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ZrB_2-SiC as a protective coating for C/SiC composites: Effect of high temperature oxidation on thermal shock property and protection mechanism

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ABSTRACT

ZrB_2-SiC coating was prepared on C/SiC composites surface by slurry method, and then the thermal fatigue behavior of ZrB_2-SiC coated C/SiC composites was studied. The composition of the coating layers was characterized by XRD, SEM and EDS. With the thickness was 200 μm, the coating was ZrB_2 and SiC. During thermal cycle between 1773 K in air and 373 K in boiling water, the weight of the ZrB_2-SiC coated composites decreased lightly. The decrease of the flexural strength during the thermal cycle was primarily due to the debonding of the fiber–matrix interfaces and the oxidation of the coated samples. Compared with the uncoated C/SiC composites, the coating played an important role in enhancing the resistance to the thermal shock.

1. Introduction

Anti-oxidation coating is a key technique for C/SiC composites [1–3]. At higher temperatures (>1473 K), the oxidation of the fiber and matrix cooperatively influences the oxidation behavior of C/SiC composites in oxygen atmosphere [4,5]. Consequently, C/SiC composites need oxidation protection when exposed to oxidizing environment at higher temperatures.

Considering its extremely high melting temperatures, high hardness, and great strength retaining characteristics at high temperatures [6], Zirconium diboride (ZrB_2) was the most interesting material for thermal shielding applications. However, boron oxide partial pressure was not negligible [7], it began to evaporate at higher temperature.

Different strategies to improve the oxidation resistance had been tested. ZrB_2-SiC ceramic had been proved as an effective coating for protecting carbon materials from oxidation at high temperature [8,9].

The anti-oxidation ability of coating was commonly tested at an invariable temperature [4,5,8,9]. The ultimate application environment of C/SiC composites usually related to a thermal cycle between low and high temperature.

The static oxidation test could not entirely reflect the protective ability of the coating, so study on the thermal fatigue behavior of ZrB_2-SiC coated C/SiC composites was very important, which had not been reported up to now.

In this paper, the oxidation behavior of ZrB_2-SiC coated C/SiC composites under thermal shock was studied, and the effect of high temperature oxidation on thermal shock property and protection mechanism was discussed.

2. Experimental procedures

2.1. Preparation of C/SiC composites

Polycarbosilane (PCS) [10] (molecular weight: 1742, soften point: 448 K) was synthesized in our laboratory. Xylene was used as solvent for PCS. Three-dimensional braided carbon fibers (T-300, ex-PAN carbon fiber, Toray) were used as the reinforcement. C/SiC composites denoted as raw samples were prepared using 9–12 cycles of infiltration of PCS-Xylene solution (mass ratio 1:1) and subsequently pyrolyzed at 1473 K under N_2 (purity: 99.99%) atmosphere [3].

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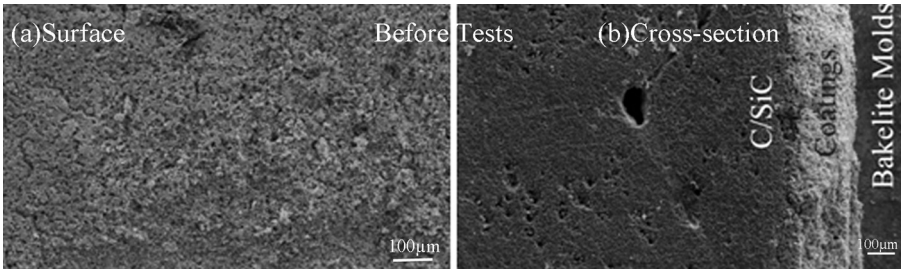

Fig. 1. SEM of ZrB_2-SiC coated C/SiC composites: (a) surface and (b) cross-section.

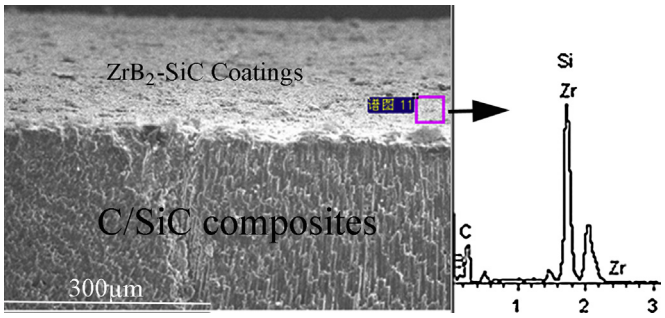

Fig. 2. EDS of ZrB_2-SiC coated C/SiC composites.

2.2. Preparation of ZrB_2-SiC coating

ZrB_2-SiC coating was fabricated in order to increase the oxidation resistance. ZrB_2 powder (2.5 mm, Dandong Chemical Engineering Institute Co. Ltd., China) with PCS-Xylene solution (weight ratio: 1:1) was pasted on the composites. The best filler was composed of 60 wt.% ZrB_2, 4 wt.% SiC, 6 wt.% PCS, 4 wt.% B and 26 wt.% DVB, and subsequently pyrolyzed at 1473 K under N_2 (purity: 99.99%) atmosphere.

2.3. Procedures for thermal shock tests

The substrate and coated samples were heated at 1773 K in air, and then cooled through boiling water. The thermal shock tests were conducted by alternating the specimens quickly between 1773 K in static air and 373 K in water. Each specimen was held in the tube furnace, which was preheated to 1773 K for 10 min and then cooled to 373 K in water for 10 min. After the temperature of the specimen dropped to 373 K in water for 10 min, it was immediately sent back to the furnace, which was held at 1773 K. The time to move the specimen from the furnace to the boiling water was approximately 5 s. Such a heating-cooling cycle indicated that the specimens were thermally shocked for one time. In order to reveal the thermal shock resistance of the composites, at least three specimens were investigated at prescribed number of 50 cycles. Details for thermal shocks tests were reported in Ref. [11].

2.4. Samples characterization

The samples were weighed after tests by an electronic balance with a sensitivity of ±0.001 g. Three-point bending tests were used to evaluate the flexural strength of C/SiC composites with the span/height ratio of 15 and a crosshead speed of 0.5 mm/min before and after oxidation.

The σ_{3b} was calculated by the following:

$$\sigma_{3b} = \frac{3PL}{2BH^2} \quad (1)$$

where P was the load in test, L was outer support span, B was specimen width, and H was specimen thickness. The samples were cut and polished to 70 mm in length, 5 mm in width and 4 mm in thickness. The length and width directions were parallel to warp and weft directions, respectively.

The micro-structures of the samples and fracture surfaces of the specimens after three-point bending tests were examined by

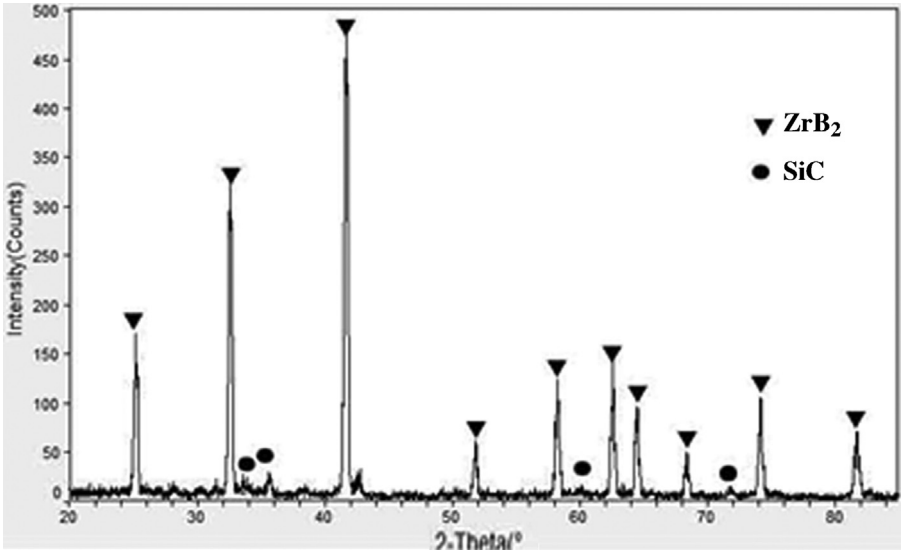

Fig. 3. XRD of ZrB_2-SiC coated C/SiC composites.