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X-ray characterization of β-SiC growth and structural modification of Si by MeV ion implantation

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Abstract. A combination of low-temperature MeV carbon ion implantation and post high-temperature annealing was used to produce a β-SiC buried layer in Si(001). The growth of the β-SiC layer and re-growth of the front Si layer upon annealing was monitored by x-ray diffraction and x-ray pole figure measurement. In the samples annealed at a temperature of 1000 °C or higher, the buried β-SiC layer has a near-perfect orientation relationship with the substrate. The structure of the front Si layer, interestingly, was modified after annealing, i.e., the forbidden Si(002) diffraction was observed. The orientation relationships among the three layers, i.e., the front Si layer, the β-SiC buried layer and the bulk substrate, were also investigated by the x-ray pole figure measurement.

1. Introduction

SiC is a promising candidate material for device fabrications due to its outstanding properties and has attracted much research interest [1, 2]. To produce successively buried SiC layers of good crystal quality and high purity, an often-employed approach is high-dose carbon implantation into Si followed by thermal annealing. This technique, namely ion beam synthesis (IBS), has been proven successful in forming polycrystalline/orientated β-SiC buried layers in silicon [3–5]. The key factor that determines the crystal quality of the β-SiC buried layer, following the literature [5], is the annealing temperature. From the experimental data published until now, it seems clear that annealing temperatures around 850 °C and 900 °C are sufficiently high to transform the implanted Si-C layer into crystalline β-SiC structure [6, 7]. However, some authors believe higher temperatures, e.g., 1000 °C [8], 1250 °C [9] and 1405 °C [10], are needed to achieve crystalline β-SiC. It is also argued that other conditions, e.g., the implantation temperature [11] and the implanted carbon dose [9], also have an influence on the β-SiC growth. More systematic works are therefore desirable for successful application of IBS to produce SiC [5].

It is known that carbon species are not homogeneously distributed in the implanted Si–C region. Figure 1 shows the concentration depth profile of carbon in Si(001) implanted with 2 MeV C^+ implantation to a dose of 1.5×10^{18} C^+ cm^{-2} by TRIM simulation. One sees that carbon is distributed in the whole implanted range, but only in a limited region is the concentration of carbon comparable to that of silicon. As the depth profile of carbon does not change much even after high-temperature annealing, β-SiC is normally formed within this layer. In the front Si layer, though the carbon concentration is far from the equiatomic,

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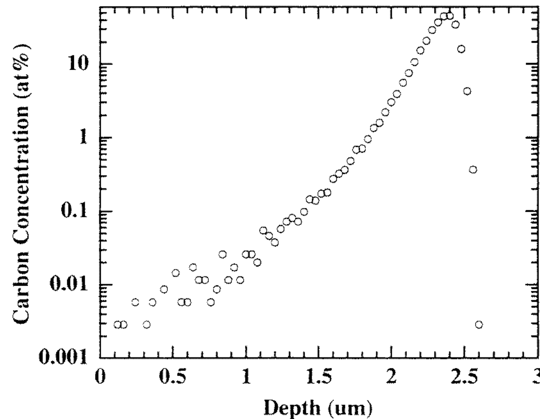


Figure 1. TRIM simulated carbon distribution in Si(001) after 2 MeV C^+ implantation to a dose of 1.5×10^{18} C^+ cm^{-2}.

it should undoubtedly have some influence on the structural recovery of Si. This, however, has not yet been reported.

In the present study, we use x-ray diffraction and x-ray pole figure measurement to monitor the growth of the β-SiC buried layer and the structural modification of the front silicon layer upon thermal annealing.

2. Experimental procedure

A silicon wafer of (100) orientation was implanted by 2 MeV carbon ions with a beam current density of about 12 μA cm^{-2} to a dose of 1.5×10^{18} C^+ cm^{-2} in a chamber with a vacuum level better than 1×10^{-7} Torr. In order to minimize the beam heating effect and thus surpress the nucleation of any crystalline SiC during implantation, the target was cooled by liquid nitrogen (LN). The temperature of the target was measured to be -90 ± 2 °C. The samples were then annealed in argon flux for 10 hours at temperatures ranging from 800 to 1200 °C to crystallize the implanted layer. After annealing, the samples were analysed by x-ray diffraction in the θ–2θ configuration with Cu Kα_1 to identify the crystal structure of the recrystallized region. The x-ray pole figure measurement was carried out with a Philips X'pert four-circle x-ray diffractometer to check the crystal quality of the SiC buried layer and the orientation relationships among the recovered front Si layer, the buried SiC layer and the bulk silicon substrate.

3. Results and discussion

Figure 2 shows the x-ray diffraction patterns of the un-implanted Si(001) substrate and the implanted sample after 10 hour annealing at 1000 °C. The diffraction patterns were collected using an x-ray diffractometer with a 2θ resolution of 0.002°. Comparing the two patterns, it is found that besides the Si(004) peak at about $2\theta = 69.1°$, there are two peaks located at about $2\theta = 41.4°$ and 33.0°, respectively. Similar diffraction patterns were also obtained for the samples annealed at 1100 and 1200 °C. The peak at about 41.4° is from the β-SiC(002) diffraction, while the peak at about 33.0° is identified to be the forbidden Si(002) diffraction,

and is designated as Si*(002). As the Si*(002) diffraction was not observed in the virgin Si(001) substrate, it is believed to result from the recovered front Si layer. For simplicity, the implanted and annealed sample can be considered as a sandwich-like structure, i.e., the re-grown front silicon layer, the buried β-SiC layer and the bulk silicon substrate.

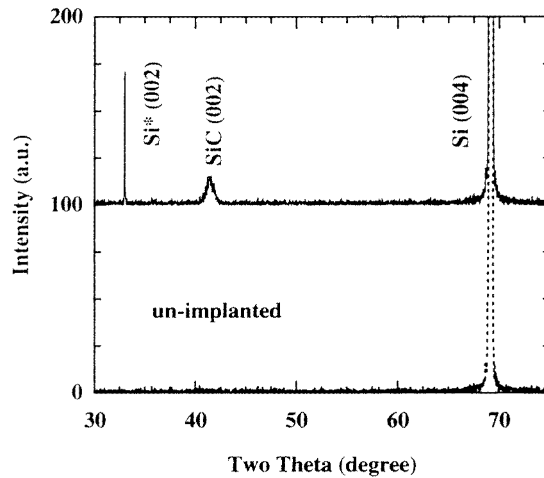


Figure 2. X-ray diffraction patterns of the un-implanted silicon substrate (lower part) and the sample annealed at 1000 °C for 10 hours (upper part).

We first focus on the buried β-SiC layer. The diffraction peak at about 41.4° in figure 2 indicates the formation of crystalline β-SiC and suggests that the buried β-SiC layer was grown epitaxially. To find out its orientation relationship with the bulk silicon substrate, x-ray pole figure measurement was carried out for the β-SiC layer and the silicon substrate. Fixing the detector at $2\theta = 35.67°$ and 28.46° respectively, the (111) pole figures of the β-SiC layer and the silicon substrate were obtained by tilting the sample from $\chi = 0°$ to 85° and doing a rotation scan from $\varphi = 0°$ to 360° with a step of 5°. Figure 3(a) and (b) shows the (111) pole figures of the β-SiC layer and the silicon substrate in the sample annealed at 1000 °C for 10 hours. It is seen that the four β-SiC{111} diffraction peaks locate correctly at the symmetrical points of {111} of the cubic structure where the angle between {111} and {001} is about $\chi = 54.74°$. In addition, the four {111} peaks are separated from each other by an azimuth rotation angle of about 90° confirming that [001] is a fourfold axis of β-SiC of a cubic structure. Comparing the (111) pole figure with that of the silicon substrate, it is found that the four {111} diffraction peaks of the β-SiC layer and the substrate differ only in their intensity, but locate at almost the same χ and φ positions. This indicates that β-SiC {111} were well aligned to that of the substrate. The (220) pole figures were also measured for both the β-SiC layer and the silicon substrate. Similarly, the {220} diffraction peaks of the β-SiC layer and the substrate differ only in their intensity, but locate at the same χ and φ positions. It is thus concluded that the buried β-SiC layer has a near-perfect orientation relationship with the silicon substrate.

We now turn to the re-growth of the front silicon layer. The diffraction peak of Si*(002) in figure 2, as was not observed in the virgin Si(001) substrate, is believed to result from the recovered Si surface layer. To find out the orientation relationships among the re-grown front silicon layer, the buried β-SiC layer and the bulk substrate, x-ray pole figure measurement was carried out for three layers. The (222) pole figure of the front Si layer,

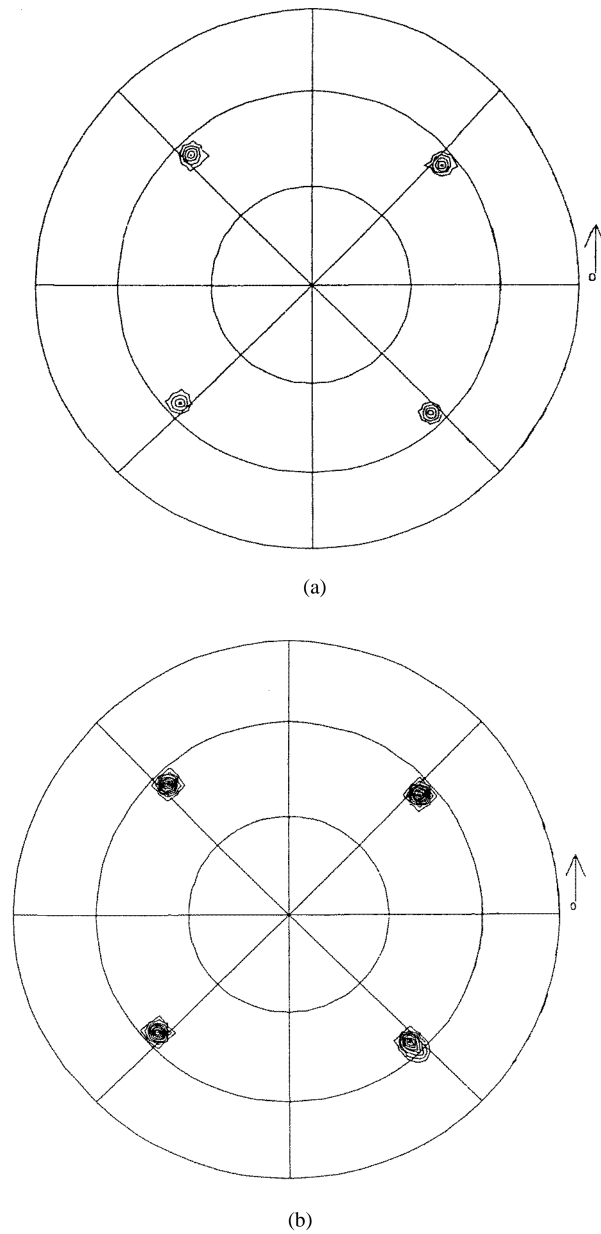
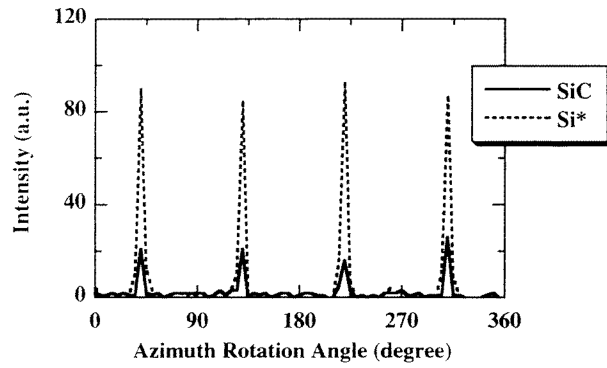
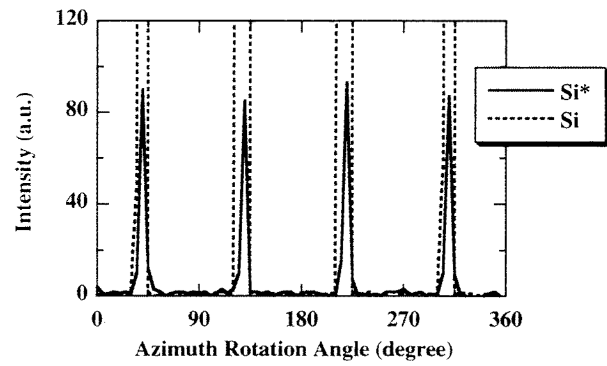


Figure 3. The (111) pole figures of (a) the β-SiC buried layer and (b) the silicon substrate in the sample annealed at 1000 °C for 10 hours.

and (111) pole figures of the β-SiC layer and the bulk silicon substrate, were obtained by a simple φ scan from 0° to 360° with a step of 1° at the exact tilt angle, i.e., $\chi = 54.74°$ for the cubic structure. It is worthwhile mentioning that the (222) diffraction is also forbidden for silicon. Figure 4(a) and (b) shows the results. It is seen that the four {111} diffraction peaks of the β-SiC layer and the silicon substrate locate at almost the same φ positions as those of the four {222} diffraction peaks of the recovered Si surface layer, though their



(a)



(b)

Figure 4. The (111) pole figures of (a) the β-SiC buried layer; and (b) the silicon substrate compared with the (222) pole figure of the recovered silicon surface layer.

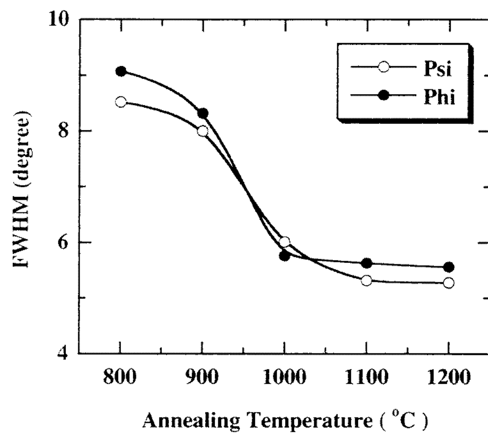


Figure 5. The FWHM values of the {222} diffraction peaks of the silicon surface layer as a function of the annealing temperature.

intensity is quite different. Therefore, the orientation relationship among the three layers is $(002)_{\mathrm{Si}^*} \parallel (001)_{\beta-\mathrm{SiC}} \parallel (001)_{\mathrm{Si}}$ and $[110]_{\mathrm{Si}^*} \parallel [110]_{\beta-\mathrm{SiC}} \parallel [110]_{\mathrm{Si}}$. In other words,

the re-grown front Si layer has also a near-perfect orientation relationship with the silicon substrate.

The recovery of the front silicon layer upon thermal annealing was investigated by checking the texture evolution, i.e. the full width at half maximum (FWHM) of the {222} peak. Figure 5 shows the FWHM of ψ and φ of the {222} diffraction peaks as a function of the annealing temperature. One sees that the FWHM of ψ and φ depends on the annealing temperature. It changed dramatically when increasing the annealing temperature from 800 to 1000 °C, i.e. from 8.52 to 6.01° for ψ and from 9.07 to 5.76° for φ. When the annealing temperature was increased from 1000 to 1200 °C, however, the FWHM for both ψ and φ changed only a little, i.e. 5.28° for ψ and 5.56° for φ. These results suggest that 1000 °C is the temperature necessary to crystallize the damaged silicon surface layer, and confirm that the structure of the front Si layer was modified after recovery, where carbon species were present.

4. Concluding remarks

An epitaxially grown β-SiC buried layer was produced in Si(001) using a combination of low-temperature MeV carbon ion implantation and high-temperature thermal annealing. Due to the presence of carbon species, the structure of the front silicon layer was modified after recovery. The orientation relationship among the front Si layer, the buried β-SiC layer and the silicon substrate was examined by x-ray pole figure measurement. The recovery of the front Si layer upon annealing was also investigated by x-ray pole figure measurement.

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