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# Effect of He pre-implantation and neutron irradiation on mechanical properties of SiC/SiC composite

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**Abstract**

Mechanical property changes of SiC/SiC (Hi-Nicalon/C/SiC) composite caused by uniform He pre-implantation up to about 170 at.ppm at 400–800 °C followed by neutron irradiation up to about $7.7\times10^{25}$ n/m$^2$ ($E_n > 0.1$ MeV) at 800 °C in HFIR were investigated by the three-point bend tests and nano-indentation tests. Degradation of the composite bend properties due to neutron irradiation was observed. The hardness increased after neutron irradiation for both the SiC-matrix and the Hi-Nicalon fiber. There was almost no change in the elastic modulus of the SiC-matrix, but there was an increase in the modulus of the Hi-Nicalon fiber after neutron irradiation. He pre-implantation had almost a negligible effect on the mechanical properties of the composite specimen.


## 1. Introduction

Silicon carbide (SiC) fiber-reinforced SiC-matrix composites (SiC/SiC composites) are being considered as a structural material for components in future fusion reactor blankets [1,2]. Displacement damage and transmutation products such as helium (He) will be produced in these materials during high energy (about 14 MeV) neutron irradiation. In a full power year, the displacement damage and He concentration in a SiC component of the blanket of the ARIES-IV concept reactor were calculated to be approximately 57 dpa and 15 380 at.ppm, respectively [2]. The He concentration per dpa in SiC will be about 10 times larger than that in other candidate materials (ferritic steels and vanadium alloys).

A relatively large number of studies of the displacement damage effect on the mechanical properties of SiC/SiC composites have been carried out. Advanced SiC/SiC composites, developed in recent years using stoichiometric and high crystalline SiC-fibers such as Hi-Nicalon Type-S [3] and Tyranno SA [4], have exhibited improved radiation resistance up to about $7.7\times10^{25}$ n/m$^2$ [5]. There have also been some studies of the effect of transmutant He on the mechanical properties of SiC/SiC composites [6–8]. Using bend tests, the ultimate fracture strength of a Nicalon CG/C/SiC composite (C: carbon) [6] and a Hi-Nicalon/C/SiC composite [7] decreased after He-implantation up to 2500 at.ppm at 900 °C and up to 150–170 at.ppm at 400–800 °C, respectively. For composite studies utilizing the nano-indentation test, both hardness and elastic modulus of the SiC-matrix and SiC-fibers (Hi-Nicalon and Hi-Nicalon Type-S) decreased after He-implantation up to 20 000 at.ppm below 100 °C

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[8]. However, the effects of He, and of displacement damage, and their synergistic effect on the mechanical property changes of SiC/SiC composites were not clearly distinguished in all cases. The purpose of this study is to investigate the effect of He pre-implantation followed by neutron irradiation on the mechanical properties of a SiC/SiC composite.

## 2. Experimental

A SiC/SiC composite with Hi-Nicalon fibers (0°/90° plain-weave) manufactured by DuPont Lanxide [9] was examined in this study. The SiC-matrix (crystalline β-SiC) was fabricated by an isothermal chemical vapor infiltration (ICVI) process. The interface material between SiC-fibers and SiC-matrix was a pyrolytic carbon (≈150 nm thickness), which was fabricated by a chemical vapor deposition (CVD) process. Hi-Nicalon is composed of β-SiC grains whose size is about 5–10 nm and have some of residual oxygen (<0.5 wt%), but substantial amounts of carbon (C/Si atomic ratio $\sim 1.39$) [3]. The geometry of the specimens for irradiation and bend tests was approximately $4^w \times 1^t \times 20^l$ mm$^3$ (machined from specimens with an original geometry of $6^w \times 2^t \times 20^l$ mm$^3$). The compression side of the bend specimens was machined to 1 mm thickness, while tension side was not machined. As a result, a CVI β-SiC overlayer remained on the tension side. Fig. 1 shows the schematic illustration of the specimen dimensions and test configurations in this study.

Helium pre-implantation was performed using the cyclotron accelerator at Tohoku University. The acceleration energy of He-ions was 36 MeV. The projected range of 36 MeV He-ions in SiC was calculated to be about 470 μm by the TRIM code [10]. Tandem type energy degrader wheels were used to obtain uniform depth distribution of He-atoms. The nominal He concentration was about 170 at.ppm. The displacement damage in the He implanted region was calculated to be about 0.009 dpa using a threshold displacement energy of 40 eV. The implantation temperature was 400–800 °C. He-ions were implanted to the tension side of the bend specimens within an area of about $4^w \times 4^l$ mm$^2$ located at the center of the specimens as shown in Fig. 1.

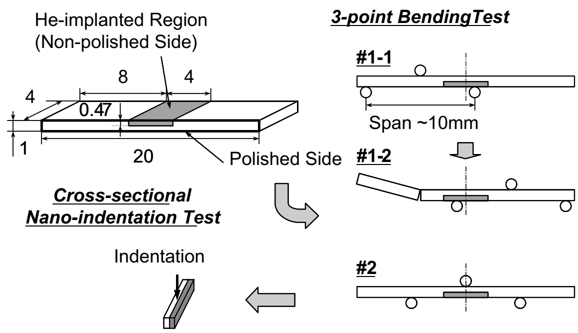


Fig. 1. The schematic illustration of the specimen dimensions and test configurations in this study.

Neutron irradiation was performed in the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory. Specimens were irradiated at 800 °C up to about $7.7 \times 10^{25}$ n/m$^2$ ($E_n > 0.1$ MeV), which corresponds to about 7.7 dpa using an assumption that the displacement energy of 40 eV for both the Si and C sublattices.

Three point bend tests were performed at room temperature in air. The support span length and cross-head speed were 10 mm and 0.5 mm/min, respectively. Configurations #1–1 and #1–2 in Fig. 1 were used for the bend test measurements of the non-He-implanted, but neutron-irradiated region. Configuration #2 was for the measurements of the He-implanted and neutron-irradiated region. Fractography observations were performed using a scanning electron microscope (SEM).

Cross-sectional nano-indentation tests were performed at room temperature using a NANO INDENTER® II (Nano-instruments) equipped with a Berkovich diamond tip. The indentation depth and loading (unloading) rate were constant, 50 nm and 50 m N/s, respectively. Specimens were mechanically sliced perpendicular to the He-implanted surface as shown in Fig. 1. The cross-section of the cut specimens for indentation was mechanically polished with a 0.5 μm diamond slurry.

Hardness $H$ and elastic modulus $E$ were evaluated using an unloading curve and the method proposed by Oliver and Pharr [11]. $H$ is calculated from the following formula:

$$H = P_{\max}/A(h_c), \tag{1}$$

where $P_{\max}$ is the maximum indentation load and $A$, which is a function of constant indentation depth $h_c$, is the contact area of the indenter with the specimen surface. $E$ is defined as follows:

$$E = \left(1 - v_s^2\right)/\left(1/E_r + (1 - v_i^2)/E_i\right), \tag{2}$$

where $v_s, v_i, E_r$ and $E_i$ are the Poisson's ratio of specimen, the indenter (0.07 for diamond), the reduced modulus and the elastic modulus of the indenter (1141 GPa for diamond), respectively. Further details of this method are given in Ref. [11].

## 3. Results

Fig. 2 shows the summary of the bend test results (ultimate fracture strength, elastic modulus and proportional limit stress – PLS) for the as-received, the non-He-implanted and neutron-irradiated (Non-He/Neutron), and the He-implanted and neutron-irradiated

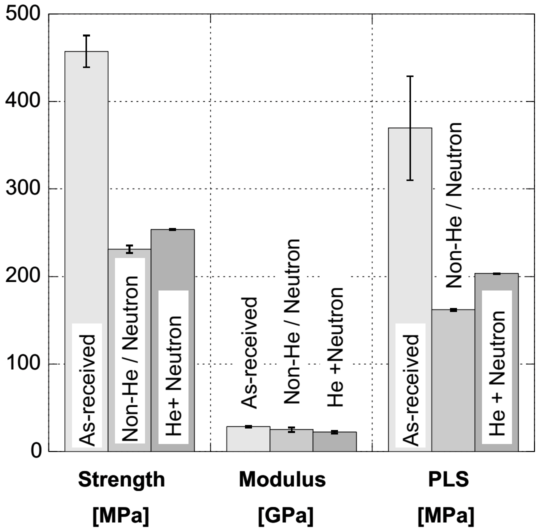


Fig. 2. The summary of the bend test results (ultimate fracture strength, elastic modulus and proportional limit stress (PLS, offset strain ~ 0.0005)) for the as-received, the non-He-implanted and neutron-irradiated (Non-He/Neutron), and the He-implanted and neutron-irradiated (He + Neutron) composites.

(He + Neutron) composites. The elastic modulus was calculated using the slope of the linear range of the stress–strain curves. The PLS was the stress value when offset strain was about 0.0005. The ultimate fracture strength, elastic modulus and proportional limit stress decreased due to neutron irradiation by about 50%, 10% and 55%, respectively. The 'He + Neutron' specimens had similar bend properties to the 'Non-He/Neutron' specimens.

Fig. 3 shows a typical observation of the fracture surface by SEM for the 'as-received', the 'Non-He/Neutron', and the 'He + Neutron' composites. He-ions were implanted from the bottom side of the pictures. The pull-out lengths of the Hi-Nicalon fibers for the 'Non-He/Neutron' composite were longer than that for the 'as-received' composite. Almost no differences of the pull-out lengths for the Hi-Nicalon fibers were observed between 'Non-He/Neutron' and 'He + Neutron' composites.

Figs. 4 and 5 show the hardness and elastic modulus of the SiC-matrix and Hi-Nicalon fiber of the 'as-received', 'Non-He/Neutron', and 'He + Neutron' composites. The data for non-He-implanted, but neutron-irradiated region and data for He-implanted and neutron-irradiated region were the average of the data points at the depth of 0–470 and 470–1000 μm from the He-implanted surface, respectively. The hardness increased by about 30% after neutron irradiation (no

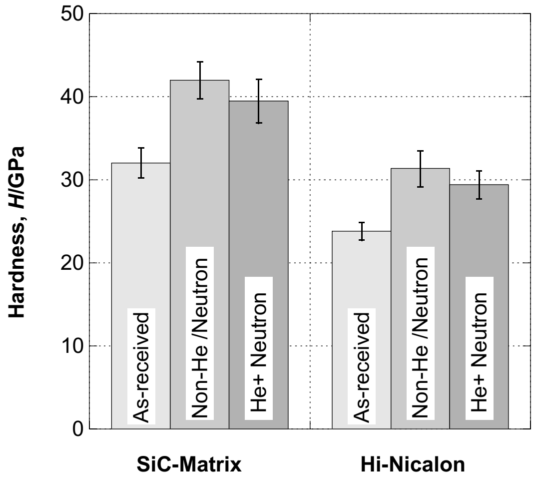


Fig. 4. The hardness of the SiC-matrix and Hi-Nicalon fiber for as-received, non-He-implanted and neutron-irradiated (Non-He/Neutron) and He-implanted and neutron-irradiated (He + Neutron) composites.

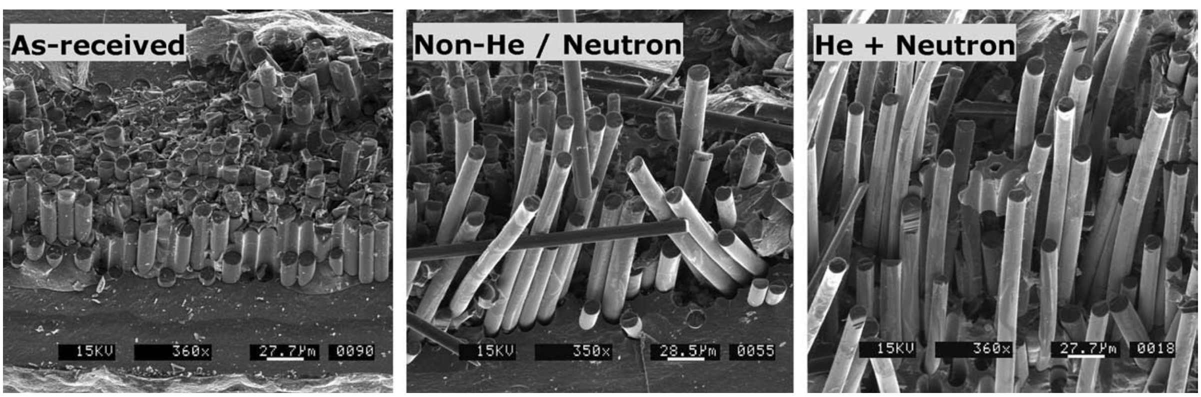


Fig. 3. Typical fracture surface observations by SEM for as-received, non-He-implanted and neutron-irradiated (Non-He/Neutron) and He-implanted and neutron-irradiated (He + Neutron) composites. He-ions were implanted from the bottom side of the pictures.

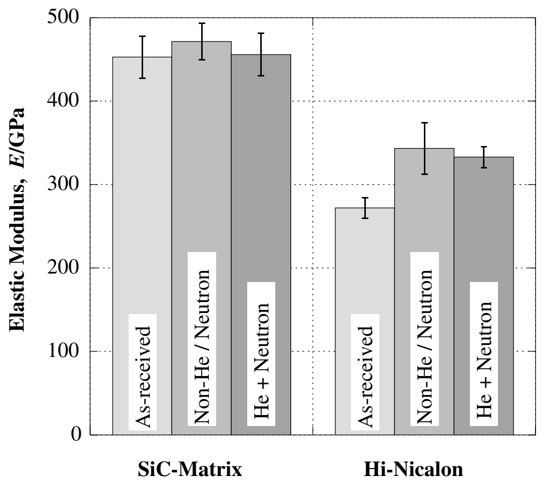


Fig. 5. The elastic modulus of the SiC-matrix and Hi-Nicalon fiber for as-received, non-He-implanted and neutron-irradiated (Non-He/Neutron) and He-implanted and neutron-irradiated (He + Neutron) composites.

helium) for both the SiC-matrix and the Hi-Nicalon fibers. Almost no change in the elastic modulus for the irradiated SiC-matrix was observed, but about a 30% increase for the irradiated Hi-Nicalon fibers was observed. The 'He + Neutron' specimens exhibited almost the same hardness and elastic modulus as the 'Non-He/Neutron' specimens.

## 4. Discussion

A 10% reduction in the elastic modulus for the composite was observed. In contrast, almost no change in the elastic modulus for the SiC-matrix and about 30% increase for the Hi-Nicalon fiber were observed. Thus, so called the sum rule defined as follows is not applicable for the neutron-irradiated composite in this study:

$$E_c = E_f \times f_f + E_m \times f_m, \qquad (3)$$

where $E_c, E_f$ and $E_m$ are the elastic modulus of the composite, fiber and matrix, $f_f$ and $f_m$ are the volume fraction of the fiber and matrix in composite, respectively. Therefore, it can be predicted that the degradation (e.g. crack and debonding) of the interface between the SiC-matrix and Hi-Nicalon fiber was occurred due to neutron irradiation. Shrinkage of Hi-Nicalon fibers during neutron irradiation was observed in previous studies [12–15]. A previous study [16] also showed the presence of microcracks and debonding of the C-interface of Hi-Nicalon/C/SiC composite due to C-ions irradiation up to about 10 dpa at 800 °C. Moreover, tensile strength of Hi-Nicalon fiber increased with neutron dose [17]. Thus, bend property degradations of Hi-Nicalon/C/SiC composite due to neutron irradiation might be related to the formation of microcracks and debonding of the C-interface induced by the shrinkage of the Hi-Nicalon fibers.

The effect of He pre-implantation treatment prior to neutron irradiation on mechanical properties was almost negligible (relative to the mechanical properties of the neutron irradiated specimens) up to a concentration of 170 at.ppm for the composite and the individual components of the composite. In previous work [8], the total number of released He atoms from the room temperature He-implanted Hi-Nicalon/C/SiC composite and carbon material during the annealing to 1500 °C were calculated to be about 30% and 80% of the original number of the implanted-He atoms, respectively. Moreover, a previous work [16] showed no cavity formation in the Hi-Nicalon/C/SiC composite due to simultaneous C- and He-ion irradiation up to about 10 dpa and 1000 at.ppm-He at 800 °C. Therefore, it can be assumed that the pre-implanted-He in the Hi-Nicalon/C/SiC composite in this study remained in the specimens (especially in the SiC-matrix and Hi-Nicalon fibers) as small clusters and single atoms after the neutron irradiation at 800 °C. However, it may be indicated that the effect of the pre-implanted He ($\sim$170 at.ppm) on the mechanical properties of Hi-Nicalon/C/SiC composite in this study was relatively smaller than that of displacement damage caused by the He pre-implantation and neutron irradiation.

## 5. Summary

Mechanical property changes of SiC/SiC composite, its matrix and fibers (Hi-Nicalon) were studied for He pre-implantation up to about 170 at.ppm at 400–800 °C followed by neutron irradiation up to about $7.7 \times 10^{25}$ n/m$^2$ ($E_n > 0.1$ MeV) at 800 °C in HFIR using bend tests and nano-indentation. The following results were obtained:

(1) The composite ultimate fracture strength, elastic modulus and proportional limit stress decreased due to neutron irradiation by about 50%, 10% and 55%, respectively. Specimens that were He pre-implanted and then neutron irradiated had similar bend properties to specimens irradiated with neutron only.
(2) The pull-out lengths of Hi-Nicalon fibers for non-He-implanted, but neutron-irradiated composites were longer than that for as-received composites. Almost no differences in pull-out lengths of Hi-Nicalon fibers were observed between non-He-implanted and neutron-irradiated composites and He-implanted and neutron-irradiated composites.

(3) The hardness increased by about 30% due to neutron irradiation for both SiC-matrix and Hi-Nicalon fiber. The specimens that were He pre-implanted and neutron irradiated had almost the same hardness properties as the specimens that were only exposed to neutrons.
(4) Almost no change in the elastic modulus of the SiC-matrix, but about a 30% increase in elastic modulus of the Hi-Nicalon fibers was observed after neutron irradiation. The specimens that were He pre-implanted and neutron irradiated had almost the same elastic modulus values as the specimens that were only neutron irradiated.

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