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Microstructural defects in He-irradiated polycrystalline α-SiC at 1000 °C

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HIGHLIGHTS

- The microstructural defects in He-irradiated polycrystalline α-SiC at 1000°C were investigated.
- The effects of grain boundaries and stacking faults on platelets and dislocation loops were discussed.

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ABSTRACT

In order to investigate the effect of the high-temperature irradiation on microstructural evolutions of the polycrystalline SiC, an ion irradiation at 1000 °C with the 500 keV He^{2+} was imposed to the α-SiC. The platelets, He bubbles, dislocation loops, and particularly, their interaction with the stacking fault and grain boundaries were focused on and characterized by the cross-sectional transmission electron microscopy (XTEM). The platelets expectably exhibit a dominant plane of (0001), while planes of (01−10) and (10−16) are also found. Inside the platelet, the over-pressurized bubbles exist and remarkably cause a strong-strain zone surrounding the platelet. The disparate roles between the grain boundaries and stacking faults in interacting with the bubbles and loops are found. The results are compared with the previous weighty findings and discussed.

1. Introduction

SiC/SiC composites are important materials for the application in the development of nuclear energy and waste technologies, due to its advanced physical and chemical properties [1,2]. In the nuclear power surroundings, the (n,α) nuclear transmutation reaction is inevitable, and the dense energetic atomic He can be produced in SiC. Because of the very low solubility of He atoms in SiC, it is a general phenomenon that He atoms would be easily trapped by vacancies to form helium-vacancy clusters, which can continually coalesce into bubbles under high temperatures [3–5]. He bubbles can cause material swelling, embrittlement, and the deleterious thermal conductivity [6,7]. Therefore, it is worth investigating the nucleation and growth of the He bubbles in He-irradiated SiC/SiC composites.

The phenomena of He-irradiated polycrystalline SiC, such as α-SiC and β-SiC, have been extensively investigated. Chen et al. investigated microstructural evolution of helium-implanted α-SiC at approximately room temperature (RT) followed by annealing ranging from 850 to 1850 °C [8]. Helium bubbles are formed in grain boundaries and the most features of the microstructure are lenticular cavities (platelets), which transform to disk-shaped arrangements of bubbles with associated dislocation loops upon high temperature annealing. In our recent study, He-irradiated polycrystalline α-SiC at room temperature followed by annealing at 1000 °C, spherical bubbles are homogenously distributed in the damage region [9]. The formation of bubbles in grain boundaries is related to the He concentration. Bubbles associated with dislocation loops can be observed. Vincent et al. investigated thermal evolution of He bubbles in He-irradiated polycrystalline SiC [10]. Grain boundaries can influence the He bubble nucleation and distribution in grain interiors. Kondo et al. investigated synergistic effects of Si and He irradiation in β-SiC at 1400 °C, and observed that the small spherical cavities homogeneously distributed in grain interiors [11], while some large cavities were formed in grain

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boundaries.

Most of the reported investigations are concerned with the thermal annealing effect on the He-irradiated SiC at RT or low temperatures. There is a lack of systematic study for the microstructural evolution in the He-irradiated polycrystalline α-SiC at high temperatures. The objective of this study is to understand the microstructural evolution of He-irradiated polycrystalline α-SiC at an elevated temperature, 1000 °C.

2. Experimental procedures

In the present study, samples were hot-pressed polycrystalline α-SiC supplied by the saint-gobain company, with a density of 3.1 g/cm^3 and grain sizes of 4–10 μm. Polycrystalline α- SiC contained approximately 90% 6H-SiC and 10% 4H-SiC. Polycrystalline α-SiC wafers were irradiated with 500 keV He^{2+} ions with a fluence of 2×10^{16} cm^{-2} at 1000 °C and the current density was approximately 0.8 μA/cm^2. He-beam was scanned horizontally and vertically for lateral homogeneity. The simulated profiles of the damage and helium deposition of polycrystalline α-SiC irradiated with 500 keV He^{2+} ions with a fluence of 2×10^{16} cm^{-2} superposed a cross-sectional TEM (XTEM) view of He-irradiated sample, as shown in Fig. 1. The peak values of displacement per atom (dpa) and He concentration were approximately 0.5 dpa and 1.26%, respectively, estimated via the Monte-Carlo code Stopping and Range of Ions in Matter (SRIM-2008) quick cascade simulations using displacement threshold energies of 20 and 35 eV for C and Si sublattices [12], respectively. It can be seen the formation of a buried damage layer at depths ranging from 1200 to 1400 nm from the surface, consistent with the location of the damage profile simulated by SRIM-2008. The irradiation was performed at the 320 kV Multi-disciplinary Research Platform for Highly Charged Ions of the Institute of Modern Physics, Chinese Academy of Sciences.

The microstructural evolution of the He-induced damage was studied via cross-sectional transmission electron microscopy (XTEM) using a FEI Tecnai G20 operated at 200 kV. Using both conventional TEM and high resolution TEM (HRTEM) the lattice defect distribution has been observed. XTEM samples were prepared by Hitachi (2000) FIB (focused ion beam) system. Before FIB process, the irradiated surface was protected with deposited a tungsten layer. The foil thickness was measured by convergent beam electron diffraction (CBED) method. The size and number density of the observed dislocation loops were measured by Nano Measurer software.

3. Results

The distribution of He-irradiation-induced bubbles was investigated by XTEM and the results are given in Fig. 2. The microstructures inside the grain (about 1 μm away from the grain boundary) are exhibited in Fig. 2a. The significant microstructural features in the damaged layer are the disc-shaped defects (platelets) and the linear-arranged small bubbles. The platelets are of nano-sized bubble clusters which are composed of the bigger ones located in the middle and the smaller ones located in the periphery, consistent with our previous report [9]. The platelets have a dominant habit plane of (0001) and minority planes of (10-10) and (10–16). These platelets have a constant thickness of about 1 nm, while the diameter varies from a few nanometers to over 100 nm. The shape of the platelets is close to a two-dimensional oblate form rather than a three-dimensional structure.

Remarkably, the platelets are surrounded by the strong strain contrasts. In the bright field image, these strong strain conditions should be caused by the over-pressurized bubbles inside the platelets. In sharp contrast to the dense bubbles in the grain interior, no bubble is observed in the stacking faults as shown in Fig. 2b, and particularly, a bubble denuded zone in width of about 500 nm is remarkably formed along the grain boundary. The bubble-free zone should be related with the He trapping effect of the boundaries. It is known that the grain boundaries can effectively trap the He atoms during the irradiation at the elevated temperature. As most of the He atoms near the grain boundaries are attracted and consume in the boundaries, barren zones of He could be formed. However, there should be some specific boundaries which have relatively weak ability to absorb or trap the He atoms and are not preferential to form He bubbles. The boundary marked in Fig. 2c and detailed in Fig. 2d, which is nearly perpendicular to the irradiated surface, can be an evidence of the inactivated boundaries for trapping the He. In this boundary, the He bubbles have a much lower density and a quite smaller, even compared with the bubbles in the interiors.

The structure of the over-pressurized platelet is detailed in Fig. 3. The bright filed image of the chosen platelet and the high-resolution image are presented in Fig. 3a and b, respectively. In

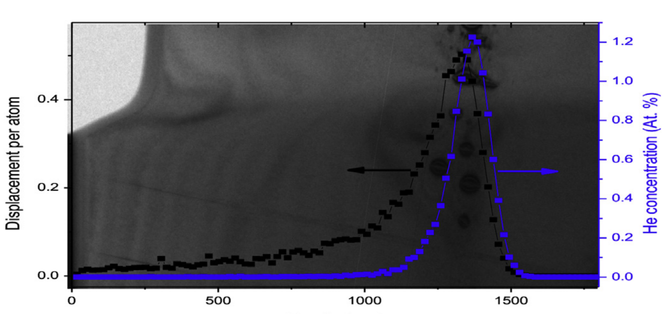

Fig. 1. XTEM bright field micrograph of polycrystalline α-SiC irradiated 500 keV He^{2+} ions to a fluence of 2×10^{16} cm^{-2} at 1000 °C, superposed the distributions of damage and helium deposition of the He irradiated polycrystalline α-SiC simulated by SRIM-2008.

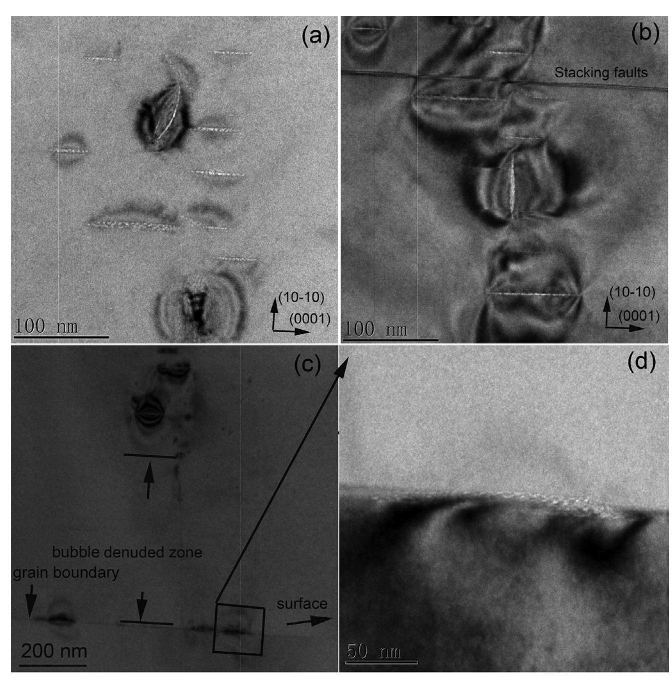

Fig. 2. Under-focused XTEM bright field micrographs of bubble formation in grain interiors (a) and near stacking faults (b), in grain boundaries (c) and (d) of the He-irradiated polycrystalline α-SiC at 1000 °C.