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Fabrication of short C fiber-reinforced SiC composites by spark plasma sintering

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Abstract

Short carbon fiber reinforced SiC matrix composites were fabricated by spark plasma sintering. Density and mechanical properties of the composites increased continuously at increasing sintering temperature and constant pressure. Cracks in the composite matrix resulted from high thermal residual stresses generated during the cooling process from the sintering temperature due to the thermal expansion coefficient mismatch between fiber and matrix. The properties of the composites were lower than those of monolithic SiC ceramics obtained with the same processing technique as the composites. Fibers provided noncatastrophic fracture behavior of the composites as evidenced by the stress-displacement curves and fracture surface of the composites.

Keywords: A. Sintering; B. Fiber; Short; C_f/SiC; Spark plasma; Thermal residual stress

1. Introduction

Silicon carbide is one of the most promising structural materials for engineering application, because of its excellent high temperature mechanical properties, high thermal conductivity and good corrosion and wear resistance. However, silicon carbide applications have been limited because of its low fracture toughness. Fibers are introduced into the silicon carbide matrix to overcome the low fracture toughness and the high flaw sensitivity of monolithic silicon carbide ceramics.

Continuous fiber reinforced SiC composites obtained by chemical vapor infiltration (CVI) [1–3], polymer infiltration and pyrolysis (PIP) [4,5], reaction sintering (RS) [6,7], and hot-pressing (HP) [8,9] have been extensively studied. Mechanics and fabrication of short fiber reinforced SiC composites have rarely been reported [10]. The use of short fiber as reinforcement is a way to reduced the cost of the composites.

Spark plasma sintering (SPS) is a relatively new sintering technique in powder metallurgy which is capable of sintering metal and ceramic powers quickly to full density at a fairly low temperature due to its unique features [11]. Nano-SiC ceramics have been fabricated by SPS [12], which would have better thermal shock than normal SiC ceramics. Fabrication of short carbon fiber reinforced SiC composites by SPS has been reported [13,14] and the densification process of composites discussed. However, the mechanical properties of the C_f/SiC composites fabricated by SPS have not been reported.

In the present work, short C fiber reinforced SiC composites were fabricated by SPS. The effects of processing parameters on the densification process and on the mechanical properties of the composites using high modulus short C fibers as reinforcement were investigated.

2. Experimental

Short C fibers (TORAY Industries Inc., Japan) with an average diameter of 6 μm, and an average length of 2–3 mm,

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were used as the reinforcement to fabricate C_f/SiC composites. To decrease the sintering temperature, a nano-β-SiC powder of average particle size of about 60 nm (Kiln Nanometer Technology Development Co. Ltd., China), was used for the matrix. Al_2O_3 (6 wt.%) (Shanghai, China) and 4 wt.% Y_2O_3 (Yuelong, China) being used as sintering aids.

The starting Nano-SiC powder added with the sintering aids was ball-milled in ethanol using SiC balls for 4 h. After drying, the powders were screened through a 100-mech sieve. Polycarbosilane (PCS) was used to improve wetting between matrix and fibers. The resulting powders and PCS were then attrition milled with xylene as solvent for 3 h to form the slurry. The short C fibers were firstly ultrasonically dispersed into the slurry in a two dimensional plane to avoid fiber to be damaged in the *Z*-direction during the sintering process. After drying, the prepared tapes were cut by doctor's knife, and then stacked. Finally, the stacked tapes were put into a graphite die with an inner diameter of 25 mm and sintered using a SPS equipment (SPS-2040, Sumitomo Coal Mining Co., Japan). The sintering temperature, measured with an infrared sensor, was increased at a rate of 150 °C/min and varied from 1550 to 1650 °C .The 25 MPa pressure was applied when the temperature reached 800 °C, the holding time at the highest temperature was 3 min. Monolithic SiC ceramics with identical matrix combination were fabricated at 1550 °C under 15 MPa by SPS for comparison with mechanical properties of the C_f/SiC composites.

The SPS samples were subsequently cut and ground into 4 mm × 1.8 mm × 20 mm specimens for three-point-bending test in an Instron-1195 testing machine, operated at a crosshead speed of 0.5 mm/min and a span of 18 mm. The density of each sample was measured by the Archimedes method. Both the polished cross section and the fracture surfaces were observed by scanning electron microscopy (SEM).

3. Results and discussion

Fig. 1 shows the densification behaviors of C_f/SiC composites prepared by Spark Plasma Sintering at 1650 °C under 25 MPa. From this figure on apparent linear shrinkage

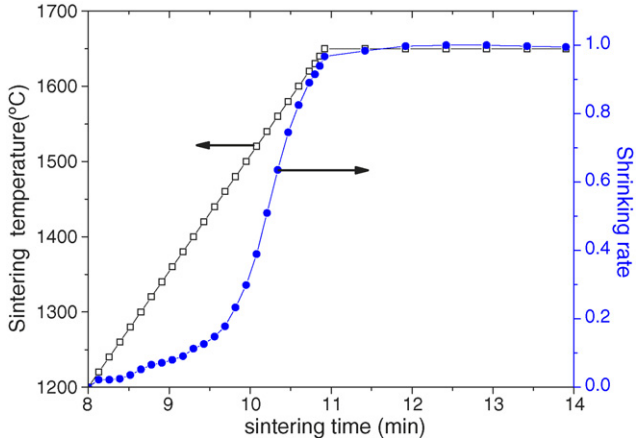

Fig. 1. Densification behavior of C_f/SiC composite by the SPS method under 25 MPa.

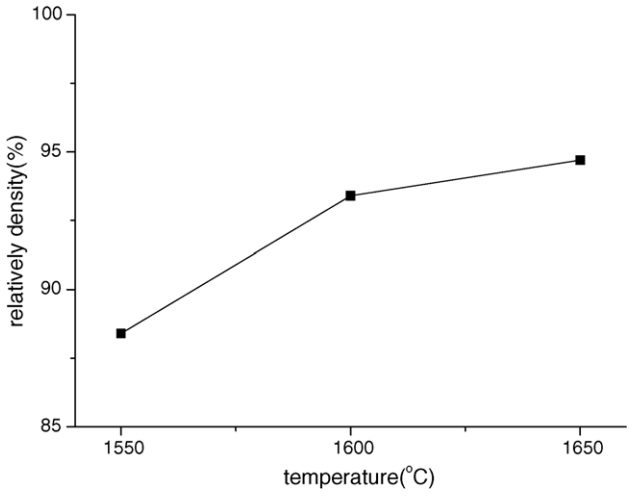

Fig. 2. Effects of sintering conditions on the densification of C_f/SiC composites by SPS.

rate on sintering temperature can be identified. With the increase of the sintering temperature, the shrinkage rate and densities of C_f/SiC composites increased rapidly from 1550 to 1650 °C, as shown in Fig. 2. By conventional hot-pressing, it is necessary to sinter the present SiC_f/SiC composite at 1800 °C or at higher temperature to attain full density [14]. Rapid densification process of the materials at low temperature became possible by SPS because of the electrical energy and effective high-temperature spark plasma [11].

Fig. 3 shows the microstructure of a polished cross-section of the composites densified at 1650 °C under 25 MPa. Carbon fibers distribute in the SiC matrix relatively homogeneously and randomly, with some cracks appearing in the matrix. The stress/displacement curves from the bending test for the SPS composites at different temperatures are shown in Fig. 4, With the increase of sintering temperature, proportional limit stress, ultimate bending strength and elastic modulus of the composites increased, indicating consistency with the data listed in Table 1, as a consequence of the raising density and the stress/displacement curves of Fig. 4 show a noncatasrophic fracture behavior. However, because of the high thermal residual stresses in the C_f/SiC composites during sintering, the bending strength of the composites was lowered compared with the strength of the monolithic SiC ceramics obtained by SPS at 1550 °C and 15 MPa (Table 1). The thermal residual stresses in the composites can be described as follows [15]:

$$\alpha^{\mathrm{T}} = k(\alpha_{\mathrm{m}} - \alpha_{\mathrm{f}})\,\Delta T \tag{1}$$

$$k = \frac{\varpi E_{\mathrm{m}} E_{\mathrm{f}}}{E_{\mathrm{m}}(1 - \upsilon_{\mathrm{f}}) + E_{\mathrm{f}}(1 + \upsilon_{\mathrm{m}})} \tag{2}$$

where α^{T} is the thermal residual stress in the composites, α_{m} and α_{f} are the thermal expansion coefficients of the matrix and the fiber, ΔT is the temperature difference between composites fabrication temperature and room temperature, E_{m}, E_{f}, υ_{m} and υ_{f} are Young's modulus and Poisson's ratios of the matrix and

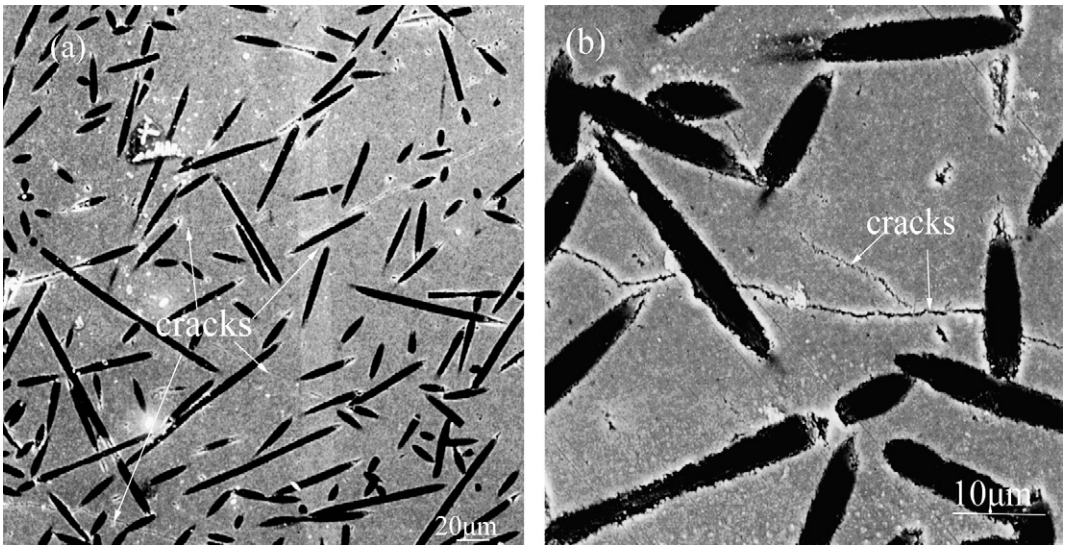

Fig. 3. SEM micrographs on the polished surface of the C_f/SiC composites at 1650 °C under 25 MPa.

fiber, respectively, and ϖ is the fiber surface roughness. The thermal expansion coefficient of the matrix can be assumed to be similar as the one of the SiC monolithic ceramics (about 4.0×10^{-6}/K) where the thermal expansion coefficient of the C fiber is about 25×10^{-6}/K in the radial direction and -1.23 to 0×10^{-6}/K in the axis direction. From (1), the matrix would be under tensile stress in the axis direction and fiber debonding may occur. When the composites were fabricated at high temperature, the matrix would create some cracks during the cooling process which are perpendicular to the fiber axis [16]. The homogeneously and randomly distributed short C fibers in the SiC matrix could make the cracks more complicated than in the unidirectional C fiber reinforced SiC composites, as the cracks might overlap, as shown in Fig. 3(b).

SEM micrographs of the fracture surfaces of the composites sintered at different temperatures under 25 MPa (Fig. 5) shows pullout of the very short fibers during the fracture of the composites. This could explain the non-linear stress-displacement behavior observed in the stress/displacement curves (Fig. 4). High rupture strength fibers would carry the load and enhance the strength of the composites by cracks initiation and deflection. However, the cracks initiated by the thermal residual

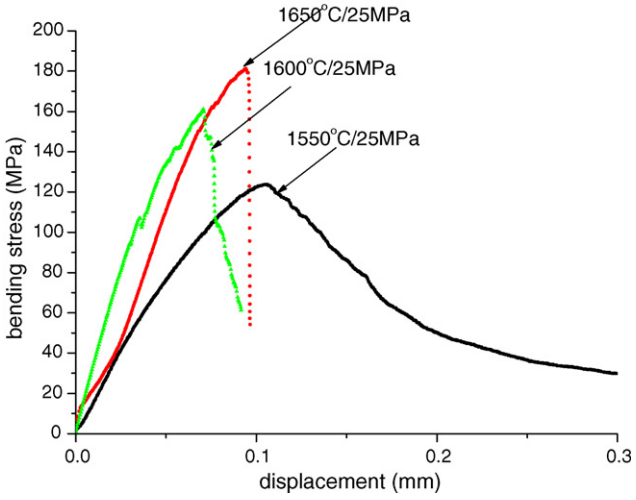

Fig. 4. Stress/displacement curves from bending test for the SPS C_f/SiC composites using at different temperature.

Table 1
Effect of sintering temperature on the mechanical properties of the composites and monolithic SiC ceramics

	Materials			
	C_f/SiC			SiC
	1550 °C/25 MPa[a]	1600 °C/25 MPa[a]	1650 °C/25 MPa[a]	1550 °C/15 MPa[a]
Density (g/cm^3)	2.65 ± 0.05	2.79 ± 0.00	2.83 ± 0.04	3.27 ± 0.01
Open porosity (%)	4.62 ± 0.03	3.32 ± 0.08	2.96 ± 0.04	0.67 ± 0.01
Proportional limit stress (MPa)	80 ± 13	131 ± 10	154 ± 94	425 ± 17
Bending stress (MPa)	118 ± 11	162 ± 12	170 ± 11	425 ± 17
Elastic modulus(GPa)	141 ± 6	188 ± 5	193 ± 7	375 ± 5

[a] Sintering condition

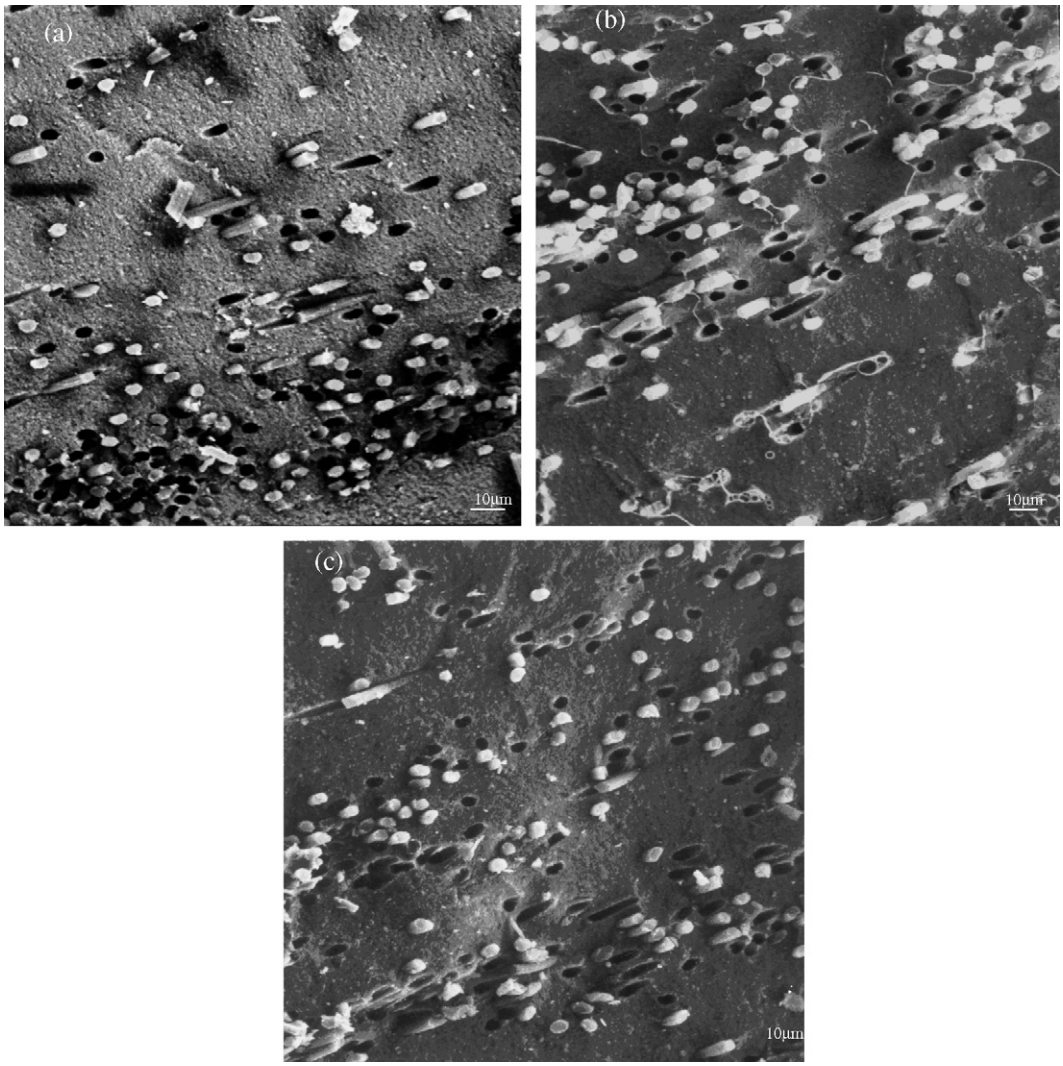

Fig. 5. Fracture surfaces of the composites at different sintering conditions: (a) 1550 °C/25 MPa, (b) 1600 °C/25 MPa, (c) 1650 °C/25 MPa.

stress in the composites would be detrimental to matrix strength, and therefore the strength of the C_f/SiC composite is lowered.

4. Conclusions

SiC composites containing 20 vol.% short carbon fiber were prepared by spark plasma sintering. At high sintering temperature, some cracks were created in the matrix because of residual thermal stresses causing the mechanical properties of the composites be lower than that of the matrix. However, the fibers in the composites could carry the load and provide noncatastrophic facture behavior to the composites.

Acknowledgement

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