

Microwave and conventional sintering of SiC/SiC composites: Flexural properties and microstructures

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Abstract

SiC/SiC composites were fabricated by the PIP process via microwave and conventional heating from 800 °C to 1200 °C. The flexural properties and microstructures of the as-fabricated SiC/SiC composites were investigated. The results indicated that the flexural strength and toughness have the same changing tendency for both conventional and microwave sintering. However, at the same PIP sintering temperature, the flexural properties of the microwave sintered SiC/SiC are higher than that of the conventional sintered ones. The higher residual strength of SiC fibers properties and more cracks in the composite matrix resulting from higher heating rates of the microwave sintering brought about the better flexural properties.

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1. Introduction

SiC/SiC composites have been developed for high temperature structural applications because of their thermal stability and excellent mechanical properties. The polymer impregnation and pyrolysis (PIP) process is considered as a fairly effective technique for fabricating SiC/SiC composites, since it can be applied to make large-scale components with complex shapes [1–4]. However, the intrinsic limitations of PIP, such as high process temperature, lengthy pyrolysis cycles and high cost, have restricted its wide applications [2,4].

As a novel heating technique, microwave heating is now more and more studied for ceramic fabrication or polymer pyrolysis process [2,4,5]. Although these research works are still on a starting stage, the notable features, such as low process temperature, rapid heating rates, tailored material properties and

microstructure have been demonstrated by ceramics development [6–11].

By now, comparison between microwave and conventional sintered SiC/SiC composites at different sintering temperatures is rarely reported. The aim of this work is to compare the mechanical properties and microstructures of the SiC/SiC composites fabricated by the PIP process using conventional and microwave heating.

2. Experimental procedure

KD-I SiC fiber bundles (their general characteristics were described in [12]) were woven into 3D (three-dimension, four-directional) preforms (fiber volume fraction: ~45%), which were coated with PyC as interface layers. Polycarbosilane (PCS, provided by National University of Defense Technology, China) was used as the polymer precursor of the SiC matrix for the PIP process, and xylene was used as a solvent for PCS. SiC fiber preforms were then impregnated with PCS solution by vacuum infiltration and then pyrolyzed by the conventional and microwave heating for 15 cycles. The pyrolysis temperature of each preform

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was 800 °C, 900 °C, 1000 °C, 1100 °C, and 1200 °C (all for 1 h and in N₂ atmosphere). The heating rates of conventional and microwave sintering were ~ 7 °C/min and ~ 40 °C/min, respectively. The SiC fiber filaments without PyC were put together with the above preforms in the microwave sintering furnace for 1 PIP cycle as heat treatment. The microwave sintering furnace (4 kW, 2.45 GHz; temperature measured by pyrometer) is manufactured by Tangshan nano-source microwave thermal instrument manufacturing Co., Ltd, China. The conventional sintering furnace (6 kW) is manufactured by Dingli Technology Co., Ltd, China.

The flexural strength was characterized by three-point bending at room temperature, with a dimension of 3 mm (B) $\times$ 4 mm (H) $\times$ 35 mm and crosshead speed of 0.5 mm/min. The fracture toughness was determined with the single edge notch beam (SENB) method with a dimension of 3 mm (B) $\times$ 6 mm (H) $\times$ 45 mm and crosshead speed of 0.05 mm/min. The morphology and microstructure of the specimens was analyzed by scanning electron microscopy (SEM, FEI Quanta-200). The SiC fiber filament (after heat treatment) strength test was measured by 'Testmetrix Micro 350' (Testmetrix Inc., UK).

3. Results and discussion

The flexural strength and fracture toughness of SiC/SiC composites sintered by conventional and microwave heating from 800 °C to 1200 °C are listed in Table 1. From Table 1, it can be observed that the flexural strength and toughness of SiC/SiC composites increase with the sintering temperature rising from 800 °C to 1100 °C and then decrease at 1200 °C for both conventional and microwave sintering.

However, the results that should be of concern are that, compared to the conventional-sintered SiC/SiC composites, the flexural properties of microwave-sintered SiC/SiC are higher at the same sintering temperature. For example, the flexure strength and flexure toughness of 1100 °C-microwave-sintered SiC/SiC composites are 11.5% and 22.6% higher respectively than that of the 1100 °C-conventional-sintered ones. And the 1100 °C-conventional-sintered SiC/SiC have almost the same flexural properties as the 1000 °C-microwave-sintered ones, which means that the microwave heating can save the sintering temperature of 100 °C, with the addition of higher heating rate (~ 7 °C/min versus ~ 40 °C/min), the microwave heating may increase the fabrication efficiency of SiC/SiC composites greatly.

Table 1
Flexural properties of SiC/SiC composites fabricated by microwave and conventional sintering.

Sintering temperature (°C)	Conventional sintering		Microwave sintering	
	Flexure strength (MPa)	Flexure toughness (MPa m ^{1/2})	Flexure strength (MPa)	Flexure toughness (MPa m ^{1/2})
800	349.6 $\pm$ 24.8	20.5 $\pm$ 1.9	425.6 $\pm$ 41.3	20.9 $\pm$ 2.0
900	364.3 $\pm$ 33.5	21.3 $\pm$ 2.1	465.8 $\pm$ 46.2	21.6 $\pm$ 2.2
1000	441.5 $\pm$ 41.7	22.7 $\pm$ 2.3	489.9 $\pm$ 47.6	25.5 $\pm$ 2.4
1100	494.8 $\pm$ 48.9	24.3 $\pm$ 2.4	551.5 $\pm$ 51.7	29.8 $\pm$ 2.8
1200	279.4 $\pm$ 19.8	15.6 $\pm$ 1.8	312.9 $\pm$ 26.3	17.9 $\pm$ 2.0

Two factors are considered to bring about the higher flexural properties of the microwave sintered SiC/SiC composites:

- (1) As one of the most important contributors of the flexural properties of the SiC/SiC composites, the KD-I SiC fibers used in this study are multiphase fibers consisted of crystalline β -SiC, amorphous Si-C-O and free carbon phase [3]. The higher heating rates of microwave heating may lower the degradation of SiC fiber properties. The residual strength of SiC fiber filament after conventional and microwave heat treatment, as shown in Fig. 1, can prove this deduction. It can be seen that the SiC filament strength after microwave heat treatment is higher than that of the conventional ones at the same temperature. Besides, the strength of KD-I SiC fiber decreases dramatically at 1200 °C, resulting in the decreasing flexural strength and fracture toughness of SiC/SiC composites sintered at 1200 °C, as shown in Table 1. Meanwhile, the tendency of the SiC filament strength is the same as that of flexural strength and fracture toughness of the SiC/SiC composites, demonstrating that the SiC fibers as reinforcement play a critical role in the mechanical properties of SiC/SiC composites.
- (2) Fig. 2 demonstrates the matrix microstructure of the conventional sintered SiC/SiC composites at 800 °C and 1100 °C. Fig. 3 displays the matrix microstructure of the microwave sintered SiC/SiC composites from 800 °C to 1100 °C.

In Fig. 2, resulting from the lower sintering rate of conventional sintering, the SiC/SiC composites have much denser matrix (Fig. 2 (a)-1 and (b)-1) with a small amount of matrix cracks (Fig. 2(a)-2 and (b)-2). In contrast, the higher heating rates of microwave heating bring about more cracks in the composite matrix (Fig. 3), which will offer more routines for the crack expanding in case of fiber rupture prematurely and absorb more extrinsic energy.

In brief, the higher residual strength of SiC fibers and more cracks in the composite matrix resulting from higher heating

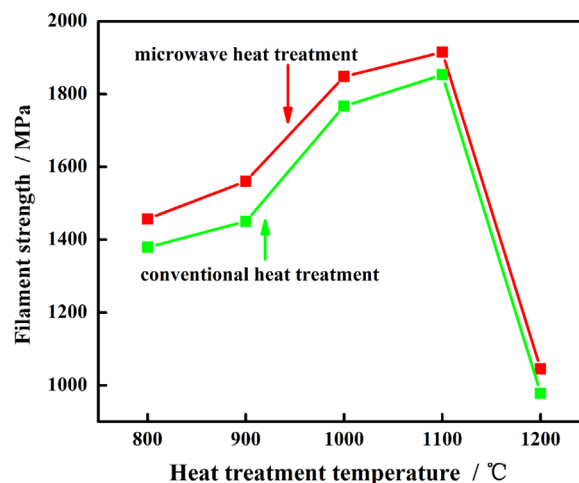


Fig. 1. The residual strength of SiC fiber filament after the conventional and microwave heat treatment from 800 °C to 1200 °C.

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