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Microstructure and mechanical properties of silicon carbide fiber-reinforced silicon carbide composite fabricated by electrophoretic deposition and hot-pressing

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A B S T R A C T

Colloidal graphite aqueous solution was used as the suspension for carbon coating on the fiber, and carbon layer was formed on SiC fiber by electrophoretic method, and SiC/SiC composite was fabricated by sheet stacking method and hot-pressing. The effect of the concentration of colloidal graphite on mechanical properties of SiC/SiC composite was investigated. Bulk density and open porosity of the composites fabricated in this study were nearly the same, and these values were independent of the concentration of colloidal graphite suspension. The carbon coating on SiC fibers was successfully formed by the electrophoretic deposition method using colloidal graphite suspension. In the case of coating with 0.10mass% of colloidal graphite suspension on SiC fibers, relatively uniform carbon coating on the fibers was observed and large fiber pullout occurred effectively during fracture.

1. Introduction

Silicon carbide (SiC) is an attractive material for future fusion reactors since it has low induced radioactivity, quick decay of activity, low heat evolution after neutron irradiation, low atomic number, good fracture resistance, excellent high-temperature mechanical and thermal properties and corrosion resistance [1–6]. Furthermore, SiC shows good resistance to high-energy neutron irradiation up to very high neutron fluences [7–11]. However, monolithic SiC ceramics show a brittle fracture behavior and low fracture toughness, and the application of SiC ceramics as the components has been limited due to its low reliability. In order to overcome this problem, SiC fiber-reinforced SiC composite (SiC/SiC), which shows a non-brittle fracture behavior and higher fracture energy, has been studied. Future fusion power reactor concepts based on the use of SiC/SiC composite have been designed by JAERI (DREAM reactor) [12], ARIES team (ARIES-I, IV and AT) [13–15] and CEA (TAURO) [16].

The authors have explored a new fabrication process using sheet stacking method and hot-pressing in order to simplify the fabrication process and to obtain dense composite with higher mechanical and thermal properties [17–20]. The dense composite with a non-brittle fracture behavior could be obtained by this process. Formation of carbon or boron nitride layer on the fiber and the SiC matrix between each fiber filament is one of the most important factors to improve the mechanical properties of the composite. Recently, polycrystalline SiC fiber (Tyranno SA) with high strength and excellent heat resistance at high temperatures has been developed, and it shows electric conductivity due to its high crystallinity. We have paid attention to electric properties of polycrystalline SiC fiber, and electrophoretic deposition method has been applied for the formation of carbon layer on the fiber and the SiC matrix between each fiber filaments [21]. In our previous study, we have successfully achieved the formation of carbon layer on SiC fiber by electrophoretic deposition method using carbon black suspension [21].

In this study, colloidal graphite aqueous solution was used as the suspension for carbon coating on the fiber, and carbon layer was formed on SiC fiber by electrophoretic deposition method, and SiC/SiC composite was fabricated by sheet stacking method and hot-pressing. Microstructure and mechanical properties of SiC/SiC composites were evaluated, and the effect of the concentration of colloidal graphite on mechanical properties of SiC/SiC composite was investigated.

2. Experimental procedures

2.1. Preparation of carbon and SiC coated Tyranno SA cloth

Two dimensionally plain-woven Tyranno SA (SiC fiber, Ube Industries, Japan) cloth was used as the reinforcement. Sizing agent on the cloth was removed by hot water. The cloth was cut into the size of 35 × 35 mm square. The suspension of graphite particles was prepared using a colloidal graphite aqueous solution (Hitasol, Hitachi Powdered Metals, Japan), and the concentration of colloidal graphite was adjusted to 0.05, 0.10 and 0.50mass%. The pH of this suspension was adjusted to 10 using small amount of *n*-butylamine. The cloth and graphite plate were settled at a distance of 10 mm in the colloidal graphite suspension as the anode and the

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cathode, respectively. The cloth was coated with graphite by electrophoretic deposition under an applied voltage of 3 V for 10 min, and then dried at 100 °C. The 10mass% of β-SiC aqueous suspension (Ibiden, Japan) with sintering aids (20mass% in total) with α-Al_2O_3 (Taimei Chemicals, Japan), Y_2O_3 (High Purity Chemical, Japan) and CaO (Kanto Chemical, Japan) was also prepared using pure water and the pH of the suspension was adjusted to 10 using *n*-butylamine. The cloth coated with graphite was dipped into the SiC matrix component suspension, and electrophoretic deposition was performed under an applied voltage of 5 V for 20 min, and then the coated cloth was dried at 100 °C.

2.2. Fabrication of SiC green sheet

The slurry for tape casting was prepared using β-SiC powder, sintering aids (20mass% in total) using α-Al_2O_3, Y_2O_3 and CaO, and some organics. The SiC sheet was prepared using a laboratory-scale tape casting equipment (DP150, Tsugawa Seiki, Japan). Details of the composition, organics in the green sheet were described in Ref. [17]. The thickness of the SiC green sheet was approximately 40–50 μm. The SiC green sheet was cut into the size of 35 × 35 mm square.

2.3. Fabrication and evaluation of SiC/SiC composite

The cloths coated with carbon and the SiC matrix component, and the SiC green sheets were stacked alternately. The stacked body was heat-treated at 300 °C for 24 h in air under a uniaxial pressure of 20 kPa in order to remove organics such as binder from the green sheets. The compact was hot-pressed at 1700 °C for 1 h in Ar under a uniaxial pressure of 40 MPa. Specimens were cut into rectangular bars ($3\ mm^w \times 2\ mm^t \times 35\ mm^l$). Bulk density was measured by Archimedes' method. Three-point bending strength was measured at room temperature with a span of 30 mm and a crosshead speed of 0.1 mm/min. The number of test pieces in each specimen for bending test was 4–5. Bending strength was calculated from the maximum load for fracture. Microstructure and fracture surface of the composite were observed by a field emission scanning electron microscope (FE-SEM).

Table 1
Bulk density, open porosity and bending strength of SiC/SiC composites using SiC fibers coated with various concentration of colloidal graphite suspension.

The concentration of colloidal graphite suspension (mass%)	Bulk density (g/cm^3)	Open porosity (%)	Bending strength (MPa)
0.05	2.88	8.73	132
0.10	2.75	9.32	117
0.50	2.90	8.52	108

3. Results and discussion

The fiber volume fraction of SiC/SiC composite fabricated in this study was 48.8–59.5%. Table 1 lists bulk density and open porosity of SiC/SiC composites fabricated in this study. Bulk density and open porosity of the composites were 2.8–2.9 g/cm^3 and 8.5–9.3%, respectively. These values were nearly the same and independent of the concentration of colloidal graphite suspension. Fig. 1 shows SEM micrographs of the cross section of the SiC/SiC composites obtained in this study. In the case of the composites using SiC fibers coated with 0.05mass% and 0.10mass% of colloidal graphite suspension, carbon and SiC phases between SiC fibers were observed. However, only carbon phase was observed between SiC fibers in the composite using SiC fibers coated with 0.50mass% of colloidal graphite suspension. Most of pores existed between each fiber filament whereas the SiC matrix was so dense. Fig. 2 shows the typical load-crosshead displacement curves of SiC/SiC composites in bending test at room temperature, and bending strength of SiC/SiC composites is listed in Table 1. Average bending strength of the composite using SiC fibers coated with 0.05mass%, 0.10mass% and 0.50mass% of colloidal graphite suspension was 132, 117 and 108 MPa, respectively. Bending strength of the composite gradually decreased with an increase in the concentration of colloidal graphite suspension. The composite using SiC fibers coated with 0.05mass% of colloidal graphite suspension showed a stepwise fracture behavior. In the case of the composite using SiC fibers coated with 0.10mass% of colloidal graphite suspension, the load gradually decreased with increasing the crosshead displacement after reaching maximum load. In the case of the composite using SiC fibers coated with 0.50mass% of colloidal graphite suspension, the load suddenly dropped after reaching the maximum load, and then the load slightly decreased with an increase in crosshead displacement.

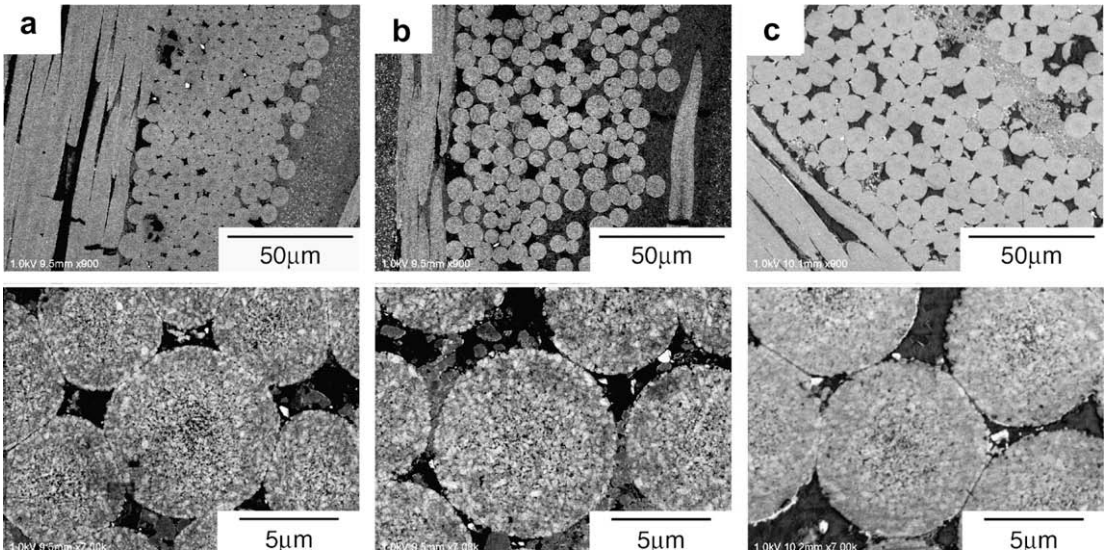

Fig. 1. SEM micrographs of the cross section of SiC/SiC composites using SiC fibers coated with various concentration of colloidal graphite suspension. The concentration of colloidal graphite suspension was (a) 0.05, (b) 0.10 and (c) 0.50mass%.

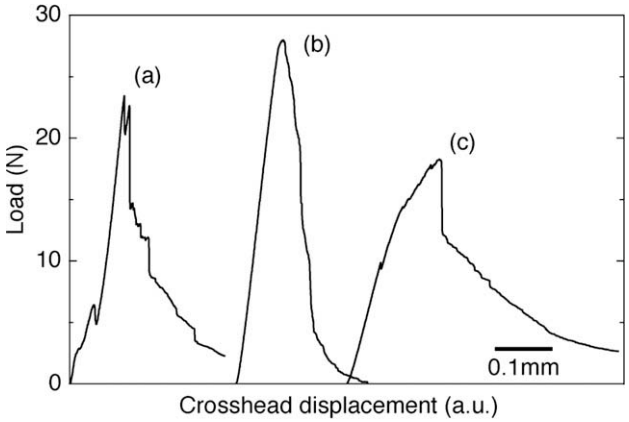

Fig. 2. The typical load-displacement curves of SiC/SiC composites using SiC fibers coated with various concentration of colloidal graphite suspension in bending test at room temperature. The concentration of colloidal graphite suspension was (a) 0.05, (b) 0.10 and (c) 0.50mass%.

SEM micrographs of the fracture surface of the composites observed from the tensile surface after bending test were shown in Fig. 3. The composite using SiC fibers coated with 0.05mass% of colloidal graphite suspension exhibited short fiber pullout, and fracture surface was almost flat. In the case of the composite using SiC fibers coated with 0.10mass% of colloidal graphite suspension, large fiber pullout was observed. The fracture surface of the composite using SiC fibers coated with 0.50mass% of colloidal graphite suspension was stepwise and showed large fiber pullout.

In order to investigate the effect of carbon coating on the SiC fiber on bending strength and fracture behavior of the composite, the carbon coating of SiC fiber after electrophoretic deposition was observed by SEM. SEM micrographs of the surface of SiC fibers coated with colloidal graphite suspension by electrophoretic deposition method are shown in Fig. 4. Carbon existed sparsely on the surface of SiC fiber coated with 0.05mass% of colloidal graphite suspension, i.e., carbon was coated non-uniformly on the fiber. It is indicated that the interface between the fiber and the matrix was not uniform and fiber pullout did not occur easily during fracture. As a result, the composite using SiC fibers coated with 0.05mass% of colloidal graphite suspension showed higher bending strength than the composites using SiC fibers coated with 0.10mass% and 0.50mass% of colloidal graphite suspension because the interface between the fiber and the matrix did not act effectively and showed higher friction during fracture. Furthermore, the difficulty in fiber pullout during fracture caused a stepwise fracture behavior and almost flat fracture surface with short fiber pullout. In the case of coating with 0.10mass% of colloidal graphite suspension, relatively uniform carbon coating on the fiber was observed. In consideration of fracture behavior and fracture surface of the composite, it seemed that the optimal interface between the fiber and the matrix was obtained by the electrophoretic deposition under this condition. In the case of coating with 0.50mass% of colloidal graphite suspension, carbon layer on the SiC fiber was so thick compared with carbon coating with 0.10mass% of colloidal graphite suspension, and colloidal graphite was filled between SiC fibers. The interface between the fiber and the matrix became so weak, and the composite using the fibers coated with 0.50mass% of colloidal graphite suspension could not sustain the applied load effectively and showed a shear fracture behavior, resulting in low bending strength and a wide load-crosshead displacement curve with lower maximum fracture load. From these results, it was concluded that the carbon coating on the fibers was successfully formed by the electrophoretic deposition using colloidal graphite suspension, and the optimal concentration of colloidal graphite suspension for relatively uniform carbon coating on the fibers was 0.10mass% in this study.

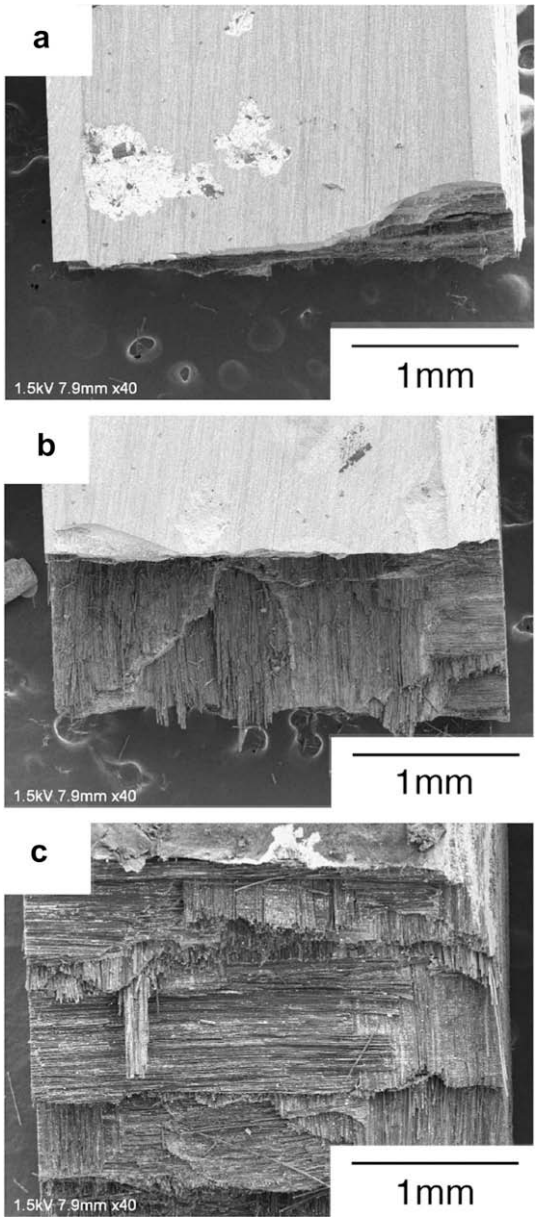

Fig. 3. SEM micrographs of the fracture surface of SiC/SiC composites using SiC fibers coated with various concentration of colloidal graphite suspension observed from the tensile surface after bending test. The concentration of colloidal graphite suspension was (a) 0.05, (b) 0.10 and (c) 0.50mass%.

4. Summary

In this study, colloidal graphite aqueous solution was used as the suspension for carbon coating on the fiber, and carbon layer was formed on SiC fibers by electrophoretic deposition method, and the SiC/SiC composite was fabricated by sheet stacking method and hot-pressing. The effect of the concentration of colloidal graphite on mechanical properties of SiC/SiC composite was investigated. Bulk density and open porosity of the composites fabricated in this study were nearly the same, and these values were independent of the concentration of colloidal graphite suspension. Bending strength gradually decreased with an increase in the

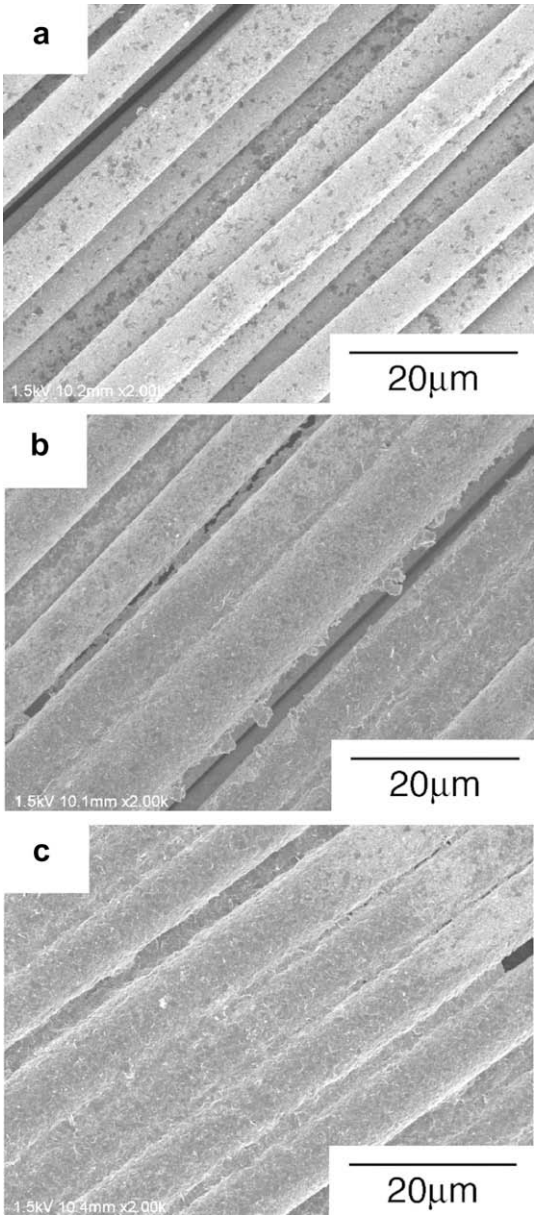

Fig. 4. SEM micrographs of the SiC fibers coated with various concentration of colloidal graphite suspension by electrophoretic deposition method. The concentration of colloidal graphite suspension was (a) 0.05, (b) 0.10 and (c) 0.50mass%.

concentration of colloidal graphite suspension. In the case of coating with 0.10mass% of colloidal graphite suspension on SiC fibers, relatively uniform carbon coating on the fibers was observed and large fiber pullout occurred effectively during fracture. From the results, it was found that the carbon coating on SiC fibers was successfully formed by the electrophoretic deposition method using colloidal graphite suspension, and 0.10mass% of colloidal graphite suspension was optimal for the formation of relatively uniform carbon coating on the fiber by the electrophoretic deposition method.

Acknowledgements

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