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Positron annihilation study of 4H-SiC by Ge^+ implantation and subsequent thermal annealing

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ABSTRACT

Positron annihilation in 800 keV Ge^+ implanted hexagonal SiC was studied by thermal annealing at temperatures ranging from 800 to 1400 °C. The variation in Doppler broadening *S* values as a function of the incident positron energy suggests a broad distribution in the depth of vacancy defects in the implanted samples. Increasing the annealing temperature triggers the accumulation of vacancies into vacancy clusters. After annealing at 1400 °C, defects in the deep region of SiC are eliminated, and Ge precipitation is believed to appear in the sample at the same time. Though Ge has a much more negative positron affinity than SiC, positron annihilation coincidence Doppler broadening measurement reveals that a preferential trapping of positrons in Ge seems impossible.

1. Introduction

Previously [1], we have reported that Ge nanocrystals can be formed in thermally grown SiO_2 films by ion-implantation technique, and size-dependent photoluminescence due to quantum size effects in the near-infrared region has been observed. Positron annihilation spectroscopy, an effective microstructure characterization method, was utilized to trace the structural evolution of silicon oxide films with Ge^+ implantation as a function of the annealing temperatures. The results not only provide an evaluation of the properties of the SiO_2 matrix but also an indirect diagnosis of the Ge nanocrystals.

From the viewpoint of optoelectronic device application demands, SiC satisfies more than SiO_2, considering its excellent electrical properties such as a large band gap and good ohmic contacts [2]. A promising approach for the development of light-emitting devices with SiC utilizes high-dose Ge implantation and subsequent annealing treatment [3,4]. Therefore, a fundamental understanding of the damage accumulation in SiC should undoubtedly be considered as the first step in the investigation of SiC-based device performance [5,6]. In this case, positron annihilation spectroscopy study is again appropriate for this study, as this technique is sensitive to open-volume type defects. Furthermore, with a variable-energy positron beam, the depth distribution properties of defects can also be examined.

2. Experiment

We implanted 800 keV Ge^+ ions with a dose of $2 \times 10^{16}\,cm^{-2}$ into *p*-type 4H-SiC at a temperature of 600 °C with a Tandem accelerator. The high implantation temperature was chosen to prevent amorphization of the crystals [7,8]. After implantation, isochronal annealing at temperatures ranging from 800 to 1400 °C was carried out, with each annealing taking up 30 min in Ar ambient.

After each step of the annealing treatment, positron annihilation Doppler broadening measurements were carried out with a magnetically guided variable-energy positron beam (0–30 keV). The annihilation γ-rays were recorded with an intrinsic Ge detector, and Doppler broadening spectra were characterized by the *S* parameter, which was determined as the ratio of the γ-ray counts in the range of 511.0 ± 0.7 keV to the total number of counts in the entire 511 keV peak (511.0 ± 4.2 keV).

To deepen the understanding of positron annihilation characteristics in the studied specimen, the obtained positron results were fit with the VEPFIT program [9], assuming that injected positrons stop and annihilate in the sample with a three- or four-layered structure with different defect concentrations. For comparison, the Ge^+ implantation process and vacancy generation were simulated with SRIM2008 code [10].

3. Results and discussion

The depth distribution of Ge^+ ions and vacancies generated in SiC following ion implantation are shown in Fig. 1, with the results obtained by SRIM calculation. The stopping probability of 7 keV

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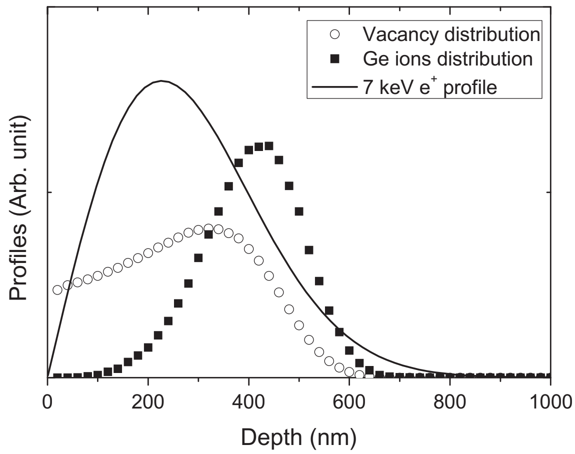

Fig. 1. Depth profile of 800 keV Ge ions and the production of vacancies, and the stopping probability of 7 keV incident positrons.

positrons in this sample is also illustrated to provide a visual comparison. The positron stopping profile was calculated by [11]

$$P(x) = 2x/x_0^2\ \mathrm{Exp}[-(x/x_0)^2] \qquad (1)$$

where $x_0 = 1.13x_{\mathrm{mean}}$ and $x_{\mathrm{mean}} = 40E^{1.6}/\rho$. ρ is the mass density of 4H-SiC in g/cm^3 unit, E is positron incident energy in keV, and x is the positron implantation depth in nm unit. The results in Fig. 1 suggest that positrons with 7-keV incident energy can be efficiently stopped in the damaged region, indicating the suitability of the positron beam for such an ion-implanted system.

Fig. 2 shows the experimentally obtained positron annihilation Doppler broadening S values as a function of the incident positron energies. Clearly, S values corresponding to 3–9 keV energies were significantly enhanced after Ge^+ implantation, and the peak value was reached at ~7 keV. In general, a reduced Doppler broadening of positron annihilation radiation corresponding to a relatively large S value is expected for an open-volume type defect, because of the reduced annihilation probability of a trapped positron with high-momentum core electrons there. Supposing that only such type of defect determines positron annihilation behavior, we can deduce that most Ge^+ implantation-generated defects should distribute around ~285 nm; that is, the mean implantation depth for 7 keV positrons. Interestingly, this coincides rather well with the SRIM simulation, which suggests a vacancy distribution peak at ~300 nm. Note that the implanted Ge mainly stopped at a relatively deeper region; that is, with a peak concentration at about 420 nm (see Fig. 1). Consequently, we infer that implantation-generated defects, but not Ge, control the final positron annihilation Doppler broadening results in as-implanted SiC.

Fig. 2 also suggests that even at higher positron energies up to 30 keV, the S values for as-implanted SiC, and also for those specimens annealed below 1400 °C, are still higher than that for the un-implanted SiC. This implies that the collision cascades accompanying Ge^+ implantation produce a high density of point defects in the SiC matrix with a broad depth distribution, extending much deeper than 650 nm as predicted by the SRIM simulation. To confirm this, we fit the experimentally obtained positron annihilation S–E data shown in Fig. 2 with the VEPFIT program. As an example, fitting results for the 1000 °C annealed sample are shown in Fig. 3. Two assumptions were tested; one divided the sample into three layers ($0 < z \leqslant 200$ nm, 200 nm $< z \leqslant 650$ nm, $z > 650$ nm), and the other into four layers ($0 < z \leqslant 200$ nm, 200 nm $< z \leqslant 650$ nm, 650 nm $< z \leqslant 1500$ nm, $z > 1500$ nm). For the former, the maximum depth of the defect distribution was set to be 650 nm, following the SRIM prediction. Above 650 nm, positrons enter a defect-free SiC bulk. Unfortunately, such an assumption failed to obtain a favorable fit at high-incident positron energies. However, a satisfactory fit of the experimental data was achieved after adding one more layer with a depth of 650–1500 nm. It should be pointed out that the positron diffusion length drawn from the VEPFIT simulation for this added layer was 20 nm, whereas for the un-implanted defect-free SiC it was 60 nm (in agreement with the reported ~40–80 nm [12] positron diffusion length for Lely-grown bulk SiC). This indicates the existence of efficient positron trapping in the deep region of this sample, even after 1000 °C heat treatment. With annealing at 1400 °C, the characteristic S values for the defect-rich region (corresponding to 3~9 keV positron energies) were significantly enhanced (see Fig. 2). This may suggest the accumulation of originally separated vacancies. The injected positrons tend to be trapped in enlarged open volumes, resulting in a further reduced Doppler broadening of annihilation radiation. In contrast, at high-incident positron energies approaching 30 keV, we observed that the S value for the 1400 °C annealed sample had recovered to that of the un-implanted SiC crystal. Certainly, this implies the reduction and even complete removal of lattice damage in the deep region of SiC.

An annealing temperature of 1400 °C is above the melting point of Ge (938 °C), and close to that of Si (1414 °C). It is also well within the commonly accepted temperature range for the recrystallization

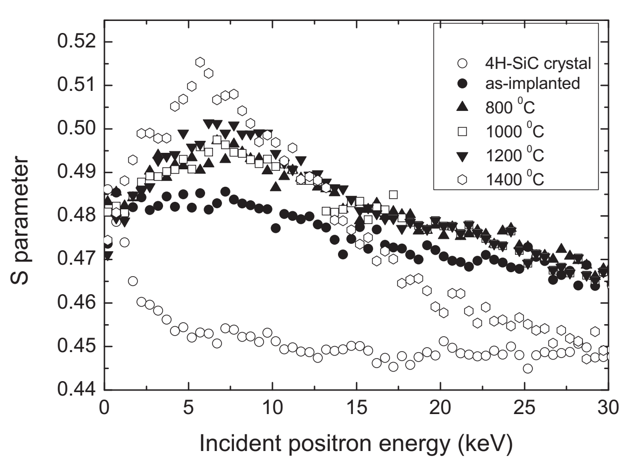

Fig. 2. Positron annihilation Doppler broadening S values as a function of the incident positron energies for 4H-SiC before and after Ge ion implantation followed by thermal annealing.

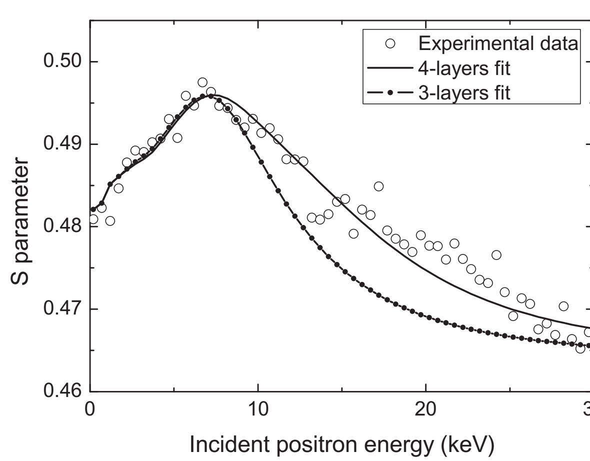

Fig. 3. VEPFIT simulation results for the 1000 °C annealed sample. Three or four layers of structural constitution were tried.

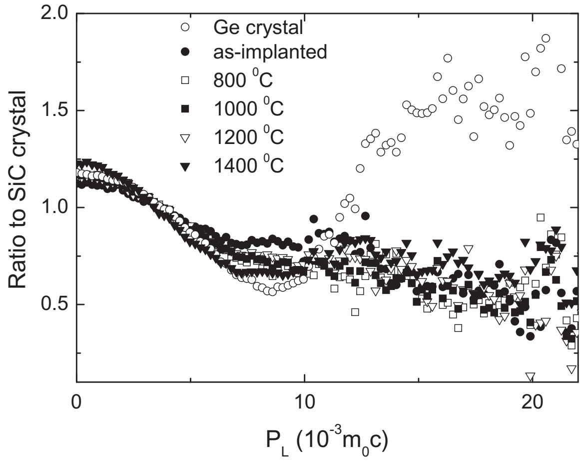

Fig. 4. Ratio curves of coincidence Doppler broadening spectra for the annealed samples relative to that of the un-implanted SiC. The result for Ge single crystal is also illustrated.

of amorphous SiC (900–1450 °C). After annealing at this temperature, it is quite possible that Ge nano-precipitates appear inside the SiC crystal [4,13]. Puska and Kuriplach et al. proposed a theory on the positron affinity calculation for elemental metals [14] and semiconductors [15]. It is believed that the positron energy level in solids becomes deeper as the positron affinity A_+ becomes more negative. Because A_+ for Ge is equal to −6.79 eV and is much less than −4.18 eV for 4H-SiC, injected positrons generally prefer to reside in Ge precipitates instead of the SiC matrix. However, there should be a prerequisite that the interface between Ge precipitates and the matrix should be coherent; for example, there should be no lattice mismatching. Our previous studies suggest that positron trapping by Ge precipitation in thermally grown silicon oxide is barely observable, though A_+ for SiO_2 is equal to −5.8 eV [16]. Unlike thermally grown silicon oxide in an amorphous state, we anticipate the existence of coherent interfaces between crystalline Ge and SiC crystal, and therefore the possibility of positron preferential trapping in Ge precipitates.

However, the ratio curves of the coincidence Doppler broadening spectra for the annealed samples relative to that of the un-implanted SiC show unexpected variations. As illustrated in Fig. 4, unlike Ge single crystal, there is absolutely no peak in the high momentum region (above $\sim 13 \times 10^{-3}\, m_0c$) of the ratio curve for the 1400 °C annealed sample. This suggests negligible positron trapping by Ge precipitates in SiC. At the same time, Fig. 4 shows that the ratios at zero momentum for all the Ge implanted samples relative to un-implanted SiC are larger than 1, which indicates the generation of vacancies due to ion implantation. Roughly speaking, these ratios increase with the rise in annealing temperature. This agrees with our postulation that the increase in the characteristic Doppler broadening *S* values for Ge-implanted SiC under high-temperature annealing is due to the accumulation of vacancies. Therefore, open-volume type defects play a dominant role in determining the positron annihilation characteristics in the studied material.

4. Summary

A positron annihilation study of Ge^+ implanted 4H-SiC crystal was conducted with a variable-energy positron beam. The results suggest that open-volume type defects, such as vacancy and vacancy clusters, are efficient positron-trapping centers not only in the as-implanted sample, but also in the high-temperature annealed samples. Upon annealing at 1400 °C, defects in the deep region of SiC start to be eliminated. Though Ge maintains a much more negative positron affinity than SiC, preferential positron trapping in Ge precipitates was not found in the current experiment.

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