The Surface Proximity Effect on the Formation of Extended Defects in Ion Beam Synthesised SiGe/Si Heterostructures

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Abstract—The relaxation of ion beam synthesised SiGe alloys occurs during solid phase epitaxial growth (SPEG) by a roughening of the amorphous/crystalline (a/c) interface, leading to the formation of extended defects. SiGe/Si structures have been formed by implantation of Ge⁺ ions at energies of 70 keV and 400 keV and doses above the critical value for strain relaxation, followed by post-amorphisation to a depth of $\sim 1 \mu\text{m}$ and regrowth at 700°C. TEM and RBS analysis of the regrown structures show that relaxation-induced stacking faults (SFs) are nucleated in the vicinity of the peak of the Ge concentration. For an implantation energy of 70 keV, SFs are the only defects observed and they extend up to the surface. For an implantation energy of 400 keV, the SFs terminate within the bulk far from the surface, while “hairpin” dislocations are also formed and extend up to the surface. These results are explained in terms of the ratio between the depth at which SFs are nucleated and the roughness of the a/c interface at that depth. Varying the defect nucleation depth by removing a surface layer from the structures implanted at 400 keV prior to SPEG does not result in a change of the size of the SFs, while “hairpin” dislocations are still formed, suggesting that the roughness of the a/c interface during regrowth is only determined by the Ge content in the alloy.

I. INTRODUCTION

Ion implantation has been proposed as one of the possible technologies to produce device worthy SiGe/Si heterostructures [1], [2]. As a consequence of ion implantation, the near surface layer is amorphised, calling for a solid phase epitaxial growth (SPEG) stage during which the accumulation of elastic strain may result in the formation of extended defects. It has been shown by Paine et al [3], [4], that the introduction of strain relieving stacking faults (SFs) is facilitated by a change from a planar (001) growth surface to one which is faceted parallel to the crystal {111} planes. This description has been confirmed by Elliman et al [5] who used time resolved reflectivity (TRR) measurements to show that the roughness ($r(z)$) of the amorphous/crystalline (a/c) interface at a particular depth (z) during regrowth is directly proportional to the germanium concentration at that depth. However, although in the work of Paine the authors reported the presence of SFs nucleated in the vicinity of the peak of the Ge depth profile, no SFs were found to extend up to the surface in fully regrown structures synthesised by implantation of 200 keV Ge⁺ ions. Instead, long threading dislocations were observed, similar to the “hairpin” dislocations previously reported by our group [6], [1] in structures synthesised by implantation of 400 keV Ge⁺ ions. On the other hand, relaxation-induced SFs have been found to extend to the surface with no “hairpin” dislocation formation

in structures formed using lower Ge⁺ implantation energy (120 keV in [7] or 160 keV in [8]).

The aim of this experiment is to identify which type of relaxation-induced extended defects is formed after regrowth of SiGe alloy layers synthesised using different values of the Ge⁺ implantation energy. In addition, the influence of the surface proximity on the type of defect formed is also investigated. The TEM and RBS results are discussed in terms of the ratio between the depth at which the relaxation-induced defects are nucleated and the roughness of the a/c interface at that depth.

II. EXPERIMENTAL DETAILS

3” p-type (10 $\Omega\text{-cm}$) (100) Cz silicon wafers were implanted with Ge⁺ ions at energies of 70 keV and 400 keV to doses of $4 \times 10^{16} \text{ Ge}^+/\text{cm}^2$ and $1 \times 10^{17} \text{ Ge}^+/\text{cm}^2$, respectively. Both Ge⁺ doses are above the critical value for strain relaxation for the implantation energy used in this study [9]. The wafers were subsequently post amorphised down to a depth of $\sim 1 \mu\text{m}$ using 500 keV $6 \times 10^{15} \text{ Si}^+/\text{cm}^2$ implanted at 150 K using liquid nitrogen. Selected samples from wafer 2 (see table I) were thinned using anodic oxidation in a 0.05 molar solution of KNO₃ in 90% ethylene Glycol and 10% de-ionised water. This solution produced anodisation coefficient of 0.25nm/V. Using platinum wire as the cathode and the samples as the anode a constant current supply of 90 mA and 100 V were used to oxidise 25 nm per run. This oxide was subsequently removed in HF solution. The process was repeated to remove layers of different thickness. All samples were finally annealed at 700°C for 20 min in flowing N₂ in an RTA unit. The various process steps are also schematically shown in figure 1.

The thickness of the remaining amorphous layers in the various samples prior to SPEG was measured by RBS. The corresponding values of the thickness removed by anodic oxidation are reported in table I. Due to non-uniformity in the efficiency of the anodisation process across the samples, surface roughening was observed after regrowth of the oxidised samples. However, the roughness values were found to be low even after several anodisation runs. After removing a surface layer of almost 200 nm the average value of the surface roughness was measured to be about 9 nm, with a maximum “peak-to-valley” distance of about 15 nm.

In addition to the thickness of the amorphous layers, the germanium doses and the germanium depth distribution parameters shown in table I were also determined by RBS

analysis, performed using 1.5 MeV He⁺ ions with an overall system resolution of 14 keV. The microstructure of the synthesised structures after regrowth was investigated by plan view and cross sectional TEM using either a JEOL 2000-FX or a Philips 400T electron microscope.

III. RESULTS AND DISCUSSION

Figure 2(a) shows a XTEM micrograph from sample 1 where a layer of crystallographic defects is observed extending from a depth of about 50 nm up to the surface. The extended defects include “V”-shaped stacking faults lying on {111} planes, with a density of the order of $1 \times 10^{10} / \text{cm}^2$. The peak germanium concentration for this sample is 11.4 at.%. A lower magnification image (not shown) also shows a layer of EOR defects at a depth of 1 μm , corresponding to the thickness of the amorphous layer formed during the post-amorphisation step. Assuming the simple relation given by Elliman [5] between the roughness of the (a/c) interface at a particular depth during regrowth and the germanium concentration at that depth, it is found for sample 1, implanted with $4 \times 10^{16} \text{ Ge}^+ / \text{cm}^2$ at 70 keV, that strain relieving stacking faults were formed at a depth of 50 nm, which is comparable to the estimated value of the roughness of the a/c interface (30 nm). This situation is schematically described in figure 2(b), where it is shown that the faceting of the a/c interface, responsible for the formation of strain relieving SFs, occurs at a depth close to the surface. Under such conditions, SFs are easily terminated at the free surface, storing all the elastic energy released in the process.

Figure 3(a) shows a XTEM micrograph from sample 2, implanted with $1 \times 10^{17} \text{ Ge}^+ / \text{cm}^2$ at 400 keV. “Hairpin” dislocations are observed in the surface region of the sample. They are formed at a depth of about 0.25 μm and extend up to the surface. This depth corresponds to the $\bar{R}_p$ of the implanted Ge⁺ ions where the Ge concentration has a maximum value of 7.8 at.%. The density of “hairpin” dislocations, determined by plan view TEM (not shown), has been found to be $(1.5 \pm 0.35) \times 10^9 \text{ disl} / \text{cm}^2$. Similar to the previous sample, a layer of EOR defects is observed at a depth of 1 μm . However, a high magnification XTEM image from the same sample (figure 5(a)), taken from a region close to the $\bar{R}_p$ of the germanium distribution profile, shows that SFs are also formed in this structure, with an average size of 30 nm, which are terminated within the bulk. No SFs were observed either above or below the “ $\bar{R}_p$ ” region. Using the same argument employed for the low energy implanted sample, it is found for sample 2 that strain relieving SFs and “hairpin” dislocations were formed at a depth of $\sim 250 \text{ nm}$, where the estimated value of the roughness of the a/c interface is 20 nm, which is more than ten times smaller. This situation is schematically described in figure 4, where it is shown that the faceting of the a/c interface, responsible for the formation of strain relieving SFs, occurs at a depth far away from the surface. Under such conditions, SFs cannot extend up to the surface, as the energy required would exceed the available elastic energy associated with the SiGe alloy layer [10]. In addition,

as the moving a/c interface passes through the peak of the germanium depth profile, crystallization proceeds in a region of decreasing germanium concentration, leading to a smoothing of the a/c interface [5]. Therefore the probability of new SFs to be nucleated close to the surface is low in agreement with the TEM results.

The effect of the proximity of the free surface on the formation of relaxation-induced defects in samples implanted with 400 keV Ge⁺ ions is described in figure 3b, c and d where XTEM micrographs from samples 2b, 2c and 2d are presented, respectively. The layer of EOR defects associated with the Si⁺ post-amorphisation step is observed in all the samples, at a depth depending on the thickness of the surface layer removed by anodic oxidation and HF etching (see table I). For a removed layer considerably thinner than $\bar{R}_p$ (samples 2b, 2c in figure 3b and c, respectively), “hairpin” dislocations are still observed, whose length is consistent with the thickness of the removed layer. On the other hand “hairpin” dislocations are no longer observed in sample 2d (figure 3d) where a surface layer of 180 nm was removed before SPEG, comparable to the depth corresponding to the $\bar{R}_p$ of the implanted Ge⁺ ions. High magnification XTEM micrographs from the same samples are shown in figure 5b, c and d, respectively. It is observed that SFs are still formed both within the bulk as well as on the surface of the samples. However, their size never exceeds 30 nm, as in the reference non-etched sample (figure 5a). These results suggest that, upon removal of a thin surface layer, the ratio between the defect nucleation depth and the a/c interface roughness decreases, allowing some of the SFs to extend up to the surface. However, the size of the SFs is not found to increase, indicating that the a/c interface roughness is not affected by the proximity of the free surface but it is determined by the Ge content only.

IV. CONCLUSIONS

SiGe/Si structures have been formed by implantation of Ge⁺ ions at energies of 70 keV and 400 keV and doses above the critical value for strain relaxation, followed by post-amorphisation to a depth of $\sim 1 \mu\text{m}$ and regrowth at 700°C. Surface layers of different thicknesses were removed from selected samples before SPEG using anodic oxidation and HF etching. From the TEM and RBS analysis it is concluded that:

- The extended defects formed upon relaxation of the strain associated with a SiGe/Si heterostructure synthesised by Ge⁺ implantation consist of SFs nucleated in the vicinity of the peak of the germanium concentration.
- The relaxation induced SFs extend up to the surface for a low Ge⁺ implantation energy (70 keV) or terminate within the bulk for a higher Ge⁺ implantation energy (400 keV) depending on the ratio between the depth at which the defects are nucleated and the roughness of the a/c interface at that depth during SPEG. For a high Ge⁺ implantation energy, “hairpin” dislocations which extend up to the surface are also formed together with the SFs.

- The size of the SFs is not affected by the proximity of the free surface to the a/c interface during SPEG, while "hairpin" dislocations are no longer observed when the thickness of the removed layer is close to the defect nucleation depth.

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Fig. 1. Schematic diagram of the processing steps for the formation of SiGe/Si structures by ion implantation and anodic oxidation.

Fig. 4. Schematic diagram illustrating the roughening of the a/c interface and the formation of SFs terminating within the bulk and "hairpin" dislocation extending up to the surface during SPEG of SiGe alloys synthesised at an implantation energy of 400 keV.

TABLE I

DETAILS OF SAMPLES AND GERMANIUM IMPLANT PARAMETERS EXTRACTED FROM RBS ANALYSIS.

Sample no.	Ge ⁺ en. (keV)	Dose (Ge ⁺ /cm ²)	$\bar{R}_p$ (nm)	Peak Ge conc. (at.%)	Post-amorphisation	Thickness removed by Anodic Oxidation (nm)	Annealing (in N ₂)
1	70	4.0e16	30	11.4±0.45	500 keV Si ⁺	—	700°C 20 min
2	400	1.0e17	245	7.8±0.3	$\phi=6e15/cm^2$ at 150 K α -thick.:980 nm	—	
2a	400	1.0e17	—	—		75	
2b	400	1.0e17	—	—		95	
2c	400	1.0e17	—	—		130	
2d	400	1.0e17	—	—		180	
2e	400	1.0e17	—	—		225

Fig. 2. (a) XTEM bright field micrograph from sample 1, implanted with 70 keV Ge^+ ions, after post-amorphisation and regrowth at 700°C for 20 min. (b) Schematic diagram illustrating the roughening of the a/c interface and the formation of SFs terminating at the surface during SPEG of SiGe alloys synthesised at an implantation energy of 70 keV.

Fig. 3. XTEM micrographs from samples implanted with 400 keV Ge^+ ions after removal of different surface layers and regrowth at 700°C. (a) Sample 2 (bright field). (b) Sample 2b (weak beam dark field). (c) Sample 2c (weak beam dark field). (d) Sample 2d (weak beam dark field).

Fig. 5. High magnification XTEM micrographs from samples implanted with 400 keV Ge^+ ions (see figure 3) after removal of different surface layers and regrowth at 700°C. (a) Sample 2 (bright field). (b) Sample 2b (dark field). (c) Sample 2c (dark field). (d) Sample 2d (dark field).