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Oxidation protection of carbon/carbon composites with SiC/indialite coating for intermediate temperatures

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Abstract

In order to improve the oxidation resistance of carbon/carbon composites at intermediate temperatures, a novel double-layer SiC/indialite coating was prepared by a simple and low-cost method. The internal SiC transition layer was prepared by pack cementation and the external indialite glass–ceramic coating was produced by in situ crystallization of ternary $MgO–Al_2O_3–SiO_2$ glass. The microstructures and morphologies of coating were determined by scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS). Oxidation resistance of the as-coated C/C composites was evaluated in ambient air at temperature from 800 °C to 1200 °C. Nearly neglectable mass loss was measured after 100 h isothermal oxidation test, indicating that SiC/indialite coating possesses excellent oxidation protection ability. The as-coated samples have a good thermal shock resistance and no obvious damage was found in the coating even after suffered more than 11 thermal cycles between test temperature and room temperature. The oxidation protection mechanism of this coating was also discussed.
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1. Introduction

Carbon/carbon (C/C) composites are attractive thermostructural materials for high-temperature application due to their low density, low coefficient of thermal expansion (CTE), high thermal conductivity, excellent thermal shock resistance and mechanical properties.[1] However, C/C composites have an inherent defect, that is, rapid oxidation in oxidizing environment above 400 °C, which restricts wide application of these materials.[2]

Multilayer coatings were proved to be an effective way to improve the oxidation resistance of these composites[3] and the choice of coating systems was highly dependent on the application temperature. Generally, SiC coating has been considered as one of the best bonding layer due to its good physical and chemical adaptability with C/C substrates.[4,5] Several coating systems, such as SiC/Y_2SiO_5,[6] SiC/Si–W–Mo[7] and SiC/mullite,[8] can effectively protect C/C composites over 1400 °C for a long time. However, some serious problems existing in the coating systems include inherent brittleness of ceramic coating and large mismatch of CTE between C/C substrates and coating. As a result, these coatings are prone to cracking under thermal cycles and the stress-induced cracks fail to self-seal below 1200 °C, which lead to the severe oxidation of C/C composites at intermediate temperatures. To solve this problem, some researchers proposed a feasible way to reduce the interface thermal stress by preparing a low CTE external coating.[9]

Some glass–ceramic materials possess unique combination of properties, such as good mechanical strength and high-temperature stability, especially, the low CTE.[10] In this work, a double-layer SiC/indialite glass–ceramic coating was prepared for protection of C/C composites at intermediate temperatures and the microstructures and oxidation resistance of the coating system were fully investigated.

2. Experimental

Small samples (10 mm × 10 mm × 10 mm) used in this work were cut from bulk felt C/C composites with a density of 1.75 g/cm^3. After being polished with 500 grits SiC paper,

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samples were ultrasonically cleaned in acetone and dried at 100 °C for 30 min. The composition for preparing SiC internal coating by pack cementation was: 65–80 wt.% Si, 10–25 wt.% graphite and 5–10 wt.% Al_2O_3. C/C samples and pack mixtures were put in a graphite crucible and heat-treated at 1800–2000 °C for 1–2 h in argon to form the internal SiC coating.

External glass–ceramic coating was prepared by slurry spraying. Glass chosen in this study fell into primary phase field of cordierite and the compositions of glass were composed of 50–75 wt.% SiO_2, 2–10 wt.% Al_2O_3, 10–20 wt.% MgO and a few amounts of CaO and TiO_2. The mixtures were ball-milled in polyurethane container for 30 min, melted in Al_2O_3 crucible at 1500 °C for 2 h to form homogenous glass, and then quenched in distilled water immediately. Followed by gas-free piece was ground to 30 μm, the fine powders were mixed with carboxymethyl cellulose to form homogeneous slurry and sprayed onto the surface of SiC coating. Finally, the samples were heated to 1400 °C for 10 min to form the glass coating and dwelled at 1000 °C for 2 h for further crystallization to form glass–ceramic coating. During cooling step, slow cooling rate was set at 3–5 K/min to minimize thermal shock.

The isothermal oxidation test was carried out in air in an electric furnace. Weight change of the samples were measured by a precision balance and recorded as a function of time. Cumulative weight change percentages of the samples were calculated using the following equation:

$$\Delta W\% = \frac{m_0 - m_1}{m_0} 100\%$$

where m_0 and m_1 were the weight of the samples before oxidation and after oxidation, respectively.

The crystalline phase of the as-coated C/C was identified with Rigaku D/max-3C X-ray diffraction (XRD). The surface and cross-section morphologies of the as-coated samples were determined by JSM-6460 scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). CTE values of glass–ceramic and C/C material were performed on DIL-402 high-temperature thermal expansion apparatus. The specimens (20 mm × 4 mm × 4 mm) of indialite glass–ceramic were obtained by the same process as external coating preparation.

3. Results and discussion

3.1. Microstructures of the coatings

Fig. 1 shows the surface and cross-section SEM images of the internal coating prepared by pack cementation. From Fig. 1a, it can be seen that internal coating exhibits a dense and homogeneous structure. However, a few microcracks can be observed in the coating, which resulted from the great resid-

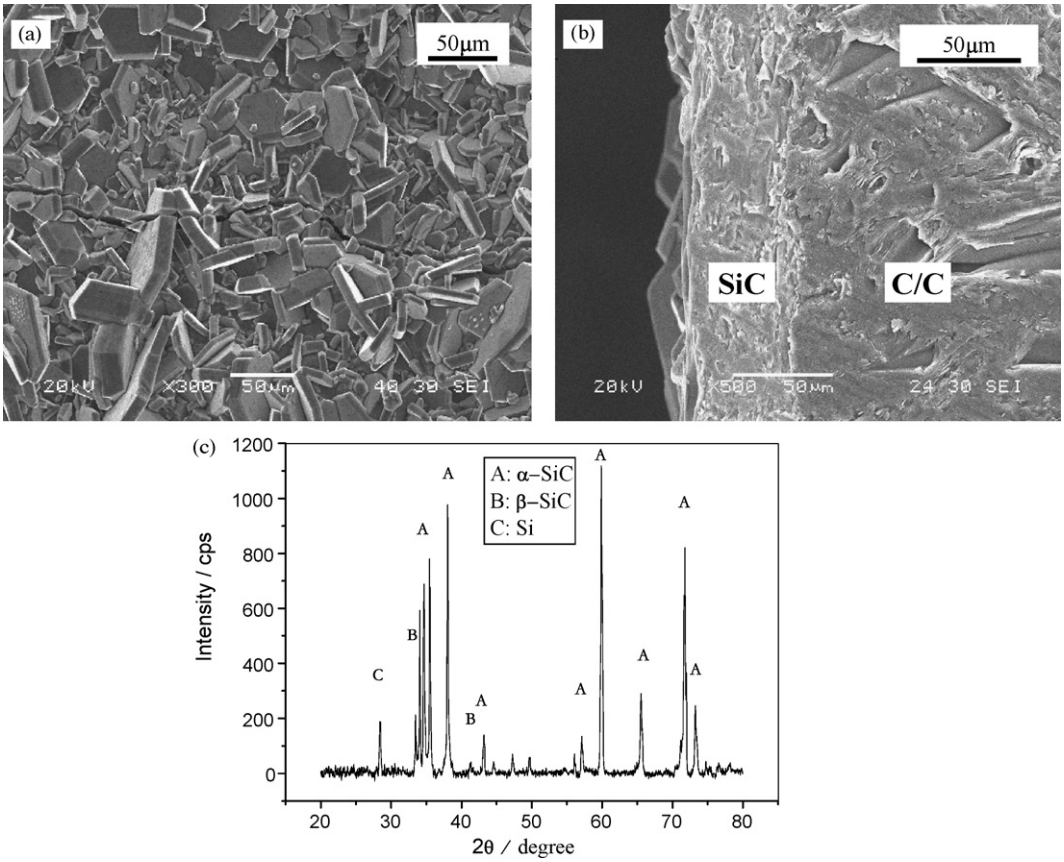

Fig. 1. Morphologies and phase analysis of SiC transition coating prepared by pack-cementation (a) surface SEM image, (b) cross-section SEM image and (c) XRD patterns.

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