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Mechanical properties of unidirectional and crossply SiC_f/SiC composites using SiC fibers with carbon interphase formed by electrophoretic deposition process

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ABSTRACT

Unidirectional (UD) and crossply (CP) SiC_f/SiC composites with different fiber volume fraction were fabricated based on EPD and sheet stacking process, and their mechanical properties were evaluated at room temperature. The UD-SiC_f/SiC composites showed a pseudo-ductile fracture behavior in bending test. Bending strength and fracture energy of the UD-SiC_f/SiC composites with high V_f (66%) was higher than those of the UD-SiC_f/SiC composites with low V_f (47%). Although the CP-SiC_f/SiC composites also fractured in a non-brittle manner, their bending strength and fracture energy were much lower than the UD-SiC_f/SiC composites. In the UD-SiC_f/SiC composites, fiber pullout was clearly observed, and the number of fiber pullout in the UD-SiC_f/SiC composites with high V_f was large rather than the UD-SiC_f/SiC composites with low V_f . On the other hand, fiber pullout did not appear in the CP-SiC_f/SiC composites with high V_f . This can be explained by the thickness of the SiC matrix-layers between SiC fiber-layers.

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1. Introduction

Continuous silicon carbide fiber-reinforced silicon carbide matrix composites (SiC_f/SiC) have been recognized as a key material as components for high temperature gas turbine, aerospace industries and future nuclear power applications (Kaya, 1999; Christin, 2002; Naslain, 2005; Beesley, 1997; Giancarli et al., 2000; Jones, 2003; Ogasawara, 2004). In fusion power application, SiC_f/SiC composites have been expected to be used as the components for fusion breeding blanket and divertor, and future fusion power reactor concepts based on the use of SiC_f/SiC composites have been designed by JAEA (DREAM reactor) (Ueda et al., 1998), ARIES team (ARIES-I, IV and AT) (Sharafat et al., 1991) and CEA (TAURO) (Giancarli et al., 1998). Recently, SiC_f/SiC composites have been paid

* Corresponding author. Tel.: +81 3 5734 3082. *E-mail address:* k-yoshida@nr.titech.ac.jp (K. Yoshida). attention as the materials for fuel cladding in fission power application instead of zirconium (Zr)-alloy because SiC has the following advantages over Zr-alloy; lower absorption of thermal neutrons (~25% less than for Zr, for the same cladding wall thickness), little corrosion and no hydrogen pickup during normal operation, strength retention and low corrosion rate at elevated temperatures, and much slower degradation in a severe accident scenario (no meltdown, low corrosion rate, less/no hydrogen generation) (Deck et al., 2012; Hallstadius et al., 2012).

Fiber/matrix interfaces in SiC_f/SiC composites are very important to toughen and strengthen the composites, and the interface must be optimized. The optimum fiber/matrix interfaces play an important role for the inhibition of reactions between fiber and matrix, and for the promotion of interfacial debonding and fiber pullout (Kerans et al., 1989; Naslain, 1993; Yano and Yoshida, 2003). At present, carbon and hexagonal-boron nitride (h-BN) coatings have been commonly applied on the surface of SiC fibers by chemical vapor deposition (CVD) or chemical vapor infiltration







(CVI) in SiC_f/SiC composites (Naslain, 1993; Kagawa, 1998). However, these processes generally need longer manufacturing time and complicated apparatuses, and they use toxic, flammable or combustible reactant gases, resulting in higher production cost and an increase in environmental load. For this reason, the novel fabrication process of high-performance SiC_f/SiC composites that is simple, environmentally benign and low-cost has been strongly requested.

Electrophoretic deposition (EPD) is a colloidal process wherein ceramic bodies are shaped directly from a stable colloid suspension by a DC electric field, and EPD process can be applied for attaining complex shaped ceramics and a coating on the substrate (Sarkar and Nicholson, 1996; Boccaccini and Zhitomirsky, 2002; Tabellion and Clasen, 2004). Present authors paid attention to EPD process, and we proposed the novel fabrication process including interfacial control of SiC_f/SiC composites based on EPD (Yoshida et al., 2007, 2009a, 2009b, 2012; Yoshida, 2010). In order to apply SiC_f/SiC composites as structural parts, fiber orientation in SiC_f/SiC composites is also important because the fiber-orientation would affect the mechanical properties of SiC_f/SiC composites. Therefore, the relation between fiber orientation in SiC_f/SiC composites and their mechanical properties must be clarified for the practical use of them as structural parts. In this study, unidirectional (UD) and crossply (CP) SiC_f/SiC composites were fabricated based on EPD and sheet stacking process, and their mechanical properties were evaluated at room temperature. In addition, the UD- and CP-SiC_f/ SiC composites with different fiber volume fraction were fabricated. and their mechanical properties were also evaluated.

2. Experimental procedures

2.1. Formation of carbon interphase and SiC matrix for unidirectional SiC fiber perform by EPD process

Polycrystalline SiC fibers (Tyranno SA, fiber diameter; 10 µm, 800 filaments/yarn, Ube Industries, Ltd., Japan) were used as the reinforcement. The SiC fibers were unidirectionally aligned and then fixed on the jig. The SiC fiber preform was immersed in hot water to remove a sizing agent on the fibers. Silver was pasted on the upside of the SiC fiber preform. Aqueous suspension of graphite particles for EPD was prepared using a commercial colloidal graphite aqueous solution (Hitasol, average particle size; 54 ± 28 nm, Hitachi Powdered Metals, Co., Ltd., Japan). Concentration of colloidal graphite suspension was set at 0.3 wt%. Small amount of *n*-butylamine was added to the suspension to adjust the pH to 10. The colloidal graphite particle is negatively charged at pH 10. The unidirectional SiC fiber preform and graphite plates with the size of 38 mm^w \times 2 mm^t \times 70 mm^l were used as the anode and the cathode, respectively. They were settled at a distance of 10 mm in the colloidal graphite suspension under an applied voltage of 3 V for 1 h. They were dried at 120 °C after the deposition of colloidal graphite particles on SiC fibers by EPD.

Beta-SiC powder (Ultrafine, average particle size; 0.32 μ m, Ibiden Co., Ltd., Japan) and sintering additives composed of α -Al₂O₃ (TM-D, average particle size; 0.23 μ m, Taimei Chemicals Co., Ltd., Japan), Y₂O₃ (RU, average particle size; 1.2 μ m, Shin-Etsu Chemical Co., Ltd., Japan) and CaCO₃ (Kanto Chemical Co., Inc., Japan) were dispersed in distilled water using a commercial dispersant (ammonium polyacrylate). The concentration of β -SiC with the sintering additives in the aqueous suspension was 10 wt%. Small amount of *n*-butylamine was added to the suspension, and the pH of the suspension was adjusted to 10. Zeta potentials of β -SiC, α -Al₂O₃, Y₂O₃ and CaCO₃ are also negative values at pH 10. The carbon-coated SiC fiber preforms and graphite plates were dipped into the SiC suspension as the anode and the cathode, respectively, and they were settled at a distance of 10 mm. SiC matrix with the sintering additives was infiltrated within the fiber preform by EPD under an applied voltage of 5 V for 1 h. Afterward, the fiber preforms were dried at 120 °C.

2.2. Fabrication of unidirectional and crossply SiC_f/SiC composites

SiC green sheet was prepared by tape casting using a laboratoryscale tape casting equipment (DP150, Tsugawa Seiki, Japan). The slurry for tape casting was prepared using submicron-sized β-SiC powder, sintering additives (20 wt% α-Al₂O₃-Y₂O₃-CaO in total) and some organics. Details of the preparation of the SiC sheet were described in ref (YoshidaBudiyanto et al., 1998). In this study, two kinds of SiC green sheet with different thickness were prepared to obtain the SiC_f/SiC composites with different fiber volume fraction. Their thickness was 40–60 µm and 90–100 µm. The SiC sheet and the unidirectional SiC fiber preform with carbon interphase and SiC matrix were pasted with polyvinyl alcohol aqueous solution and stacked alternately, and then warm-pressed at 120 °C for 10 min under a uniaxial pressure of 2 MPa. Fig. 1 shows the schematic illustration of stacking direction of SiC fibers for UD- and CP-SiC_f/ SiC composites. In the case of UD-SiC_f/SiC composites, fibers were aligned in 0° direction. For CP-SiC_f/SiC composites, SiC fibers were aligned in 0° and 90° direction alternately. The stacked green body was heat-treated at 300 °C for 24 h in air in order to remove organics. The compact was hot-pressed at 1700 °C for 1 h in argon flow under a uniaxial pressure of 40 MPa.

2.3. Characterization and evaluation of unidirectional and crossply SiC_f/SiC composites

The SiC_f/SiC composite was cut into rectangular bars (4.0 $\text{mm}^w \times 2.4-2.8 \text{ mm}^t \times 35 \text{ mm}^l$). For the UD-SiC_f/SiC composites, they were cut into rectangular bars along the fiber direction. Bulk density and open porosity of specimens were measured by Archimedes' method. Bending strength of specimens was measured by three-point bending test 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 five. Bending strength of the SiC_f/SiC composites was calculated from the maximum load for fracture. Fracture energy of the SiC_f/SiC composites was calculated from the area of load—displacement curve in bending test divided by twice the fracture surface area. Microstructure and



Fig. 1. Schematic illustration of stacking direction of SiC fibers for UD- and CP-SiC_f/SiC composites.

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