

Study on microstructure and mechanical properties of He and H ion irradiated 6H-SiC



Q. Bai^a, L. Li^b, F.F. Cheng^c, R. Bin^d, T. Fa^d, E. Fu^a, S.D. Yao^{a,*}

^a State Key Laboratory of Nuclear Physics and Technology, School of Physics, Peking University, Beijing 100871, China

^b Department of Physics and Electronics, School of Science, Beijing University of Chemical Technology, Beijing 100029, China

^c Key Lab of Beam Technology and Material Modification of Ministry of Education, College of Nuclear Science and Technology, Beijing Normal University, Beijing 100875, China

^d Chinese Academy of Engineering Physics, Mianyang 621900, China

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ABSTRACT

6H-SiC single crystal wafers were irradiated by 200 keV He ions, 100 keV H ions, and 200 keV He ions followed by 100 keV H ions, with the radiation fluences in the range of 10^{16} ions cm^{-2} , respectively. After ion irradiation, the samples were annealed at 1273 K in N_2 atmosphere. XRD, RBS/C, and nano-indentation measurements were carried out to evaluate the damage of the irradiated samples. XRD results show that new diffraction peaks from radiation damage appear at lower angles next to the main diffraction peaks, which indicates that radiation process caused the increase of lattice parameter in the damage region. RBS/C results show that complete amorphization does not occur even under 6×10^{16} cm^{-2} He ions followed by 8×10^{16} cm^{-2} H ions irradiation indicated by $\chi_{\text{min}} = 17.63\%$. Damage produced by He ion irradiation at a fluence of 6×10^{16} ions cm^{-2} is greater than that produced by 3×10^{16} cm^{-2} He ions and followed by 4×10^{16} cm^{-2} H ions irradiation, which implied that He ions irradiation effect plays a dominant role in the process of damage producing. The nano-indentation measurements show that hardness variation depends on the irradiation fluences: high fluence radiation leads to the decrease of the hardness while low fluence radiation leads to the increase of the hardness. After annealing, the hardness of all the irradiated samples increases. Possible mechanisms are discussed for explaining these phenomena.

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1. Introduction

Silicon carbide (SiC) has been investigated as potential fusion structural material for many years because of its excellent properties such as microstructural and chemical stability, high efficiency of thermal conductivity, low activation and high-temperature strength [1–3]. In fusion reactors, He and H ions are produced by 14.1 MeV neutron irradiation-induced transmutations and they will simultaneously induce the radiation damage in SiC [4]. Radiation damage induced by single-ion irradiation in SiC has been studied extensively. Katoh et al. [5] reviewed irradiation effects in SiC for fission and fusion applications. According to the review, helium has strong interaction with vacancies and this will change the development of both vacancies and interstitial clusters. Jiang et al. [6] studied the lattice disorder and recovery of 6H-SiC irradiated with 2.0 MeV Au ions and concluded that high-energy heavy-ion beam induced recovery is similar on both the Si and C

sublattices in 6H-SiC. Leclerc et al. [7] studied the swelling of 4H-SiC under irradiation with 50 keV He ions and found that both elastic strain and He bubbles contribute to the swelling. Tromas et al. [8] studied the change of mechanical properties of 4H-SiC implanted with 50 keV He ions and found that amorphization decreases the hardness of irradiated samples. The effects of many other kinds of ions, such as C ion, Fe ion, Ne ion, As ion and Cs ion irradiation on SiC materials have been studied as well [9–13]. Compared with the extensive study of single-ion irradiation, the study for dual-ion irradiation is limited. Under dual-ion irradiation, defects produced by different ions will interact with each other and thus the effect of the dual-beam ion irradiation will be different with the effect of single-ion irradiation. Taguchi et al. [14] studied the synergistic effects of the implanted helium and hydrogen in SiC composites and found that H might have an effect to prevent the migration of He and inhibit the growth of He bubbles. Although synergistic effects has been known by researchers, the influences of the transmutation products He and H simultaneous irradiation on the properties such as microstructure and mechanical properties are not well understood [5].

* Corresponding author.

E-mail address: sdiao@pku.edu.cn (S.D. Yao).

In our study, 6H-SiC was irradiated by 200 keV He ions and followed by 100 keV H ions. Irradiation with only 200 keV He ions and only 100 keV H ions was also performed to study the different effects of single-ion irradiation and dual-ion irradiation. After irradiation, thermal annealing treatment at 1273 K was performed on the as-irradiated 6H-SiC samples. The purpose of our research is to study the changes of microstructure and mechanical properties of 6H-SiC under irradiation with He ions, H ions and He followed by H ions and the damage recovery after annealing. Synergistic effects of the He and H ions and the role of individual ion in determining the change of the properties are discussed.

2. Experimental

6H-SiC single crystal wafers with the size of 1 cm × 1 cm were divided into four groups. The irradiation on the samples includes 200 keV He ions, 100 keV H ions, and 200 keV He ions followed by 100 keV H ions at room temperature, respectively. The irradiation fluences was chosen in the range of 3×10^{16} – 6×10^{16} cm⁻² for He ions and 4×10^{16} – 8×10^{16} cm⁻² for H ions. In order to reduce channeling effects, the direction of the incident beam was set to 7° away from the normal direction of the sample surface. The irradiation fluences for each group are shown in Table 1. The projected range R_p and straggling range ΔR_p predicted by SRIM 2008 code [15] are 697.4 nm and 81.4 nm for He ions and 583.6 nm and 52.6 nm for H ions, respectively. The threshold displacement energies of 35 eV and 22 eV for Si and C [16] was applied in the SRIM calculations. After irradiation, each sample was cut into two parts, and one part was annealed at 1273 K in N₂ atmosphere for one hour.

X-ray diffraction (XRD) measurements were carried out on the Beijing synchrotron radiation facility 1W1A system with the wavelength $\lambda = 1.548$ Å. The θ - 2θ scanning mode was used near (0006) reflection plane. Rutherford backscattering-channeling spectrometry (RBS/C) measurements were performed on the 2×1.7 MV tandem accelerator at Peking University using a 2.023 MeV He⁺ collimating beam. An Au-Si barrier detector was fixed at the backscattering angle of 165° to record the backscattered ions. Nano-indentation measurements were carried out on Nano Indenter G200 in CSM mode using Berkovich indenter. The maximum penetration depth was about 1000 nm. Five different points were chosen for each sample in the test in order to get an average hardness value.

3. Results and discussion

3.1. X-ray diffraction

XRD spectra of virgin and irradiated 6H-SiC are shown in Fig. 1(a). The diffraction intensity was plotted in logarithmic scale. It's found that satellite peak appears due to ion irradiation at lower angle side next to the main Bragg peak ($\theta_{\text{Bragg}} = 35.804^\circ$) in the irradiated samples. The main Bragg peak comes from the coherent diffraction of the X-ray reflected by the original SiC (0006) lattice plane, so the difference between the main Bragg peak position and satellite peak position is used to determine the damage caused by

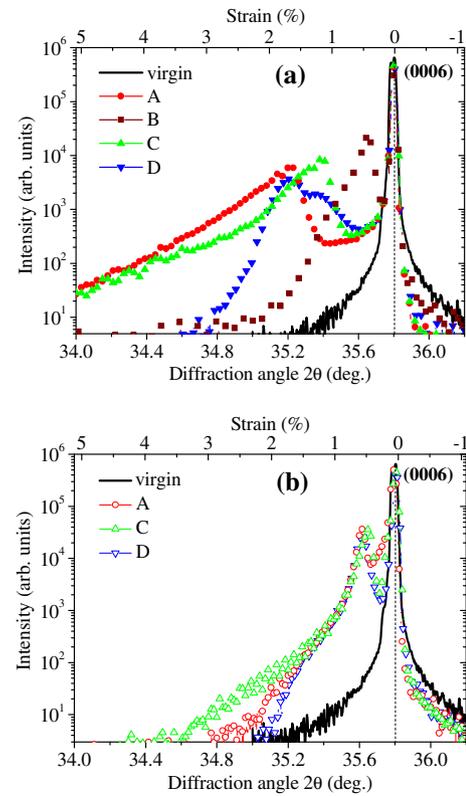


Fig. 1. XRD θ - 2θ scan spectra of virgin and irradiated 6H-SiC in (0006) plane (a) before annealing and (b) after annealing. The radiation fluence: A: 6×10^{16} He ions cm⁻²; B: 8×10^{16} H ions cm⁻²; C: 3×10^{16} He ions cm⁻², 4×10^{16} H ions cm⁻²; D: 6×10^{16} He ions cm⁻², 8×10^{16} H ions cm⁻².

ion irradiation. Based on Bragg's law, the low angle position of the satellite peak indicates that the ion irradiation process caused the dilatation of the original SiC lattice along the direction perpendicular to the sample surface. This effect has been reported in previous studies [7,11,17]. These satellite damage peaks reflect the variation of radiation-induced elastic strain (ε) which can be used to quantify the degree of the lattice deformation. The mean value of the elastic strain in the near surface region can be deduced from the position of the satellite peak. According to the Bragg equation (Eq. (1)) and the lattice plane spacing equation (Eq. (2)) for hexagonal crystal system

$$d_{hkl} = \frac{\lambda}{2 \sin \theta} \quad (1)$$

$$d_{hkl} = \frac{1}{\sqrt{\frac{3}{4} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}}} \quad (2)$$

where h, k, l represent the crystal plan indices. We obtain the actual value of lattice parameter c_{exp} from Eq. (2). The mean value of elastic strain

$$\varepsilon = \frac{c_{\text{exp}} - c_0}{c_0} \quad (3)$$

where c_0 is the lattice parameter of virgin 6H-SiC.

The comparison of the mean value of elastic strain shows that the sample irradiated by H ions at 8×10^{16} cm⁻² (sample B) has the minimum mean value of elastic strain about 0.42% in the near surface region. The mean value of elastic strain in the sample irradiated by He ions at 3×10^{16} cm⁻² and followed by H ions at 4×10^{16} cm⁻² (sample C), is about 1.15% and in the sample irradiated only by He ions at 6×10^{16} cm⁻² (sample A), is about 1.65%.

Table 1
The radiation fluence for each group of sample.

Group No.	He ions fluence/cm ²	H ions fluence/cm ²
A	6e16	0
B	0	8e16
C	3e16	4e16
D	6e16	8e16

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