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Ablation behavior of functional gradient ceramic coating for porous carbon-bonded carbon fiber composites

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ABSTRACT

To improve the ablation resistance of low density carbon bonded carbon fiber (CBCF) composites at high temperature, a novel functional gradient ceramic coating was prepared. The coating exhibits a two layer structure with a compact HfB_2-$MoSi_2$ outer layer and an inner Si-CBCF layer with gradient transition structure. The high temperature ablation resistance of the coated CBCF composites was investigated with an arc-jet testing at temperature 1500–2200 °C. The results showed that the prepared functional ceramic coating exhibited good ablation resistance and good thermal shock resistance after four ablation cycles. After ablation, the surface coating exhibit three different ablation regions due to the different ablation temperature. The formation of a dense silica glass layer embedded with HfO_2 particles or a molten HfO_2 layer was responsible for the good ablation resistant to the functional gradient ceramic coating.

1. Introduction

Carbon-bonded carbon fiber (CBCF) composites are one kind of porous carbon-carbon (C/C) composites, with low densities in the range of 0.1–0.5 g/cm^3 and high open porosities over than 70% [1–3]. They are consisted of a layered fiber network structure in which the fibers are bonded at their intersections by discrete pyrolytic carbon matrix. The unique properties including lightweight, low thermal conductivity, high temperature stability and low cost make them attractive candidates for high-temperature heat insulation applications [4,5] and thermal insulation applications in aerospace [3,6,7].

Unfortunately, monolithic CBCF composites are sometimes inappropriate for specific applications due to their poor mechanical strength and extreme oxygen sensitivity above approximately 400 °C. As for mechanical properties, our previous work proved that the compressive strength increased with density following exponential functions [8]. Moreover, the fiber length and the fiber orientation have big effects on the mechanical properties of CBCF composites. The mechanical properties of CBCF composites could be enhanced by increasing fibers or altering characteristics of fibers and bonding points. Recently, lots of researchers have focused on the matrix modification to improve the mechanical properties of CBCF composites by the addition of carbon-based matrix, ceramic-based matrix or mixed matrix [9–13]. For example, CBCF composites with in-situ grown carbon nanotubes (CNTs) [11] and SiC nanowires (SiC_{NW}) [12] were fabricated through precursor impregnation and pyrolysis (PIP) methods. Results showed that the CNTs and SiC_{NW} can bridge the carbon fibers and matrix, which is beneficial to the enhancement of mechanical properties of CBCF composites. SiC modified CBCF composites were prepared by dispersion and flocculent approach [9]. The compressive strength and compression modulus of the SiC modified CBCFs were improved compared with unmodified CBCFs. SiOC modified CBCF composites were prepared by PIP methods [13]. It was found that the compression strength increase from 3.0 to 7.09 MPa in the XY direction and 1.02–3.78 MPa in the Z direction when the density increasing from 0.38 to 0.94 g/cm^3. Meanwhile, these matrix modification methods can improve anti-oxidation resistance of CBCFs as well. Such as SiOC modified CBCF composites exhibited better oxidation resistance than original CBCF composites due to the formation of amorphous SiO_2 products [13]. However, the least weight loss of SiOC/CBCF composites after oxidation at 1000 °C for 120 min was as high as 21.5%, which hardly meet the application needs of lightweight structural materials. Preparation an oxidation coating on

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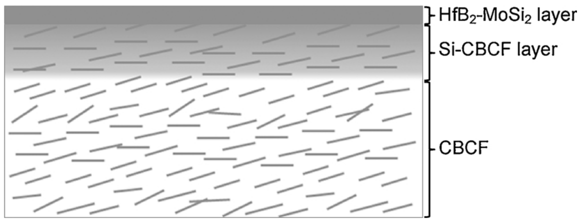

Fig. 1. The schematic diagram of functional gradient ceramic coating.

the surface of CBCF composites is considered as an efficient method to solve this problem. Various Si based coatings including SiC, $MoSi_2$, $TaSi_2$ are widely investigated as an attractive coating material because of its SiO_2 glassy film products [14,15]. SiO_2 can act as an oxidation inhibitor due to its low oxygen diffusion coefficient at high temperature. However, these coatings lose effectiveness at higher temperature due to the rapid volatilization of SiO_2 and active oxidation of SiC [16]. Ultra-high temperature ceramics (UHTCs), such as zirconium carbides and zirconium borides, have high melting point and good oxidation resistance at high temperature. Xu develop a double-layer ZrB_2-based ceramic coating on the surface of matrix modification CBCF composites [17].This ZrB_2-based ceramic coating had a good oxidation resistance at 1500–1600 °C and the dense glass layers were forming under the surface coatings which could protect CBCF composites from erosion during high temperature oxidation. However, with the ablation temperature increasing to 1700 °C, the coating exhibited severe ablation. To our knowledge, report on the ultra-high temperature oxidation resistance of CBCF composites is limited and their surface temperatures are always less than 1600 °C.

The aim of this work is to develop a high temperature anti-oxidation coating, which could protect CBCF composites at temperature above 1700 °C A functional gradient ceramic coating with two layers was designed and fabricated. First, an inner Si-CBCF layer with gradient transition structure was prepared by brush-infiltration and pyrolysis method. Then, a surface compact HfB_2-$MoSi_2$ coating was prepared through low pressure plasma spray technique. The microstructure and oxidation behavior of the coated CBCF composites were analyzed and discussed in detail.

2. Experimental

2.1. Preparation of functional gradient ceramic coatings

Rayon-based carbon fibers with 0.8 mm length were used to prepare CBCFs by dispersion and filtration technique. Details of the preparations were given elsewhere [8]. The cylindrical samples (φ25.4 × 50 mm) used as substrates were cut from CBCF composites with a density of 0.25 g/cm^3 and a total open porosity of ∼85%. Samples were cleaned ultrasonically with ethanol and dried at 120 °C for 2 h. The schematic diagram of functional gradient coating is shown in Fig. 1. There are two layers and each layer has different functions. The outer layer is a dense HfB_2-$MoSi_2$ coating which affords high temperature oxidation resistance. It was prepared by low pressure plasma spray technique (LPPS). Commercially available HfB_2 and $MoSi_2$ powders with a mean size of ∼10 μm (∼99%) were supplied from Northwest Institute of Non-ferrous Metal Research, China. HfB_2-25 vol% $MoSi_2$ mixed powders were homogenized by ball milling. A plasma spray system was applied to deposit the coatings as the same described elsewhere [18]. During this process, argon was used as the carrier gas. The spraying voltage, spraying current, powder feed rate and the spraying distance was 70 V, 750 A, 20 g/min and 180 mm, respectively. The inner layer is a gradient ceramic oxidation layer, namely Si-CBCF layer. This layer acts as a transition layer to alleviate the mismatch between the inner layer and the outer layer. It was prepared by slurry brushing and infiltration process using SiBCN ceramic precursor. All surface of samples were brushed to introduce the Si-based matrix for 2–3 cycles. After curing at 200 °C, the impregnated samples were pyrolyzed at 1500 °C for 120 min under a flowing Ar atmosphere. The thickness of this infiltration layer can be controlled through changing the brush and pyrolysis cycles, which is different to the vacuum slurry infiltration methods [13]. Under these two layers is the original CBCF composite, which plays a key role of thermal insulation and lightweight due to its low density and high porosity.

2.2. Ablation tests

The ablation behaviors of the as-prepared CBCF composites with functional gradient ceramic coatings were evaluated using simulated

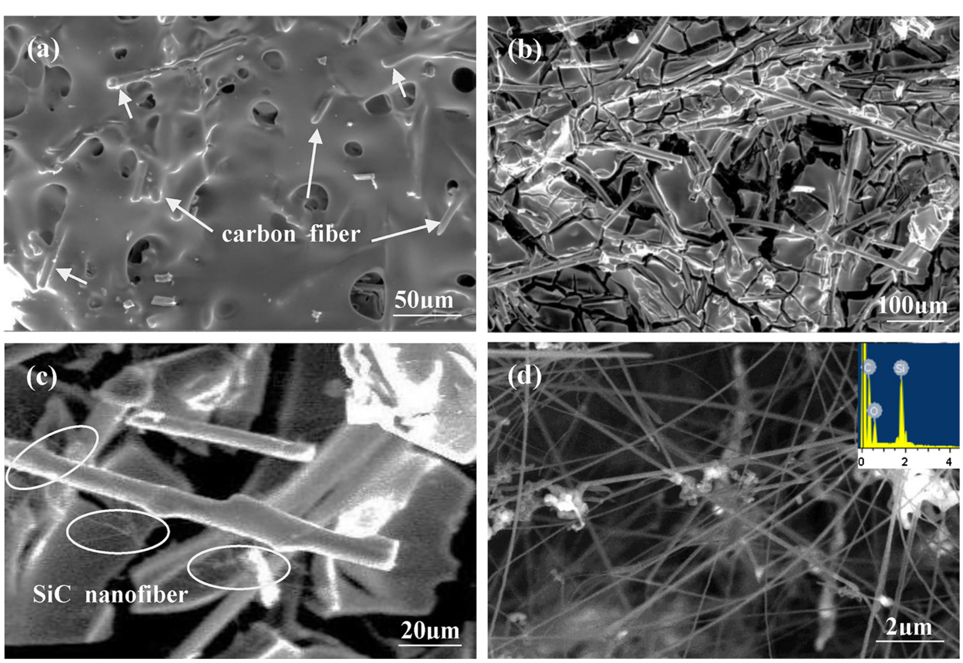

Fig. 2. Typical surface images after preparing of the inner Si-CBCF layer: (a) before pyrolysis; (b) after pyrolysis; (c) high magnification in (b); (d) high magnification in (c); the insert in (d) is the EDS of SiC nanofiber.

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