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Interface strength of SiC/SiC composites with and without helium implantation using micro-indentation test

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Abstract

Helium implantation effects on interface strength of SiC/SiC composite were studied using the micro-indentation fiber push-out method. Helium implantation was carried out with an accelerator at about 400 K. Total amount of implanted helium was approximately 10 000 appm. Increase of the fiber push-in load was observed in as-implanted specimen. After post-implantation-annealing at 1673 K for 1 h, the change of the fiber push-in load by helium implantation was not observed. Effects of helium implantation on the interface are discussed. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

Silicon carbide (SiC) has been considered as one of the candidate structural materials for fusion reactor applications due to its low induced activation by 14 MeV-neutron and its high-temperature strength. To improve the brittleness of monolithic SiC, SiC fiber reinforced SiC composites (SiC/SiC) have been developed.

Under fusion reactor environment, the relatively high helium generation rate of SiC to displacement damage is a characteristic factor. The He/dpa ratio is estimated to be 150 appm/dpa in the first wall region, and helium generation rate will be up to 1600 appm/MWy/m^2 [1,2]. Total amount of helium in the SiC/SiC material at the first wall will be above 10 000 appm after several years of irradiation. Helium solubility is nearly zero in most of the materials, hence it is conceivable that helium may precipitate at grain boundary or fiber/matrix interface as bubbles at high temperature in which helium has enough mobility. Mechanical properties of fiber reinforced composites strongly depend on the fiber/matrix interface strength [3,4], therefore, the investigation of helium effects on the fiber/matrix interface strength is important to reveal the irradiation response of SiC/SiC composites under fusion reactor environment.

There are several ways to introduce helium into materials. The method of helium implantation by accelerator is successfully used and can introduce high concentration helium into most materials without any additional elements such as boron. On the contrary, problems of the implantation method are relatively a small volume of implanted material which is determined by the irradiation energy of the ions. A micro-indentation test has been successfully used to evaluate the mechanical property of such a small specimen [5]. The micro-indentation tests have also been used to estimate the interface strength of various type of fiber reinforced composites [6]. Though fiber debonding in SiC/SiC composite after neutron irradiation caused by fiber shrinkage and degradation of toughness was reported [3,4], there are few data of helium effect on interface of composites.

In order to study helium effects on mechanical properties of SiC/SiC composites under fusion reactor environment, measurement of the interface strength of helium implanted SiC/SiC composite has been carried out in this work.

2. Experimental procedure

The SiC/SiC composite specimens used (ID: USSD01) were made by DuPont. These composites consist of eight plies of 2-dimentional (0–90°) woven

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Hi-Nicalon^{TM} fiber clothes. Carbon coating on Hi-Nicalon fibers was performed by chemical vapor infiltration (CVI) method and the thickness of coating layer was about 1.2 μm. The β-SiC matrix was fabricated on the SiC clothes by CVI method.

The specimens were sliced into about 600 μm sections perpendicular to one direction of the Hi-Nicalon tows with a low speed diamond saw and thinned with mechanical polishing to a thickness of 120 μm. Finally, specimen surface was polished by using 1 μm diamond paste to remove small scratches induced by polishing process. The specimen size for testing is about $2 \times 3 \times 0.12$ mm^3.

Helium implantation was carried out with a Dynamitron type accelerator at the faculty of Engineering of Tohoku University. The specimen was irradiated with helium ion beam through an energy degrading system combined with oxide dispersion beam monitoring materials to obtain homogeneous depth distribution of helium in the sample. Acceleration energy was 3.00 and 2.95 MeV. A rotating wheel energy degrader consists of 11 aluminum-foil sections. Calculated helium distribution and displacement damage profile by TRIM code [7] is shown in Fig. 1. Using the energy degrading system and two irradiations with 3.0 and 2.95 MeV helium ions, homogeneous distribution area of helium was obtained in the range from 1 to 5.5 μm from the surface. Total amount of helium concentration was about 10 000 appm on the average and displacement damage was about 0.3–0.7 dpa in this area. Implantation atmosphere was a vacuum of 1×10^{-4} Pa and implantation temperature was 403 ± 40 K measured with an infrared thermometer. Post-implantation annealing was performed at 1673 K in a vacuum of 1.0×10^{-3} Pa for 1 h. Surface observation of the specimen was performed with an optical microscope and a scanning electron microscope (SEM).

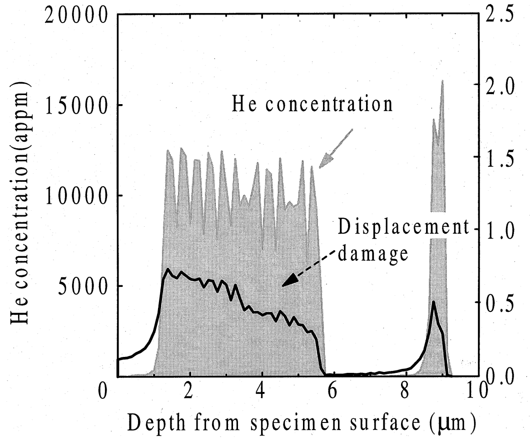

Fig. 1. Calculated distribution of helium concentration and displacement damage in Hi-Nicalon fiber after helium implantation. (Using TRIM-code)

Micro-indentation test was carried out on fiber with Akashi MZT-4. A Berkovich type indentation tip was used. The peak load of indentation test was set to 100 gf and the loading rate was 10 gf/s. To use fiber push-out method [8], the stage of the specimen was grooved to the width of 70 μm. The specimen was fixed on the grooved stage with resin. The load–displacement (*P–h*) curve of fiber push-out test was derived from indentation test of a fiber on the groove. From the *P–h* curve, two particular values were obtained, that is, push-in load and push-out load. Push-in load was determined from the curvature change as compared with the normal parabolic *P–h* curve, and push-out load was obtained from rapid displacement change of the *P–h* curve.

3. Results and discussion

Surface morphology change such as blistering was not observed in as-implanted specimen. However, surface morphology change was observed after post-implantation-annealing at 1673 K for 1 h. Fig. 2 shows an optical micrograph of the annealed specimen. Left and right sides of the picture shows unimplanted and implanted area, respectively. Unimplanted fibers look dark probably because of thermal etching, but the morphology of implanted fibers did not change by the annealing. This thermal etching seems to come out from grain growth of the fiber from a result of SEM observation. On the contrary, surface morphology of SiC matrix after annealing was observed both in helium implanted area and unimplanted area. Helium implantation effects on surface morphology change after annealing in the matrix were not observed.

Fig. 3 shows SEM micrographs of the specimens after the fiber push-out test. These composites have two interfaces, one is the interface between SiC matrix and carbon layer, and the other is the interface between

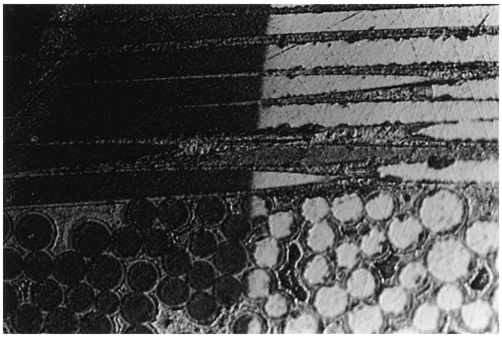

Fig. 2. Optical microscope image of helium implanted SiC/SiC composite after annealed at 1673 K.

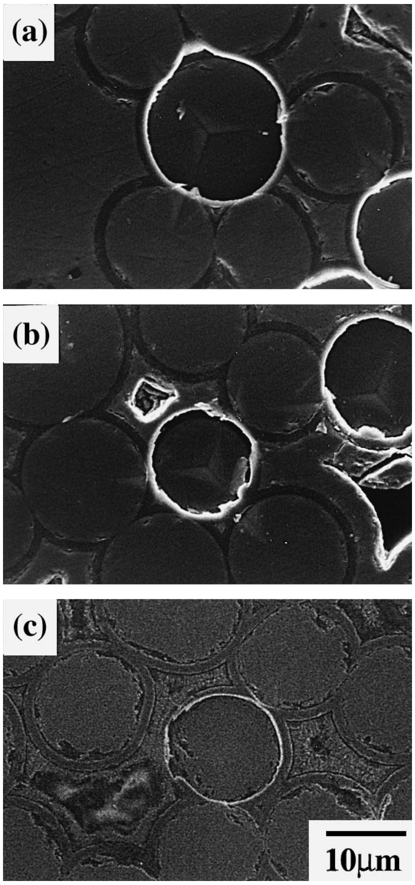

Fig. 3. SEM image of specimen surfaces after fiber push-out test: (a) unimplanted; (b) as-implanted; (c) after annealed at 1673 K.

carbon layer and fiber. Fig. 3(a) shows the surface of the unimplanted specimen. Debonding occurred almost in the interface between carbon layer and fiber. In the case of as-implanted specimen which is shown in Fig. 3(b), debonding of SiC matrix and carbon layer was frequently observed. Similar tendency was observed in the post-implantation-annealed specimen as shown in Fig. 3(c). These results mean that the strength of SiC matrix/carbon interface decreased after helium implantation and annealing compared to that of carbon layer/SiC fiber interface.

Fig. 4 shows the fiber push-in load as a function of diameter of fiber. Circle and triangle symbols of Fig. 4 indicate the results of unimplanted and helium implanted specimens, respectively. In the case of fiber push-out test, push-in load corresponds to the initiation of fiber debonding process and push-out load corresponds to the fiber sliding. Since helium implantation area was in the range from 1 to 5.5 μm, helium implantation effect mainly acts at the beginning of the fiber debonding process. In this implantation condition, therefore, it is useful to regard fiber push-in load as the parameter of helium effects on interface strength. In the result of as-implanted specimen (Fig. 4(a)), though it varies considerably, linearity was found out. The value of push-in load of helium implanted interface is higher than that of unimplanted interface. On the other hand, in the 1673 K annealed specimen (Fig. 4(b)), no linearity was found and helium effects were not clearly observed. The push-in load both of implanted and unimplanted specimens was significantly decreased after annealing.

The normalized push-in load (P_n^{in}) was used to compare helium effects on fiber debonding behavior. Since the average diameter of SiC fiber of the specimen was about 14 μm, P_n^{in} was calculated as follows; $P_n^{in} = P^{in}/d_f \times 14$, where P^{in} is fiber push-in load, d_f is the fiber diameter. Fig. 5 shows the average P_n^{in} with its standard deviation of as-implanted and 1673 K post-implantation-annealed composite in comparison with helium implanted and unimplanted ones. In the results of as-implanted specimen, remarkable increase of the P_n^{in}

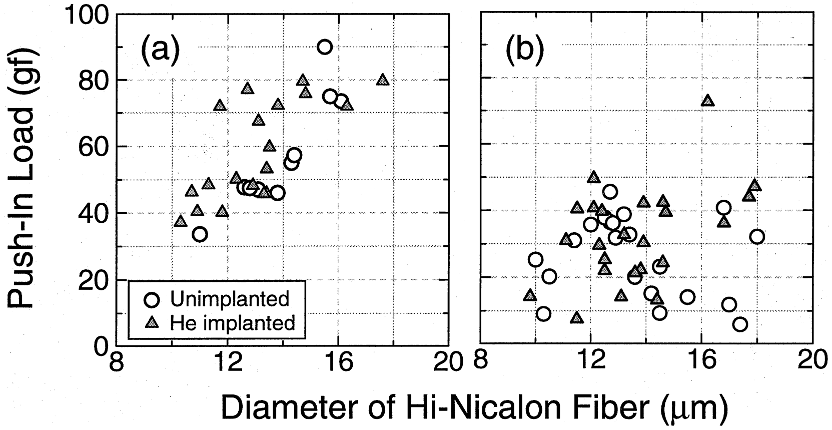

Fig. 4. Fiber push-in load as a function of diameter of Hi-Nicalon fiber: (a) as-implanted; (b) after annealed at 1673 K for 1 h.

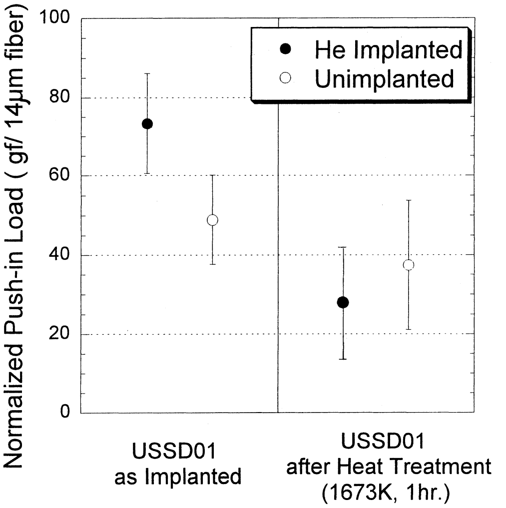

Fig. 5. Effect of helium implantation on fiber push-in load of the composite. The bar of each data shows the standard deviation.

by helium implantation was found. Whereas, in the results of 1673 K annealed specimen, change of P_n^{in} by helium implantation was not clearly observed. Previous works showed that the amount of swelling in monolithic SiC by neutron irradiation increased with decreasing irradiation temperature with a saturation in room temperature, linear expansion of about 1% [9]. An additional experiment was carried out to confirm the swelling by the same helium implantation condition using beam masking and step-height measurement. The result showed that swelling of the matrix was 3.7% and the swelling of the fiber was 2.0% after the 10 000 appm helium implantation at 400 K. The details of the results will be published elsewhere [10]. Based on these results, helium implantation effects on the fiber push-in load may be attributed to compressive stress caused by swelling of fiber and matrix. Decrease in helium implantation effects on P_n^{in} after annealing may be due to the recovery of the swelling in fiber.

Micro-structural observation of helium implanted and annealed specimens will be published elsewhere [11]. The result of micro-structural observation showed that the helium bubble formed only in the SiC matrix after the heat treatment (1673 K, 1 h.), but bubbles were not observed in the interface or SiC fiber. Therefore, the difference in debonding position after indentation may not be caused by helium bubble formation. Helium bubble swelling of matrix, shrinkage of fiber and carbon layer by recovery of defect structure and helium release probably occurred during the annealing. This behavior depends on temperature. Further investigation is required to clarify the debonding mechanism.

Surface morphology change of SiC fiber by 1673 K annealing is also considered as helium implantation effect on micro-structure. The thermal etching may be caused by the interaction of impurity oxygen and extra carbon in SiC fibers. The suppression of morphology change of SiC fiber by helium implantation might be attributed to the suppression of mobility of oxygen and extra carbon by their trapping with He-vacancy clusters which were induced by the helium implantation.

In spite of such large amount of helium, interface debonding assisted by helium bubble formation was not observed in this experimental condition. The results show that helium effects on interface may not be so serious, but helium assisted swelling and difference of helium behavior in each part of SiC/SiC will be important to apply to SiC/SiC component in fusion reactor systems.

4. Summary

Helium effects on SiC/SiC composites are investigated by helium implantation (10 000 appm) and fiber push-in method. Surface morphology change was not observed in as-implanted specimen, but helium implantation effects on the change of surface was observed in fiber after 1673 K annealing. It may be attributed to stability of the micro-structure of SiC fiber by helium implantation.

Fiber push-in load increased by helium implantation in as-implanted specimen. This may be caused by compressive stress due to swelling of SiC fiber and SiC matrix by helium implantation, however, detailed microstructural observation is required to confirm the above. The fiber push-in load after the annealing of helium implanted and unimplanted specimens decreased, but interface debonding by helium bubble formation was not observed in these experimental conditions.

Acknowledgements

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