

A glass coating prepared by pulse arc discharge deposition for oxidation protection of carbon/carbon composites



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ABSTRACT

Pulse arc discharge deposition (PADD) was developed to prepare an oxidation protective borosilicate glass coating for SiC pre-coated carbon/carbon composites (SiC-C/C). The phase compositions, microstructure and oxidation resistance of the as-prepared glass/SiC coating were investigated. The results show that pulse arc discharge deposition is an effective route to achieve a dense and crack-free outer coating, which is attributed to a low temperature with pulse technique and arc discharge sintering process combined. The glass/SiC multilayer coating can effectively protect C/C composites from oxidation in air at 1773 K for 292 h with a weight loss of 2.04%.

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1. Introduction

Carbon/carbon (C/C) composites are promising candidate materials for applications in aerospace industry due to their exceptional properties such as low density and good thermal shock resistance as well as excellent mechanical properties [1,2]. Unfortunately, the oxidation of C/C composites above 723 K in oxidizing atmosphere greatly limits their applications as high temperature structural materials [3,4]. Coating is considered to be an effective method for protecting C/C composites from oxidation at high temperature [5]. SiC is usually used as a bonding layer due to its good chemical and physical compatibility with C/C composites [6]. However, microcracks usually exist in SiC coating due to the mismatch of thermal expansion coefficient between the coating material ($\alpha_{\text{SiC}} \approx 5 \times 10^{-6} \text{ K}^{-1}$) and the C/C substrate ($\alpha_{\text{C/C}} \approx 1 \times 10^{-6} \text{ K}^{-1}$) [7]. It is an effective way to use glasses as crack sealants [8]. Borosilicate glass coatings have been prepared by different methods, which possess good oxidation resistance and self-sealing properties at high temperature [9]. A self-sealing glass coating for C/C composites was prepared by slurry method [10], which can protect C/C composites from oxidation in air at 1573 K for 150 h with a weight loss less than 1%. Moreover, the glass coatings were also prepared by other

methods, such as in situ formation method [11] and plasma spray [12]. However, these methods will result in the generation of thermal stress in the coatings and the thermal damage for C/C composites at high temperature.

Recently, pulse arc discharge deposition (PADD) has been demonstrated to be an effective technique to prepare a crack-free and dense coating due to a low temperature with pulse technique and arc discharge sintering process combined. An $\text{AlPO}_4\text{-SiC-MoSi}_2$ composition coating was successfully deposited by PADD [13], which can protect C/C composites from oxidation in air at 1873 K for 44 h with a weight loss less than 2%.

In the present work, a borosilicate glass coating was deposited on SiC-C/C composites by PADD with periodic deposition. The phase compositions, microstructure of the as-prepared glass coating were characterized. The oxidation resistance and the failure mechanism of the multilayer coated C/C composites were investigated.

2. Experimental

2.1. Specimens and coating preparation

Small specimens (10 mm × 10 mm × 10 mm) were cut from bulk 2D C/C composites with a density of 1.75 g/cm³ were used as substrates. The specimens were hand-abraded with 320-grit SiC paper, then cleaned with distilled water and dried at 333 K for 2 h. The SiC bonding layer was prepared on C/C specimens by pack

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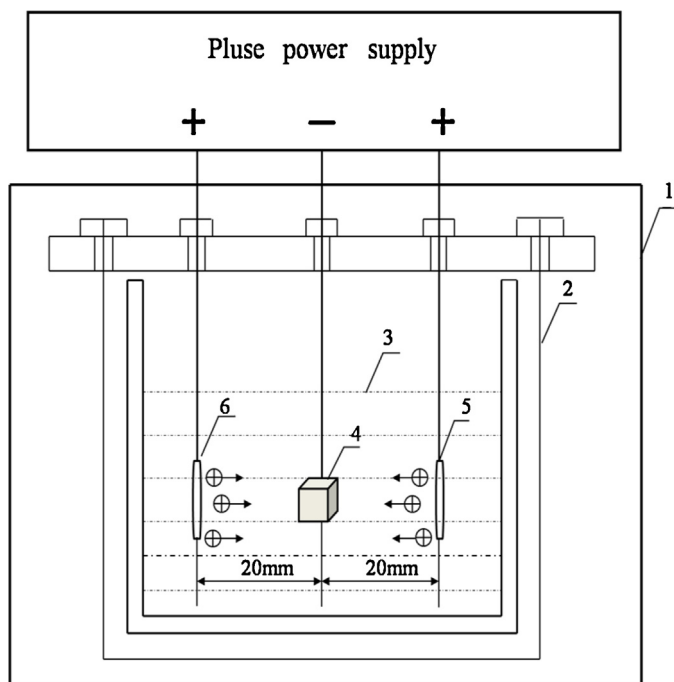


Fig. 1. The device diagram of pulse arc discharge deposition system: 1-dryer; 2-hydrothermal autoclave; 3-suspension; 4-cathode (sample); 5,6-anode (graphite substrate).

cementation. Details for the preparation procedure of the bonding SiC layer were reported in Ref. [14].

The composition of borosilicate glass was 65–75 wt.% SiO₂, 20–30 wt.% B₂O₃, 2–10 wt.% Al₂O₃. The powders were mixed by tumbling in a ball mill and heated at 1373–1473 K for 2 h in a corundum crucible. Then, the corundum crucible was cooled naturally down to room temperature in furnace and taken out from the furnace. The as-received glass was milled 36 h to attain the glass powder with a size of 500 nm.

For the pulse arc discharge deposition process, glass powders (500 nm) with the mass of 3 g were dispersed in 150 ml isopropanol with an ultrasonic bath for 40 min and a later magnetic stirring for 8 h. Next, 0.225 g crystalline analytical reagent iodine used as charge agent was dissolved in the above suspension with magnetic stirring for 12 h. Protons were produced by the reaction between iodine and isopropanol shown in Eq. (A.1) [14]. The above suspension was transferred to a hydrothermal autoclave and the SiC–C/C substrate was fixed on the cathode of the autoclave. After sealed, the autoclave was put into a furnace. The voltage, pulse duty ratio, the pulse frequency and furnace temperature were kept at 400 V, 70%, 2000 Hz and 373 K, respectively. After 25 min deposition, the autoclave was taken out of the furnace and cooled down to room temperature naturally. Subsequently, the specimen was taken out from the autoclave and dried at 353 K in air for 2 h.



2.2. Pulse arc discharge deposition system

Fig. 1 shows a device diagram of pulse arc discharge deposition system. The anode of the autoclave is a graphite substrate (20 mm × 10 mm × 3 mm) and the SiC–C/C substrate is fixed on the cathode of the autoclave. The suspension is used to deposit coating for deposition process. The device of PADD is same with hydrothermal electrophoretic deposition device except the power. Pulse power supply takes the place of DC power supply in hydrothermal

electrophoretic deposition process [15] and periodic arc discharges take place in a symmetrical electric field [16].

2.3. Characterization

The as-coated specimens were heated at 1773 K in air in an electrical furnace to investigate the isothermal and thermal cycle oxidation behavior. During the tests, the weight of the samples was measured at room temperature by electronic balance with a sensitivity of ±0.1 mg. The mass loss was calculated by Eqs. (A.2) and (A.3). The end mass loss is from average value of the group of five samples after oxidation at high temperature for a certain time.

$$\Delta W\% = \frac{m_0 - m_t}{m_0} \times 100\% \quad (\text{A.2})$$

$$v_{\text{oxidation}} = \frac{m_0 - m_t}{S \cdot t} \times 100\% \quad (\text{A.3})$$

$$\Delta W = \frac{m_0 - m_t}{S} \times 100\% \quad (\text{A.4})$$

where m_0 is the original mass of the coated C/C composites, m_t is the mass of the coated C/C composites after oxidation at high temperature for a certain time, g ; t is the oxidation time, h and S is the surface area of the specimen, cm². $\Delta W\%$ is percentage of weight loss, ΔW is weight loss per unit area, g m⁻² and $v_{\text{oxidation}}$ is weight loss rate, g m⁻² h⁻¹.

The morphology, crystallite structure and the element composition of the glass coatings were analyzed by a scanning electron microscope (SEM, JSM-6390A) with energy-dispersive spectroscopy (EDS) and a X-ray diffractometer (XRD, Rigaku D/max-3C).

3. Results and discussion

3.1. XRD analysis and surface morphologies of the coating

Fig. 2(a) shows surface XRD pattern of SiC bonding layer by pack cementation. It reveals that the bonding layer prepared by pack cementation is mainly composed of α -SiC phase, less β -SiC phase and Si phase. The surplus silicon is helpful to relax the thermal expansion and improve bonding strength between SiC layer and the glass coating. From Fig. 2(b), it can be found that a loose structure SiC layer with some microholes, microcracks and big particles are obtained after the pack cementation process.

The surface SEM micrograph of the as-prepared glass coating by PADD is shown in Fig. 3(a). It shows that a very dense and crack-free coating is achieved. No clear microcracks and microholes are observed on the double-layer coating surface. This may be due to the arc discharge sintering for the densification of the as-prepared glass/SiC coating. By X-ray diffraction analysis shown in Fig. 3(b), the amorphous nature of glass between 15° and 25° of 2θ can be found. Additionally, the appearances of SiO₂, Al₂SiO₅ and B₂O₃ were supposed to be due to the crystallization of the glass phase during cooling to room temperature [17]. From the cross-section SEM micrograph (Fig. 3(c)), it can be observed that a dense and homogeneous glass is achieved without penetrative cracks and obvious interface between the SiC layer and the glass coating. This possibly due to the preparation method of PADD with a low temperature. Additionally, the thickness of the glass/SiC coating is about 180 μm .

Fig. 4 displays the EDS element line scan analysis of the cross-section of the glass/SiC coating. It shows the concentration distributions of C, O, Al and Si elements along the coating cross direction. The element line scan analysis demonstrates that the double-layer coating sample could be divided into three parts, designated as A, B and C. Part A is the C/C composites matrix, Part B

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