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UHTC coating reinforced by HfC nanowires against ablation for C/C composites

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ABSTRACT

To improve the ablation resistance of the coated carbon/carbon composites, HfC nanowires acting as reinforcement materials were introduced into ZrB_2-SiC/SiC multilayer coating. Synthesis, morphology, mechanical properties and ablation resistance of the coating were researched. A porous HfC nanowire layer was uniformly covered on SiC coating. Both of the toughness and interface bonding strength of ZrB_2-SiC coating with HfC nanowires were higher than those of the coating without HfC nanowires. HfC nanowires could effectively inhibit the cracking and peeling of outer coating during ablation. After oxyacetylene torch ablation for 90 s, the mass ablation rate of the coated sample without and with HfC nanowires was 0.20 mg/s and −0.12 mg/s, respectively.

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1. Introduction

Because of the superior mechanical properties at high temperature, carbon/carbon (C/C) composites are broadly applied as thermal structural materials [1–4]. In practical applications, C/C composites should bear chemical ablation and mechanical denudation in ultra-high temperature (above 2273 K) environment along with severe combustion gas flow. Unfortunately, the rapid oxidation of C/C composites above 773 K will degrade their mechanical properties. So, it is crucial to increase the oxidation and ablation resistance of C/C composites [5,6]. Preparing an external coating is an effective method to prevent oxidation and ablation of C/C composites in such severe environment [7,8]. Because of its relative lower coefficient of thermal expansion (CTE), SiC is usually used as a transition layer to decrease the mismatch of CTE between C/C substrate and outer coating [2].

Ultra-high temperature ceramics (UHTCs) (HfC, ZrC, HfB_2, ZrB_2 and so on) are appropriate ablation resistance coating materials for C/C composites [9]. Among these UHTCs, ZrB_2 has high melting point (3313 K), good chemical stability, good ablation resistance and so on [10–12]. The oxidation products of SiC and ZrB_2 could form silicate glass between 1273 K and 2073 K, so adding appropriate SiC could increase the ablation resistance of ZrB_2 [13]. Up to now, ZrB_2-SiC coating has been successfully synthesized on C/C composites. Yao [14] and Zhang [15] synthesized ZrB_2-SiC/SiC multilayer coating by supersonic atmosphere plasma spray (SAPS), which could effectively prevent C/C composites from oxyacetylene torch ablation. Wang [16] prepared ZrB_2-SiC coating on C/C composites through three-step process including pressing, pyrolysis and reactive silicon infiltration, and then analyzed the ablation performance of the coating. Previous research proved that ZrB_2-SiC multiphase coating could prevent C/C composites from ablation in severe environment. Among all of the preparation methods, SAPS is a very high-efficiency and low-cost method to prepare a dense ZrB_2-SiC coating [14,15]. The thickness and composition of the coating could be controlled by adjusting the spraying parameters.

Nevertheless, compared with inner coating, the CTE of outer coating is much larger, which could lead to the generation of cracks in outer coating during preparation and ablation [17,18]. SiC nanowires have been reported as the reinforcing materials to inhibit the outer coating cracking. Chu [19] and Li et al. [20] prepared ceramic coatings reinforced by SiC nanowires, both of the isothermal oxidation and thermal shock performance of the coatings were improved by introducing SiC nanowires. Li et al. [21] reported that the ablation resistance of HfC coating was also increased due to the incorporation of SiC nanowires. However, the melting point of SiO_2 (2000*K*) is lower than the ablation temperature (above 2273 K), so SiC nanowires in the coating would be oxidized and melted during ablation, which would weaken their toughing effect. The melting point of HfC nanowires (4263 K) is much higher [22]. Besides, HfO_2 also possesses high melting temperature (3083 K). So HfC nanowires are more suitable to be used in ablation coatings. So far, no research about the mechanical properties and ablation performance of HfC nanowire-toughened ablation coating has been reported.

In the present study, ZrB_2-SiC coating with HfC nanowires was synthesized on SiC-coated C/C composites. The effects of HfC nanowires on the microstructure, toughness, interface bonding strength and ablation performance of ZrB_2-SiC outer coating were researched.

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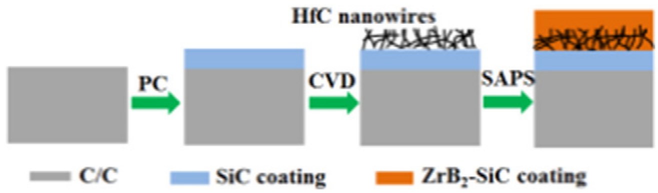

Fig. 1. Diagram of the fabrication process of HfC nanowire-toughened ZrB_2-SiC/SiC coating on C/C composites.

Table 1
Spraying parameters for ZrB_2-SiC coating.

Content	Parameters
Spraying current (A)	380–420
Spraying voltage (V)	110–150
Primary gas Ar (L/min)	70–80
Carrier gas Ar (L/min)	8–15
Second gas H_2 (L/min)	3–5
Powder feed rate (g/min)	20
Spraying distance (mm)	100
Nozzle diameter (mm)	5.5

2. Experimental procedures

2.1. Materials and processing

Cylindrical C/C specimens (φ30 × 10 mm) with a density of about 1650 kg/m^3 were used as substrates. Before preparing coating, the samples were ultrasonically cleaned in distilled water and then dried at 373 K.

Schematic diagram of the coating preparation process is shown in Fig. 1. To begin with, pack cementation was employed to prepare SiC inner coating on C/C specimens using 65–80 wt.% Si, 10–25 wt.% graphite and 5–15 wt.% Al_2O_3 as raw materials. The preparation temperature was 2073–2373 K and the heat treatment time was 2 h. Detailed experimental process was described in Ref. [19].

Then, HfC nanowires were synthesized on SiC coating by chemical vapor deposition (CVD). The SiC-coated C/C substrates were covered with catalyst particles ($Ni(NO_3)_2$) before deposition through means of soaking. $HfCl_4$ powders acting as hafnium source were put in the low-temperature evaporation chamber; CH_4, H_2 and Ar served as carbon source, reducing gas and inert gas, respectively. The high-temperature deposition chamber was heated to 1373–1473 K and maintained for 3 h. The deposition pressure was set at 10–20 KPa. The flow rates of CH_4, H_2 and Ar were 200–300, 1000–2000 and 400–800 mL/min, respectively. After deposition, HfC nanowires were obtained on SiC coating.

At last, ZrB_2-SiC outer coating was synthesized by SAPS. ZrB_2 (70–80 vol.%) and SiC (20–30 vol.%) powders acting as raw materials were firstly ball-milled for 3 h. To improve the flowability of the powders, a slurry composed of 48 wt.% distilled water, 2 wt.% polymeric binder and 50 wt.% mixture powders was agglomerated using a spray drier. Table 1 shows the detailed spraying parameters [15]. So, the desired HfC nanowire-toughened ZrB_2-SiC coating was obtained. The pure ZrB_2-SiC without HfC nanowires was also synthesized by the similar SAPS process.

2.2. Mechanical and ablation property tests

Hardness and elastic modulus of the ZrB_2-SiC coatings were measured through nano-indentation test using Nano-Indenter™ XP (MTS System Corp. USA) system with a diamond Berkovich indenter. Penetration depth was constant (500 nm). During indentation, the computer recorded the change of load along with the displacement changing. Hardness and elastic modulus of the coatings could be calculated through Oliver and Pharr method [23].

Fracture toughness (K_c) of the coatings was measured using a micro-hardness tester with a Vickers indenter. A load of 0.2 kg was applied on the polished coating section to induce coating cracking.

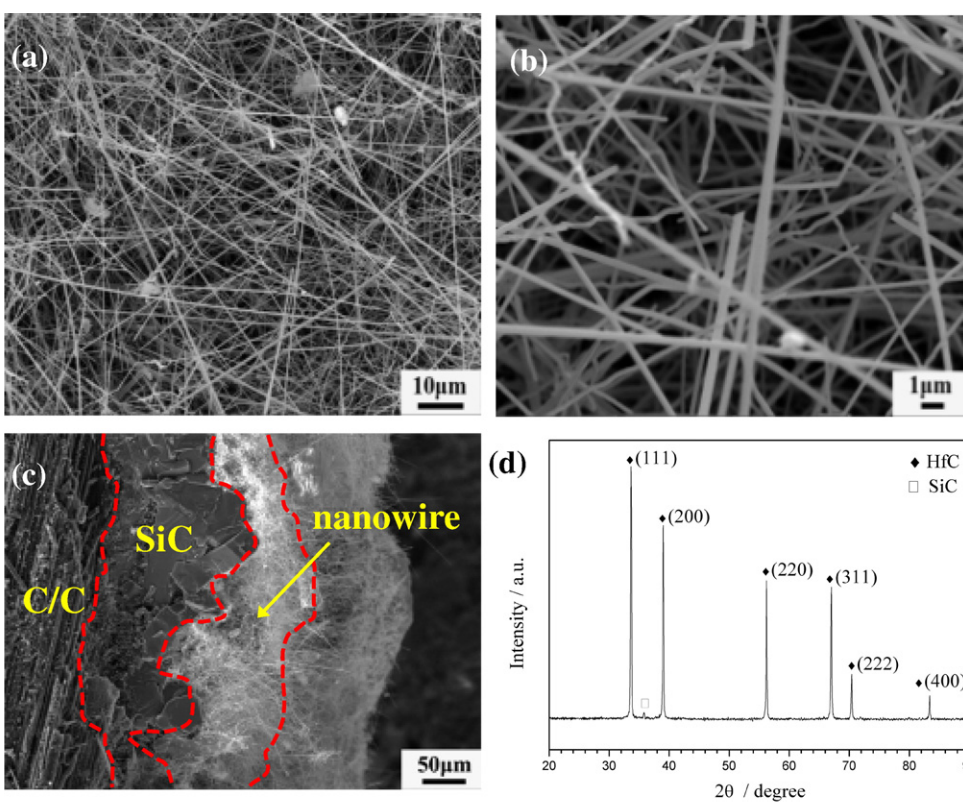

Fig. 2. SEM images (a, b, c) and XRD pattern (d) of the prepared HfC nanowires on SiC coating.

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