

# Current Status of SiC<sub>f</sub>/SiC Composites Material in Fusion Reactor

Han-ki Yoon\*<sup>1</sup> and Sang-pill Lee<sup>1</sup>

<sup>1</sup>Department of Mechanical Engineering, Dong-eui University, 995 Eomgwangno, Busanjin-gu, Busan, 614-714, Korea

[hkyoon@deu.ac.kr](mailto:hkyoon@deu.ac.kr)

## Abstract

The characterization of monolithic SiC and SiC<sub>f</sub>/SiC composite materials fabricated by NITE and RS processes was investigated in conjunction with the detailed analysis of their microstructure and density. The NITE-SiC based materials were fabricated, using a SiC powder with average size of 30 nm. RS- SiC<sub>f</sub>/SiC composites were fabricated with a complex slurry of C and SiC powder. In the RS process, the average size of starting SiC particle and the blending ratio of C/SiC powder were 0.4 μm and 0.4, respectively. The reinforcing materials for /SiC composites were BN-SiC coated Hi-Nicalon SiC fiber, unidirectional or plain woven Tyranno SA SiC fiber. The characterization of all materials was examined by the means of SEM, EDS and three point bending test. The density of NITE-SiC<sub>f</sub>/SiC composite increased with increasing the pressure holding time. RS-SiC<sub>f</sub>/SiC composites represented a great decrease of flexural strength at the temperature of 1000 °C.

## INTRODUCTION

The development of high temperature materials in the fusion power plant have been extensively studied for the production of main parts such as divertor, first wall and coolant channel. [1,2] SiC fiber reinforced SiC matrix composites (SiC<sub>f</sub>/SiC) are recognized as promising candidates for the blanket module in the gas cooling reactor, because it has excellent mechanical or physical properties such as high temperature strength, fracture toughness, thermal conductivity and irradiation resistance. In the last decade, the many research groups made an effort for the fabrication of SiC<sub>f</sub>/SiC composites and their property evaluation. Both the reaction sintering (RS) and the liquid phase sintering (LPS) processes are recognized as attractive methods for making high density SiC<sub>f</sub>/SiC composites with good thermal conductivity. In the RS process, the molten silicon remains as residual silicon phases, because it is easy to fill the pores or

voids dispersed in the porous preform. [3,4] The LPS process, which associated with the transformation of additive materials into the some eutectoids between starting particles, can provide a high density and an excellent crystallinity for the matrix of SiC<sub>f</sub>/SiC composites. [5-7] The LPS process generally performs at the sintering temperature lower than that of solid state sintering, owing to the eutectic temperature of additive materials. However, the performance of LPS-SiC materials is greatly affected by the type or compositional ratio of additive materials and the size of starting powder. Recently, the control of SiC grain size is tried to improve the mechanical properties of monolithic SiC and SiC<sub>f</sub>/SiC composite materials. Especially, the NITE (Nano-Infiltration Transient-Eutectic Phase) process, which means LPS process by the induction of nanosize SiC powder, is used for the fabrication of SiC based materials. [8]

The purpose of present study is to investigate the efficiency of NITE process for monolithic SiC and SiC<sub>f</sub>/SiC composite materials with nanosize SiC particles. The high temperature strength of RS-SiC<sub>f</sub>/SiC composites was also examined, in conjunction with the detailed analysis of their microstructure.

## Experimental

The SiC powder (Marketch International Inc., USA) with average size of about 30 nm was used for NITE-SiC based materials. The typical composition of this nano-powder is as follows: SiC > 95%, Free C 1~2% and O 1~1.5%. The mixture of Al<sub>2</sub>O<sub>3</sub> (High Purity Chemical, Japan) and Y<sub>2</sub>O<sub>3</sub> (High Purity Chemical, Japan) particles was used as a sintering additives. The blending ratio of Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> powder for the NITE process was fixed as 1.5. The mixed powder of SiC, Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> particles was infiltrated into the fabrics of unidirectional or plain woven Tyranno SA SiC fiber. The NITE-SiC based materials were sintered by hot pressing in argon atmosphere. The Tyranno SA SiC fiber was coated with the carbon layer thickness of 0.25 μm. The NITE-SiC and NITE-SiC<sub>f</sub>/SiC composite materials were fabricated at the temperatures of 1780 °C and 1900 °C under an applied pressure of 20 MPa for 60 min, respectively. The holding time for the NITE-SiC materials was also changed up to 30 min. The volume ratio of Tyranno SA SiC fiber was constant as 15% in the composite system.

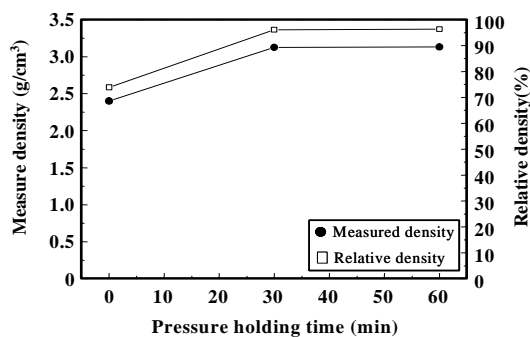
RS-SiC<sub>f</sub>/SiC composites were fabricated by the infiltration of molten Si into the porous perform of fabric structure. The fabricating temperature and holding time for the infiltration of molten Si were 1450 °C and 2 hours in vacuum atmosphere, respectively. The porous preform was prepared by injecting C/SiC complex slurry into a braiding fabric of BN/SiC coated Hi-Nicalon SiC fiber. The volume fraction of SiC fiber in the

composite system was fixed as 30 %. The complex slurry was a mixture of SiC powder, carbon powder and water. The thickness of BN and SiC layers was about 0.7  $\mu\text{m}$ , respectively. Average sizes of commercial SiC and carbon particles were 4  $\mu\text{m}$  and 85 nm, respectively. The compositional ratio of carbon and SiC particles was fixed at 0.4 wt %.

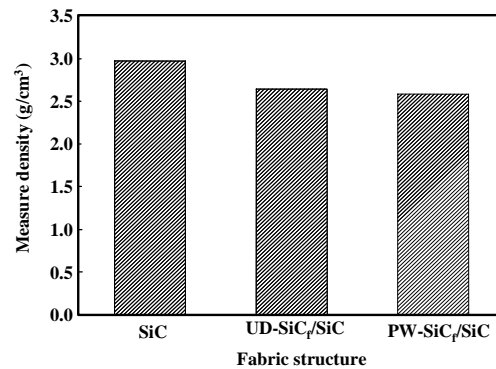
In order to examine the correlation between mechanical property and microstructure, the microstructure constituents of all materials were analyzed using SEM with EDS. The surface of test sample for the microstructure observation was etched, after the mechanical polishing by diamond powders. The density of all materials was determined by the Archimedes' method. The mechanical properties of RS-SiC<sub>f</sub>/SiC composites were investigated by three point bend tests. The three point bend test was carried out at elevated temperatures (25  $^{\circ}\text{C}$ , 1000  $^{\circ}\text{C}$ , 1100  $^{\circ}\text{C}$  and 1300  $^{\circ}\text{C}$ . The test sample was heated for about 4 hours up to each temperature before three point bend tests. The dimension of test samples was 1(t) $\times$ 4 $\times$ 25 mm<sup>3</sup>. The span length and the crosshead speed for the three point bend test were 18 mm and 0.5 mm/min, respectively.

## Results and discussion

Fig. 1 shows the sintered density of NITE-SiC materials fabricated with a different holding time of applied pressure in the hot press process. The relative density of LPS-SiC materials, which was determined as a ratio of measured density to theoretical density, was also shown in this figure. The sintering temperature of NITE-SiC materials was 1900  $^{\circ}\text{C}$ . The induction of applied pressure was effective for the improvement of sintered density for NITE-SiC materials. However, NITE-SiC materials represented a similar density level beyond the holding time of 30 min. NITE-SiC materials had a high density of about 3.1 g/cm<sup>3</sup>, which corresponds to about 95 % of theoretical density,



*Fig. 1 Sintered density of NITE-SiC materials fabricated with a different holding time of applied*



*Fig. 2 Density of NITE-SiC/SiC composites with a different fabric structure*

when fabricated at the pressure holding time of 60 min.

Fig. 2 showed the sintered density of NITE-SiC<sub>f</sub>/SiC composites with a different fabric structure. NITE-SiC<sub>f</sub>/SiC composites were fabricated at the temperature of 1780 °C under an applied pressure of 20 MPa for 60 min. The sintered density of NITE-SiC materials was also shown in this figure. NITE-SiC<sub>f</sub>/SiC composites represented an average density of about 2.6 g/cm<sup>3</sup>, regardless of fabric structure. The density of NITE-SiC materials greatly decreased with the induction of SiC fabrics. This is due to the insufficient formation of SiC matrix in the intra-fiber bundle region. Especially, in the case of plain woven fabric structure, it is very difficult to create the matrix at the cross section of fiber bundle.

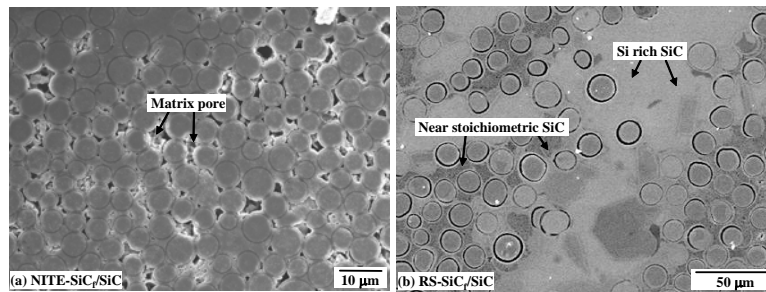


Fig. 3 Microstructure of NITE-SiC<sub>f</sub>/SiC and RS-SiC<sub>f</sub>/SiC composites.

Fig. 3 shows the intra-fiber bundle microstructure of NITE-SiC<sub>f</sub>/SiC and RS-SiC<sub>f</sub>/SiC composites. NITE-SiC<sub>f</sub>/SiC composites were fabricated with plain woven fabric structure. NITE-SiC<sub>f</sub>/SiC composites exhibited large amount of matrix pores around SiC fiber in the microstructure. Such a sintering defect is considered as another factor to

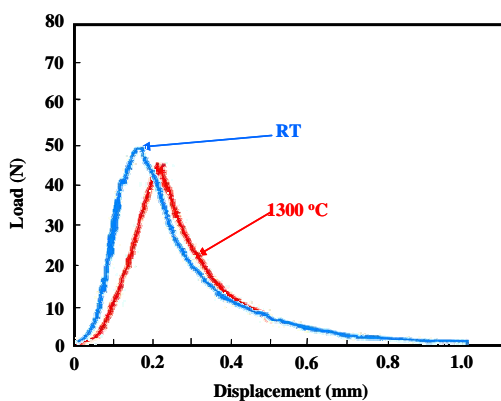


Fig. 4 Representative load-displacement curve of SiC<sub>f</sub>/SiC composites obtained from the three point bending test.

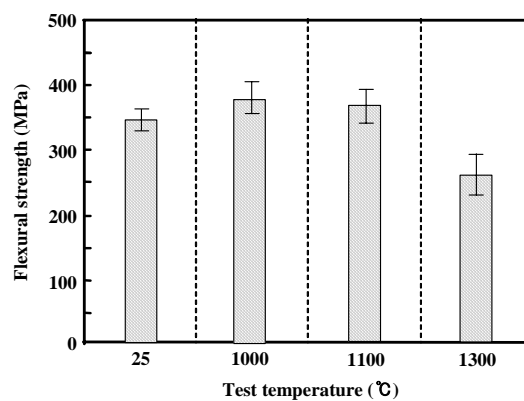


Fig. 5 Effect of test temperature on the average flexural strength of RS-SiC<sub>f</sub>/SiC composites

decrease the sintered density. (See Fig. 2) On the contrary, RS-SiC<sub>f</sub>/SiC composite showed a dense morphology without matrix cracks or shrinkages, even if there was severe deformation of SiC fibers. This is because the molten Si easily flows between SiC fibers during RS process, and then fills large matrix pores and openings. RS-SiC<sub>f</sub>/SiC composites also had a good density of about 2.8 Mg/m<sup>3</sup>. However, from the results of EDS analysis, the matrix of RS-SiC<sub>f</sub>/SiC composite was mainly composed of two types of SiC phases. In other words, a large amount of Si rich SiC with a Si/C ratio of about 1.7 was formed in the matrix region, compared to that of near stoichiometric SiC with a Si/C ratio of about 0.9. Such a chemical fluctuation in the matrix is due to the fine dispersion of unreacted C and residual Si phases. [3]

Fig. 4 shows the representative load-displacement curve of RS-SiC<sub>f</sub>/SiC composites obtained from the three point bending test. The maximum load and the fractured displacement of RS-SiC<sub>f</sub>/SiC composites also tended to decreased at the test temperature of 1300 °C. However, RS-SiC<sub>f</sub>/SiC composites clearly represented a pseudo fracture behavior with stable crack propagation beyond the maximum load, regardless of test temperatures. Such a fracture behavior is related to the variation of the crack propagation path. In other words, the crack propagation path was greatly changed by the creation of interfacial delamination and fiber pull-out, since the crack continuously reinitiates on the tensile surface of subsequent fibers after the crack is arrested at the interface.

Fig. 5 shows the effect of test temperature on the average flexural strength of RS-SiC<sub>f</sub>/SiC composites. The flexural strength of RS-SiC<sub>f</sub>/SiC composites greatly decreased at test temperatures higher than 1100 °C, even if it retained up to the test temperature of 1000 °C. RS-SiC<sub>f</sub>/SiC composites represented the flexural strength of about 330 MPa at the room temperature. On the other hand, RS-SiC<sub>f</sub>/SiC composites had the flexural strength of about 260 MPa at the test temperature of 1300 °C. This is due to the damages of SiC matrix and interfacial coating layer by the thermal exposure history corresponding to the duration of three point bend tests. The strength of RS-SiC<sub>f</sub>/SiC composites is also affected by the degradation of Hi-Nicalon SiC fiber, since the Hi-Nicalon SiC fiber generally leads to a strength reduction at the temperature higher than 1000 °C.

## SUMMARY

(1) NITE-SiC materials represented a good density of about 3.1 g/cm<sup>3</sup>, at the sintering temperature of 1900 °C, which corresponds to about 95 % of theoretical density. On the

contrary, NITE-SiC<sub>f</sub>/SiC composites represented a low density of about 2.6 g/cm<sup>3</sup>, due to insufficient formation of SiC matrix in the intra-fiber bundle region.

(2) RS-SiC<sub>f</sub>/SiC composites exhibited large amount of Si rich SiC phase with a Si/C ratio of about 1.7 in the intra-fiber bundle region, even if it had a good morphology with a density of about 2.8 Mg/m<sup>3</sup>.

(3) The flexural strength of MI-SiC/SiC composites showed about 330 MPa at the room temperature, accompanying a pseudo fracture behavior. Such a strength level decreased with the increase of test temperatures, due to the degradation of Hi-Nicalon SiC fiber and the damage of SiC matrix and interfacial coating layer.

## REFERENCES

- [1] B. Riccardi, L. Giancarli, A. Hasegawa, Y. Katoh, A. Kohyama, R.H. Jones and L.L. Snead, Issue and advances in SiC/SiC composites development for fusion reactors, J. Nucl. Mater. Vols. 329-333, pp.56-65 (2004).
- [2] A. Kohyama, M. Seki, K. Abe, T. Muroga, H. Matsui, S. Jitsukawa and S. Matsuda, "Interactions between fusion materials R&D and other technologies", J. Nucl. Mater. Vols. 283-287, pp.20-27 (2000).
- [3] S. P. Lee, Y. Katoh, J. S. Park, S. M. Dong, A. Kohyama, S. Suyama and H. K. Yoon, "Microstructural and Mechanical Characteristics of SiC/SiC Composites with modified-RS process", J. Nucl. Mater. Vols. 289, pp.30-36 (2001).
- [4] S. Suyama, Y. Itoh, A. Kohyam and Y. Katoh, "Effect of residual silicon phase on reaction-sintered silicon carbide", Ceramic Transactions, Vol. 144, pp.181-185 (2002).
- [5] H. K. Yoon, H. C. Jung, "Microstructure Effect on Bending Strength Characteristics of LPS-SiC Ceramic", Journal of Ocean Engineering and Technology, No. 20, Vol. 5, pp. 77-81 (2006).
- [6] S. P. Lee, Y. S. Shin, B. H. Min, D. S. Bae, J. S. Park and A. Kohyama, "Fabrication of liquid phase sintered SiC based materials and their characterization", Fusion Engineering and Design, Vol. 81, pp.963-967 (2006).
- [7] J. S. Park, Y. Katoh, A. Kohyama, J. K. Lee, J. J. Sha and H. K. Yoon, Tailoring the microstructure of hot-pressed SiC by heat treatment, J. Nucl. Mater. Vols. 329-333, pp.558-561 (2004).
- [8] Y. Katoh, A. Kohyama, T. Nozawa and M. Sato, SiC/SiC composites through transient eutectic-phase route for fusion applications, J. Nucl. Mater, Vols. 329-333, PP. 587-591 (2004).