing the best results obtained for samples implanted with low dose and low energy ($10^4$ cm$^{-2}$) and those obtained from other groups[57], for low dose and high energy implantations ($10^3$ cm$^{-2}$) there is a difference of one order of magnitude between them. We believe that this difference is, at least partially, due to undesirable effects such as thermal stresses produced during the local area implantation and the presence of contaminants in the silicon substrates which are due to sputtering of metal components within the accelerator vacuum system.

8.3.1 Effects of thermal stresses on the nucleation of defects in SIMOX.

In both the wafers implanted at the University of Surrey and at Eaton, we have observed that anomalous defect densities (see Fig. 7.11) were present in areas near the ion implanted boundary (IIB). We believe that these highly defective areas appear as a
consequence of large stress fields caused both by the pinning of the silicon overlayer and by the formation of large thermal gradients (see section 7.2). The effects of large stresses on the generation of crystallographic defects in SIMOX have been recently discussed by Nejim et al [170] and by Venables et al [51]. Venables and co-authors have argued that large stress fields can assist in the nucleation and climb of dislocation loops. These stress fields can cause plastic deformation of the silicon matrix by generation of dislocations, either heterogeneously by the capture and multiplication of dislocations from other sources or homogeneously by the condensation of silicon interstitials[171]. Homogeneous nucleation occurs following a clustering of silicon interstitials which align in the stress field and collapse into a dislocation loop [172]. The observation of defect bands entering the implanted area along <110> directions (see Fig. 7.25) is strong evidence of the deformation process caused by these large stress fields.

We believe that, for the wafers prepared at Eaton Corporation (see table 7.3), the nucleation of defects caused by this process was localized in the regions close to the implanted/non-implanted boundary because almost the whole 6 inch wafer was implanted. However, for the wafers prepared at the University of Surrey, the effect of large stress fields on the nucleation of defects affects the whole of the implanted area because only a 6.45 cm² area of the 3 inch diameter wafer was implanted. Most probably, this is one of the reasons why the lowest defect densities observed in wafers prepared at the University of Surrey was 30 times higher than those obtained from wafers prepared at Eaton Corporation. Therefore, we think that to further reduce the defect density it will be necessary to implant the whole wafer. In doing so, thermal and mechanical stresses will be reduced leading to lower defect densities.
8.3.2 Effects of contamination on the formation of crystallographic defects.

Carbon and metals contaminants (such as Fe, Ni, etc) can be accidentally introduced during the implantation process. Carbon is likely to be incorporated into the wafer surface by a "knock on" process of hydrocarbons deposited on the surface of the wafer, which originate from the vacuum pump oils while metals can be sputtered from the walls of the implanter or mounting clips during the implantation process[173].

Recently, the role of carbon in the nucleation of defects has been studied[174]. It has been suggested that C absorbs silicon interstitials by the formation of beta SiC precipitates. This process leads to volume contraction preventing the development of stress caused by self-interstitial supersaturation[175]. If a supersaturation of vacancies is present, the formation of beta SiC precipitates will further increase the vacancy supersaturation, enhancing the condensation of vacancies and the formation of dislocation loops through the collapse of vacancy aggregates. Therefore, the detrimental or beneficial role of carbon in the formation of defects will depend upon the concentration and type of point defects in the material.

On the other hand, the detrimental effects of metal contamination are widely known[176]. The formation of crystallographic defects due to the presence of metal contamination may occur through the nucleation of dislocation loops induced by the metal silicide precipitate. These dislocation loops are nucleated due to the plastic deformation of the silicon matrix caused by the extra volume of the new phase (see section 3.3).
In chapter 7, we have shown that a high concentration of Cu (see Fig. 7.26) was observed in silicon wafers supplied by Wacker after high temperature treatments. We have shown that decorated SF (5.0x10^5 cm^-2) were formed below the buried oxide layer and in the non-implanted area of the silicon wafer. In addition, we have also shown that Cu was not introduced during the implantation process since it was already observed in non-implanted wafers after high temperature processing (1300°C, 6 hours in Ar + 1/2% O_2). These results suggest at first sight that the high Cu concentration originated from contamination of the furnace tube. However, if this was the case, we would expect that the incorporation of Cu would occur homogeneously along the silicon surface and therefore, we would expect to find similar SF densities inside and outside the implanted area. Since no SF were observed in the implanted area (< 10^5 cm^-2) this assumption seems unlikely. Another possibility is that Cu was already present in the bulk of the silicon wafer before annealing and during the thermal treatment diffuses towards the silicon surface. This assumption could explain why SF were not observed in the silicon overlayer if we consider that the BOX layer acts as a barrier for the diffusion of Cu. This assumption is further supported by the results obtained from Wacker wafers annealed in a different furnace (Eaton laboratories) in which a similar SF density of 5.0x10^5 cm^-2 was observed. If the formation of SF was furnace dependent we would expect to find a completely different SF density. Since the wafers were supplied by the same vendor it is probable that the Cu contamination in these wafers was the same, giving rise to similar SF densities.

These findings suggest that further reduction of defect densities could be achieved by the reduction of contaminant levels using a cleaner implanter environment and also by using silicon substrates more amenable to extremely high temperature.
Chapter Nine:

Conclusions and Suggestions for future work

In this project we have extensively characterized thin SIMOX materials prepared by two different methods namely, sacrificial oxidation of thick SIMOX materials and low energy and low dose oxygen implantations. For this purpose we have specially developed a new defect etchant capable of delineating stacking faults and threading dislocations in thin silicon films (≥500 Å). Detailed analysis of large areas of thin film SIMOX was possible due to the speed with which the samples could be prepared and etch pit densities obtained. Determination of the lateral distributions of crystallographic defects in the implanted and non-implanted areas of the wafers was also carried out. Furthermore, in conjunction with XTEM and plan view TEM analysis, the depth distribution of the crystallographic defects was also determined. From these observations it was possible to determine the nature and origin of the different crystallographic defects, and by so doing, control their final density. The main conclusions drawn from these extensive analyses are presented below.
The main defects observed in oxidised SIMOX samples were OISF and threading dislocations. The OISF densities found in the samples analysed in this project varied from \(5.0 \times 10^2\) to \(5.0 \times 10^5\) \(\text{cm}^{-2}\) and were found to depend upon the density of nucleation sites prior to the oxidation. The nucleation and growth of the OISF have also been studied and it has been determined that the main nucleation sites are SFP/SFT that are present before oxidation and lie close to the Si/SiO\(_2\) interface. The formation of SFP in SIMOX occurs during the high temperature annealing (typically 1300°C) and although few mechanisms of formation have been proposed in the literature we have suggested that they arise from the interaction of Frank dislocation loops pinned by large SiO\(_2\) precipitates. Annihilation of the SFP/SFT from the Si/SiO\(_2\) interface by prior amorphisation has shown that the effective annihilation of these defects enables the substrates to be thinned whilst maintaining an extremely low OISF density \((5.0 \times 10^2\) \(\text{cm}^{-2}\)). It has also been observed that the growth of OISF in SIMOX is an activated process with an activation energy of \(1.5\) eV \(\pm\) 0.5 eV. Furthermore, an estimate of the number of interstitials captured by the OISF has been made in order to determine the minimum number of interstitials ejected from the growing oxide during thermal oxidation. It has been shown that the number of ejected interstitials increases with oxidation temperature in close agreement with the model proposed by Dunham et al [147]. Therefore, in order to avoid the formation of long OISF \((<5\) \(\mu\)m\), we suggest that dry oxidation treatments at moderate temperatures \((900^\circ\text{C} \text{ or} 950^\circ\text{C})\) be used to thin the silicon overlayers of thick SIMOX materials.

9.1 Crystallographic defects in SIMOX thinned by sacrificial oxidation

The main defects observed in oxidised SIMOX samples were OISF and threading dislocations. The OISF densities found in the samples analysed in this project varied from \(5.0 \times 10^2\) to \(5.0 \times 10^5\) \(\text{cm}^{-2}\) and were found to depend upon the density of nucleation sites prior to the oxidation. The nucleation and growth of the OISF have also been studied and it has been determined that the main nucleation sites are SFP/SFT that are present before oxidation and lie close to the Si/SiO\(_2\) interface. The formation of SFP in SIMOX occurs during the high temperature annealing (typically 1300°C) and although few mechanisms of formation have been proposed in the literature we have suggested that they arise from the interaction of Frank dislocation loops pinned by large SiO\(_2\) precipitates. Annihilation of the SFP/SFT from the Si/SiO\(_2\) interface by prior amorphisation has shown that the effective annihilation of these defects enables the substrates to be thinned whilst maintaining an extremely low OISF density \((5.0 \times 10^2\) \(\text{cm}^{-2}\)). It has also been observed that the growth of OISF in SIMOX is an activated process with an activation energy of \(1.5\) eV \(\pm\) 0.5 eV. Furthermore, an estimate of the number of interstitials captured by the OISF has been made in order to determine the minimum number of interstitials ejected from the growing oxide during thermal oxidation. It has been shown that the number of ejected interstitials increases with oxidation temperature in close agreement with the model proposed by Dunham et al [147]. Therefore, in order to avoid the formation of long OISF \((<5\) \(\mu\)m\), we suggest that dry oxidation treatments at moderate temperatures \((900^\circ\text{C} \text{ or} 950^\circ\text{C})\) be used to thin the silicon overlayers of thick SIMOX materials.
9.2. Crystallographic defects in low energy, low dose SIMOX.

9.2.1. SIMOX wafers produced at the University of Surrey.

The main crystallographic defects present in the silicon overlayer of thin film structures prepared at the University of Surrey are threading dislocations. The threading dislocation density in samples implanted with a dose of $3.3 \times 10^{17} \text{ O}^+ \text{ cm}^{-2}$ at an energy of 70 keV varied from $2.0 \times 10^8 \text{ cm}^{-2}$ to $3.0 \times 10^4 \text{ cm}^{-2}$ depending upon the implantation temperature. Indeed, it was observed that the threading dislocation density decreases by 660 times when the implantation temperature is increased from 550°C to 700°C. The same trend was observed in samples implanted with $5.0 \times 10^{17} \text{ O}^+ \text{ cm}^{-2}$ at an energy of 90 keV when the dislocation density decreases from $1.0 \times 10^8 \text{ cm}^{-2}$ to $3.0 \times 10^6 \text{ cm}^{-2}$ when the implantation temperature is increased from 550°C to 700°C. However, it was observed that for the wafers implanted at the highest temperatures (650°C-700°C) a non uniform distribution of threading dislocations across the implanted area was present. We have suggested that the presence of different defect densities inside the same wafer, was either due to the pinning of the silicon overlayer at its periphery by the surrounding bulk silicon or due to the presence of thermal gradients in the wafer during the implantation. These assumptions were validated by preliminary X-ray diffraction analyses which showed the existence of large stress fields near the ion implanted boundary (IIB) in the as-implanted samples.

The relatively high threading dislocation densities can also be attributed to the presence of contaminants or to the non adequate control of the implant conditions (such as particulates, current stability) at the University of Surrey.
9.2.2 SIMOX wafers produced at Eaton Corporation.

The main crystallographic defects present in the silicon overlayer of the specific wafers examined were threading dislocations and SF complexes. The threading dislocation density varied from $1.0 \times 10^4$ cm$^{-2}$ to $5.0 \times 10^6$ cm$^{-2}$, while the density of the SF complexes varied from $8.0 \times 10^6$ cm$^{-2}$ to $5.0 \times 10^9$ cm$^{-2}$. The lowest threading dislocation densities ($1.0 \times 10^4$ cm$^{-2}$) were found in samples implanted using singly charged molecular oxygen ($O_2^+$) with a dose of $2.2 \times 10^{17}$ O$^+$ cm$^{-2}$ and energy of 30 keV ($Ti=530$ °C). These results are very interesting since they show that it is possible to obtain low defect density SIMOX materials using low energy and low dose oxygen implants. Furthermore, complementary analyses by XTEM showed that no silicon islands (e.g. $< 10^7$ cm$^{-2}$) were present in the buried oxide layer suggesting that these materials could be used, in the near future, as the substrate for low power CMOS integrated circuits. Indeed, O$^+$ implants at 30 keV with doses of $2.2 \times 10^{17}$ O$^+$ cm$^{-2}$ produce, after annealing ($1300$ °C for 6 hours), an ultra thin silicon overlayer (500 Å) containing a low density of threading dislocations ($1.0 \times 10^4$ cm$^{-2}$).

9.3 Suggestions for future work.

In this project we have successfully characterized SIMOX materials and optimised the processing conditions such as implantation temperature ($700$ °C) and oxidation temperature ($900$ °C) to achieve reduced densities of crystallographic defects in thin SIMOX structures. Annihilation of the SF complexes located near the Si/SiO$_2$ interface has been achieved using a post amorphisation process. However, further improvements in the quality of the silicon overlayer are required in order to achieve dislocation free...
SIMOX structures. For this purpose, we suggest that the nature and origin of SFP/SFT have to be further studied. The determination of the nature (intrinsic/extrinsic) of SFP/SFT in SIMOX by high resolution TEM analysis could give us more indications about the actual mechanism of formation of these defects. Another experiment that could be carried out in order to determine the mechanism of formation of SFP/SFT is to study the effects of quenching on the SFP/SFT density. This could be easily done by changing the cooling down rates following the high temperature treatments. This experiment could be used to further improve the thermal annealing process in order to produce SIMOX structures with lower SF complex densities.

We also suggest that in a future project, the effects of thermal and mechanical stresses on the nucleation of defects in low energy and low dose SIMOX has to be further studied. Implantations of the whole surface of the wafer would presumably reduce these effects and therefore, comparison with partially implanted wafers or patterned wafers would give us an indication about the influence of the thermal/mechanical stresses on the formation of defects. Furthermore, the effects of contaminants on the nucleation of SF in the silicon substrate need also to be studied. This could be done by implanting ultra pure FZ-silicon wafers and comparing the results with those obtained with the Wacker Cz-silicon wafers.

Finally, the quality of the SIMOX wafers examined in this project has been found to be very sensitive to the details of the implantation and thermal processing. For this reason it is suggested that any future experimental work should be a joint project involving a SIMOX wafer vendor so that tight control of the processing conditions can be maintained from batch to batch and wafer to wafer when meaningful defect densities could be determined.
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Appendix A

SOI technologies

During the last thirty years several SOI technologies have been developed. Among the most important ones we can name, for instance, Silicon on Sapphire (SOS), Full Insulation of Porous Oxidized Si (FIPOS), Zone Melt Regrowth (ZMR), Epitaxial Lateral Oxidation (ELO), Wafer Bonding and Separation by Implanted Oxygen (SIMOX).

In Silicon on Sapphire (SOS), a thin epitaxial film of silicon is grown on a single-crystal alpha-aluminum oxide (sapphire) substrate by chemical vapour deposition (CVD). SOS was the first SOI technique applied to VLSI, however, due to the low quality of the silicon overlayer particularly near the Si/Al₂O₃ interface, it is now being replaced by alternative substrates.

In Full Insulation of Porous Oxidized Silicon (FIPOS), porous silicon is formed by electrochemical etching silicon islands in a HF aqueous electrolyte. The oxidation rates of porous and monocrystalline silicon differ by an order of magnitude and therefore, during oxidation, the oxide is formed mainly underneath the silicon islands. The practical drawback of this approach is that the electrochemical etching necessary to form porous silicon formation is not compatible with other IC fabrication steps. However, interest has recently shifted to the study of light emission from porous silicon. As a consequence its importance as a SOI material has decreased considerably.

In Zone Melt Regrowth (ZMR), the process starts with deposition of polycrystalline silicon over SiO₂. Then the film is implanted with silicon. A channelled silicon ion beam
amorphises most of the polysilicon layer leaving intact grains with major crystallographic axes that are aligned along the channelling direction. These grains are used as seeds for a solid-state regrowth, which take place during a subsequent thermal treatment. The main drawback of the technique is the poor quality of the silicon overlayer and residual defects at the grain boundary interfaces.

**Epitaxial lateral overgrowth (ELO)**, consists of the growth of silicon by chemical vapour deposition (CVD) within windows in a SiO$_2$ mask. If no nucleation of silicon on the SiO$_2$ layer takes place, the silicon grows vertically to the mask level and then laterally over the SiO$_2$ mask. The lateral growth enables small SOI islands to be formed. The main drawback of the technique, is indeed, the small size of the islands.

The formation of SOI substrate by **Wafer Bonding** uses the principle that two surfaces in close contact may be bonded (Van der Walls forces) during a high temperature anneal. This basic idea has evolved towards silicon wafer bonding with a thermal SiO$_2$ layer on top. The main drawback of the technique is that wafer thinning processes have to be applied, and consequently, due to its limitations, lateral non-uniformities of the thickness of the Si overlayer will occur. However, during the last few years a new thinning process based on plasma etching[6] has been developed, and is now being used as the main method to produce thick (> 1.0 µm) SOI substrates.

**In SIMOX** the formation of a buried SiO$_2$ layer is obtained by high energy and high dose O$^+$ implantations. To facilitate dynamic annealing the wafers are maintained at a high temperature (> 600°C) during O$^+$ implantation. Due to the radiation damage caused by the incident O$^+$ ions ions, a post-implantation thermal annealing is needed both to annihilate the defects and to redistribute the implanted oxygen. The presence of residual
defects (dislocations, stacking faults, and SFT) in the silicon overlayer are the main
disadvantage of this technique. In Table 1 we summarise the main advantages and
drawbacks of the different SOI technologies[7].

<table>
<thead>
<tr>
<th>Technique</th>
<th>Advantages</th>
<th>Drawbacks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon on Sapphire (SOS)</td>
<td>1. Conventional processing</td>
<td>1. Cannot be used for 3D integration.</td>
</tr>
<tr>
<td>Zone melt and regrowth (ZMR)</td>
<td>1. Conventional processing</td>
<td>1. Limited applicability due to the smallness of the stripes</td>
</tr>
<tr>
<td></td>
<td>2. Application: 3D</td>
<td></td>
</tr>
<tr>
<td>Full Insulation of Porous Oxidized Si (FIPOS)</td>
<td>1. Low defect density.</td>
<td>1. Process noncompatible with IC processing.</td>
</tr>
<tr>
<td></td>
<td>2. Application: CMOS, bipolar.</td>
<td>2. Limited applicability due to smallness of the islands.</td>
</tr>
<tr>
<td>Epitaxial Lateral Oxidation (ELO)</td>
<td>1. Conventional processing.</td>
<td>3. Cannot be used for 3D integration.</td>
</tr>
<tr>
<td></td>
<td>2. low defects/warpage</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. Applications : bipolar, 3D integration.</td>
<td></td>
</tr>
<tr>
<td>Wafer Bonding</td>
<td>1. Low defect density</td>
<td>1. Thinning necessary for CMOS high speed circuit.</td>
</tr>
<tr>
<td></td>
<td>2. Application: 3D integration.</td>
<td>2. Low overgrowth ratio (small areas)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3. Seeding necessary.</td>
</tr>
<tr>
<td>Separation by Implanted OXygen (SIMOX)</td>
<td>1. Conventional processing.</td>
<td>1. Residual defects in the Si overlayer.</td>
</tr>
<tr>
<td></td>
<td>2. Good quality SOI; low defect density/warpage</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. Stage of development mature complex circuits demonstrated</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4. Application for CMOS, bipolar.</td>
<td></td>
</tr>
</tbody>
</table>

Table 1 - Main advantages and drawbacks of SOI technologies.

References


210 Appendix A
Appendix B

Chemical defect etching

1. Basic principles

A) types of reactions

Chemical etching can be described as the dissolution process of a material into an undersaturated medium. The term dissolution covers many types of reactions. Focusing our attention to the dissolution of solids in liquids, we can name three different dissolution processes. Firstly, we can have the dissolution of a molecular crystal into an organic solvent. In this reaction intermolecular crystal forces are overcome by solute-solvent interaction, and although the environment of a molecule in solution is different from that in the crystal the molecule itself is unchanged. A second type of dissolution is encountered with ionic crystals. The characteristic properties of the solid result from the electrostatic interaction of the regular array of ions. In solution, the ions are separated and properties characteristic of the solid are lost. Thirdly, there is dissolution brought about by chemical change. More frequently oxidation is required to convert the solid into a soluble material. This is expressed in general terms as the extraction of electrons from the material. The resultant material has then to be removed from the surface, usually with the accompaniment of dissolution. This kind of reaction is the most common in semiconductors and metals and therefore, it is the most important for the work discussed in this thesis.

The rate of dissolution of a solid (semiconductor) in a given etchant is primarily controlled by the speed of transfer of the active ions in the solution to the dissolving
namely an initial oxidation followed by dissolution of the oxide, the effectiveness of the oxidation will depend on the ability of the surface to adsorb the oxidising ions in the solution. This will undoubtedly be influenced, among other things, by the atomic nature of the surface, such as crystallographic orientation.

**B) Kinematic description.**

The kinematic aspects of crystal etching is generally considered to proceed through the retreat of monomolecular steps across the surface of the crystal\[1\]. The active sites are taken to be the places along the steps where single molecular rows end. These places, called "kinks", are where individual molecules may be removed. When a perfect crystal face is exposed to an etchant, dissolution begins by the nucleation of unit pits of one molecule depth. These units pits grow as steps retreat across the crystal through the action of the kinks. On such a perfect crystal face the nucleation of unit pits is random. On a real crystal, defect such as dislocations may be preferential sites for nucleation of unit pits and repeated nucleation at a dislocation leads to the formation of an etch pit. The formation of visible dislocation etch pits depends on the nucleation rate for unit pits at a dislocation and the rate of motion of the steps across the crystal surface. These two quantities are reflected in the step velocity normal to the surface at a dislocation $V_n$ and parallel to the surface $V_s$ (see Fig. 1).
C) Nucleation Process

The driving force for the nucleation of an etch pit is the energy around the dislocation. This 'localized' energy consists of the core energy plus a fraction of the total elastic strain energy. A model to determine the general features and important parameters of such a nucleation process was proposed by Cabrera et al. [2]. In their model, they assumed that the nucleus lies along the core of the dislocation and that a cross-section of the nucleus perpendicular to the dislocation is circular.

The change of free enthalpy due to the formation of a two-dimensional nucleus is given by the equation[3]:

$$\Delta G_R = \Delta G_B - \Delta G_F - \Delta G_Z$$

(B1)
The term $\Delta G_b$ describes the work required for the formation of the (circular) periphery of the pit and is given by the expression:

$$\Delta G_B = 2\pi r \rho$$  \hspace{1cm} (B2)

where $r$ is the radius of the pit and $\rho$ the specific free boundary tension. $\Delta G_F$ is the work gained by the transition from the solid to the solute,

$$\Delta G_F = \frac{\pi r^2 h \Delta \mu}{\Omega}$$  \hspace{1cm} (B3)

with $\Omega = \text{atomic volume}$
$h = \text{height of step}$
$\Delta \mu = \text{chemical potential difference between crystal and surrounding crystal.}$

Finally, $\Delta G_Z$ describes the gain in strain energy and depends largely on the type of dislocation, the distance from the dislocation line, $r$, and the radius of the dislocation core $r_0$[2]:

$$\Delta G_Z = A h \ln \left(\frac{r}{r_0}\right) + B \quad (r \geq r_0)$$  \hspace{1cm} (B4)

For a pure edge dislocation, $A$, is given by:

$$A_E = \frac{Gb^2}{4\pi(1-\nu)}$$  \hspace{1cm} (B5)

and for a pure screw dislocation is:

$$A_S = \frac{Gb^2}{4\pi}$$  \hspace{1cm} (B6)

The term $B$ in eq. B4 is the core energy and it must be considered because the driving force for the formation of an etch pit is actually the release of the core energy

214  \hspace{1cm} Appendix B
immediately at the point of emergence of a dislocation. The relationship between $\Delta G_R$ and $r$ is shown graphically in Fig. 2.

\begin{figure}[h]
\centering
\includegraphics[width=0.7\textwidth]{figure2.png}
\caption{Free enthalpy of formation of a pit near a dislocation site ($\Delta G_R$) and free enthalpy of formation of a pit in a perfect surface ($\Delta G_{\text{ideal}}$).}
\end{figure}

Rewriting Eq. 1, we obtain for a screw dislocation:

$$\Delta G = 2\pi r \rho - \frac{\pi r^2 h \Delta \mu}{\Omega} - \frac{hGb^2}{4\pi} \ln\left(\frac{r}{r_0}\right)$$

(B7)

The first derivative, $\frac{d(\Delta G)}{dr}$, yields two solutions when equated to zero (the two equilibrium points):

$$r_{\text{max}} = \frac{\rho \cdot \Omega}{2 \Delta \mu h} (1 + \sqrt{1 - \xi})$$

(B8)

and:

$$r_{\text{min}} = \frac{\rho \cdot \Omega}{2 \Delta \mu h} (1 - \sqrt{1 - \xi})$$

(B9)

Appendix B
where: \( \xi = \frac{Gb^2h^2\Delta \mu}{2\pi^2\rho^2\Omega} \)

For \( \xi = 1 \), we obtain:

\[
r_{\text{max}} = r_{\text{min}} = \frac{\rho \cdot \Omega}{2\Delta \mu h} = \frac{r_c}{2}
\]  

(B10)

The critical radius of the nucleus, \( r_c \), is derived from the treatment of a dislocation-free surface as follows:

\[
\Delta G_{\text{ideal}} = \Delta G_{B} - \Delta G_{F} = 2\pi r p - \frac{\pi r^2 \Delta \mu}{\Omega}
\]  

(B11)

\[
\left(\frac{d\Delta G}{dr}\right)_{r=r_c} = 0
\]

provides:

\[
r_c = \frac{\rho \Omega}{\Delta \mu h}
\]

Eq. (B11) yields with \( r_c \), the following expression:

\[
\Delta G_c = \frac{\pi \rho^2 \Omega}{\Delta \mu h}
\]  

(B12)

For \( \xi < 1 \), the critical free enthalpy for nucleation at the site of a screw dislocation is:

\[
\Delta G^*_R = \Delta G_{r_{\text{max}}} - \Delta G_{r_{\text{min}}}
\]  

(B13)

and with Eqs. (B8) and (B9) we obtain:

\[
\Delta G^*_R = \pi \rho r_c \sqrt{1 - \xi} + \frac{hGb^2}{4\pi} \ln \left( \frac{1 - \sqrt{1 - \xi}}{1 + \sqrt{1 + \xi}} \right)
\]  

(B14)

for \( r > r_0 \) and \( \xi < 1 \).

Appendix B
Equation (B14) yields the decrease of the work of nucleation at a dislocation site compared with a perfect surface when setting $\xi \leq 1$. Then:

$$\Delta G^*_R = \Delta G_{\text{ideal}} \sqrt{1 - \xi}$$  \hspace{1cm} (B15)

The numerical value of $\xi$ is a function of the undersaturation. Thus etch pits occur when the undersaturation in the immediate vicinity of the surface is smaller than a critical undersaturation which causes spontaneous dissolution at a dislocation site. The presence of inhibitors decrease the numerical value of $\xi$, thus, decreasing the free enthalpy required for nucleation.

2. Silicon etchants.

Several preferential etchants can be used to delineate crystallographic defects in silicon (see table B1). The dissolution process of some of them are briefly discussed below.

A) The $\text{CrO}_3\text{H}_2\text{O-HF}$ and $\text{K}_2\text{Cr}_2\text{O}_7\text{H}_2\text{O-HF}$ systems.

The dissolution process in $\text{CrO}_3\text{H}_2\text{O-HF}$ and $\text{K}_2\text{Cr}_2\text{O}_7\text{H}_2\text{O-HF}$ etchants is based on the oxidation of the silicon by the oxidizing agent (CrO$_3$ or K$_2$Cr$_2$O$_7$) and the subsequent dissolution of the oxide by the HF acid. For the $\text{CrO}_3\text{HF-H}_2\text{O}$ system (Sirlt, Shimmel, Yang etch) the behaviour of the etchant in terms of preferential attack and formation of well defined etch pits, will depend on the concentration ratio $m$ (given in term of %/M)[3]. If $m$ is too high or even too low, the etchant will lose both its capability of preferential etching and to form well defined etch pits, respectively. This can be explained by the fact that the formation of a dislocation etch pit arises mainly from the
strain field associated with a dislocation (see heading 1C). The strain field produces a surface energy difference, $\Delta G_R$, between the site of the dislocation and the surrounding perfect crystal. If the CrO$_3$ concentration is too high the strong oxidising power of the solution tends to minimise the surface potential to a negligible extent. As a result preferential etching does not occur. Instead, if the concentration of CrO$_3$ is too low, etching due to HF dissolution is the dominant effect and the etch pits become ill defined. The same principle applies to the K$_2$Cr$_2$O$_7$ system (Secco etch). However, due to the low solubility of K$_2$Cr$_2$O$_7$ in water, the concentration of the oxidizing agent can not rise to a value high enough to give well defined etch pits. This is one of the reasons why the Secco etch gives rise only to isotropic (circular) etch pits.

**B) The HNO$_3$-HF-H$_2$O system**

The dissolution mechanism of silicon in HF-HNO$_3$-H$_2$O occurs mainly through an electrolytic mechanism that allows the corrosion of the silicon by the formation of local anodic and cathodic sites on the surface of the semiconductor[4]. In addition to the electrolytic attack, a chemical reaction also takes place on the silicon surface. The electrolytic reactions on the cathodic and anodic sites may be written as:

$$4e^+ + Si + 6F^- \rightarrow SiF_6^-$$

$$4e^- + 8H^+ + 4NO_3^- \rightarrow N_2O + 4H_2O$$

The local chemical attack is a reduction-oxidation reaction followed by the dissolution of the silicon dioxide formed. These reactions can be written as:
Si + 4 HNO₃ → SiO₂ + 4 NO₂ + 2 H₂O

SiO₂ + 6 HF → H₂SiF₆ + 2 H₂O

The formation of well defined etch pits will depend on the nature and quantity of the inhibitors in the solution. The role of impurities is to decrease the shift velocity \( v_t \) in order to obtain a very small ratio \( v_t/v_n \) (see Fig. 1) giving rise to well defined etch pits (see Fig. 1B).

<table>
<thead>
<tr>
<th>Silicon Etchant</th>
<th>Formula</th>
<th>Characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Secco [5]</td>
<td>2 HF(49%): 1 K₂Cr₂O₇ (0.15 M)</td>
<td>Preferential etching in all Si planes. Etch pits not well defined.</td>
</tr>
<tr>
<td>Schimmel [6]</td>
<td>2 HF(49%): 1 CrO₃ (1 M)</td>
<td>Preferential etching in all Si planes. Etch pits not well defined.</td>
</tr>
<tr>
<td>Sirtt [7]</td>
<td>4 HF(49%): 5 CrO₃ solution (50 g CrO₃ for 100 g D.I. H₂O)</td>
<td>Preferential etching in (111) and (110) planes. Etch pits well defined.</td>
</tr>
<tr>
<td>Yang [3]</td>
<td>1 HF(49%): 1 CrO₃ solution (1.5 M)</td>
<td>Preferential etching in all planes. Etch pits well defined.</td>
</tr>
<tr>
<td>Wright [8]</td>
<td>60 ml HF(49%): 30 ml HNO₃: 30 ml CrO₃: 60 ml HAc: 60 ml H₂O</td>
<td>Preferential etching in (111) and (110). Etch pits well defined.</td>
</tr>
<tr>
<td>Dash [9]</td>
<td>1 HF(49%): 3 HNO₃(70%): 10 HAc (glacial)</td>
<td>Preferential etching in all planes. Etch pits not well defined.</td>
</tr>
<tr>
<td>MEMC [10]</td>
<td>36 HF(49%): 25 HNO₃(70%): 18 HAc(glacial): 21 H₂O plus 1 g of Cu (NO₃)₂: 3 H₂O per 100 ml of mixed acid</td>
<td>Preferential etching in all planes. Etch pits well defined.</td>
</tr>
</tbody>
</table>

*Table B1 - Preferential etchants for silicon.*

219

*Appendix B*
References

Appendix C

Dislocations and plastic flow in the Diamond Cubic structure

1. Introduction.

The first observation of the plastic flow of silicon and germanium crystals was made by Gallagher [1] in 1952. They observed that although these crystals were completely brittle at room temperature, they become increasingly ductile above 60% of their absolute melting temperatures, that is about 450°C for Ge and 750°C for Si. Since the plastic glide of crystals operates by the movement of dislocations, they attributed the low-temperature brittleness of diamond structure crystals to the high frictional resistance in shearing the directional covalent bond. This frictional force against the movement of dislocations is generally known as the Pierls-Nabarro force [2].

2. Lattice defects in the Diamond structure.

A. Geometry

The diamond structure (D.S.) [3] can be derived from the fcc structure by doubling every atom of the fcc and shifting the second lattice by a/4[111] (see fig. 1 a). Thus, the close-packed \{111\} planes of the fcc lattice become pairs of \{111\} planes in the diamond structure as is shown in Fig. 1 b, or in a two-dimensional projection, in Fig. 1c.
It is observed, that if ABCABC is the stacking order of the \{111\} planes in the first fcc sublattice of the D.S. and abcabc is the stacking order of \{111\} planes in the second produced by the a/4[111] shift from the fcc, then aB, bC, cA, are closely spaced, double planes connected by three times as many bonds as are between the three times wider spaced Aa,Bb, Cc pair of planes. Since the Bravais lattice of the D.S. still is the fcc, the slip system of the D.S. is expected to be similar to that of the fcc. Therefore, one expects the D.S. to glide on \{111\} planes in <110> directions. Indeed, experimental results have
shown that the \{111\} planes are the slip planes in silicon\[1\] and germanium\[1\] and that the slip direction is \langle110\rangle.

**B. Perfect dislocations in the D.S.**

Perfect dislocations in D.S. have \(\mathbf{b} = \frac{1}{2} \langle 110 \rangle\) Burgers vectors and \{111\} glide planes\[4\]. The structure of perfect dislocations in the D.S. structure was first considered by Honstra \[5\] in 1958. He described various types of dislocations such as the 60° and the screw dislocation, among others. Fig. 2 a) shows the representation made by Honstra of the 60° dislocation (60° is the angle between the dislocation line \(\xi\) and the Burgers vector \(\mathbf{b}\)). It is observed that a dense row of "dangling" bonds are present along the edge of the extra half plane. Fig. 2 b) shows the representation made by Honstra of a 'screw dislocation. It is observed that the screw dislocation, contrary to the 60° dislocation, has no "dangling" bonds.

---

**Fig. 2 a- continued**
More recently, Hirth and Lothe[4] proposed that, due to the double occupancy of the fcc bravais lattice, there are two different sets of dislocations in the diamond cubic lattice, with one member of each set having a Burgers vector identical to one dislocation in the other set.

In order to see the distinction between these two sets of dislocations let us consider the simple case of a $60^\circ$ dislocation (see Fig. 4 a). From Fig. 4 a, it is observed that the extra plane terminates between layers of the *same letter index*, i.e. B and b. Therefore, glide of this dislocation will occur between layers B and b (or equivalent pairs). However, it is also possible that the extra plane, instead of terminating between layers of the same letter could terminate between layers of different index, for example between layers a and B (see Fig. 3). In this case, glide of this dislocation will occur between layers a and B.

Hirth and Lothe, named these two different set of dislocations as being the "glide" (dislocations which glide between layers a and B) and the "shuffle" (dislocations which glide between layers B and b) set. According to Hirth and Lothe, perfect dislocations of
the "glide" set can dissociate into glissible Shockley partials[6] whilst perfect dislocations of the "shuffle" set can not dissociate directly into partials, because such dissociation would produce a high-energy fault of the type:

\[ CcAaB \rightarrow aBbCc \]

\[ \text{Fig. 3 - 60° dislocation of the "glide" set (According to Hirth and Lothe [4])} \]

**C. Partial dislocations**

Frank[6] suggested that the stability of a dislocation depended on the strain energy associated with it and that, in a first approximation, the energy associated with a dislocation was proportional to the square of its Burgers vector. He, therefore, proposed that a dislocation with Burgers vector \( b_1 \) could dissociate into two other dislocations \( b_2 \) and \( b_3 \) if:

\[ b_1^2 > b_2^2 + b_3^2 \]  

(C1)
Eq. (C1) is generally known as the Frank energy criterion[6].

The dissociation of perfect dislocations into partial (imperfect) dislocations occurs as a method to reduce the energy of the dislocation according to the Frank criterion. The dissociation process always creates a narrow stacking fault between the two partials. This configuration is generally known as an extended dislocation. (see Fig. 4)

\[ b_1 = \frac{1}{2}[\overline{1}01] \quad b_2 = \frac{1}{4}[\overline{2}11] \quad b_3 = \frac{1}{4}[\overline{1}12] \]

**Fig. 4 - Dissociation of a perfect dislocation into Shockley partials.**

In the D.S. structure, Shockley partials[7] can be created by the dissociation of a perfect \((b = \frac{1}{2} < 110 >)\) dislocation of the 'glide' set (see heading B).

The dissociation reaction can be written as:

\[ \frac{1}{2}[110] \rightarrow \frac{1}{6}[211] + \frac{1}{6}[12\overline{1}] \]  

**Perfect**  **Shockley**  **Shockley**

Another possible dissociation reaction, is the formation of a Frank sessile[8] and a Shockley partial. This reaction can be written as:

\[ \frac{1}{2} < 110 > \rightarrow \frac{1}{3} < 111 > + \frac{1}{6} < 211 > \]  

**Perfect**  **Frank**  **Shockley**
Experimental evidence of the existence of extended dislocations in silicon [9] and germanium [10] has been widely reported, confirming that dissociation of perfect dislocations occurs in D.S. crystals.

**Thompson tetrahedron**

In fcc and similar structures (such as the D.S.), dissociation reactions can be easily written using the notation proposed by Thompson[11].

Thompson suggested that by joining the atoms in a one-eighth unit cell by straight lines, a imaginary tetrahedron (see Fig. 5) containing the four possible \{111\} glide planes could be formed.

![Fig. 5 - A one-eighth unit cell of edge length 1/2a₀ of an fcc lattice, showing the Thompson tetrahedron ABCD.](image)

From Fig. 5, it is easily observed that the edges of the tetrahedron correspond to the six \<110\> glide directions of the fcc structure. The atom at the origin is labelled D and the others are labelled ABC in the clockwise order indicated in Fig. 5. The midpoints of the faces opposite to A, B, C and D are labelled \(\alpha, \beta, \gamma\).
and δ respectively. If the tetrahedron is open up at D, it can be folded out into the planar arrangement displayed in Fig. 6.[11]

As it is clear in Fig. 6, the letters a, b, c and d represent the \{111\} planes; the perfect dislocations \( \frac{1}{2} <110> \) are represented by pairs of Roman letters- for example \( \frac{1}{2}[011] \) becomes BD; and Shockley partials are represented by Greek-Roman pairs, for example \( \frac{1}{6}[211] \) becomes Dβ. Finally, the Frank partials are represented also by Greek-Roman letters, for example \( \frac{1}{3}[111] \) becomes 5D.

Fig 6- A Thompson tetrahedron opened up at a corner D.
Silcox and Hirsch [12] proposed that the stacking fault tetrahedron could be formed by the expansion of a triangular Frank dislocation loop (δD) (see Fig. 7). They proposed that the <110> sides of the triangular Frank loop could dissociate according to the reaction:

\[ \delta D \rightarrow \delta \beta + \beta D \]  

(C4)

where δβ (b = \frac{1}{\sqrt{2}}[110]) is a low energy stair-rod dislocation[12] and βD is a Shockley partial. This dissociation reaction can take place at the three sides of the triangular loop, yielding the configuration of Fig. 7 b. According to Silcox and Hirsch the Shockley partials bows out in their slip plane as they are repelled by the stair rods. Taking into account the fact that opposite ends of the loops are of opposite signs, the Shockley partials attract each other in pairs to form stair-rods along DA, BA and CA according to the reaction:

\[ \beta D + \gamma D \rightarrow \gamma \beta \]  

(C5)

The attraction forces lead to the development of configurations depicted in Fig. 7 c and Fig. 7 d. Therefore, the final result is a tetrahedron bounded completely by stacking faults and stair-rod dislocations.
Fig. 7 - Various stages (a-d), in the formation of a stacking-fault tetrahedron from a Frank dislocation loop (according to Silcox and Hirsch[12])

Appendix C
References

Appendix D

Preparation of thin samples for TEM observations.

1. Preparation of silicon samples for plan view observations.

The thin film silicon samples analysed by plan view TEM were prepared by the "float off" method[1]. Briefly the following sample preparation procedure was used:

1. Cleave SIMOX wafers into small samples (typically 5x5 mm).
2. Immerse the SIMOX samples in HF(40%) to dissolve the BOX layer (typically 8 hrs.)
3. Rinse in DI water.
4. The silicon thin film floats to the surface and is scooped up using a 3 mm Cu grid.
5. Remove the excess water using filter paper.
6. Dry in air and place a second grid on top of the support grid and glue them with a small spot of silver dag.
2. Preparation of samples for cross section observations.

The samples prepared for XTEM analyses were thinned to electron transparency using ion beam milling[2]. Briefly, the procedure was as follows:

1. Cleave SIMOX wafers into small samples (typically 3x10 mm²).
2. Glue two samples together (top surfaces in contact) using an epoxy resin.
3. Cut the glued sample into 500 μm thin sections using a diamond saw.
4. Grind and polish the 500 μm sections down to a thickness of about 50 μm.
5. Mount the 50 μm thin section onto a 3 mm Cu grid using silver dag.
6. Load the sample into the ion beam miller (Model: Gatan 600).
7. Bombard the sample using a dual Ar⁺ beam accelerated to 5 keV with an angle of incidence of 15° until electron transparency of the sample is obtained.

References


Appendix E

Density and length of the OISF in the sacrificial oxidised SIMOX samples.

A list of all the sacrificial oxidised SIMOX together with the length and density of OISF is presented in tables 1 to 8. We also list the thermal oxide thicknesses, the estimated number of silicon atoms consumed by the oxidation ($N_{\text{ox}}$), and the estimated number of interstitials captured by the OISF ($N_{\text{OISF}}^{\text{Si}}$, see section 8.2.2 for more details). In order to facilitate the interpretation of the data, we have grouped the sacrificial oxidised samples in different tables corresponding to different SIMOX wafers (table 1 to 5). Moreover, in table 6, 7 and 8, we group the sacrificial oxidised SIMOX samples (1100°C for 4 hours) pre-amorphised with BF$_2$, Si$^+$ and Ge$^+$ ions, respectively. From table 1 to 5, it is observed that the $N_{\text{OISF}}^{\text{Si}}$ values increase with temperature and vary significantly for different wafers (see section 8.2.2 for more details).
<table>
<thead>
<tr>
<th>Sample</th>
<th>Wafer</th>
<th>Oxidation temperature (°C)</th>
<th>Oxidation time (hrs.)</th>
<th>Thermal Oxide thickness (Å)</th>
<th>Si overlayer after oxidation (Å)</th>
<th>OISF density x10² (cm⁻²)</th>
<th>OISF length (μm)</th>
<th>N_x x10¹⁵cm⁻²</th>
<th>N_w x10¹⁶cm⁻²</th>
</tr>
</thead>
<tbody>
<tr>
<td>B1/1</td>
<td>B1/1</td>
<td>850</td>
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Table 1 - List of sacrificially oxidised SIMOX prepared from wafer B1 (initial thickness: Ti = 2100Å).
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<th>Thermal Oxide thickness (Å)</th>
<th>Si overlayer thickness (Å)</th>
<th>OISF density x10^5 (cm^-2)</th>
<th>OISF length (µm)</th>
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Table 2- List of sacrificially oxidised samples prepared from wafer B2 (initial thickness: Ti = 2500 Å).
### Table 3- List of sacrificially oxidised samples prepared from wafer B3

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Table 4- List of sacrificially oxidised samples prepared from wafer B4 (initial thickness, Ti =2100 Å).
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<th>Oxidation temperature (°C)</th>
<th>Oxidation time (hrs.)</th>
<th>Thermal Oxide thickness (Å)</th>
<th>Si overlayer thickness (Å)</th>
<th>OISF density (x10^3) cm(^{-2})</th>
<th>OISF length (μm)</th>
<th>(N_{ox}) x10(^{15}) cm(^{-2})</th>
<th>(N^0_{ox}) x10(^{16}) cm(^{-2})</th>
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Table 5- List of sacrificial oxidised samples prepared from wafer B5 (initial thickness: Ti = 2100 Å).
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Table 6 - List of sacrificial oxidised samples implanted with BF⁺ ions.

Appendix E
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<th>Ion implanted</th>
<th>Energy (keV)</th>
<th>Dose (x10¹⁸ cm⁻²)</th>
<th>Oxidation temperature (°C)</th>
<th>Oxidation time (hrs)</th>
<th>OISF density x10⁴ cm⁻²</th>
<th>N₂o x10¹⁷ cm⁻²</th>
<th>N²off x10⁶ cm⁻²</th>
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<td>1</td>
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*Table 7- List of sacrificial oxidised samples previously implanted with Si ions.*
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<th>Oxidation temperature (°C)</th>
<th>Oxidation time (hrs.)</th>
<th>OISF density (x10⁶ cm⁻²)</th>
<th>N_{ox} x10⁷ cm⁻²</th>
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</table>

Table 8 - List of sacrificial oxidised SIMOX samples previously implanted with Ge⁺ ions.
Publications

Part of this thesis has been published in several conferences and journals:


