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Irradiation-induced microstructural evolution and swelling of 3C-SiC

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ABSTRACT

In this study, an ion-irradiated single crystal 3C-SiC under fluences of up to 20 dpa at 400–1350 °C was examined using synchrotron based X-ray diffraction and high resolution transmission electron microscopy. Interstitial clusters, dislocation loops, Frank loops, stacking fault loops, and voids in 3C-SiC were investigated. The high resolution TEM results show that clusters collapsed to {111} small loops when their size reached few nm with increasing temperature, and gradually develop into Frank loops with an added atomic layer along {111} at 1000 °C.

Interplanar spacing information of single crystal SiC was obtained from synchrotron XRD radial scan measurements. Irradiation-induced volume swelling at 400–1350 °C was measured, and the anisotropic ($a = b < c$) swelling behavior of SiC was confirmed. In addition, humps on the right side of SiC (002) were observed, which suggested that C^{+}/Si^{+}-Si⟨100⟩ and/or C^{+}/Si^{+}-C⟨100⟩ dumbbells gave rise to diffuse scattering.

1. Introduction

Cubic silicon carbide (3C-SiC) is a promising structural and cladding material, and is used in fusion reactors and advanced fission reactors due to its attractive mechanical and thermal properties [1]. Even though several studies have reported the irradiation-induced microstructural evolution [2–5] and swelling of SiC [6–9], the contribution of defects to the swelling of irradiated 3C-SiC is less understood. If left unchecked, the swelling resulting from voids or point defects may lead to unwanted degradation of the mechanical properties of 3C-SiC [10]. In addition, 3C-SiC exhibits excellent electrical properties, which make it an ideal candidate for use in electronic devices [11]. However, 3C-SiC-based electronic devices have not achieved their expected performance so far, mainly because of defects formed during the manufacturing process [12]. Therefore, in order to use SiC in electronic devices, a fundamental understanding of the swelling and microstructural evolution of this material is crucial.

In previously published literature [13], various types of defects (e.g. black spots, dislocation loops, Frank loops, and voids) observed under different irradiation temperatures in irradiated 3C-SiC were shown, and the size and density of the defects were quantified. Although the types of irradiation-induced defects present in different temperature regimes were roughly classified, studies on the precise microstructural configuration, which provide a better understanding of the evolution of radiation damage in 3C-SiC, have been limited. Furthermore, irradiation-induced swelling, which is related to microstructural evolution, is a principal phenomenon that determines the behavior of damaged structural materials. At temperatures where vacancies were sufficiently mobile ($T/T_m > 0.35$), the swelling of SiC resulted from vacancy accumulation in the voids [14] or bubbles [15]. However, swelling behavior was also observed at lower irradiation temperatures, where no voids were observed in transmission electron microscope (TEM) images. It is generally accepted that this low-temperature swelling regime (about 250–1000 °C) is the result of point defects (interstitials and vacancies) and/or point defect clusters [16]. Therefore, the main type of defects that cause the swelling effect are different at temperatures below and above 1000 °C.

Modern synchrotron X-ray sources with high intensity and small spot sizes are capable of distinguishing details of the primary damage state, such as small defects in irradiated materials that are too small to be seen by TEM. In a previous study conducted by our research group [17], we developed experimental techniques to characterize irradiation-induced volume swelling. In this work, we have presented a better visualization of the irradiation-induced microstructures, and have accurately determined the swelling of

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ion-irradiated SiC under a high dose (~20 dpa) with temperatures ranging from 400 °C to 1350 °C. The purpose of this paper is to confirm the configuration of different defects and their contribution to the swelling of irradiated 3C-SiC. Based on the results obtained, we have proposed a possible mechanism for the radiation induced swelling in SiC.

2. Material and methods

2.1. Materials

The material used for this work was a single crystal 3C-SiC wafer (by NOVA SiC, France). A 3C-SiC crystal with surface orientation (002) was grown using the CVD process on a Si substrate. The thickness of the SiC epitaxy layer and Si substrate was 1.17 and 450 μm, respectively. Twins and stacking faults formed in the 3C-SiC layers grown on silicon were mainly due to the large lattice mismatch and the difference in the thermal expansion coefficients between Si and SiC. Owing to the tremendous difference in size between irradiation-induced defects (<20 nm) and pre-existing defects (>200 nm), they were easily distinguishable in the TEM images.

2.2. Irradiation conditions

Ion beam irradiation was performed at the DuET facility at Kyoto University, Japan. 5.1 MeV Si^{2+} ion with a fluence of 5.65×10^{17} ion/cm^2 was implanted for inducing displacement damage at irradiation temperatures of 400, 600, 800, 1000, 1200, and 1350 °C. In Fig. 1, the depth distribution versus displacement per atom of incident Si ion was simulated by SRIM code simulation, and the displacement energies for Si and C were set at 35 eV and 21 eV, respectively. For examination of the microstructure, the displacement damage level, calculated at a depth of 0.6 μm from the irradiated surface, was 20 dpa, and the damage rate was about 2.7×10^{-3} dpa/s averaged over the damage range.

2.3. Microstructural investigation

Thin foil specimens were prepared for TEM analysis by mechanical polishing and 3–5 keV Ar ion-milling methods. The microstructures of the irradiated region were characterized using a JEOL-2010F 200 keV TEM and JEOL-ARM200F 200 keV spherical aberration corrected scanning TEM. The number density of the defects in the specimen were quantified base on the thickness estimated by electron energy loss spectroscopy (EELS) log ratio method. The size of the defect was directly measured from the TEM images. The defect density was calculated by counting the number of defects in selected areas of the TEM images. The thickness of the selected area was estimated by using the intensity of zero loss and plasma loss in the EELS spectra. Furthermore, high-resolution TEM (HRTEM) images were post-processed using a HREM filter in Gatan Digital Micrograph software to de-noise and improve images.

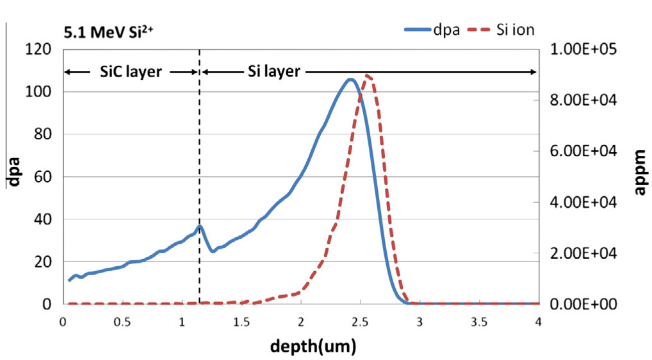

Fig. 1. Depth profile for the atomic displacement damage profile.

2.4. Synchrotron radiation based X-ray diffraction

Single crystal specimens were cut into 5 × 5 mm pieces and measured with a synchrotron source with a wiggler beamline BL-17B1 in the National Synchrotron Radiation Research Center (NSRRC), Taiwan. The photon energy was 8 keV with a flux estimated to be 10^{11} photons/s and a spot size of 0.5 × 0.5 mm. The surface orientation of different crystals was determined using an eight-circle diffractometer, which enabled the measurement of the radial intensity scan (θ–2θ scan) along different SiC ($h\,k\,l$) planes. Diffraction peaks were fitted with a nonlinear least squares fit of a Lorentzian convolved with a slit on a quadratic background. Peak centroids were determined to a precision of ±0.002°.

3. Results and discussion

3.1. Microstructural changes in irradiated 3C-SiC

Fig. 2 shows electron micrographs on the evolution of defects with elevated temperatures. The bright field TEM images in Fig. 2 were all taken along the ⟨1 1 0⟩ incident beam direction of 3C-SiC. In Fig. 1(a), only small features were observed, as indicated by the dark contrast. These observable microstructures are often described as black spot defects, and are most likely to be clusters of interstitial atoms (Fig. 3(a)). A high-resolution image of the same specimen is shown in Fig. 3(b). The darker region shown in the HRTEM images may be the result of the overlap of atoms on the primitive site and interstitial atoms, which cluster in various configurations (e.g. tetrahedral interstitials, antisite defects, and dumbbells). These experimental results are in agreement with images simulated by Gao and Weber [18].

In the specimens irradiated under 20 dpa at 600 °C (Fig. 2(b)) and 800 °C (Fig. 2(c)), a variety of defects including black spots and small loops were observed. HRTEM images of the same specimens in Fig. 2(b) and (c) are presented in Fig. 3(c) and (e), respectively. It is likely that interstitial clusters (black spots defects) change into small loops with increasing temperature. In Fig. 3(d), the size of dark region is more than 10 nm. Generally, in covalent ceramics, such a large cluster should not be stable in the temperature. Magnified images of the defects under 20 dpa at 600 °C (Fig. 2(d)) and 800 °C (Fig. 2(f)) show that clusters collapsed to {1 1 1} loops when their size reached few nm with increasing temperature. Therefore, the dark region surrounding loops should be the strain caused by the arrangement of stacking sequence (formation of a loop). The large dark region may also allow us to assume the loops were not the "coherent" Frank loops. Furthermore, C and Si interstitials migrate to the ideal atom site in 3C-SiC and prefer to agglomerate on the {1 1 1} plane under high irradiation temperatures. The irradiation times were almost identical for the specimens in the study, hence, the interstitials may migrate even faster or farther at higher temperatures.

In the HRTEM images, each bright spot corresponds to the signal for the Si and C columns due to the limited point to point resolution. In order to de-noise and improve the HRTEM images, they were post-processed by proper image processing, Fast Fourier transform, Fourier filter, and inversed Fast Fourier transform with the HREM filter in the Gatan Digital Micrograph software. With an irradiation temperature of up to 1000 °C, the defects formed were mostly {1 1 1} planar defects, as shown in Fig. 2(d). A Frank interstitial loop is shown in Fig. 4(c), with a ...ABC|B|ABC... stacking