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A HRXRD and nano-indentation study on Ne-implanted 6H–SiC ☆

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ABSTRACT

Specimens of 6H–SiC single crystal were irradiated at room temperature with 2.3 MeV neon ions to three successively increasing fluences of 2×10^{14}, 1.1×10^{15} and 3.8×10^{15} ions/cm^2 and then annealed at room temperature, 500, 700 and 1000 °C, respectively. The strain in the specimens was investigated with a high resolution XRD spectrometer with an ω-2θ scanning. And the mechanical properties were investigated with the nano-indentation in the continuous stiffness measurement (CSM) mode with a diamond Berkovich indenter. The XRD curves of specimens after irradiation show the diffraction peaks arising at lower angles aside of the main Bragg peak Θ_{Bragg}, indicating that a positive strain is produced in the implanted layer. In the as-implanted specimens, the strain increases with the increase of the ion fluence or energy deposition. Recovery of the strain occurs on subsequent thermal annealing treatment and two stages of defects evolution process are displayed. An interpretation of defects migration, annihilation and evolution is given to explain the strain variations of the specimens after annealing. The nano-indentation measurements show that the hardness in as-implanted specimens first increases with the increase of the ion fluence, and a degradation of hardness occurs when the ion fluence exceeds a threshold. On the subsequent annealing, the hardness variations are regarded to be a combined effect of the covalent bonding and the pinning effect of defect clusters.

1. Introduction

Due to its superior high-temperature strength, high thermal conductivity, chemical inertness and small neutron capture cross section, silicon carbide (SiC) is suitable for use as structural components in nuclear fusion reactors, or as an encapsulating material for nuclear fuel in light water reactors and gas-cooled fission reactors, and also in radioactive nuclear waste disposals. The accumulation of lattice damage and nuclear transmutation products (H, He, heavier inert gas atoms, etc.) due to irradiation and their impact on mechanical properties has been a serious concern for the application of silicon carbide in these environments [1–3]. A better understanding of the behavior of evolution of defects and mechanical properties during or after irradiation is thus of vital importance.

Previous studies on inert gas atoms in silicon carbide were mainly focused on microstructural evolution after helium implantation [4–10], but fewer efforts were paid on effects of implantation with heavier inert gas ions. Compared with helium implantation, more displacements of lattice atoms can be produced in one collision cascade by an incident heavier inert-gas ion. Moreover, energies for the migration, clustering of heavier inert gas atoms and their interaction with defects should be different from those of helium atoms, and can results in different behavior from that of helium atoms in materials. In the present study, changes of microstructures and mechanical properties after energetic Ne-ion implantation and subsequent thermal annealing treatment are investigated, and underlying mechanisms are discussed.

2. Experimental

The specimens used in this study were *n*-type $(0001)_{Si}$ single-crystal 6H–SiC wafer (commercial standard) supplied by Cree Research Inc. The irradiation with neon ions ($^{20}Ne^{8+}$ with a kinetic energy of 2.3 MeV) was performed at room temperature at the 320 kV high-voltage Platform equipped with an ECR (Electron Cyclotron Resonance) ion source in Institute of Modern Physics, CAS, Lanzhou. Three successively increasing fluences of 2×10^{14}, 1.1×10^{15} and 3.8×10^{15} Ne ions/cm^2 were chosen, corresponding to the peak damage levels of 0.05, 0.28 and 0.95 dpa (displacement per atom) at a depth of 1500 nm and to the peak Ne atomic concentrations of 90, 480 and 1650 appm at a depth of 1620 nm according to Monte-Carlo code SRIM 2006 [11] (a specific gravity of 3.21 g/cm^3 and threshold displacement

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energies of 20 and 35 eV for C and Si sub-lattices [6–8], respectively, were adopted). During the irradiation the normal of the samples were tilted from the incident direction by 7° to reduce the channeling implantation. A low beam-current of 4.1 μA was used to limit additional beam heating during implantation. After the irradiation, each specimen was cut into four pieces, and three of them were then isochronally annealed at 500, 700 and 1000 °C, respectively, in a vacuum of 1×10^{-3} Pa for 60 min.

High-resolution XRD measurements were carried out in the Bragg (reflection) geometry on a four-circle diffractometer with the pure Cu Kα1 line of wavelength λ = 1.5405 Å of D8 Discovery of BRUKER. An ω-2θ scanning mode was used near the (00012) Bragg reflection plane with a resolution of 0.005°. A Ge (220) double-crystal monochromatic was chosen to ensure the resolution.

Nano-indentation measurements were carried out using a diamond Berkovich indenter (triangular based pyramid) in CSM mode of a Nano-Indenter G200 produced by MTS Inc. The maximum penetration depth was about 1.7 μm and the load was kept for 10 s at the maximum depth. The contact area between indenter and sample surface is $A(h_c) = 24.56h_c^2$, where h_c is the indenter penetration depth. Each specimen was tested for six times at different points and the results presented in this paper were their average values. Because of the deviation from ideal shape of diamond indenter and the surface effect of the specimens, the data from surface to 150 nm were discarded.

The strain magnitude of specimens after irradiation can be calculated from the formula:

$$\varepsilon = \frac{c_i - c_0}{c_0} \tag{1}$$

where the c_0 and c_i are the lattice parameter along c axis of the un-irradiated and the irradiated SiC, respectively. For the un-irradiated SiC, c_0 = 15.117 Å. The lattice parameter of c_i of the hexagonal crystal system can be obtained from the formula:

$$c_i = \frac{\lambda}{2\sin\theta}\sqrt{\frac{4}{3(a_i/c_i)^2}(h^2 + hk + k^2) + l^2} \tag{2}$$

where the h, k, l are the crystal plane indices $(h, k, -(h+k), l)$ of hexagonal system. The parameters of a_i and θ are the lattice parameter along a axis and diffraction angle.

3. Results and discussion

3.1. The HRXRD measurement

The XRD curves of the un-implanted and the as-implanted SiC specimens to fluences of 2×10^{14}, 1.1×10^{15} and 3.8×10^{15} ions/cm^2 are shown in Fig. 1. Even at the lowest ion fluence, peaks of diffraction intensity arising at lower angles aside the main Bragg peak Θ_{Bragg} $(-(\Delta d/d)_N = 0)$ are observed, and are attributed to damaged layers of the crystals, where the main Bragg peak Θ_{Bragg} results from the crystal underneath the perturbed layer. The position of the peaks at lower angle side was used to quantify the irradiation-induced elastic strain. Their positions suggest a dilatation of lattice parameter occurring along the direction perpendicular to the specimen surface. A similar dilatation of lattice was observed in previous experiments of SiC irradiated under different conditions [7–9]. On the other hand, the peak beside the main Bragg peak Θ_{Bragg}, attributing to the damage in the near surface region [12], moves towards the lower angle side with the increase of ion fluence, indicating that the strain increases gradually with the increase of ion fluence. The strains (estimated with Eq. (1)) and the total nuclear collision energy losses in the near surface region (from an estimation with SRIM 2006) are given in Fig. 2, corresponding to ion fluences of 2×10^{14}, 1.1×10^{15} and 3.8×10^{15} ions/cm^2, respectively. It is noticed that the strain increases with the increase of the nuclear collision energy depositions.

More over, as displayed in Fig. 1, the peak corresponding to the peak damage region (dpa peak) can be observed only in the specimen irradiated to the lowest ion fluence of 2×10^{14} ions/cm^2, while tails of diffuse scattering were observed in the specimens irradiated to the higher ion fluences of 1.1 and 3.8×10^{15} ions/cm^2. The absence of diffraction peaks corresponding to the peak damaged region in these two specimens can be ascribed to the diffuse scattering from the highly defective region rather than the diffraction [8,13]. Strain can therefore not be estimated precisely in this case. We noticed that the present results are different from the XRD results from SiC helium-implanted at room temperature [9], where a peak of intensity corresponding to the peak damage region can be clearly seen in a specimen implanted with 160 keV He ions to a fluence of 1×10^{16} ions/cm^2 which deposited a total nuclear collision energy loss close to that in the specimen of the mediate fluence (1.1×10^{15} Ne ions/cm^2) in the present study. An interpretation for the difference is that more defects were produced and survived under the irradiation with heavier ions (Ne), possibly due to more complex nuclear collision cascades [3], or due to proposed more effective stabilization of defect clusters by heavier inert gas atoms [14]. The peak damaged region in the spec-

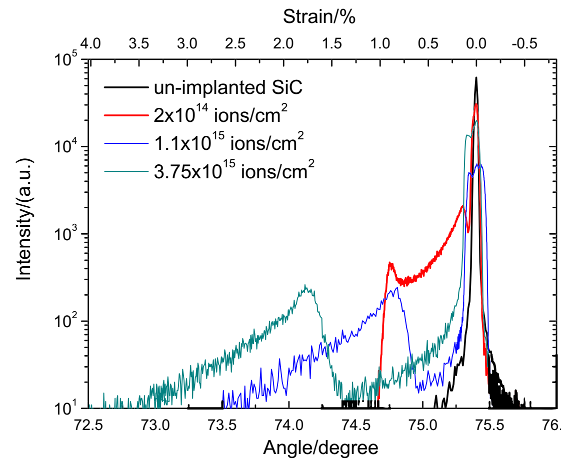

Fig. 1. Experimental ω-2θ scanning on the (00012) Bragg planes of un-implanted SiC and Ne-implanted SiC to fluences of 2×10^{14}, 1.1×10^{15} and 3.8×10^{15} ions/cm^2, respectively. The quantity on the top-axis indicates the elastic-strain level.

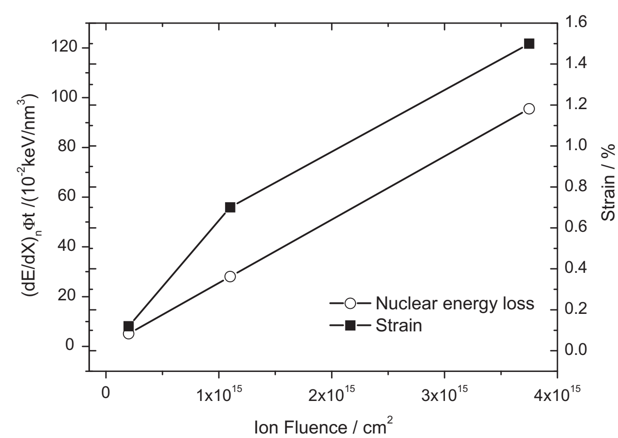

Fig. 2. The strain and the total nuclear energy deposition in the near surface region as a function of the ion fluence.