

Fabrication of carbon fiber reinforced ceramic matrix composites with improved oxidation resistance using boron as active filler

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Abstract

Boron was introduced into C_f/SiC composites as active filler to shorten the processing time of PIP process and improve the oxidation resistance of composites. When heat-treated at 1800 °C in N₂ for 1 h, the density of composites with boron (C_f/SiC-BN) increased from 1.71 to 1.78 g/cm³, while that of composites without boron (C_f/SiC) decreased from 1.92 to 1.77 g/cm³. So when boron was used, two cycles of polymer impregnation and pyrolysis (PIP) could be reduced. Meanwhile, the oxidation resistance of composites was greatly improved with the incorporation of boron-bearing species. Most carbon fiber reinforcements in C_f/SiC composite were burnt off when they were oxidized at 800 °C for 10 h. By contrast, only a small amount of carbon fibers in C_f/SiC-BN composite were burnt off. Weight losses for C_f/SiC composite and C_f/SiC-BN composite were about 36 and 16 wt%, respectively.

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1. Introduction

The most commonly used ceramic matrix composites (CMCs) are those comprising carbon or SiC fibers in a carbon or SiC matrix (C/C, C/SiC, SiC/SiC composites). C/SiC composites are potential candidates for a variety of applications in aerospace fields, including rocket nozzles, heat shields and aeronautic jet engines.^{1,2} However, the principal obstacle for wider applications, especially for long-term applications, of carbon-containing materials is known to be their relatively poor oxidation resistance.³ Oxidation of carbon starts at temperatures as low as 450 °C, and its lifetime is dramatically limited beyond this temperature. The lifetime of CMCs which are partly made of carbon is strongly depended on the efficiency of the anti-oxidation systems used to reduce the oxygen permeability.⁴ Extensive studies have been undertaken to improve the oxidation

resistance of carbon fiber reinforced CMCs and most are related to the application of boron-bearing species.^{5–8} One important property of boron-bearing species is to form fluid oxide phases (B₂O₃ or B–M–O ternary phase) over a broad temperature range (600–1200 °C for B₂O₃) when heated in an oxidizing atmosphere.⁹ Compared to multilayered self-sealing matrix fabricated by CVI method,⁷ boron-based particles introduced by slurry infiltration method have shortened the processing time and lowered the processing cost. A simple way to introduce boron-based particles is to impregnate a fiber perform with slurry consisting of boron-based particles.¹⁰

In the present work, carbon fiber reinforced ceramic matrix composites were fabricated by active-filler-controlled polymer pyrolysis (AFCOP) process.¹¹ Boron was introduced into the carbon fiber bundles as active filler to form some boron-bearing species by in situ reactions during the heat-treatment process. The influences of boron on microstructures as well as the oxidation resistance of the composites were studied in comparison with those of C_f/SiC composites fabricated by PIP using SiC particles as inert filler.

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Table 1
Properties of the carbon fiber reinforced ceramic matrix composites.

Sample	Raw material ratio (wt%)	Fiber fraction (%)	Density (g cm^{-3})	Open porosity (%)	Bending stress (MPa)	Elastic modulus (GPa)
S	55(SiC)+45(PCS)	45%	2.11	7.2	88.5 ± 30.4	60.9 ± 4.5
S*	45(B)+55(PCS)	44%	1.98	8.4	86.5 ± 9.6	52.5 ± 4.5

2. Experimental procedure

Nano-SiC (Kai'er Nanometer Technology Development Co., Ltd., Hefei, China) or boron (Tangshan WeiHao Magnesium Powder Co., Ltd., Tangshan, China) powders were mixed with PCS (National University of Defense Technology, Changsha, China) by ball-milling for 48 h, using xylene as solvent to form homogenous slurries. Table 1 listed the ratios of raw materials in the slurries. 2D woven carbon fiber plain fabrics (Mitsubishi Rayon, 200 g/m^2) were first deposited with PyC interphase at 1050°C using methane as precursor and the average thickness of the PyC interphase is about 100 nm. The source gases were first introduced into the reaction chamber with a pressure of 10 kPa, then the exhausts were evacuated to below 300 Pa using vacuum pumps within 5 s after holding for 20 s at 1050°C . The coated fabrics were impregnated with the aforementioned slurries and stacked in a graphite die after drying. Pressures were applied to control their thicknesses according to a fiber volume of about 45% in the hot-pressing furnace at 200°C . Composite derived from the boron-containing slurry was denoted as S* and the other was denoted as S. After further densified by several cycles of PIP (5 cycles for S and 3 cycles for S*) using PCS as the polymer with the pyrolysis temperature of 800°C , the samples were heat-treated (nitrided) at 1800°C in N_2 atmosphere for 1 h. Then PIP was performed again to fill the open pores with the pyrolysis temperature of 1100°C .

After being cut and ground into $5 \text{ mm} \times 2 \text{ mm} \times 20 \text{ mm}$, the specimens were deposited with a SiC coating of about $10 \mu\text{m}$ by CVD to isolate the cross-sections from the air for oxidation. SiC coating was deposited at 1100°C and 5 kPa using MTS and H_2 as source gases with the molar ration of H_2 to MTS was 10. Oxidation tests were performed in a muffle furnace. The specimens were pushed into the furnace when the temperature reached 800°C and then taken out quickly to weight the mass after each 2.5 h oxidation. The weight losses of composites in air from room temperature to 1000°C were also measured by TG.

The densities and open porosities of samples were measured by the Archimedes method. Mechanical properties were measured by three-point-bending tests with $5 \text{ mm} \times 2 \text{ mm} \times 40 \text{ mm}$ specimens in an Instron-5566 machine, operated at a crosshead speed of 0.5 mm/min and a span of 24 mm. The microstructures of composites both before and after oxidation were observed by electron probe microanalyzer (EPMA, JXA-8100, JEOL, Tokyo, Japan) equipped with wave-length dispersive spectroscopy (WDS).

3. Results and discussion

The properties of both composites are summarized in Table 1. It can be obtained that the density of composite S is higher

than that of S*. This may be a result of lower density of h-BN (2.27 g/cm^3) than that of SiC (3.20 g/cm^3). It is also indicated that composite S* has a lower elastic modulus though there is only very small difference in the bending stresses, which may be accounted by the introduced h-BN into composites and h-BN is often introduced into composites to decrease their elastic modulus.^{12–14}

The densities of samples after each PIP cycle are calculated according to their weights and volume in that there is almost no change in the volumes of samples in both PIP process and nitridation process. The density variation versus PIP cycles is plotted in Fig. 1. It is interesting to find from these two curves that the introduction of active fillers has a strong influence on the densification process of the composites. In the nitridation process, the density of S decreases from 1.92 to 1.77 g/cm^3 while that of S* increases from 1.71 to 1.78 g/cm^3 . The density decrease of S may be caused by the weight loss of carbon fibers and decomposition of Si–O–C existing in the matrix. The fiber reinforcements used in this work will lose some active components when the fibers were heat-treated at high temperature and the weight loss is about 3.5%. In addition, as a result of the oxygen introduced into the PCS structure during its synthesis, Si–O–C phase will be formed. At temperatures higher than 1400°C , decomposition of amorphous Si–O–C phase begins with the evolution of CO and SiO.¹⁵ When S* is concerned, boron will react with

Table 2
Changes in density and open porosity for both composite S and S* occurring in the nitridation process.

Sample	Density (g cm^{-3})		Open porosity (%)	
	Before	After	Before	After
S	1.92	1.77	8.7	24.7
S*	1.71	1.78	12.2	20

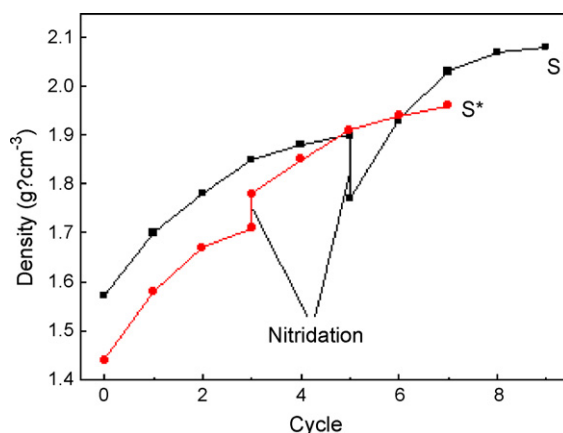


Fig. 1. Influences of boron on the density variations of composites.

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