



## Oxidation behavior of C/C composites with SiC/ZrSiO<sub>4</sub>–SiO<sub>2</sub> coating



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**Abstract:** A SiC/ZrSiO<sub>4</sub>–SiO<sub>2</sub> (SZS) coating was successfully fabricated on the carbon/carbon (C/C) composites by pack cementation, slurry painting and sintering to improve the anti-oxidation property and thermal shock resistance. The anti-oxidation properties under different oxygen partial pressures (OPP) and thermal shock resistance of the SZS coating were investigated. The results show that the SZS coated sample under low OPP, corresponding to the ambient air, during isothermal oxidation was 0.54% in mass gain after 111 h oxidation at 1500 °C and less than 0.03% in mass loss after 50 h oxidation in high OPP, corresponding to the air flow rate of 36 L/h. Additionally, the residual compressive strengths (RCS) of the SZS coated samples after oxidation for 50 h in high OPP and 80 h in low OPP remain about 70% and 72.5% of those of original C/C samples, respectively. Moreover, the mass loss of SZS coated samples subjected to the thermal cycle from 1500 °C in high OPP to boiling water for 30 times was merely 1.61%.

**Key words:** C/C composite; SiC/ZrSiO<sub>4</sub>–SiO<sub>2</sub> coating; oxygen partial pressure; anti-oxidation; thermal shock; residual compressive strength

### 1 Introduction

Carbon/carbon (C/C) composites possess many advantages such as light weight, high strength and modulus at the elevated temperature [1,2]. They are considered to be the potential candidates for high-temperature structures. However, the C/C composites are prone to oxidation in a oxidizing environment above 400 °C [3]. Thus, many investigations on curing Achilles' heel of C/C composites have been carried out for several decades [4–7].

Currently, the coating technology has been regarded as an effective way to protect the C/C composites against oxidation [8]. It is generally known that the silicon carbide (SiC) coating possesses good physical and chemical compatibility to C/C matrix, and is widely used as bonding layer between C/C and outer ceramic layer to provide extra protection for C/C composites [9,10]. However, a single SiC layer cannot provide a long-term protection for C/C matrix [6]. Hence, great efforts were concentrating on the silicon-based multilayer coatings in order to improve the anti-oxidation of C/C composites [11,12]. The zirconium silicate (ZrSiO<sub>4</sub>) has been

considered as a candidate material to apply at high-temperature due to its excellent chemical stability and low coefficient of thermal expansion ( $4.1 \times 10^{-6}/^{\circ}\text{C}$  at 1400 °C) [13,14]. SUN et al [15,16] and LIU et al [17] developed a ZrSiO<sub>4</sub>/SiC coating by hydrothermal electrophoretic deposition which showed a good anti-oxidation as well as the SiC–Si–ZrSiO<sub>4</sub> coating systems. It also proved that the ZrSiO<sub>4</sub>–SiC coating systems offered good solutions for the oxidation protection of C/C composites. However, according to the work of LIU et al [17], SUN et al [15,16] and LI et al [1], the preparation methods for coating systems were pretty complicated. Additionally, most of their investigations were limited in the isothermal oxidation in ambient air. It is well known that the C/C composites suffered to different environments with different oxygen partial pressures (OPP) presented diversity in properties at high temperature during the potential applications [18,19]. Hence, the silicon-based coating systems subjected to different OPP at high temperature should be comprehensively studied. In the present work, the SZS coating was simply prepared by combination of pack cementation for SiC inner coating and slurry painting then sintered for ZrSiO<sub>4</sub>–SiO<sub>2</sub> out coating. The anti-

oxidation of SZS coating at 1500 °C in high OPP (air flow rate: 36 L/h) and low OPP (ambient air), was systematically investigated. Additionally, the microstructures, residual compressive strength after oxidation test and thermal shock resistance from 1500 °C in high OPP (air flow rate: 36 L/h) to boiling water were studied to evaluate the performance of the SZS coating system. The current study aims to provide some valuable information for the further research in the protection of C/C composites at the elevated temperature under different oxygen partial pressures.

## 2 Experimental

### 2.1 Preparation of coatings

The small specimens (10 mm × 10 mm × 10 mm) with the density of 1.67 to 1.70 g/cm<sup>3</sup> as substrates were cut from the bulk 2.5-dimensional C/C composites, which were prepared by chemical vapor infiltration. The C/C specimens were polished by using 200, 600 and 1000 grit SiC paper, successively, then followed by water cleaning and drying at 120 °C for 2 h. The powder composition for pack cementation included: 60%–65% Si, 5%–10% carbon black, 10%–30%  $\beta$ -SiC and 0–5% Al<sub>2</sub>O<sub>3</sub> (mass fraction). All the powders (analytical grade) were mixed by ball-milling for 2 h in ethanol solution. The C/C composite specimens and the powder mixtures were placed in a graphite crucible, and heated to 1750 °C in an argon atmosphere and then held at 1750 °C for 2 h to form the SiC coating. After the preparation of SiC layer, the ZrSiO<sub>4</sub>–SiO<sub>2</sub> coating was obtained on the as-prepared SiC coating surface by slurry painting and sintering method. The slurry painting composition in this procedure included: 20%–60% SiO<sub>2</sub>, 30%–60% ZrO<sub>2</sub> and 0–5% sinter aids (mass fraction). All the powders were analytically graded and mixed by ball-milling with a ethanol solution containing 3% polyvinyl alcohol for 2 h. After slurry painting process, the coated specimens were dried at 100 °C and then sintered at 1500 °C under argon atmosphere for 1 h to form the ZrSiO<sub>4</sub>–SiO<sub>2</sub> coating.

### 2.2 Isothermal oxidation test

The samples with SiC and SZS coatings were tested at 1500 °C in low OPP (ambient air) or high OPP (air flow air: 36 L/h) in a corundum tube furnace (tube diameter: 10 cm, Length: ~100 cm) to investigate the isothermal oxidation behavior. The cumulative mass change rates ( $\Delta w$ ) of the specimens were described by the following expression and were reported as a function of time:

$$\Delta w = \frac{m_i - m_0}{m_0} \times 100\% \quad (1)$$

where  $m_0$  and  $m_i$  are measured by an electronic balance with a sensitivity of  $\pm 0.1$  mg and represent the masses of the specimens before and after oxidation, respectively.

### 2.3 Thermal shock test

The thermal shock test was performed on the SZS coated C/C composites samples from 1500 °C in high OPP to boiling water. The specimens were placed into the corundum tube furnace and heated at 1500 °C for 3 min. Subsequently, the specimens were poured into the boiling water (about 100 °C) to cool down quickly to room temperature. Finally, after 2 h drying at 100 °C in the oven, the masses of specimens were measured by electronic balance with sensitivity of  $\pm 0.1$  mg, and the mass loss was calculated by Eq. (1). The above steps were repeated 30 times.

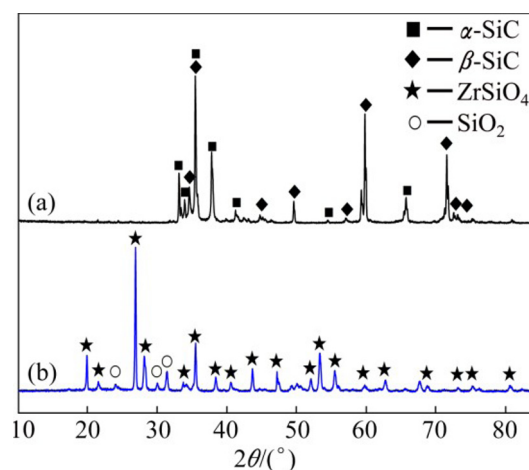
### 2.4 Characterization

According to ASTM C1258–97, the compressive tests for the residual strength of specimens after oxidation were carried out on a universal testing machine (Instron 1196) with a loading rate of 1 mm/min. The microstructures and the phase composition of the SiC and SZS coatings were characterized by scanning electron microscopy (SEM, FEI Nova Nano SEM–230) with energy dispersive X-ray spectroscopy and X-ray diffraction (XRD) analyzer (Rigaku Ltd., Japan), respectively.

## 3 Results and discussion

### 3.1 Phase compositions and microstructures of coatings

Figure 1 shows the XRD patterns of the as-prepared SiC coating and SZS coating. As shown in Fig. 1, the SiC coating is composed of  $\alpha$ -SiC and  $\beta$ -SiC, and the SZS coating is composed of ZrSiO<sub>4</sub> and residual SiO<sub>2</sub> after the



**Fig. 1** XRD patterns of SiC and SZS coatings: (a) SiC coating; (b) SZS coating after sintering

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