Nuclear Instruments and Methods in Physics Research B 326 (2014) 345–350

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

Evolution of defects in silicon carbide implanted with helium ions

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ARTICLE INFO

Article history:
Received 26 June 2013
Received in revised form 11 September 2013
Accepted 2 October 2013
Available online 15 January 2014

Keywords:
Silicon carbide
Helium
Implantation
Defect
Radiation damage

ABSTRACT

Effects of accumulation of radiation damage in silicon carbide are important concerns for the use of silicon carbide in advanced nuclear energy systems. In the present work lattice damage in silicon carbide crystal (4H type) implanted with 100 keV $^4He^+$ ions was investigated with Rutherford backscattering spectrometry in channeling geometry (RBS/c) and positron beam Doppler broadening spectrometry (PBDB). Helium implantation was performed at the specimen temperature of 510 K to avoid amorphization of the SiC crystal. Fluences of helium ions were selected to be in the range from 1×10^{16} to 3×10^{16} ions cm^{-2}, around the dose threshold for the formation of observable helium bubbles under transmission electron microscopes (TEM).

The RBS/c measurements show distinctly different annealing behavior of displaced Si atoms at doses below or above the threshold for helium bubble formation. The RBS/c yield in the peak damage region of the specimen implanted to 3×10^{16} He-ions cm^{-2} shows an increase on the subsequently thermal annealing above 873 K, which is readily ascribed to the extra displacement of Si atoms due to helium bubble growth. The RBS/c yield in the specimen implanted to a lower ion fluence of 1.5×10^{16} He-ions cm^{-2} decreases monotonously on annealing from ambient temperatures up to 1273 K. The PBDB measurements supply evidence of clustering of vacancies at temperatures from 510 to 1173 K, and dissociation of vacancy clusters above 1273 K. The similarity of annealing behavior in PBDB profiles for helium implantation to 1×10^{16} and 3×10^{16} ions cm^{-2} is ascribed to the saturation of trapping of positrons in vacancy type defects in the damaged layers in the specimens helium-implanted to the two dose levels.

1. Introduction

Due to its superior high-temperature strength, high chemical inertness and low long-term radioactivity, silicon carbide (SiC) is considered for application as structural components in nuclear energy engineering or as electronics in harsh environments [1,2], where the buildup of lattice damage and nuclear transmutation product helium in silicon carbide is a serious concern [3–5]. In the past decades a number of studies concerning diffusion and thermal desorption of helium, formation of gas bubbles and dimensional change of helium-doped SiC were carried out by several groups, where helium was introduced in the material by neutron irradiation (with B-doping) or by direct helium implantation [6–17]. One of interests from the practical point of view is the rather high resistance of fibers and nano-crystals of silicon carbide to irradiation [16,17]. A rather high dose threshold of formation of nano-metric helium bubbles at near-surface region was indicated in helium-implanted SiC crystal investigated with transmission electron microscopy (TEM) in our previous study [18,19].

In the present work, we investigated the defect evolution in helium-implanted silicon carbide around the dose threshold of helium bubble formation, by using Rutherford Backscattering Spectrometry in channeling geometry (RBS/c) and positron beam Doppler broadening spectrometry (PBDB). RBS/c technique was proven to be an efficient tool to investigate interstitial-type defects [13], distortion and deformation in a crystal. A quantitative analysis of defect concentrations in crystals irradiated with energetic ions was made possible by combing RBS/c and a Monte Carlo simulation [20,21]. While positron annihilation spectrometry (PAS) is capable of detecting mono-vacancy, di-vacancy or simple vacancy clusters in high sensitivity [22]. By using slow positron beam technique, information of depth distribution of defects in the near surface region can be obtained.

2. Experimental

Specimens cut from a 4H-SiC wafer (research standard, n-type, (0001)-oriented, 0.2 mm in thickness, supplied by Cree Research Inc.) were used in the study. The specimens were implanted with

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http://dx.doi.org/10.1016/j.nimb.2013.10.064

100 keV $^4He^+$ ions in an ion implanter to three successively increasing fluences of 1.0×10^{16}, 1.5×10^{16} and 3.0×10^{16} ions cm^{-2}, which correspond to peak damage levels of 0.3, 0.45 and 0.9 dpa (displacement per atom), and to peak helium concentrations of 0.7, 1.1 and 2.2 at.%, respectively, according to the Monte-Carlo code SRIM-2012 [23]. The highest helium concentration is slightly above the threshold for helium bubble formation, while the other two doses are lower than the dose threshold, as proposed by our previous TEM observation [18]. During helium implantation, the specimen temperature was kept at about 510 K to avoid amorphization of the crystal due to damage accumulation [4]. The incident helium ion beam was along a direction 7° off the normal of the specimens so as to minimize channeling implantation.

The specimens for RBS/c analysis (helium-implanted to 1.5×10^{16} and 3.0×10^{16} ions cm^{-2}) were subsequently cut into smaller pieces (3 × 3 mm in dimensions), and some of them were thermally annealed at temperatures in the range from 773 to 1273 K, respectively, for 30 min in a vacuum of 2×10^{-5} Pa. The as-implanted and the annealed specimens were analyzed with a probe beam (2 MeV $^4He^+$) in an experimental terminal of a tandem electrostatic accelerator in Peking University. A non-implanted 4H-SiC specimen was also tested for reference. A three-axis goniometer was used to control the orientation of the specimens. The probe beam was incident along ⟨0001⟩ axis of the specimens while the backscattered He ions were detected at an angle of 165° from the ion incident direction with a surface-barrier Si detector. A standard specimen of Au/Y/Co multilayered thin films grown on a silicon wafer was used to calibrate the multichannel analyzer, which was used to acquire backscattering spectra in both random and channeling geometry from each specimen. An ion charge of 30 μC was accumulated during the recording of each spectrum.

After the RBS measurements, some of the tested specimens were used to prepare cross-sectional TEM samples by using a small cleavage technique [24]. The samples were investigated with a transmission electron microscope of JEOL 2010F.

Specimens for positron annihilation Doppler broadening spectra measurement (helium-implanted to 1.0×10^{16} and 3.0×10^{16} ions cm^{-2}, with dimensions of 15 × 15 mm) were tested in the ^{22}Na slow positron beam line located in the Key Laboratory of Nuclear Analysis Technology, Institute of High-energy Physics in Beijing. The incident energy of the positron beam is variable from 0.1 to 30 keV. Details of the facility were described in a previous paper [25]. The specimens either in as-implanted state or in annealed states (at temperatures successively increasing from 773 to 1273 K, 30 min for each temperature) were tested. A non-implanted 4H-SiC specimen was also tested for reference. The shape parameter ($S(E)$) and wing parameter ($W(E)$) that describe the shape of the 511 keV annihilation line were calculated. These parameters are dependent on the width of the gamma ray peak due to annihilation of positrons with electrons [26]. The central region (S parameter) and the wing (W parameter) of the spectrum were defined as $|511 - E_\gamma| \leq 0.44$ keV and $2.5 \leq |511 - E_\gamma| \leq 4.0$ keV.

3. Results and discussion

3.1. RBS/channeling measurement

The RBS/channeling technique supplies a unique insight of the depth distribution of lattice damage in the near surface region of crystals. The measured energy spectra of backscattered $^4He^+$ ions from specimens helium-implanted to two fluences (1.5×10^{16} and 3.0×10^{16} ions cm^{-2}) are shown in Fig. 1 (a) and (b), respectively. Energy spectra in both random and channeling geometry from a non-implanted specimen are given for reference. There is a remarkable increase in the backscattering yield corresponding to the damage peak in Si sub-lattice in the as-implanted specimens. The backscattering yield changes significantly on subsequent thermal annealing treatment, and shows different temperature dependence for the two dose levels, which is addressed in detail in the follows.

Depth profiles of relative disorder in Si sub-lattice from the decoding of the RBS/c spectra are given in Fig. 2. The depth information was derived by using the electronic stopping power data supplied by SRIM-2012 code, while the relative disorder was taken as the proportion $[Y - Y_c]/[Y_r - Y_c]$ (where Y is the backscattering yield in channeling geometry from helium-implanted specimens, while Y_r and Y_c are the backscattering yield in random and channeling geometry, respectively, from the non-implanted specimen). Depth profiles of estimated atomic displacement damage (in dpa) and helium concentration (in at.%) from SRIM-2012 code are given in Fig. 2 (a). The annealing behavior of relative disorder in Si sub-lattice is found to be significantly different for the two helium ion fluences. For lower ion fluence (Fig. 2 (b)), the relative disorder in Si sub-lattice decreases rapidly on annealing at 773 K, and reaches a saturation level about 1/6 of the as-implanted state on annealing at higher temperatures up to 1273 K, indicating that the defect evolution is dominated by the recombination process of Frenkel pairs produced by helium implantation, except that a low concentration of complex defects were finally survived and

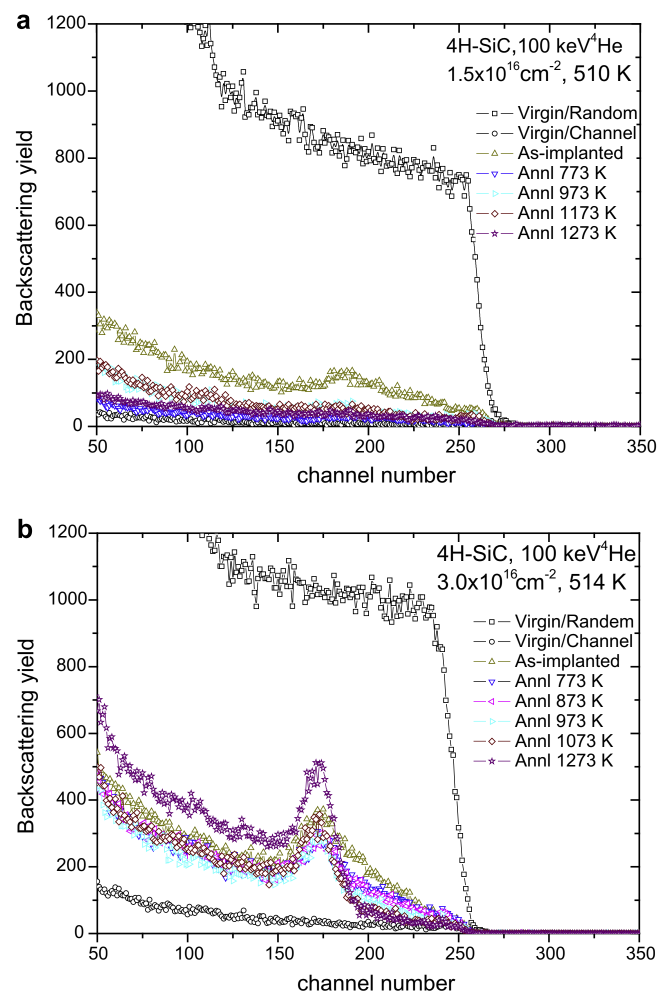

Fig. 1. Energy spectra of backscattered ions from RBS/channeling with 2.0 MeV $^4He^+$ probe beam, corresponding to helium implantation to (a) 1.5×10^{16} ions cm^{-2} and (b) 3.0×10^{16} ions cm^{-2} in 4H-SiC specimens with 100 keV $^4He^+$ ions at about 510 K. Non-implanted SiC specimens were used for reference.