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Study of the damage produced in 6H-SiC by He irradiation

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ABSTRACT

Lattice damage and evolution in 6H-SiC under He^+ ion irradiation have been investigated by the combination of Rutherford backscattering in channeling geometry (RBS/C), Raman spectroscopy, UV–visible spectroscopy and transmission electron microscopy (TEM). 6H-SiC wafers were irradiated with He ions at a fluence of 3×10^{16} He^+cm^{-2} at 600 K. Post-irradiation, the samples were annealed in vacuum at different temperatures from 873 K to 1473 K for isochronal annealing (30 min). Thermally annealed He irradiated 6H-SiC exhibited an increase in damage or reverse annealing behavior in the damage peak region. The reverse annealing effect was found due to the nucleation and growth of He bubbles. This finding was consistent with the TEM observation. The thermal annealing brought some recovery of lattice defects and therefore the intensities of Raman peaks increased and the absorption coefficient decreased with increasing annealing temperature. The intensity of Raman peak at 789 cm^{-1} as a function of annealing temperature was fitted in terms of a thermally activated process which yielded activation energy of 0.172 ± 0.003 eV.

1. Introduction

Silicon carbide (SiC) has remarkable physical, optical, and electronic properties [1,2]: it exhibits much higher blocking voltage, increased switching frequencies, lower power losses, and higher operational temperatures. SiC has thus gotten great interest in high-power, high temperature, and high-frequency applications [3,4]. In addition, SiC has a low cross-section for neutron capture and thus exhibits a low induced activity in neutron irradiation ambient. Its excellent structural, chemical and mechanical stability make SiC as an ideal structural component for working in harsh environments [5,6], i.e., in fusion reactors and cladding materials for gas-cooled fission reactors. Energetic He atoms can be formed through nuclear reactions. He atoms agglomerate and coarsen into bubbles in the He- irradiated SiC have been extensively investigated [7]. He bubbles can deteriorate structural properties by inducing crack, creep [8,9]. In addition, there are some applications of He-induced cavities in semiconductors, such as fabricating silicon-on-insulator, controlling electric behaviors. The investigation of helium ion-induced lattice damage formation and evolution under annealing in SiC is important for device fabrication and application in high-radiation environment.

Previous studies of microstructures mainly concentrated on damage created by He irradiation in 6H-SiC at room temperature [10]. Less attention has been paid to He irradiation at high temperatures [7,11]. Zhang et al. [12] studied the relation of implantation temperature and damage accumulation in Al-implanted 4H-SiC. They thought that significant dynamic recovery occurred at 450 K, because defect migration and clustering were very active when ion implantation was performed beyond 450 K. In addition, the damage accumulation on structural properties was mainly measured by transmission electron microscopy. In contrast, non-destructive spectroscopic methods, such as Raman and UV–visible spectroscopy, gave simple and accurate information on defect formation and structural transformations [13].

In the present work, we have investigated the damage production and annealing behavior in He irradiated 6H-SiC at 600 K by RBS/C, Raman, UV–visible transmittance and TEM.

2. Experimental details

In the present experiment, 6H-SiC wafers (research standard, n-type, oriented <0001>Si surface with 0.3 mm thickness, one side was polished) were supplied by the Cree Research Inc. The 6H-SiC wafers were irradiated with He^+ with 100 keV at a fluence of 3×10^{16} He^+/cm^2 with a current density of about 0.8 $\mu A/cm^2$. The sample holder was kept at 600 K during He irradiation. In order to minimize channeling effects, wafers were 7–8° tilted from the

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normal incidence. After He irradiation, wafers were isochronally annealed in a tube furnace at temperatures of 873 K, 1073 K, 1273 K and 1473 K, respectively, for 30 min in vacuum condition ($<1 \times 10^{-3}$ Pa).

The irradiated as well as un-irradiated samples were analyzed by Rutherford backscattering spectrometry in channeling geometry using 2.022 MeV He^+ ions and a backscattering angle of 165°. The RBS/C measurements were carried out at the State Key Laboratory of Nuclear Physics and Technology, Peking University. The Raman spectrometry was carried out with a JY-HR800 spectrometer, in the backscattering configuration. In the experiment, the Raman spectra were obtained by using the 514.5 nm line of the Ar^+ laser as excitation source. The exciting wavelength should be able to penetrate the entire thickness of the 6H-SiC wafer. The UV–visible transmittance was performed using a Lambda 900 (Perkin Elmer Inc.). Cross-sectional microstructures were characterized with Hitachi H-700 TEM operating at 200 keV andJEOL 3010 operating at 300 keV. High resolution TEM microstructures in the damaged region were observed by HVEM operating at 1250 kV in Hokkaido University. Cross-sectional samples for the TEM observation were prepared by standard mechanical polishing. When the thickness of the samples reached about 40 μm, ion milling with a Gatan-691 was performed. Ion milling was operated at room temperature under a pressure lower than 1×10^{-3} Pa. The energy of Ar ions was 4 keV and incident ion angle was ±5°. All the experiment measurements were performed at room temperature.

3. Results and discussion

3.1. Rutherford backscattering-channeling spectrometry

The RBS-channeling technique gives a unique insight of the depth distribution of defects produced in the near surface layer of the samples. The RBS spectra were carried out along the <0001 > direction of the He irradiated samples. Fig. 1 shows the channeled spectra of the backscattered ions from the samples [14]. In comparison, the random and channeled spectra of the un-irradiated sample were also presented. Because Rutherford backscattering spectrometry along channels is sensitive to very small atomic displacements from the crystalline lattice sites and the main interaction is with interstitial-like defects, the emergence of a damage peak in the channeling spectra indicates the presence of interstitials-like defects in the lattice. The evaluation of the damage

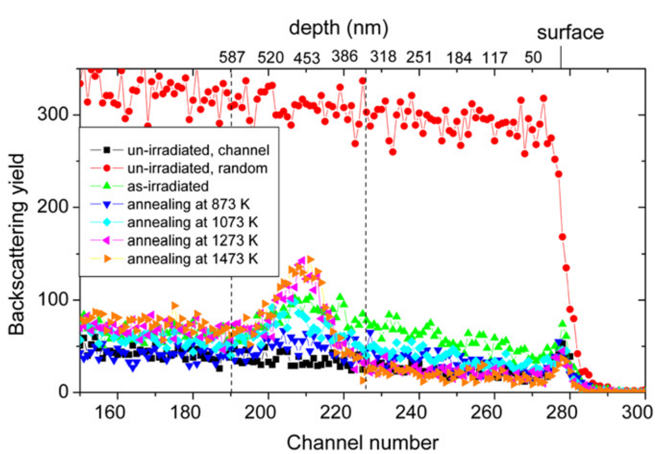

Fig. 1. Aligned RBS spectra for the 6H-SiC samples under 100 keV He irradiation to a fluence of 3×10^{16} He^+cm^{-2} followed by isochronal annealing at 873 K, 1073 K, 1273 K and 1473 K. Random and channeling spectra from an un-irradiated sample were also included. The solid lines are guide for the eyes.

profile is more precise if one uses the Si signal due to the overlap of the surface C signal with the Si bulk signal and the lower backscattering cross section of C. As shown in Fig. 1, the channel spectrum of the near surface was lower than the random spectrum when the sample was irradiated with 3×10^{16} He^+cm^{-2}.According to the simulation of He irradiation in SiC with 3×10^{16} He^+cm^{-2} using the stopping and range of ions in solids (SRIM) 2008 code [15], the damage peak is over 1.0 dpa (displacement per atom). It can easily form amorphization (0.3–0.4 dpa) when the He irradiation is performed at room temperature [16], RBS/C displays here that dynamic annealing is significant at 600 K, which is consistent with the report of Zhang et al. [11].

In order to study on the evolution of He irradiation-induced lattice damage, the derived depth distribution of the relative damage in the silicon sub-lattice was also given in Fig. 1. The depth distribution of the relative damage could be divided into two regions. One region was the layer extending from the surface to about 345 nm and the relative damage in the silicon sub-lattice decreased with increasing annealing temperature, indicating the recovery of lattice damage under annealing. The finding was consistent with the results of Raman spectroscopy and UV–visible transmittance spectra (see Fig. 2 and Fig. 3). The other region was the layer range 345 nm–587 nm, corresponding to the maximum He-induced lattice damage shown in the interval of two broken lines. The depth of the damage peak coincides with the result of cross-sectional transmission electron microscopy (XTEM), as shown in Fig. 4(a). Furthermore, the intensity of the damage peak decreased with annealing temperatures up to 873 K, indicating that the annealing process was controlled by defect recombination. Upon annealing temperatures above 873 K, the lattice disorder increased with increasing annealing temperature, indicating that microstructural evolution changed with annealing temperature. The XTEM investigation of microstructures in helium-irradiated 6H-SiC was performed, as discussed later.

3.2. Raman scattering using 514.5 nm excitation

The frequencies of the folded transversal and longitudinal phonon modes for the acoustic (FTA) and optical (FTO) branches of the dispersion curves of 6H-SiC have been investigated by Nakashima and Tahara [17]. In contrast with the sample irradiated at room temperature, the Raman spectra shapes of He^+ hot irradiation at 600 K before annealing did not present the strong broadening peaks located near 500 cm^{-1}, 800 cm^{-1} and 1400 cm^{-1}(not shown). The Raman results demonstrated that there was no amorphous layer in the He irradiated sample, consistent with the result of RBS/C. Moreover, the effect of annealing temperature on the Raman scattering was investigated, as shown in Fig. 2(a). Three sharp lines were observed at 767 cm^{-1}, 789 cm^{-1} and 967 cm^{-1} in the wave number range 650 cm^{-1} to 1150 cm^{-1}. The 767 cm^{-1} and 789 cm^{-1} lines are assigned to E_2 (TO) vibration modes, while the 967 cm^{-1} line is assigned to A_1 (LO) vibration mode. The energy positions of these features in the Raman spectra were obtained by fitting Lorentzian type line profiles to the experimental data. The spectrum of E_2 (TO) phonon at 789 cm^{-1} monitored the crystalline quality of the sample, as shown in Fig. 2(a). The E_2 linewidth of the He irradiated 6H-SiC decreased with increasing annealing temperature except after annealing at 873 K due to the large deviation between Lorentzian profile and the experimental result in the as-irradiated sample. In addition, the E_2 intensity of the He irradiated 6H-SiC increased with annealing temperature. The integrated intensity A of E_2 was normalized to the value A_{cryst} of the crystalline material, i.e., $A_{norm} = A/A_{cryst}$. The value of A_{norm} increased exponentially with temperature except after annealing at 1473 K. The data were fitted by a function of an Ahrrenius-type giving activation energy of