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# An *in-situ* TEM study of the effects of 6 keV He ion irradiation on Si and SiO<sub>2</sub>

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**Abstract.** Using the new MIAMI\* facility at the University of Huddersfield, an *in-situ* TEM study has been carried out on the effects of helium implantation on a tri-layer system consisting of monocrystalline silicon (c-Si), silicon dioxide (SiO<sub>2</sub>) and polycrystalline silicon (poly-Si). Co-irradiation of these three components enabled direct comparisons to be made of differences between the response of c-Si and poly-Si to the He irradiation; in particular, differences in the development of interstitial clusters and bubbles and eventual amorphisation. For implantations to high fluences (>10<sup>17</sup> ions/cm<sup>2</sup>), very significant levels of porosity were observed to build up in the Si, leading to changes in the width of the Si layers of up to 29%. Although no helium bubbles were formed in the SiO<sub>2</sub> layer, a very significant dimensional change (but in this case an observed shrinkage) also occurred in this material. Finally, room temperature amorphisation of the Si was observed at high fluences, beginning at somewhat lower fluences in the poly-Si than in the c-Si. A brief discussion of the origins of these effects is presented.

\*Microscope and Ion Accelerators for Materials Investigations

## 1. Introduction

In order to compare the development of porosity in c-Si and poly-Si as a result of implantation with helium ions, we have prepared specimens from a tri-layer structure consisting of poly-Si (100 nm) on a SiO<sub>2</sub> layer (50 nm) on bulk crystalline silicon using standard cross-sectional TEM specimen preparation techniques. These thin tri-layers were then irradiated at 6 keV with He<sup>+</sup> at room temperature *in situ* in a TEM at the MIAMI facility. This approach enabled us to subject the two materials to identical treatments (flux, fluence, temperature) and thus make accurate comparative measurements. We report on the development of radiation damage in the form of interstitial clusters and He bubbles, on the swelling due to the radiation damage and the differences in the amorphisation of the poly-Si and c-Si at high fluences. We also present and discuss changes in the SiO<sub>2</sub> layer (shrinkage) due to the ion irradiation which indicate that viscoelastic processes previously observed for irradiation at higher energies (MeV) also occur at low energies.

## 2. Experimental

The tri-layer specimens were prepared at the Scottish Microelectronic Centre at the University of Edinburgh by depositing a 114nm layer of poly-Si onto a 57 nm thermally grown amorphous SiO<sub>2</sub> layer on a {100} Cz crystalline Si wafer [1]. Cross-sectional specimens were then prepared at the University of Salford by means of mechanical (tripod) polishing and ion-beam thinning using a Gatan Model 691 PIPS system with 4 keV Ar<sup>+</sup> ions incident at 4° to the specimen surface.

Full details of the MIAMI in-situ TEM/ion-accelerator facility can be found elsewhere [2]. The experiments presented here were performed at room temperature, with 6 keV He<sup>+</sup> ions at an ion flux of  $\approx 10^{13}$  ions/cm<sup>2</sup>/s. It is important to note that the specimens were irradiated in this cross-sectional, tri-layer form so that each of the layers was exposed to an identical flux of He ions. (The angle between ion and electron beams is 25°). Images were recorded by means of a 1.4 megapixel Gatan ES500W digital camera mounted above the viewing chamber. The real space images were post-processed into time-lapse video sequences of the experiments. Diffraction patterns were recorded using a bottom-mounted 4 megapixel Gatan Orius SC200 digital camera.

## 3. Results and Discussion

Figure 1a) is a bright-field micrograph of the specimen prior to irradiation in which the 114 nm wide poly-Si layer can be seen at the top of the image and is separated from the c-Si layer by a 57 nm layer of SiO<sub>2</sub>. The micrographs in figure 1b) to 1e) show the changes that resulted from irradiation with 6 keV He<sup>+</sup> ions.

### 3.1. Interstitial clusters

The implantation of He into c-Si at room temperature leads to the formation of interstitial clusters. These are visible in bright-field TEM in all orientations and, due to the fact that these defects are made visible by the interaction of the electron beam with their strain field whose extent is much larger than the cluster itself, they are generally visible by TEM at fluences lower than those at which bubbles are visible. The clusters can be seen in the c-Si layer in figure 1b) as small regions of dark contrast. However in the poly-Si layer, clusters are only seen to form in the largest grains and in much of this layer are never observed. Grains larger than approximately 40 nm behave similarly to monocrystalline silicon and interstitial clusters form in them; in grains below this size interstitial clusters do not form. The implication of this observation is that, in small grains, interstitials diffuse to the nearby grain boundary before encountering other interstitials and nucleating interstitial clusters.

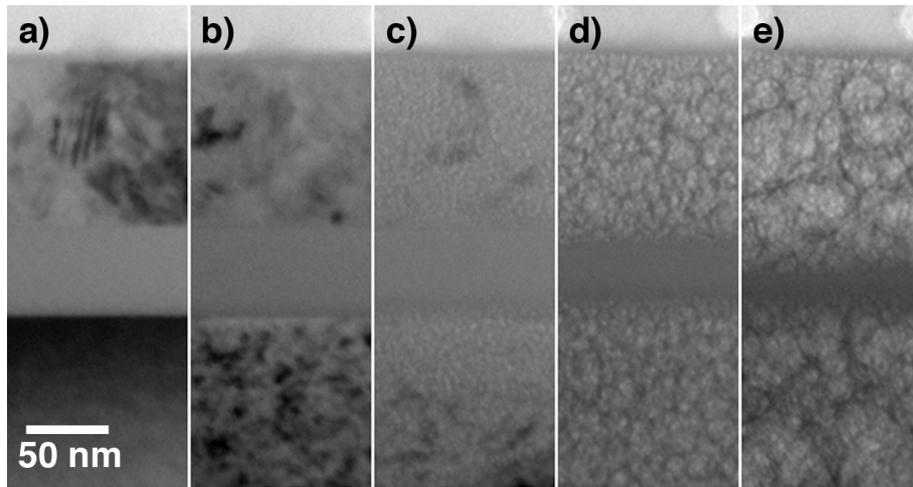
### 3.2. He bubbles

Nanometre-sized He bubbles, identified by contrast reversal on passing through focus, are first discernible at fluences of  $\approx 7 \times 10^{16}$  ions/cm<sup>2</sup> in both Si layers and grow in size with increasing fluence; however, the diffraction contrast from interstitial clusters renders it more difficult to observe the bubbles in the c-Si layer. Figure 1c shows the bubble morphology following irradiation to a fluence of  $1.3 \times 10^{17}$  ions/cm<sup>2</sup>. It is not possible to measure the bubble diameters with any degree of accuracy at this stage as the size observed in the TEM varies significantly with degree of defocus and the bubbles are not visible at focus — but they are approximately 1–2 nm in diameter.

There is no measurable difference in bubble size in the two layers and no grain boundary decoration (formation of larger bubbles) is observed. Also, unlike the case reported for bubbles in some metals [3,4], a denuded region close to grain boundaries does not occur. Figures 1d and 1e show the specimen following implantation to fluences of 2.3 and  $3.1 \times 10^{17}$  ions/cm<sup>2</sup> respectively and in these images also, no significant differences can be seen in the morphology of the poly-Si and c-Si layers.

### 3.3. Swelling of the Si layers

In addition to the contrast due to the interstitial clusters and helium bubbles, larger scale changes are also visible in figure 1. Specifically, changes to the width of the poly-Si and SiO<sub>2</sub> layers occur as the fluence is increased – the poly-Si layer increases and the SiO<sub>2</sub> layer decreases in width.



**Figure 1.** TEM micrographs of the c-Si/SiO<sub>2</sub>/poly-Si tri-layer (the c-Si layer is at the bottom of the images) following irradiation to nominal fluences of 6 keV He ions of: a) 0; b)  $5.0 \times 10^{16}$ ; c)  $1.3 \times 10^{17}$ ; d)  $2.3 \times 10^{17}$  and e)  $3.1 \times 10^{17}$  ions/cm<sup>2</sup>. (Bright-field images).

The swelling is clearly measurable in the poly-Si layer and in figure 1e) the poly-Si layer is 29% wider than in the unirradiated material. Assuming an isotropic swelling, this corresponds to a volume swelling of 115%. If the swelling results entirely from the displaced material from the He bubbles, with no additional contribution from elastic distortion around interstitial defects, then this corresponds to a porosity of 53%. This may be a reasonable assumption as, at this stage, the silicon has been amorphised (see below). Although the swelling cannot be measured in the c-Si layer, the similar bubble morphology leads us to the conclusion that a similar level of swelling occurs in this layer also.

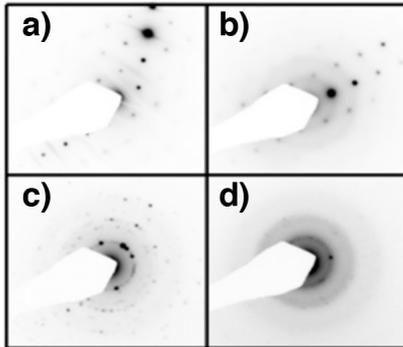
#### 3.4. Shrinkage of the SiO<sub>2</sub> layer

Helium bubbles do not form in the SiO<sub>2</sub> due to the high mobility of He in SiO<sub>2</sub> which enables the He to diffuse out of the layer [5]; however, in figure 1 the SiO<sub>2</sub> layer can be clearly seen to shrink with increasing He fluence. The shrinkage, at the highest fluence of  $3.1 \times 10^{17}$  ions/cm<sup>2</sup> is 68%.

There is a body of literature on the effects of ion irradiation on SiO<sub>2</sub> with ions of much higher energies (MeV) [6,7]. It is well established that SiO<sub>2</sub>, under mechanical stress and under ion irradiation, undergoes viscoelastic flow so as to reduce the applied stress to zero. The dimensional changes to the SiO<sub>2</sub> layer in the current experiments can be explained in the following manner: the swelling in the silicon layers on either side of the oxide layer then applies stress to the oxide layer at both interfaces. The SiO<sub>2</sub> layer will, therefore, expand in both orthogonal directions parallel to the interface and conservation of mass requires that the width of the layer decrease in the unconstrained orthogonal direction, giving rise to the observed shrinkage. We, therefore, believe that our experiments provide evidence for the occurrence of ion-beam induced viscoelastic flow processes of stressed SiO<sub>2</sub> under irradiation with 6 keV helium ions. Assuming isotropic swelling in both of the Si layers, the observed shrinkage of the SiO<sub>2</sub> layer width would be expected to be twice the observed linear swelling in the Si layers. The actual values (29% and 68%) may indicate a degree of anisotropy in the Si swelling (greater in the direction of ion incidence) but further studies will be required to test this.

#### 3.5. Si Amorphisation

Experiments in which selected area diffraction patterns were recorded as a function of fluence indicate a difference in the amorphisation behaviour of c-Si and the poly-Si with the later amorphising at lower fluences. This can be seen in figure 2.



**Figure 2.** Selected area diffraction patterns before and after irradiation with 6 keV He to a fluence of  $2.5 \times 10^{17}$  ions/cm<sup>2</sup>. a) c-Si and c) poly-Si before irradiation; b) c-Si and d) poly-Si after irradiation. A much greater degree of amorphisation is seen in the poly-Si than in the c-Si as evidenced by the intensity of the amorphous rings in d) compared with those in b) and by the greater intensity of the crystalline spots in b) compared with those in d).

Although there is ambiguity in the scientific literature regarding the possibility of amorphising Si using light ions such as He, both Reutov and Sokhatskii [8] and Siegele and Weatherly [9] have reported such amorphisation with higher energy ions (17 and 20 keV respectively). The present results constitute the first evidence for amorphisation of silicon at an energy as low as 6 keV.

#### 4. Conclusions

Irradiation of poly-Si and c-Si with 6 keV He<sup>+</sup> ions at room temperature results in the formation of identical helium bubble distributions in both materials and, at high fluences, leads to significant porosity and swelling (53% and 115% respectively). The formation of interstitial clusters is, however, suppressed in poly-Si and this is assumed to be due to absorption of Si interstitials by the grain boundaries. A surprising conclusion, is thus that the presence of interstitial clusters appears to have no effect on the nucleation and growth of helium bubbles in Si.

The large degree of swelling in the two Si layers exerts a tensile stress in both orthogonal directions at the SiO<sub>2</sub> interfaces and this effect, coupled with ion-beam-induced viscoelastic flow of the oxide so as to minimise the stress leads to a shrinkage of the width of the SiO<sub>2</sub> layer in the direction directly observed in our experiments. These experiments constitute the first indication that ion-beam-induced viscoelastic processes in SiO<sub>2</sub> can occur during irradiation using ions with energies below the MeV range.

Both c-Si and poly-Si have been observed to amorphise as a result of room temperature irradiation with 6 keV He ions with the poly-Si amorphising at lower fluences. This is assumed to be due to preferential nucleation of the amorphous material at the grain boundaries.

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