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Helium implantation effects on mechanical properties of SiC_f/SiC composites

A. Hasegawa [a,*], M. Saito [a], K. Abe [a], R.H. Jones [b]

[a] *Department of Quantum Science and Energy Engineering, Tohoku University, Aramaki-Aza-Aoba, Aoba-ku, Sendai 980-77, Japan*
[b] *Pacific Northwest National Laboratory, P.O. Box 999, Richland, WA 99352, USA*

Abstract

Helium effects on mechanical properties of SiC/SiC composites were studied to apply the composites on structural materials of a fusion reactor. Two types of 2D-SiC/SiC composites were examined in this work. Helium was implanted using 36 MeVα particles with an energy degrader system to obtain a uniform helium depth distribution in the specimen. The implantation temperature was 400–800°C and the helium concentration after implantation was about 150–170 appm. Three-point bending test was carried out at room temperature. Results of bending test showed that a small decrease of bend strength was observed in the composite which had the higher bend strength before implantation. Helium implantation effect was not clearly observed in the lower bend strength composite. SEM observation results on the fracture surface gave no evidence for fiber coating failure. The decrease of bend strength after helium implantation may be attributed to a degradation of mechanical properties of the fibers by implanted helium. © 1998 Elsevier Science B.V.

1. Introduction

Silicon carbide (SiC) materials have been proposed for structural materials in fusion reactors because of their low induced activity by neutron irradiation and high temperature strength. Since monolithic SiC is very brittle as a structural material, SiC reinforced SiC matrix composites have been developed to increase its toughness. The SiC composite system which has received the most attention for fusion energy applications, is the continuous fiber rein forced and chemically vapor infiltrated (CVI) composites. Recent status of SiC composites for fusion was reviewed by Snead et al. [1].

The key elements affecting ceramic composite performance are the fiber reinforcement and the fiber/matrix interface. Regarding SiC-based fibers for SiC/SiC composites, Nicalon™ fibers have been widely used. Nicalon is a polymer-derived Si–C–O fiber [2] but its physical and dimensional properties have been shown to be unstable at relatively low neutron fluence [3,4]. In order to improve the high temperature stability of Nicalon fiber, reduced oxygen Hi-Nicalon™ fibers were developed by Nippon Carbon [2]. These fibers are synthesized from polycarbosilane using electron beam curing and pyrolysis. Hi-Nicalon fiber showed higher degree of structural stability after high fluence of neutron irradiation [5,6], therefore Hi-Nicalon is considered a promising candidate reinforced fiber for fusion applications.

To improve SiC/SiC performance, several kinds of fiber coating were studied. The most widely utilized interface in the CVI SiC/Nicalon system is carbon. There is an optimum carbon coating thickness to obtain higher strength of SiC composite. It was typically 0.1 to 0.2 μm [7] but its optimum thickness and coated materials under irradiation environment has not been clarified yet because sufficient irradiation data have not been obtained on new fibers which have high performance such as Hi-Nicalon.

In a fusion reactor environment, helium will be produced at the first wall at the rate of about 1500–2000 appm He/(MW a m^2) in SiC by transmutation reactions and displacement damage will be 10–15 dpa/(MW a m^2), depending on the details of the blanket structure and on neutron spectrum [1,8]. The He/dpa ratio of SiC will be higher than that of other candidate alloys such as vanadium and ferritic steels [1]. Since helium in materials under

* Corresponding author. Fax: +81-22 217 7925; e-mail: akira.hasegawa@qse.tohoku.ac.jp.

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irradiation environment accelerates swelling and creep, and precipitates at interfaces such as grain-boundary and fiber/matrix interfaces, mis-match and interface debonding between fiber and matrix may occur in SiC/SiC composite under fusion reactor conditions. On the other hand, the strength of fiber/matrix interface will influence the fracture strength and toughness of fiber reinforced composites. Therefore, behavior of helium gas atoms and their effect on mechanical properties of SiC/SiC composite will be important for its application to structural materials of fusion reactors. The purpose of this work is to study helium effects on the bending properties of SiC composites which are reinforced with SiC weaves made of Hi-Nicalon fibers using helium implantation methods by accelerators.

2. Experimental

In this work, we studied He effects on two types of 2D-SiC_f/SiC composites reinforced by SiC weaves made of Hi-Nicalon fiber. Specimen code names are JSS01 and USSD01 respectively. Both JSS01 and USSD01 were fabricated by the chemical vapor infiltration (CVI) method. The JSS01 is the first batch of Japanese reference material of SiC/SiC composite fabricated by Ube Industries. The Hi-Nicalon cloths were stacked and impregnated in polymer solution to form pre-formed composites. It was heat-treated at 1000°C to form thin carbon coating layer on the fibers, then SiC matrix was deposited by CVI process at about 1100°C. The strength of the JSS01 at room temperature was in the rage of 100 to 220 MPa. The details of fabrication and its strength at room temperature and higher temperature will be presented elsewhere [9]. The USSD01 was fabricated by Dupont in USA. Carbon coating on the fiber was carried out by CVD process and SiC matrix was deposited by CVI. Its bend strength at 550°C in argon was 550 ± 30 MPa. The details were reported elsewhere [10]. Major differences of the specimens were thickness and processing method of carbon coating on SiC fibers. The thickness of the carbon coating of JSS01 was approximately 100 nm and it was coated by solution impregnation. The thickness of the carbon coating layer of USSD01 was 1.2 μm and it was processed by CVD.

Because a relatively large volume was required to evaluate bending strength, high energy helium ion implantation was used to prepare helium implanted bend bars. Specimen size of bend bar was 4 mm width, 1 mm thickness and 18 mm length. The numbers of 2D woven SiC cloth layers thorough the thickness of the bend bar were 3 in JSS01 and 4 in USSD01. The number of fiber bundles along the specimen width was 3 in both specimens.

Helium implantation was carried out using a cyclotron at Tohoku University. The accelerated energy of the helium ions was 36 MeV and the projected range in SiC was approximately 470 μm which was calculated by TRIM code [11]. To obtain uniform helium depth distribution, tandem type degrader wheels were used [12]. The total amount of implanted helium concentration calculated from the irradiation fluence was 150–170 appm, depending on irradiation position. Displacement damage in the He implanted area was approximately 0.09 dpa using $E_d = 45$ eV. The implantation temperature was measured by an infrared pyrometer and it was 400–800°C for different specimens. The He implanted area was on the tensile surface. Since the number of specimen was limited, four specimens were irradiated and tested for each composites.

Three-point bending test was carried out at room temperature. The span length was 14 mm and the cross head speed was 0.2 mm/min. After the bending test, fractography observation was carried out by SEM.

3. Results and discussion

Fig. 1 shows load–deflection curves of JSS01 and USSD01. Because of space limit of the bending fixture,

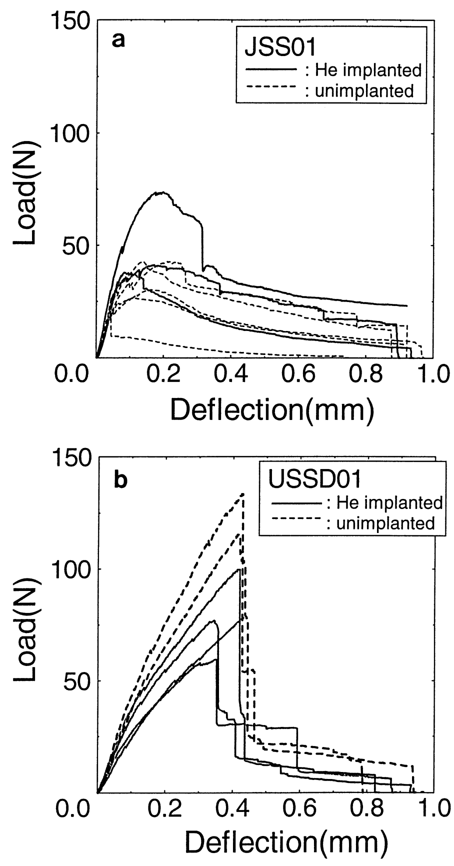

Fig. 1. Load–deflection curves of SiC/SiC composites. Dashed lines are data of unimplanted specimens, solid lines are data of He implanted specimens. (a) JSS01, (b) USSD01. Implanted temperature was in the range of 400–800°C.

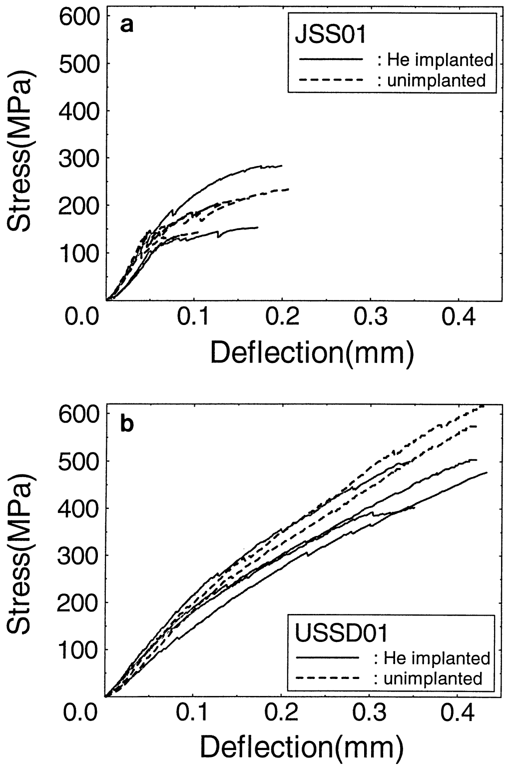

Fig. 2. Stress–deflection curves of SiC/SiC composites up to maximum strength. Dashed lines are data of unimplanted specimens, solid lines are data of He implanted specimens. (a) JSS01, (b) USSD01.

the bend test was stopped before complete failure of tested specimen, so work-of-fracture could not be obtained in this experiment. Fig. 2 shows the stress–deflection curves up to the maximum strength. The stress level of first matrix cracking (FMC) in both composites was in the range of 100 to 150 MPa for JSS01 and it was below 100 MPa for USSD01. A helium implantation effect was not clearly seen in the FMC behavior. After FMC, the stress–deflection curve and maximum bending strength of JSS01 had wide scatter probably because of inhomogeneity of the composite; therefore helium implantation effects were not clearly seen.

On the other hand, a decrease of the maximum bend strength by helium implantation compared to data scattering of unimplanted specimen was observed in USSD01. The minimum strength of irradiated USSD01 specimen may be caused by temperature increase during implantation compared to other ones. The implantation temperature of other three was in the range of 400–600°C; however the implantation temperature of the weakest one was in the range of 500–800°C. The temperature difference may be attributed to thermal diffusivity difference caused by thin cracks between Hi-Nicalon weaves or pores in the matrix which were formed during CVI process. The slope of the stress–deflection curve after FMC was decreased by helium implantation. This may be attributed to helium effects on modulus and fracture behavior of fiber bundles during bending test of USSD01. The higher implantation temperature may have decreased the modulus and strength of the USSD01.

A large load drop after maximum strength which was caused by fiber bundles failure was observed, and a relatively low load stage was continued at higher deflection until specimen failure occurred. During this stage fiber pullout mainly occurred. Helium implantation effects were not observed in JSS01, but a reduction of load during the pull out period by helium implantation was observed in USSD01 as is shown in Fig. 1.

Fig. 3 shows the microstructure of the unimplanted specimen after the bending test. The upper side of these pictures is the tensile-stress-loaded side. Helium was injected in this side. Large cracks were observed along cross line of the fiber bundles in both materials. Small cracks

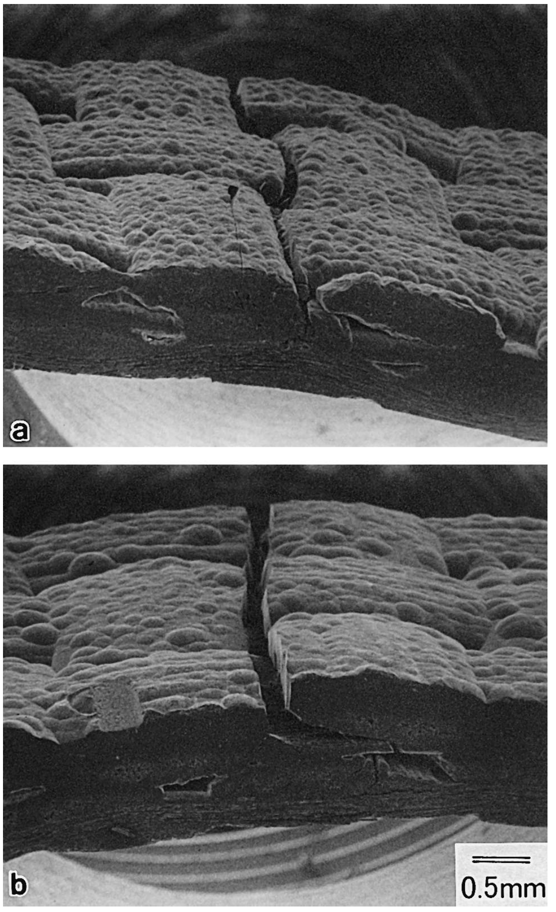

Fig. 3. SEM micrographs of bend tested unimplanted specimens. (a) USSD01, (b) JSS01.

and fine debonding cracks between fiber/matrix was observed in USSD01, but large cracks and large delaminated surface between SiC weaves was observed in JSS01.

Details of SEM micrographs of ruptured surface of USSD01 are shown in Fig. 4(a, b, c). The helium ions were injected from the lower side of the pictures. The surfaces of the pullout fibers were rough. The rough blanket type image around the fibers is carbon coating layer. These results showed the debonding between fiber/matrix occurred in the coated carbon layer. Although a decrease of load during fiber pullout period was observed in USSD01, helium implantation effects of the surface morphology of these fibers were not observed. The results shows that interface debonding by implanted helium did not occur in this implantation conditions. Fig. 4(d) shows a typical surface of fibers in JSS01. The fiber surface was flat and smooth in both unimplanted and helium implanted specimens. It shows that the bonding strength of fiber and carbon coating layer of JSS01 is relatively lower than CVD coated carbons such as USSD01. In addition, helium implantation effects on the surface of pullout fiber were not observed in JSS01.

The difference of unimplanted bending strength between the two composites may be attributed to fiber/matrix interface strength which was dependent on the thickness of carbon coating on the fibers and the quality of coating. In the case of JSS01, the coating layer did not have enough strength and thickness, and optimization of process conditions of coating is required for JSS01. The strength also depends on insufficient filling up of the SiC matrix between SiC weaves during CVI process. The density of USSD01 was 2.42 g/cm^3 and that of JSS01 was 2.2 g/cm^3. The porosity of matrix also influences the strength of the composites. In the case of JSS01, the strength level of the composite may be too low to show the helium effects.

The mechanism responsible for the decrease of bending strength and pullout load of USSD01 by helium implantation was not clarified in this work, but the former may be attributed to reduction of modulus and strength of fibers by helium implantation. To clarify the helium effects, mechanical testing of helium implanted fiber and microstructural observation of helium implanted composites with TEM is required.

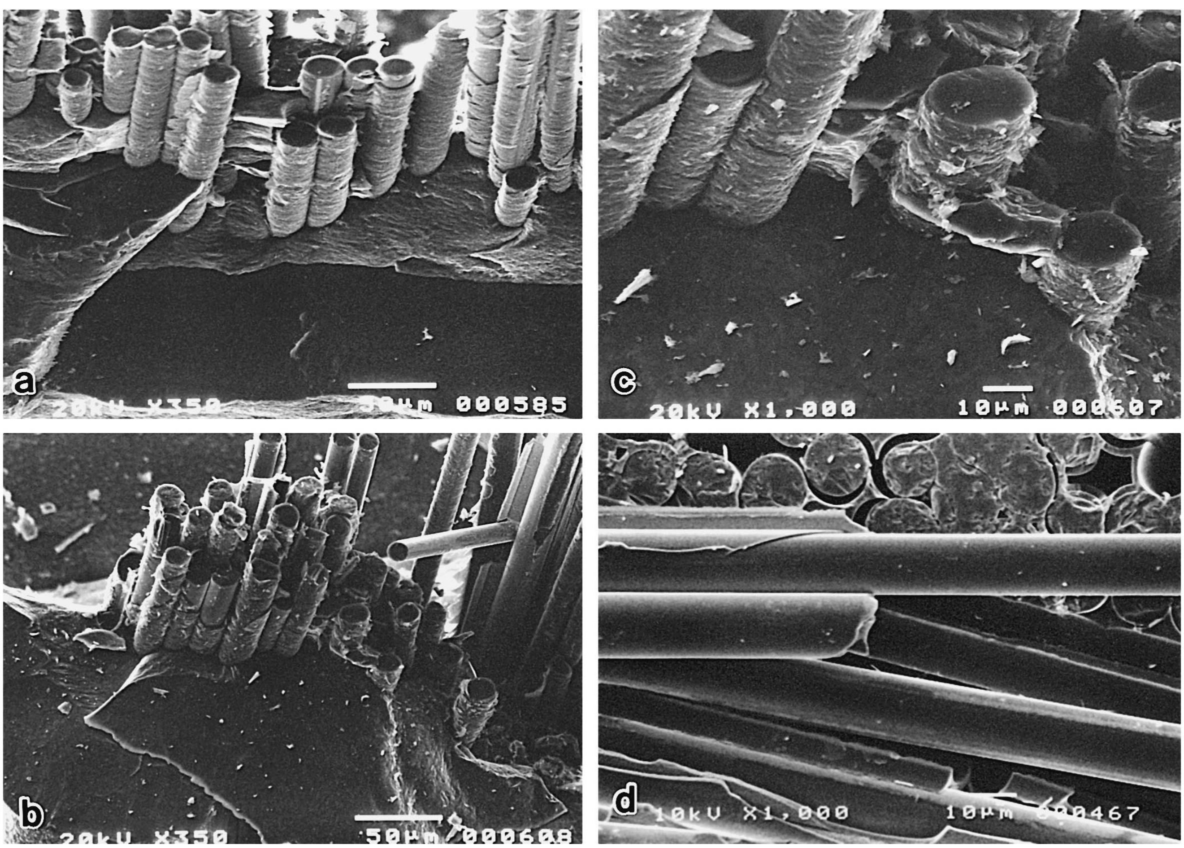

Fig. 4. SEM micrographs of fracture surface of bend tested specimens. (a) USSD01: unimplanted, (b) USSD01: He implanted, (c) USSD01: He implanted, (d) JSS01: unimplanted.

4. Summary

Helium effects on bending behavior of 2D-SiC/SiC composites reinforced by Hi-Nicalon fibers were studied using helium implantation by accelerator. The following results were obtained.

(1) Helium effects on bending strength at room temperature was observed in USSD01 after 150–170 appm helium implantation. A decrease of the maximum strength and a decrease of load during fiber pullout were observed, relative to unirradiated specimens.

(2) SEM observation showed that surface morphology of pullout fiber of USSD01 was not changed by helium implantation. Reason of the strength change of the composites may not be attributed to interface debonding of fiber. It may be attributed to degradation of mechanical properties of fiber by helium implantation.

(3) Bend strength change by helium implantation was not observed in JSS01. Morphology of pull out fiber surface was flat and smooth. Relatively lower strength of JSS01 may be attributed to insufficient strength of fiber/matrix interface and it was the reason of the response of helium implantation.

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