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Short communication

A hot-pressing reaction technique for SiC coating of carbon/carbon composites

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Abstract

A hot-pressing reactive sintering (HPRS) technique was explored to prepare SiC coating for protecting carbon/carbon (C/C) composites against oxidation. The microstructures of the coatings were analyzed by X-ray diffraction and scanning electron microscopy. The results show that, SiC coating obtained by HPRS has a dense and crack-free structure, and the coated C/C lost mass by only 1.84 wt.% after thermal cycles between 1773 K and room temperature for 15 times. The flexural strength of the HPRS-SiC coated C/C is up to 140 MPa, higher than those of the bare C/C and the C/C with a SiC coating by pressure-less reactive sintering. The fracture mode of the C/C composites changes from a pseudo-plastic behavior to a brittle one after being coated with a HPRS-SiC coating.

Keywords: A. Hot pressing; A. Sintering; D. SiC

1. Introduction

Oxidation resistance is a key requirement for carbon/carbon (C/C) composites for applications in an oxygen-containing environment at high temperature [1–3]. To prevent C/C composites from oxidation, SiC coating was widely used due to its excellent oxidation resistance and good compatibility with C/C composites [4,5]. Presently, SiC can be coated on the surface of C/C composites by several methods, such as pack cementation [6], chemical vapor deposition (CVD) [7] and laser-induced chemical decomposition (LICD) [8]. Among these methods, pack cementation was usually used for providing a strong interface bonding between SiC coating and C/C composites [9]. However, cracks will be formed inevitably in this coating during the cooling process from high temperature to room temperature due to the mismatch of thermal expansion between SiC and C/C composites, which offer entrance channels for oxygen and result in the failure of the coating [5,7].

Hot-pressing reactive sintering (HPRS) is a technique in which both the chemical reactions of the starting materials and densification occur in single step. This technique can be economical due to low cost starting powders and relatively low sintering temperature, and it also leads to the possibility of densifying the materials without additives [10,11]. As far as the authors know, no literature has been published about using HPRS technique to prepare ceramic coatings for C/C composites. Though HPRS technique is not practical to prepare coatings for complex shaped C/C composites, it can be applicable to regular shaped ones by designing suitable moulds. In the present work, HPRS technique was proposed to prepare SiC coating for C/C composites. The microstructures and oxidation protective ability of the coatings were investigated, and the effect of SiC coating on the flexural property of the coated C/C composites was also studied.

2. Experimental

The substrates were cut from bulk 2D C/C composites with a density of 1.75 g/cm^3. Powder compositions for the HPRS process were 65–80 wt.% Si and 20–35 wt.% graphite. These powders were mixed by a blender for 2 h. C/C specimens were packed by these mixtures in a graphite crucible, and were pressed by a graphite pressing head. The pressure was controlled by 250 kPa. Then the graphite crucible was heated to 1873–2073 K and held at that temperature for 2 h under

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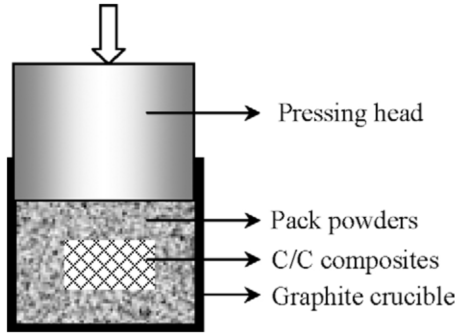

Fig. 1. Sketch of preparing SiC coating for C/C composites by HPRS.

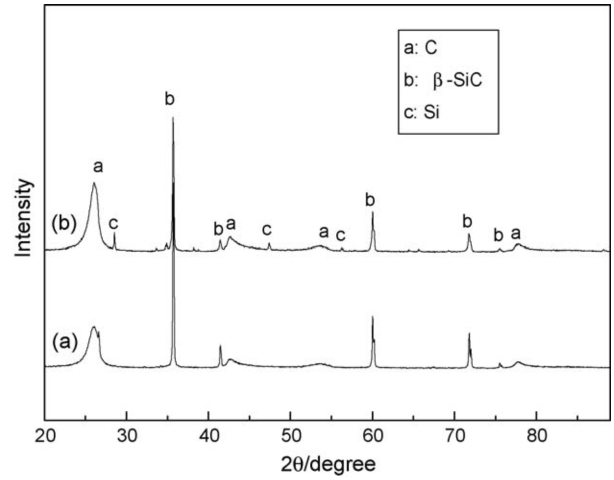

Fig. 2. XRD patterns of the surface of the C/C samples with coatings prepared by PRS (a) and HPRS (b).

slight argon flow. During this process, Si will melt and react with C/C composites to form SiC coating. The sketch of preparing SiC coating for C/C composites by HPRS is shown in Fig. 1. For comparison, another kind of SiC coating was prepared by pressure-less reactive sintering (PRS) with the same starting powders and heat-treatment temperature.

To investigate the thermal stress resistance of the coatings, the thermal cycling test between 1773 K and room temperature was performed. The samples were weighed at room temperature by electronic balance with a sensitivity of ± 0.1 mg during thermal cycling tests. To evaluate the mechanical properties of the samples, three-point bending tests were carried out in a servohydraulic machine of 8871 (INSTRON CO., Ltd., USA). The span was 40 mm and the crosshead speed was 0.5 mm/min. Five samples for each kind of sample were tested and the final flexural properties were obtained by computing the average values of five samples.

The morphologies and crystalline structures of the coatings were analyzed by JSM-6460 scanning electron microscopy (SEM) and Rigaku D/max-3C X-ray diffraction (XRD).

3. Results and discussion

Fig. 2 shows XRD patterns of the surface of the coated samples. From Fig. 2(a), the diffraction peaks of graphite and cubic β-SiC are detected from the surface of the coating obtained by PRS. Graphite is corresponding to the C/C substrate, and β-SiC comes from the coating. Fig. 2(b) displays that a new phase of Si was generated in the coating by HPRS. During the preparation of coating, melted Si will react with C/C to form SiC coating. Under pressure, some of liquid Si in the pack powders was congregated to the surface of the samples and left in the SiC coating, which is advantageous to relax the stress at the end of the cracks and heal up the cracks in the coating.

Fig. 3(a) displays SEM image of the coating prepared by PRS. This coating has a porous structure with some microcracks, resulted from bigger coefficient of thermal expansion of SiC coating than that of C/C composites. Due to its loose structure, this coating might provide a poor oxidation protective ability for C/C composites. From Fig. 3(b), the as-received coating prepared by HPRS possesses a dense and crack-free structure. With pressure, the coatings will have compressive stress, which can effectively make up for the tensile stress induced by the shrinkage of SiC coating during the cooling process from high temperature to room temperature. The thermal stress in SiC coatings can be relaxed by the introduction of pressure.

SEM images of the cross-section of the SiC coated samples are shown in Fig. 4. From Fig. 4(a), there are large numbers of holes in the SiC coating prepared by PRS, owing to the difficult sintering of SiC ceramic without pressure. From Fig. 4(b), SiC coating prepared by HPRS is denser than that without pressure,

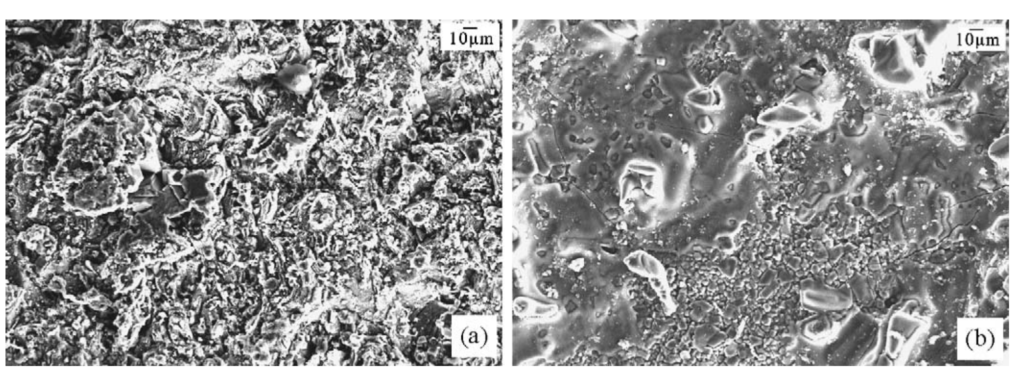

Fig. 3. SEM images of the surface of the coatings prepared by PRS (a) and HPRS (b).