Available online at www.sciencedirect.com
ScienceDirect
Journal of Nuclear Materials 367–370 (2007) 753–757
journal of nuclear materials
www.elsevier.com/locate/jnucmat

# Cavity formation in Tyranno-SA $SiC_f$/SiC composite irradiated with multiple-ion beam at elevated temperatures

H.T. Keng [a], S.W. Li [a], S.W. Wu [a], Ji-Jung Kai [a,*], Fu-Rong Chen [a], Yutai Katoh [b], A. Kohyama [c]

[a] *Department of Engineering and System Science, National Tsing Hua University, Hsinchu 300, Taiwan*
[b] *Metals and Ceramic Division, Oak Ridge National Laboratory, Oak Ridge, TN, USA*
[c] *Institute of Advanced Energy, Kyoto University, Uji, Kyoto 611-0011, Japan*

**Abstract**

The microstructural evolution of Tyranno-SA $SiC_f$/SiC composite irradiated at 800 °C and 1000 °C was investigated by transmission electron microscopy. The irradiation was performed using multiple-ion beam in order to simulate the D-T fusion reactor environment. In this study, we irradiated $SiC_f$/SiC composite made from advanced SiC fibers namely, Tyranno-SA, using dual-ion beam (6 MeV $Si^{3+}$ and 1.13 MeV $He^+$) and triple-ion beam (6 MeV $Si^{3+}$, 800 keV $He^+$, and 280 keV $H^+$). The effects of He and H atoms on the cavity formation during irradiation are discussed. Cavities were found mainly on the grain boundary of $SiC_f$/SiC composite, and cavity coarsening was observed after higher temperature (1000 °C) irradiation. Hydrogen atoms in the $SiC_f$/SiC composite will enhance cavity nucleation.


## 1. Introduction

Silicon carbide fiber–reinforced Silicon carbide composites are major candidates for use as advanced structural materials for future fusion power reactors due to its low radioactivity and high temperature strength [1]. One of the main concerns about using these materials in the fusion environment is the stability of the microstructure during irradiation, that could affect their mechanical properties at high temperature [2].

The $SiC_f$/SiC composite used in this experiment was made with low oxygen, high crystalline and stoichiometric composition Tyranno-SA SiC fiber [3]. In fusion reactor, gas production is estimated to be about 2000 appm He/(MW/$m^2$) and 800 appm H/(MW/$m^2$) corresponding to a gas/dpa ratio of 150 appm He/dpa and 60 appm H/dpa [1]. Helium atoms are almost insoluble in SiC and easily trapped by vacancy clusters produced by neutron cascade. The solubility of hydrogen in the SiC is also low. Hydrogen is always trapped at both Si- and C-sites, and the mobility of hydrogen is high at elevated temperatures because the bonding of Si- and C- would be broken [4]. Previous studies [5] show that hydrogen may enhance cavity formation by increasing the number of nucleation sites. One purpose of

* Corresponding author. Tel.: +886 3 5742855; fax: +886 3 5716770.
*E-mail address:* jjkai@ess.nthu.edu.tw (J.-J. Kai).

0022-3115/$ - see front matter © 2007 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2007.03.087

this study is also to clarify the effect of hydrogen on cavity formation in $SiC_f/SiC$ composites.

## 2. Experiment

The uni-directional $SiC_f/SiC$ composites with Tyranno-SA fibers examined in this study were received from the Institute of Advanced Energy, Kyoto University, Japan. The thickness of carbon layer coating on the fibers is about 1 μm. The matrix of these $SiC_f/SiC$ composites were fabricated using the chemical vapor infiltration (CVI) method. The structure of the matrix is fcc β-SiC with many stacking faults in the grains SiC (see Fig. 1). They were residual defects formed during the CVI fabrication process.

Three irradiation conditions were employed in this study. Fig. 2 shows the depth distribution of 6 MeV $Si^{3+}$ and 1.13 MeV $He^{+}$ dual-ion irradiation in $SiC_f/SiC$ composite calculated by the TRIM98-code. The irradiation doses in the examined area (2.4 μm from the surface) were 10 dpa and 100 dpa, respectively, and irradiation temperatures were at 800 °C and 1000 °C, respectively. Fig. 3 shows the depth distribution of 800 keV $He^{+}$ and 280 keV $H^{+}$ dual-ion irradiation in $SiC_f/SiC$ and the fluence were He/H = 15000 appm/6000 appm in the examined area at 800 °C and 1000 °C, respectively. Fig. 4 shows the depth distribution of 6 MeV $Si^{3+}$, 800 keV $He^{+}$, and 280 keV $H^{+}$ triple-ion irradiation in $SiC_f/SiC$. The irradiation dose is 10 dpa in the examined area (1.56 μm from the surface), and the irradiation temperature is 800 °C. The damage rate was about $4 \times 10^{-4}$ dpa/s.

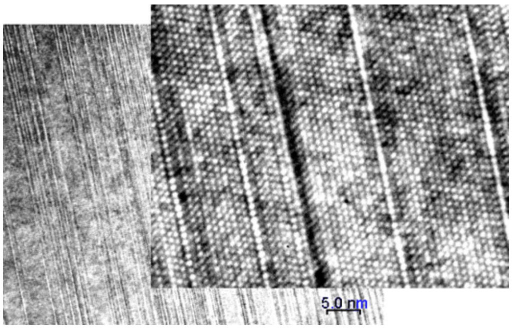


Fig. 1. The high resolution TEM image in the grains of $SiC_f/SiC$ matrix.

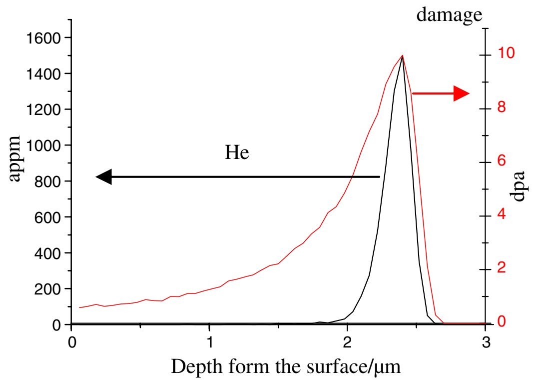


Fig. 2. The depth distribution of the displacement damage, He in SiC calculated with TRIM-code.

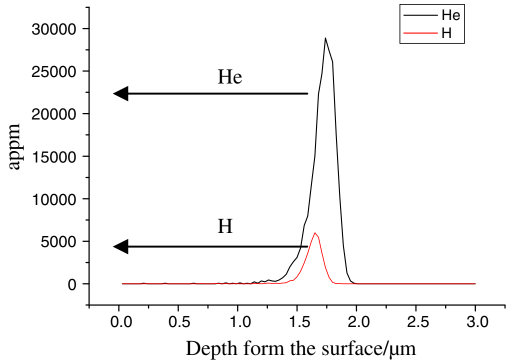


Fig. 3. The depth distribution of the H, He in SiC calculated with TRIM-code.

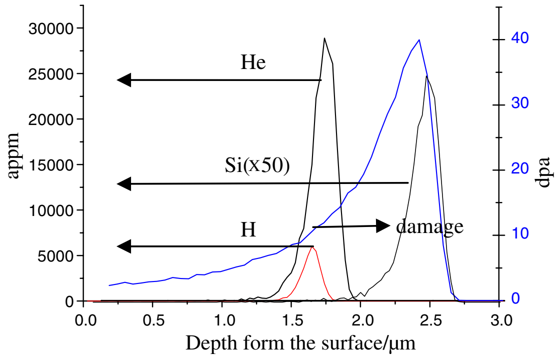


Fig. 4. The depth distribution of the displacement, H and He in SiC calculated with TRIM-code.

## 3. Results and discussion

Fig. 5 shows TEM micrographs of the irradiated region in matrix and fiber following $dual_{Si–He}$-ion and $triple_{Si–He–H}$-ion irradiation at 800 °C to 10 dpa. Cavities were found mostly along grain boundaries. Cavities were observed in SiC matrix irradiated with dual-ion and triple-ion at 800 °C. However, in the Tyranno-SA SiC fiber, cavities were

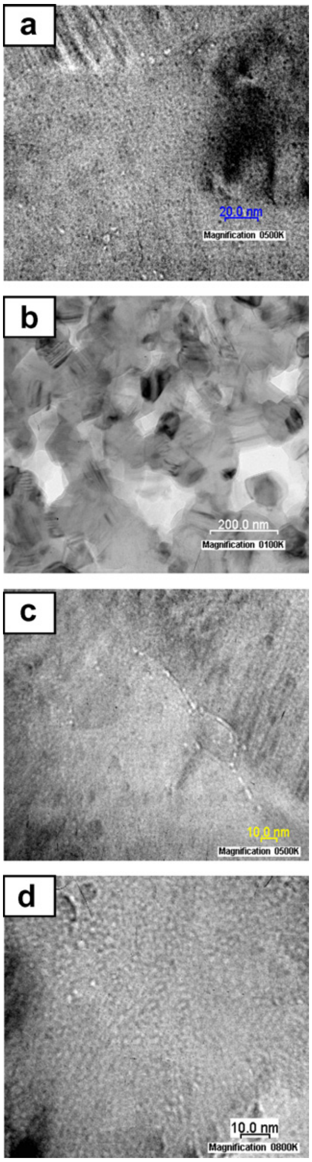


Fig. 5. TEM images of the multiple-ion irradiated region which show the distribution of bubbles. (a) matrix, and (b) fiber, after $dual_{Si–He}$-ion irradiation; (c) matrix, and (d) fiber, after triple-ion irradiation.

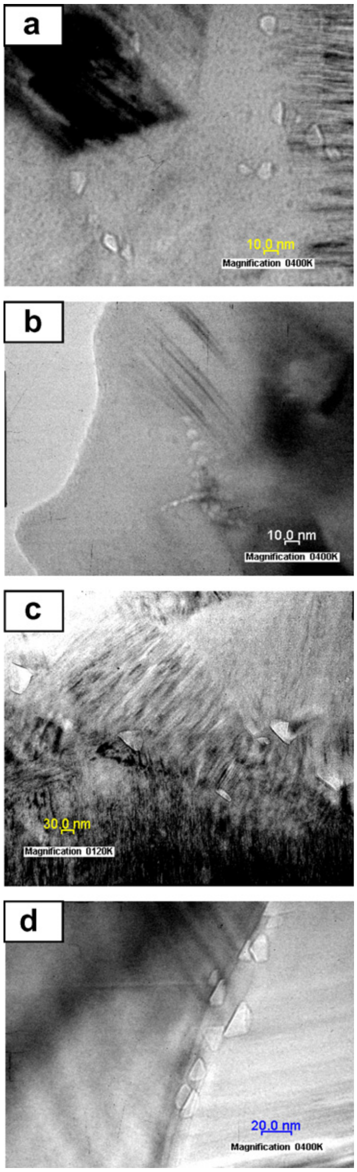


Fig. 6. TEM images of dual-ion irradiated SiC/SiC. (a) matrix, and (b) fiber, after 100 dpa irradiation at 800 °C; (c) matrix, and (d) fiber, after 100 dpa irradiation at 1000 °C.

observed only in the $triple_{Si–He–H}$-ion-irradiated sample. The average cavity size in the SiC matrix after triple-ion beam irradiation is about 1.0 nm. In the $dual_{Si–He}$-ion irradiation experiment, the average size of cavities is 1.2 nm. The size of cavities in matrix is almost the same in both irradiation conditions, but the cavity density is higher in the $triple_{Si–He–H}$-ion irradiation case.

Fig. 6 shows cavities in matrix and fiber of $SiC_f$/SiC after $dual_{Si–He}$-ion irradiation to 100 dpa at 800 °C and 1000 °C, respectively. The average diameter of cavities in matrix and fiber after irradiation to100 dpa at 1000 °C were much larger than those at 800 °C. This result indicates that higher irradiation temperature enhances the growth of cavities, accompanied with decrease in number density.

Comparing the $dual_{Si–He}$-ion beam irradiation experiment results of 100 dpa and 10 dpa at 800 °C, cavities were found only in the Tyranno-SA fiber in the specimen irradiated to 100 dpa.

Higher dose and helium concentration enhances cavity nucleation in the Tyranno-SA fiber.

Hydrogen atom diffuses much faster than helium atoms in SiC matrix [6]. Therefore, hydrogen atoms can enhance cavity formation at lower irradiation temperature. The experiments with dual$_{He–H}$-ion beams at 800 °C and 1000 °C with 15000 appm/6000 appm were performed. Fig. 7 shows microstructure in matrix and fiber of Tyranno-SA $SiC_f$/SiC composites after dual$_{He–H}$-ion beam at 800 °C and 1000 °C. Comparison between present work and previous work [7] indicates that cavities could form at much lower temperatures (800 °C) in the dual$_{He–H}$-ion implanted SiC matrix than the specimen implanted with He-ion alone (1200 °C).

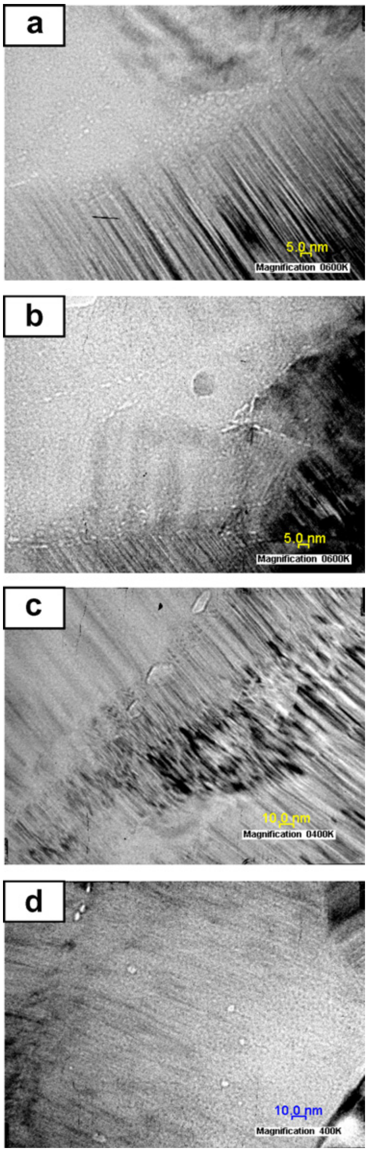


Fig. 7. TEM images of dual$_{He–H}$-ion implanted region of SiC (a) matrix and (b) fiber after dual$_{He–H}$-ion beam at 800 °C (c) matrix and (d) fiber after dual$_{He–H}$-ion beam at 1000 °C.

Table 1
Summary of cavity size and number density of Tyranno-SA/SiC irradiated at 800 °C and 1000 °C

| Conditions | Size | | Density | |
|---|---|---|---|---|
| | Mat | Fib | Mat | Fib |
| Dual$_{Si–He}$/10 dpa/800 °C | 1.2 | X | 0.85 | X |
| Triple$_{Si–He–H}$/10 dpa/800 °C | 1 | 0.85 | 1.2 | 3 |
| Dual$_{Si–He}$/100 dpa/800 °C | 9 | 5 | 0.26 | 0.45 |
| Dual$_{Si–He}$/100 dpa/1000 °C | 37 | 15 | 0.14 | 0.27 |
| Dual$_{He–H}$/15 K-6 K appm/800 °C | 2 | 1 | 3.4 | 5.6 |
| Dual$_{He–H}$/15 K-6 K appm/1000 °C | 10 | 2 | 0.9 | 2.7 |
| Single$_{He}$/10 K appm/1200 °C[a] | 3 | X | N | X |

Size, nm; density, $\times 10^{22}$(/m$^3$); X, no cavity; N, not investigated.
[a] J. Nucl. Mater. 329–333 (2004) 518.

Table 1 summarizes the average size and number density of cavities for Tyranno-SA $SiC_f$/SiC irradiated with multiple-ion beams in different conditions. From these data, it is suggested that hydrogen atoms play an important role in nucleation of cavities. Hydrogen atoms could diffuse in SiC toward grain boundaries much faster than He atoms at 800 °C. High concentration of hydrogen at grain boundaries enhances the nucleation of cavities. Actually, cavities were found in the Tyranno-SA SiC fiber after dual$_{He–H}$-ion irradiation but not found after single$_{He}$-ion irradiation [7], and in Tyranno-SA SiC fiber we found a high density of small cavities in the triple$_{Si–He–H}$-ion irradiated sample but not following dual$_{Si–He}$-ion irradiation. This observation is similar to the results of Tauguchi et al. [8].

The average size of cavities in the Tyranno-SA $SiC_f$/SiC irradiated at 1000 °C was much larger than that at 800 °C. This is probably due to the greater diffusivity of helium and hydrogen atoms at 1000 °C. Helium and hydrogen atom can diffuse to grain boundaries, leading to enhancement of the nucleation and growth of cavities. This could result in increase in average size and decrease in number density of cavities. There were no cavities found in the amorphous carbon interlayer which is attributed to faster diffusion rates of He and H atoms in this layer and the cavity embryos may not be able to grow into nuclei in this region.

## 4. Summary

The microstructure development of cavity formation in Tyranno-SA $SiC_f$/SiC composite after multiple-ion irradiation up to 10 and 100 dpa at 800 and 1000 °C was investigated by TEM. The following results were obtained:

(1) Cavities were found mainly on the grain boundary of $SiC_f$/SiC composites. Grain boundaries are preferred as nucleation sites for cavity formation both in Tyranno-SA SiC fiber and SiC matrix.
(2) Hydrogen atoms in the $SiC_f$/SiC composites will enhance cavity nucleation due to its high diffusivity in this material.
(3) Comparison between the results of $dual_{He–H}$-ion beam and $dual_{Si–He}$-ion beam irradiation to 100 dpa at 800 °C and 1000 °C, showed cavity coarsening effects after higher temperature irradiation.
(4) Cavities were found in the Tyranno-SA fiber after $triple_{Si–He–H}$-ion beam (10 dpa) at 800 °C.
(5) The cavity density in the Tyranno-SA SiC fiber is higher than in the SiC matrix. This is due to its smaller grain size in the fiber which offers many more possible cavity nucleation sites.

## Acknowledgement

This work is financially sponsored by the ROC National Science Council under the Project No. NSC 93-2212-E-007-021.

## References

[1] L.L. Snead, R.H. Jones, A. Kohyama, P. Fenici, J. Nucl. Mater. 233–237 (1996) 26.
[2] T. Nozawa, T. Hinoki, Y. Katoh, A. Kohyama, J. Nucl. Mater. 307–311 (2002) 1173.
[3] T. Hinoki, L.L. Snead, Y. Katoh, A. Hasegawa, T. Nozawa, A. Kohyama, J. Nucl. Mater. 307–311 (2001) 1157.
[4] Y. Yamauchi, Y. Hirohata, T. Hino, Fus. Eng. Des. 39&40 (1998) 427.
[5] K. Hojou, S. Furuno, K.N. Kushita, H. Otsu, K. Izui, Nucl. Instrum. and Meth. B 91 (1994) 534.
[6] G.A. Esteban, A. Perujo, F. Legarda, L.A. Sedano, B. Riccardi, J. Nucl. Mater. 307–311 (2002) 1430.
[7] T.S. Duh, K.M. Yin, J.Y. Yan, P.C. Fang, C.W. Chen, J.J. Kai, F.R. Chen, Y. Katoh, A. Kohyama, J. Nucl. Mater. 329–333 (2004) 518.
[8] T. Tauguchi, N. Igawa, S. Miwa, E. Wakai, S. Jitsukawa, L.L. Snead, A. Hasegawa, J. Nucl. Mater. 355 (2004) 508.