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Effect of displacement damage up to 50 dpa on microstructural development in SiC/SiC composites

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Abstract

The effect of displacement damage up to 50 dpa on microstructural development in SiC/SiC composites with Hi-Nicalon Type S SiC fiber was investigated. He bubbles were observed in the matrix of SiC/SiC composites irradiated to more than 10 dpa in ion irradiations at 1000 °C. The average size of He bubbles increased with increasing displacement damage. Almost all the He bubbles were formed at the grain boundaries in the matrix irradiated to 10 dpa. On the other hand, He bubbles were also formed within the grains of the matrix irradiated to 50 dpa. The matrix irradiated to 50 dpa in a fusion reactor relevant condition had the largest average size of He bubbles. The average size of He bubbles in the matrix irradiated to 50 dpa decreased with increasing the amount of implanted H to more than a fusion reactor relevant condition. He bubbles were formed in fibers irradiated to more than 10 dpa.

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1. Introduction

Continuous silicon carbide (SiC) fiber reinforced SiC matrix (SiC/SiC) composites are known to be attractive candidate materials for first wall and blanket components in fusion reactors due to their high temperature strength and low-residual radioactivity to neutron irradiation [1–4]. In the fusion environment, helium (He) and hydrogen (H) are produced as transmutation products and He bubbles can be formed in the SiC by irradiation of 14-MeV neutrons [5,6]. Authors have previously reported the synergistic effect of He and H as transmutation products on swelling behavior and microstructural development in the temperature range of 800 to 1300 °C at doses up to 10 dpa [7–9]. The previous works indicated that He bubbles were formed by the synergistic effect of displacement damage, He and H atoms, and that the amount of implanted H affects the average size and number density of He bubbles.

The SiC/SiC composites for fusion reactors may be irradiated to more than 50 dpa at the end of life [10,11]. However, the effect of He and H atoms as

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transmutation products on the formation and growth of He bubbles in SiC/SiC composites irradiated to more than 50 dpa has not been investigated. In this study, the effect of displacement damage up to 50 dpa on microstructural development in SiC/SiC composites was investigated.

2. Experimental procedure

2.1. Materials

The SiC/SiC composites used in this study have 2D plain-weave fabrics of Hi-Nicalon Type S SiC fiber fabrics as reinforcement. The SiC/SiC composites were fabricated using the forced thermal gradient chemical vapor infiltration (F-CVI) process at Oak Ridge National Laboratory. The details of the fabrication procedure are described elsewhere [12,13]. A carbon layer was deposited on the SiC fiber as an interphase layer between the matrix and the fiber. The matrix formed by this process consists of a high purity, faulted form of β-SiC. Such composites typically have approximately 15% porosity. However, on the scale important for the microstructural studies presented here, the matrix can be considered fully dense and stoichiometric.

2.2. Irradiation

Simultaneous ion-beam irradiations were carried out at the TIARA (Takasaki Ion Accelerators for Advanced Radiation Application) facility of JAEA. The specimens were irradiated at 1000 °C by 6.0 MeV Si^{2+} ions, 1.0 MeV He^{+} ions and/or 340 keV H^{+} ions. Various combinations of ions were examined in this study as summarized in Table 1. The irradiation conditions in this study are almost consistent with the conditions in a fusion reactor [3,5]. The implantation of He^{+} and H^{+} ions was conducted using an aluminum foil energy degrader in order to control He and H distribution in the depth range of about 1.0–1.8 μm from the specimen surface. The displacement damage, He, H and Si concentration as a function of depth from the surface in SiC calculated by the SRIM code [14] is shown in Fig. 1. The displacement threshold energies of Si and C were assumed to be 35 and 20 eV, respectively [15]. The irradiation was performed up to 50 dpa at the depth of 1.4 μm as shown in Fig. 1. The resultant He/dpa and H/dpa ratios were 130 and 40 appm/dpa, respectively, which would correspond to a region of the first wall of a fusion power reactor.

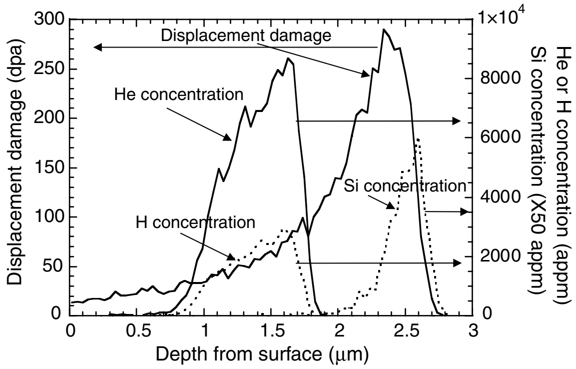

Fig. 1. Displacement damage, He, H and Si concentration as a function of depth from the surface in SiC calculated by SRIM code.

2.3. Microstructure observation

Focused ion-beam processing was used to prepare thin foil specimens for transmission electron microscopy (TEM) observation. Microstructure observation was conducted with TEM (JEOL 2000 FX) operated at 200 kV.

3. Results and discussion

The low-magnification TEM microphotograph of the SiC/SiC composite irradiated by simultaneous triple ion-beams at 1000 °C is shown in Fig. 2. In earlier work, the surface of the SiC fiber and matrix irradiated to 10 dpa at 1300 °C had no step [8]. The surface of the SiC fiber and matrix irradiated to 50 dpa at 1000 °C in this study, however, had step

Table 1
Irradiation condition of simultaneous ion-beams

Condition ID	Kind of ions	Displacement damage (dpa)	He concentration (appm)	H concentration (appm)
Dual	$Si^{2+} + He^{+}$	10 or 50	1300 or 6500	0
Triple	$Si^{2+} + He^{+} + H^{+}$	10 or 50	1300 or 6500	400 or 2000
Triple(H × 10)	$Si^{2+} + He^{+} + H^{+}$	10 or 50	1300 or 6500	4000 or 20000

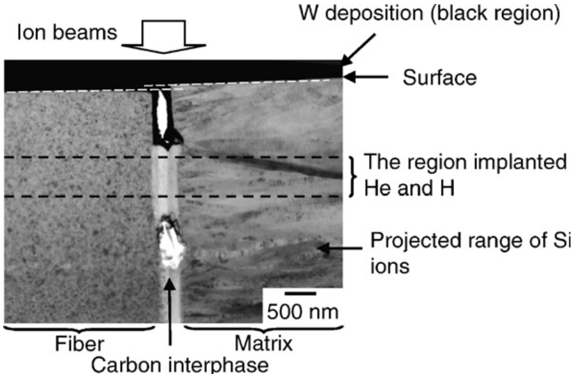

Fig. 2. Low-magnification TEM microphotograph of the SiC/SiC composite irradiated to 50 dpa by simultaneous triple ion-beams at 1000 °C. The W deposition (black region) was used to shield the surface of SiC/SiC composites against Ga ion irradiation during focused ion-beam processing.

height. The surface of the matrix was slightly higher than that of the SiC fiber. The SiC fibers consist of fine SiC grains, carbon phase and small pores, while the matrix consists of only dense SiC grains. The number of grain boundaries in the SiC fibers was also larger than that in the SiC matrix because the grain size of SiC fibers was smaller than that of the SiC matrix. More of the large number of defects induced by ion-beams irradiation were trapped in the grain boundary, the carbon phase and the small pore of the SiC fiber than in the SiC matrix. Therefore, the swelling rate of the matrix and the SiC fiber may be slightly different. The carbon interphase disappeared in higher than 100 dpa irradiation region since it was damaged due to high displacement damage by simultaneous ion-beam irradiation. On the other hand, the fiber/matrix interphase debonding, which leads to rapid degradation of strength and thermal conductivity, did not occur for any combinations of ion-beams irradiation up to 50 dpa. This result indicated that the SiC/SiC composites using Hi-Nicalon Type S appear to have excellent dimensional stability under simultaneous ion-beam irradiations up to 50 dpa at 1000 °C.

The cross-sectional TEM microphotographs of the SiC matrices irradiated to 10 and 50 dpa at about 1.4 μm depth from the surface are given in Fig. 3. He bubbles are observed in the matrices of SiC/SiC composites irradiated to more than 10 dpa at 1000 °C. Almost all the He bubbles were formed at the grain boundaries in the matrix irradiated to 10 dpa. On the other hand, He bubbles were also formed in the grains of matrices irradiated to 50 dpa. The effect of displacement damage on the average size of He bubbles is shown in Fig. 4. The average size of He bubbles increased with increasing displacement damage. The reason is that the amounts of vacancies, He and H increased with increasing displacement damage. The average size of He bubbles at the grain boundaries in the matrix irradiated under the Dual irradiation condition was almost saturated at 10 dpa. On the other hand, under the Triple irradiation condition, the average size of He bubbles in the matrices was almost directly proportional to displacement damage. Hojou et al. reported that a large amount of implanted H is contained in the form of H

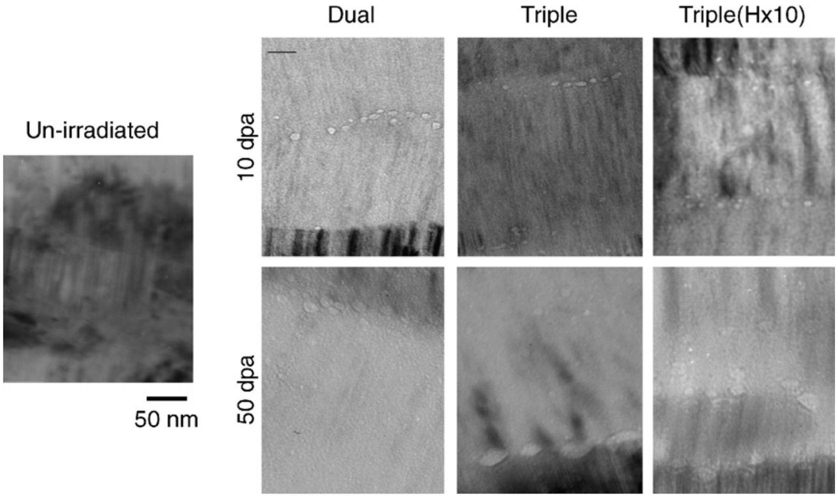

Fig. 3. Cross-sectional TEM microphotographs of the SiC matrices irradiated to 10 and 50 dpa at about 1.4 μm depth from the surface with different irradiation types.

molecules and C–H compound in bubbles [16]. The size of He bubbles increased with increasing pressure in the bubbles. These results reveal that the existence of H accelerates the growth of He bubbles. Fig. 5 shows the effect of the amount of implanted H on the average size of He bubbles in the matrix irradiated to 10 and 50 dpa. The average sizes of He bubbles in the matrices irradiated to 10 dpa under the Dual and Triple irradiation conditions were almost the same and they were larger than that in the matrix irradiated under the Triple(H × 10) irradiation condition. On the other hand, the matrix irradiated to 50 dpa by the Triple irradiation condition (a fusion reactor relevant condition) had the largest average size of He bubbles. Also, the average size of He bubbles decreased as the amount of implanted H increased to more than a fusion reactor relevant condition. As mentioned above, the availability of H accelerates the growth of He bubbles. However, at 10 dpa, the existence of H hardly accelerated the growth of He bubbles because the amount of implanted H was very small (approximately 400 appm) under the Triple irradiation condition. At 50 dpa, the average size of He bubbles increased because of the existence of sufficient H in the matrix. In the case of H oversupply, the implanted H might inhibit the recombination of He clusters and the growth of He bubbles since the irradiation temperature of 1000 °C was enough for the implanted H to diffuse in SiC and release from SiC [17].

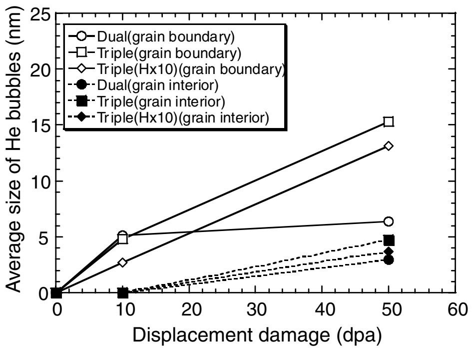

Fig. 4. Effect of displacement damage on average size of He bubbles in the matrices irradiated up to 50 dpa at 1000 °C.

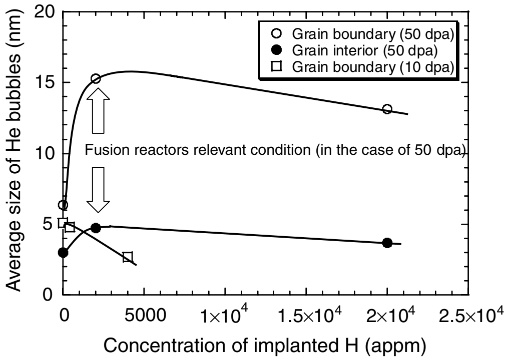

Fig. 5. Effect of the amount of implanted H on the average size of He bubbles in the matrices irradiated to 50 dpa.

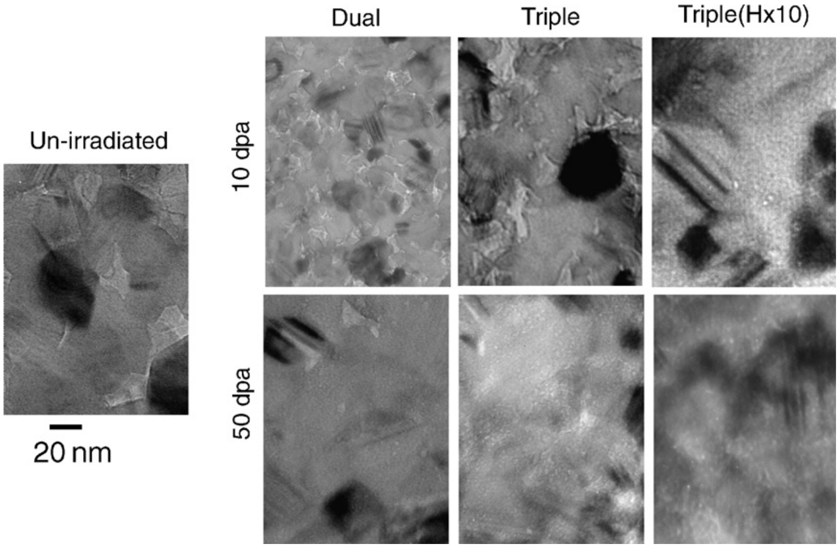

Fig. 6. Cross-sectional TEM microphotographs of the SiC fibers irradiated to 10 and 50 dpa at about 1.4 μm depth from the surface with different irradiation types.

The cross-sectional TEM microphotographs of the SiC fiber irradiated to 10 and 50 dpa at about 1.4 μm depth from the surface are given in Fig. 6. At displacement damage of 10 dpa, no He bubbles were observed in the fiber irradiated under the Dual irradiation condition. On the other hand, He bubbles were observed in the fiber irradiated to 50 dpa under the Dual irradiation condition. He bubbles were formed in the fiber irradiated to more than 10 dpa under the Triple irradiation condition. There are pores and carbon phases among the SiC grains in the SiC fibers, in which He interstitials and vacancies induced by ion-beams irradiation were trapped. However, since the amount of defects such as He interstitials and vacancies increased with increasing displacement damage, not all defects were trapped in the pores and carbon phases. The defects that were not trapped could combine together and form He bubbles. The average size of He bubbles in SiC fibers irradiated to 10 dpa was almost the same as that of the fibers irradiated to 50 dpa. On the other hand, the number density of He bubbles in SiC fibers irradiated to 50 dpa was much larger than that irradiated to 10 dpa.

4. Conclusion

The effect of displacement damage up to 50 dpa on microstructural development in the SiC/SiC composites was investigated. The SiC/SiC composites with Hi-Nicalon Type S SiC fiber were irradiated up to 50 dpa by simultaneous ion-beams of Si, He and H at 1000 °C.

(1) He bubbles were observed in the matrix irradiated to more than 10 dpa at 1000 °C. The average size of He bubbles increased with increasing displacement damage.
(2) Almost all the He bubbles were formed at the grain boundaries in the matrix irradiated to 10 dpa. On the other hand, He bubbles were also formed in the grains of the matrix irradiated to 50 dpa.
(3) The matrix irradiated to 50 dpa under the Triple irradiation condition (a fusion reactor relevant condition) had the largest average size of He bubbles. The average size of He bubbles decreased when the amount of implanted H increased to more than a fusion reactor relevant condition.
(4) He bubbles were formed in the fibers irradiated to more than 10 dpa under the Triple irradiation condition. The average size of He bubbles in the fibers irradiated to 10 dpa was almost the same as that in the fibers irradiated to 50 dpa. On the other hand, the number density of He bubbles in the fibers irradiated to 50 dpa was much larger than that in the fibers irradiated to 10 dpa.

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