Effect of SiC Nanorods on Mechanical and Thermal Properties of SiC Composites Fabricated by Chemical Vapor Infiltration

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ABSTRACT

To reduce residual pores of composites and obtain a dense matrix, SiC_f/SiC composites were fabricated by chemical vapor deposition (CVI) using SiC nanorods. SiC nanorods were uniformly grown in the thickness direction of the composite preform when the reaction pressure was maintained at 50 torr or 100 torr at 1,100°C. When SiC nanorods were grown, the densities of the composites were 2.57 ~ 2.65 g/cm³, higher than that of the composite density of 2.47 g/cm³ for non-growing of SiC nanorods under the same conditions; grown nanorods had uniform microstructure with reduced large pores between bundles. The flexural strength, fracture toughness and thermal conductivity (room temperature) of the SiC nanorod grown composites were 412 ~ 432 MPa, 13.79 ~ 14.94 MPa m^{1/2} and 11.51 ~ 11.89 W/m·K, which were increases of 30%, 25%, and 25% compared to the untreated composite, respectively.

Key words : SiC_f/SiC, SiC nanorod, Densification, Mechanical property, Thermal property

1. Introduction

SiC-based ceramics are considered as materials for high temperature parts of gas turbines or core parts of reactor cores that require high temperature structural properties due to their excellent mechanical and thermal properties, chemical stability at high temperature, and good neutron radiation resistance.¹⁻⁴⁾ Monolithic SiC, however, has brittle fracture behavior, limiting its use as a construction material. To overcome this shortcoming, Continuous Fiber Reinforced Ceramic Composites (CFCCs) have been developed.⁵⁻⁶⁾ SiC fiber-reinforced SiC-matrix (SiC_f/SiC) composites are mainly fabricated using the Chemical Vapor Infiltration (CVI) method,⁷⁻⁸⁾ the Si melt infiltration method,⁹⁾ the Polymer Impregnation and Pyrolysis (PIP) method,¹⁰⁾ hot pressing method,¹¹⁻¹³⁾ and hybrid processes that combine two of these methods.¹⁴⁻¹⁵⁾

SiC_f/SiC composites contain SiC fibers as reinforcing materials and SiC as a matrix phase, and both materials are known to exhibit excellent heat and radiation resistance when they contain β-SiC with an excellent stoichiometric ratio, a high-crystallinity phase, and small amounts of impurities. Among the various composite fabrication methods, the CVI method is advantageous in that it is possible to

fabricate SiC_f/SiC composites with a high-crystallinity matrix phase and excellent stoichiometric ratio. The CVI process performs at low temperatures and pressures, thus minimizing any damage that might occur to the fibers used. Accordingly, CVI-SiC_f/SiC composites have excellent heat resistance, radiation resistance, and mechanical properties. However, these composites contain residual large pores in them, which cause to degrade their mechanical and thermal properties.⁷⁾ To address this, new processes that use SiC whiskers or nanowires have been developed.¹⁶⁻¹⁸⁾

In this study, SiC nanorods grew on SiC fibers before filling the matrix to improve the residual pore problems in the CVI-SiC_f/SiC composites. An attempt to change the size and structure of large pores by growing SiC nanorods could be resulted in improving the behaviors of matrix filling. To this end, SiC nanorods were grown with reaction pressure and temperature to establish optimum growth conditions and to evaluate the effects on mechanical and thermal properties. In addition, a pyrolytic carbon (PyC) layer was further deposited on the SiC nanorods as a means of reinforcing the matrix phase, and the reinforcing effect of these coated SiC nanorods on the matrix phase was analyzed.

2. Experimental Procedure

In this study, a plain-woven SiC fabric of Tyranno-SA3 (PSA-S17I16PX, Ube Ltd., Japan) was used, and preforms were made by stacking 15 sheets of disc-shaped SiC fabric with a diameter of 2 inches in 0° and 90° directions. At the interface of the fiber and matrix, pyrolytic carbon was

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Table 1. Growth Conditions of SiC Nanorods

Specimen	Growth Conditions	Temperature (°C)	Pressure (torr)	Time (h)
WG 001		1100	10	5
WG 002		1100	50	5
WG 003		1100	100	5
WG 004		1000	50	5
WG 005		1200	50	5
WG 006		1100	25	5

deposited at 1,100°C and 90 torr in a chemical gas reactor using CH₄ as the raw material gas. The thickness of the PyC layer was set to 150–200 nm, within which its mechanical properties are known to be excellent.¹⁹⁾

SiC nanorods must be uniformly widely distributed throughout the fiber and grow thin and long to aid in subsequent chemical vapor infiltration processes. The present study attempted to determine the optimal conditions for the SiC nanorods to grow, with the reaction pressure and temperature set as process variables. Test conditions for the SiC nanorod growth experiment, along with the specimen names used, are summarized in Table 1. In addition, after depositing a pyrolytic carbon layer on the SiC nanorod, an experiment was conducted to investigate the effect of enhancing fracture toughness by the interface layers between nanorods and matrix. CH₃SiCl₃ (MTS: Methyltrichlorosilane, SIGMA-ALDRICH) was used as a raw material for the infiltration process because it is an organometallic compound that can be easily used to stoichiometrically form the deposition layer, given that the content ratio of Si to C is 1:1. The deposition process was performed at 1,000°C for 50 h.

A scanning electron microscope (Sirion, Fei, USA) was used to observe the microstructure of the fabricated composites at each stage so that the matrix-phase filling behavior could be examined, especially with regards to the level of SiC nanorod growth, the degree of densification of the matrix phase, and damage behavior of the fibers and nanorods at the fracture surfaces. The uniformity of nanorod growth was assessed based on the diameter of the SiC nanorods at the inlet and outlet regions for reaction gases, measured at more than 20 spots and averaged using microstructure images and an image analyzer. After the reaction, a phase analysis of the SiC nanorods and the matrix phase was conducted using an X-ray diffractometer (X-Ray Diffraction: XRD, SmartLab, Rigaku, Japan) with a scan range of 20°–80°, a scan speed of 5°/min, a step size of 0.02°, and a power of 45 kV 200 mA.

Also, to assess the effect of the SiC nanorods on the properties of the SiC_f/SiC composites, the flexural strength, fracture toughness, specific heat, and thermal diffusivity were measured. To this end, two types of specimens with different SiC nanorod growth conditions, as well as specimens in

which SiC nanorods were not formed, were prepared. These disk-shaped specimens, with a diameter of 6 mm and a thickness of 1 mm, were used to measure thermal diffusivity with a laser flash analyzer (LFA 467, NETZSCH, Germany). Also, a differential scanning calorimeter (DSC204 F1 Phoenix, NETZSCH, Germany) was used to measure the specific heat in the range from room temperature to 300°C. The thermal conductivity was obtained using Eq. (1) below.

$$K = \alpha \times \rho \times C_p \quad (1)$$

Here, K = Thermal conductivity

α = Thermal diffusivity coefficient

ρ = Density

C_p = Specific heat

A universal testing machine (UTS, Instron 4465, USA) was used to measure the mechanical properties; the three-point flexural strength was measured, and the fracture toughness was determined using a single edge V-notch beam and Eq. (2) and (3). The width, length, and breadth of the specimen were 30 mm × 3 mm × 3 mm with a tolerance of ± 0.1 mm.

$$K_{IC} = 3 P (S - S_0) \alpha^{1/2} Y / BW^{1.5} * 2 (1 - \alpha)^{1.5} \quad (2)$$

$$Y = 1.9887 - 1.326(a/W) - (3.49 - 0.68(a/w) + 1.35(a/W)^2) \alpha(1 - \alpha)(1 + \alpha)^{-2} \quad (3)$$

Where, P = max load

S = Outer span

S₀ = Inner span

A = Crack length

$\alpha = a / W$

W = Width of the specimen

B = Thickness of the specimen

3. Results and Discussion

3.1. Growth Behavior of SiC Nanorods

SiC nanorods were grown during five hours of reaction

Fig. 1. XRD patterns of SiC preforms containing SiC nanorods grown under different conditions.

Fig. 2. Microstructures of SiC preforms containing SiC nanorods grown at 1,100°C under different pressures: (a) 10 torr (WG 001) (b) 25 torr (WG 006) (c) 50 torr (WG 002) (d) 100 torr (WG 003).

with the reaction pressure and temperature set as process variables. The phase analysis results of the SiC nanorods are presented in Fig. 1. As shown in these XRD analysis results, at reaction conditions of 1,000–1,200°C and 10–100 torr, only β -phase SiC was formed and grown regardless of the reaction pressure and temperature. Also, a low-intensity peak which was not observed in the SiC preform specimen in which SiC nanorods had not been formed, was found to exist at a diffraction angle lower than that of the (111) peak (labeled as s.f. in Fig. 1). This peak is associated with stacking faults and frequently observed in materials with a large aspect ratio, such as SiC nanorods, nanowires, and whiskers.^{20,21} This type of peak is known to occur by the SiC nanorods grow in the [111] direction because of the low surface energy of the {111} plane, and easy insert of the stacking fault in the [111] plane perpendicular to the growth direction. Stacking fault formation of nanowires or whiskers by CVD has been reported in previous studies and has also been reported in whisker growth by solid-gas reactions such as carbothermal reactions.^{19,22–24} As can be seen in the microstructure of Fig. 2, the SiC nanorods grow evenly in the radial direction on the surface of the SiC fiber. The growth behavior of the SiC nanorods was investigated with respect to the varying reaction pressure at the fixed reaction temperature, and with respect to the varying reaction temperature at the fixed reaction pressure, respectively. The quantitative criteria for determining the adequacy of SiC nanorod growth are uncertain. The growth of SiC nanorods in the preform can increase the SiC deposition efficiency in

a matrix fill process. However, rapid deposition may cause clogging effects, making it difficult for the reaction gas to penetrate into the preform, thereby leaving the matrix filling process with large pores. In addition, it is difficult to obtain a uniform microstructure in the thickness direction of the specimen during the SiC nanorod growth and matrix-phase filling process when the reaction conditions are different around the specimen at the reactant feed inlet and outlet portions. In the present study, the Forced-CVI process was applied, which improves the efficiency of matrix-phase filling, using the pressure difference between the inlet and outlet of the reaction gases. Given the characteristics of the process, the two regions cannot have uniform reaction conditions. Accordingly, the formed SiC nanorods varied in characteristics, and an attempt was made to find the optimal reaction conditions to minimize such non-uniformity.

To this end, the difference in diameters of SiC nanorods grown in the inlet and outlet regions of the reactor was analyzed using an image analyzer to compare the uniformity in a relative manner. Fig. 2 is the microstructure of SiC nanorods grown at 1,100°C with the reaction pressure to 10, 25, 50, and 100 torr, respectively, and Fig. 3 is the comparison of the differences between the average diameters of nanorods grown in inlet and outlet region. At reaction pressures of 10 and 25 torr, the average diameter difference of SiC nanorods between the inlet and the outlet regions was relatively large as 3311 nm and 3695 nm, respectively, while, at pressures of 50 and 100 torr, the average diameter difference was small as 94 nm and 466 nm, respectively.

Fig. 3. Comparison of diameters of SiC nanorods grown at 1,100°C with different reaction pressures in gas inlet area and gas outlet area.

Also, the average diameter of the SiC nanorods was found to be smaller by 1/10 to 1/7. Thus, it was thought that the matrix-phase filling process could be effectively performed by setting the reaction pressure to 50 or 100 torr.

On the other hand, the growth behavior of the SiC nanorods was observed while varying the temperature from 1,000°C to 1,100°C and 1,200°C at the fixed deposition pressure of 50 torr. Fig. 4 shows the microstructure of SiC nanorods grown with reaction temperature. In Fig. 5, the difference between the average diameters of SiC nanorods grown on the preform surfaces of the reactor inlet and outlet regions was compared.

At 1,000°C (Fig. 4(a) and (b)) and 1,200°C (Fig. 4(e) and (f)), the microstructure showed non-uniformity near the path along which the nanorods were formed and grown. The average diameter difference between the inlet and outlet SiC nanorods was relatively larger than was observed at

Fig. 4. Microstructures of SiC preforms containing SiC nanorods grown at different temperatures: (a), (b) 1000°C (WG 004), (c), (d) 1100°C (WG 002), and (e), (f) 1200°C (WG 006).

Fig. 5. Comparison of diameters of SiC nanorods grown at different temperatures at 50 torr in gas inlet area and gas outlet area.

1,100°C, (i.e., 94 nm at 1,100°C; 4312 nm at 1,000°C; 2404 nm at 1,200°C). As a result, the optimal growth conditions for SiC nanorods were determined, as follows: a reaction temperature of 1,100°C with reaction pressures of 50 and 100 torr.

3.2. SiC Matrix Filling and Property Evaluations

The matrix filling process was performed at 1,000°C for 50 h in both the preform specimens without SiC nanorods formed, and the specimens in which SiC nanorods were formed at 1,100°C and 50 torr (or 100 torr). For some of the preform specimens that contained SiC nanorods, the nanorods were additionally coated with PyC, and the reinforcing effect of the coating was examined. The density of the final SiC_f/SiC composite was 2.47 g/cm³ without growing nanorods, and 2.65 g/cm³ and 2.57 g/cm³ with growing nanorods and PyC-coated nanorods, respectively. Fig. 6 shows the microstructure of the vertical cross-section of these composites. Fig. 6(a) indicates that the composite specimens without any nanorods ended up with large pores between bundles that were left unfilled by the matrix phase (indicated with arrow symbols). In the composite specimens with nanorods, however, fewer of these pores existed between bundles, and the matrix phase was uniformly formed with only a small number of tiny pores, as shown in Fig. 6(b) and (c). As similarly observed in previous studies that used SiC whiskers,^{19,25)} it was thought that SiC nanorods divided the large pores contained in the preform into smaller ones, and these newly formed small pores served as new deposition sites for the SiC matrix phase. This process was considered to have promoted the densification and uniformity of the composites. As shown in Fig. 7, the flexural strength of the composite specimens without any SiC nanorods was the lowest, at 328.74 MPa. When SiC nanorods were formed, the flexural strength of the composites ranged from 410 to 430 MPa, regardless of whether the nanorods had been coated with PyC. As mentioned regarding microstructure in Fig. 6, it

Fig. 6. Cross-sectional microstructures of SiC_f/SiC composites prepared at 50 torr and 1,100°C for 50 h: (a) non-nanorods, (b) nanorods and (c) PyC coated nanorods.

was thought that the formation of SiC nanorods resulted in increased density, fewer large pores, and relatively more uniform structures. These factors are considered to have increased the flexural strength of the composites. As shown in Fig. 8, the fracture toughness was 11.99 MPa·m^{1/2} when without SiC nanorods were formed, and the fracture toughness increased to 14.94 and 13.79 MPa·m^{1/2} when typical nanorods were formed and when PyC-coated nanorods were formed, respectively. However, the additional enhancement of fracture toughness expected by coating PyC on SiC

Fig. 7. Flexural strength of other types of SiC_f/SiC composites.

Fig. 8. Fracture toughness of other types of SiC_f/SiC Composites.

nanorods was not obviously observed in this study. This is ascribed to the fact that the PyC coating resulted in a decrease in both the density and uniformity of the microstructure. Fig. 9 presents the microstructure of the fracture surfaces of the composites fabricated using the preforms that contained PyC coated nanorods. Fig. 9(a) shows images of pulled-out SiC fibers and the pores formed due to these pulled-out fibers (indicated with arrow symbols), while Fig. 9(b) illustrates how cracks tend to deflect and propagate within the matrix phase (indicated with arrow symbols). However, such phenomenon as the pull out of whiskers on the matrix observed when whiskers were grown instead of nanorods was not observed in this study.²⁵⁾ Further microstructural analysis is needed to clarify whether such a phenomenon occurs in practice.

The thermal conductivity measurement was conducted on the composites without any SiC nanorods formed, the composites where typical SiC nanorods were formed at 50 torr, and the composites where PyC-coated SiC nanorods were

Fig. 9. Fracture surface of SiC_f/SiC composites with PyC coated nanorods showing (a) SiC fiber pull-out and (b) crack deflection (arrow marks).

Fig. 10. Specific heat of SiC_f/SiC composites measured by DSC.

formed. The specific heat was measured using a differential scanning calorimeter while increasing the temperature to 300°C, and the measured results are shown in Fig. 10. The thermal conductivity was calculated by inputting the thermal diffusivity (Fig. 11(a)) measured using the laser flash method, the measured specific heat, and the density in Eq. 1, and the results are presented in Fig. 11(b). It was found that the specific heat tended to increase with increasing

Fig. 11. Thermal diffusivity (a) and conductivity (b) of SiC/SiC composites.

temperature, but the variation was small; most measurements were similar regardless of the specimen preparation conditions applied. The thermal conductivity was found to range from 9.51 to 12.75 $\text{W/m}\cdot\text{K}$. This is similar to that reported elsewhere²⁶⁾ for 2D-SiC/SiC composites. The figure was the lowest when SiC nanorods were not formed and the highest when typical SiC nanorods were formed.

Balandin²⁷⁾ reported that the thermal conductivity of PyC coated layers exhibited anisotropy and thus were 10^3 higher in the in-plane direction than in the cross-plane direction; however, within the temperature range of 0–300°C, the thermal conductivity was hardly affected by the measurement temperature, when measured in the same in-plane direction. Notably, when measured in the cross-plane direction, the thermal conductivity was found to be 6–8 $\text{W/m}\cdot\text{K}$ within the temperature range from room temperature to 300°C, indicating that the figure was not significantly affected by the measurement temperature. The composites fabricated in the present study were two-dimensional structures of SiC fabric layers stacked on top of each other, and the thermal diffusivity was measured along the cross-plane direction of the PyC layers that were coated on the fibers or nanorods. This was considered to be the reason why the

thermal conductivity of the composites was not significantly affected by whether or not the SiC nanorods contained in them were coated with PyC. The thermal diffusivity of a composite is known to increase with increasing density,²⁸⁾ and a similar trend was observed in the present study, as shown in Fig. 11(a). As shown in Fig. 11(b), the thermal conductivity increased in the order of increasing density, i.e., from the composites without any nanorods formed to the composites where PyC-coated SiC nanorods were formed and further to the composites where typical nanorods were formed. In this regard, the observed improvement in the thermal conductivity was considered to be due to the effects of SiC nanorod formation, which increased the density and microstructural uniformity of the composites.

4. Conclusions

SiC nanorods were found to effectively improve the uniformity of preforms in the thickness direction when formed and grown at a temperature of 1,100°C and a pressure of 50 or 100 torr. The formation of SiC nanorods in SiC/SiC composites divided large pores into smaller ones, improved the matrix filling effect, and increased the density. The density of the composites was 2.47 g/cm^3 when no nanorods were formed. The figures were 2.57 g/cm^3 and 2.65 g/cm^3 when pyrolytic carbon (PyC)-coated nanorods were formed and when typical nanorods were formed, respectively. When SiC nanorods were formed the flexural strength of the composites was 412–432 MPa, and the fracture toughness was 13.79–14.94 $\text{MPa}\cdot\text{m}^{1/2}$. These figures were 30% and 25% higher than the flexural strength and fracture toughness of the composites without any SiC nanorods formed, respectively, when all other conditions were the same. In the present study, no reinforcing effect was observed by SiC nanorods coated with PyC. Further research is needed to clarify whether such an effect exists in practice. When SiC nanorods were formed, the room temperature thermal conductivity of the composites was 11.51 ~ 11.89 $\text{W/m}\cdot\text{K}$, and it increased up to 25% compared to the composite prepared without SiC nanorod growth under the same conditions. The specific heat of the composites was not significantly affected by the manufacturing conditions applied, while the thermal conductivity was considered to be affected by the effects of nanorod formation, which increased the density, reduced larger pores into smaller ones, and improved the microstructural uniformity.

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