

Oxidation Behavior of $\text{Yb}_2\text{Si}_2\text{O}_7$ Modified SiC/SiC Mini-composites

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Abstract: Silicon-carbide-fiber-reinforced silicon-carbide-ceramic-based matrix (SiC/SiC) composites possess excellent properties such as low density, high strength and high temperature resistance, showing a potential application for structural components in the aerospace field, but their oxidation behavior remains largely unknown. In this study, $\text{Yb}_2\text{Si}_2\text{O}_7$ modified SiC/SiC ($\text{SiC/SiC-Yb}_2\text{Si}_2\text{O}_7$) mini-composites were prepared by introducing $\text{Yb}_2\text{Si}_2\text{O}_7$ as anti-oxidation phase into SiC fiber bundles *via* Sol-Gel and depositing SiC matrix by chemical vapor deposition (CVD). Influence of $\text{Yb}_2\text{Si}_2\text{O}_7$ on microstructure, mechanical property and oxidation behavior of SiC/SiC mini-composites was investigated. The results showed that after oxidation in air at 1200 and 1400 °C for 50 h, the tensile strength retentions of SiC/SiC mini-composites were 77% and 69%, respectively, and the fracture morphology exhibited flat. The $\text{Yb}_2\text{Si}_2\text{O}_7$ introduced by Sol-Gel partially distributed in layers, contributing to the toughening of the material. On the fracture surface, there was interlayer debonding, which extended energy dissipation mechanism of SiC/SiC mini-composites. Tensile strength of SiC/SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$ mini-composites at room temperature was 484 MPa. After oxidation in air at 1200 and 1400 °C for 50 h, the tensile strengths decreased to 425 and 374 MPa, resulting in retention rates of 88% and 77%, respectively. It displayed typical non-brittle fracture characteristics. The interface oxygen content of SiC/SiC mini-composites at the fracture surface was higher than that of SiC/SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$ mini-composites, indicating that introduction of $\text{Yb}_2\text{Si}_2\text{O}_7$ could alleviate oxygen diffusion towards the interface, and therefore improve the oxidation resistance of SiC/SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$ mini-composites.

Key words: SiC/SiC mini-composite; matrix modification; $\text{Yb}_2\text{Si}_2\text{O}_7$; oxidation behavior

SiC/SiC composites have emerged as a promising choice and development pathway for the high temperature structural components of next-generation aero engines^[1-2]. This is owing to their outstanding properties, including low density and high-temperature resistance, which facilitate weight reduction and enhance service temperature capability^[3]. However, the high-temperature structural components in service face a challenging environment marked by elevated temperatures and water-oxygen coupling environment, imposing stricter demands on the oxidation resistance of materials^[4]. Presently, efforts to enhance the oxidation

resistance of SiC/SiC composites mainly concentrate on the environmental barrier coatings (EBCs)^[5-6]. Nonetheless, the intrinsic oxidation resistance of the composite remains unaddressed, leaving it vulnerable to oxidation erosion after coating degradation. Therefore, enhancing the intrinsic oxidation resistance of SiC/SiC composites is crucial for extending the service life of the materials^[7].

Rare earth silicates, characterized by a low thermal expansion coefficient, low oxygen diffusion coefficient, and low water vapor volatilization rate, are emerging as a novel optimization system for EBCs^[8]. Currently, there are numerous reports on the modification of SiC/SiC

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composites with rare earth silicates to enhance their oxidation resistance properties^[9-11]. Boakye *et al.*^[12] fabricated SiC/SiC mini-composites featuring $\text{Y}_2\text{Si}_2\text{O}_7$ interfaces using an *in-situ* coating method combined with polymer impregnation pyrolysis technique. Oxidation resistance tests revealed that the $\text{Y}_2\text{Si}_2\text{O}_7$ coating remained stable and maintained the crucial function of the weak interface between the fiber and matrix after exposure to steam at 1000 °C for 100 h^[13]. Wang *et al.*^[14] prepared layered- $\text{Y}_2\text{Si}_2\text{O}_7$ -modified SiC/SiC composites using the chemical vapor infiltration (CVI) method. Results demonstrate that $\text{Y}_2\text{Si}_2\text{O}_7$ tends to accumulate on the oxidation surface, forming a protective layer during the oxidation process. Notably, the $\text{Yb}_2\text{Si}_2\text{O}_7$ exhibits greater structural stability than $\text{Y}_2\text{Si}_2\text{O}_7$, as it does not undergo the phase change at high temperatures. This offers the advantages of high melting point, low density, and low oxygen diffusion coefficient^[15]. These characteristics are beneficial for the application of SiC/SiC composites.

In this study, SiC/SiC mini-composites modified by $\text{Yb}_2\text{Si}_2\text{O}_7$ were prepared by Sol-Gel and CVD methods to enhance oxidation resistance. SiC/SiC mini-composites refer to unidirectional composites. Microstructure of the $\text{Yb}_2\text{Si}_2\text{O}_7$ modified SiC/SiC mini-composite was characterized. Effects of introducing $\text{Yb}_2\text{Si}_2\text{O}_7$ on the mechanical properties and oxidation resistance of SiC/SiC mini-composites were discussed.

1 Experimental

1.1 Preparation of $\text{Yb}_2\text{Si}_2\text{O}_7$ modified SiC/SiC mini-composites

$\text{Yb}_2\text{Si}_2\text{O}_7$ modified SiC/SiC mini-composites were prepared using a combination method of Sol-Gel and CVD. Ytterbium nitrate pentahydrate ($\text{Yb}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, AR, 99.99%, Shanghai Adamas Reagent Co., Ltd., China), tetraethyl orthosilicate ($(\text{C}_2\text{H}_5)_4\text{SiO}_4$, TEOS, AR, 99%, Shanghai Adamas Reagent Co., Ltd., China) and hydrochloric acid (HCl, AR, 36.0%–38.0%, Sinopharm Chemical Reagent Co., Ltd., China) were dissolved in ethanol (AR, 99.7%, Shanghai Titan Scientific Co., Ltd., China). In brief, the molar concentration of $\text{Yb}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ was 0.4 mol/L and the molar ratio of $\text{Yb}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ to TEOS was 1 : 1.2. The mixture solution was vigorously stirred at room temperature for 2 h, and then catalyst hydrochloric acid was added to promote the formation of sol. After stirring at 70 °C for several hours and aging at room temperature for several days, the uniform and transparent sol was obtained.

As depicted in Fig. 1, preparation of $\text{Yb}_2\text{Si}_2\text{O}_7$ modified SiC/SiC mini-composites includes fiber treatment, interfacial deposition, SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$ multilayer matrix preparation, and final SiC matrix preparation. The SiC

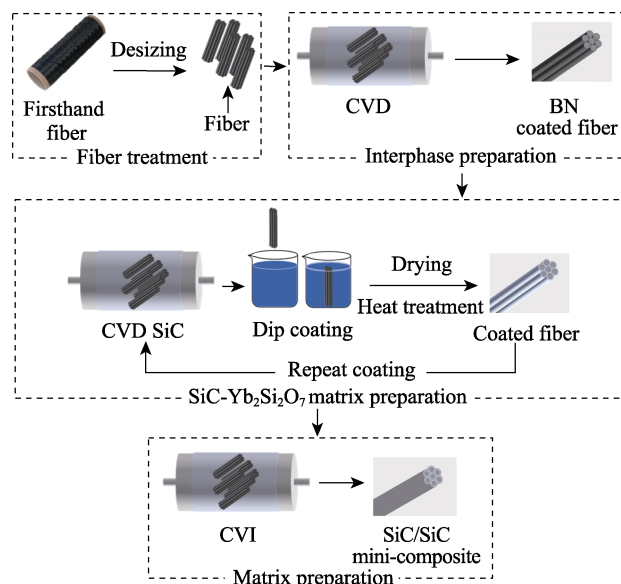


Fig. 1 Schematic diagram of preparation of $\text{Yb}_2\text{Si}_2\text{O}_7$ modified SiC/SiC mini-composites

fiber bundle was wound onto a frame graphite mold to maintain its straightened state. After desizing, a BN interface was deposited onto the surface of SiC fibers, succeeded by deposition of SiC as a protective layer using CVD. Subsequently, the preform was immersed in $\text{Yb}_2\text{Si}_2\text{O}_7$ sol along the fiber direction and left for more than 2 min. Then, using the dip-coating method at a constant speed, $\text{Yb}_2\text{Si}_2\text{O}_7$ gel film was formed on the fiber preform surface as the solvent evaporated. The $\text{Yb}_2\text{Si}_2\text{O}_7$ coating was prepared after heat treatment at 1200 °C for 1 h under an argon atmosphere. The process of preparing SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$ multilayer matrix was repeated three times. Finally, SiC matrix was deposited until the material was compacted to obtain the mini-composites (SiC/SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$).

1.2 Test and characterization

The polished cross-section morphology and tensile fracture morphology of SiC/SiC mini-composites were observed by field emission scanning electron microscope (SEM, Magellan 400, FEI, USA). The mechanical tests were carried out on MTS servo-hydraulic machine with a load displacement control of 0.2 mm/min. Different mini-composites were placed in a tubular furnace and heated to 1200 and 1400 °C at rates of 8 °C/min below 700 °C and 5 °C/min above 700 °C. There were more than three parallel samples of mini-composites oxidized at each temperature. The cooling rate was programmed by the furnace to cool slowly to 700 °C at a rate of 5 °C/min, followed by natural cooling to room temperature within the furnace. The total oxidation time duration was 50 h. Throughout the high-temperature air oxidation process, both ends of the tube furnace remained open.

The oxidation kinetics were analyzed using a parabolic power law as follows^[16]:

$$\frac{w_i - w_0}{w_0} = \sqrt{kt} \quad (1)$$

where w_i is the sample weight after oxidation for i h, w_0 is the sample weight before oxidation, t is the oxidation time, and k is the parabolic rate constant. The number of samples used in each test was more than three parallel samples.

2 Results and discussion

Fig. 2 illustrates the morphology and structure of the fibers and mini-composites. As shown in Fig. 2(a), the SiC protective layer is deposited by CVD onto BN layer to protect BN interface during the introduction of Yb₂Si₂O₇ *via* Sol-Gel. The step was taken to prevent the oxidation of BN due to the presence of oxygen-containing functional groups in the precursor sol during the Yb₂Si₂O₇ preparation. The boundary between BN interface and SiC protective layer is clear, and the layer thickness is uniform. The Yb₂Si₂O₇ layer prepared *via* Sol-Gel is grown *in situ* on the surface of SiC layer (Fig. 2(b)). Cross-section morphologies of SiC/SiC mini-composites and modified SiC/SiC-Yb₂Si₂O₇ mini-composites are shown in Fig. 2(c-f). The Yb₂Si₂O₇ layer, evident in the white region, distributes outside the BN interface and SiC protective layer, demonstrating a multi-layer matrix morphology in local areas. However, achieving a uniform distribution of the Yb₂Si₂O₇ phase becomes increasingly difficult with increasing preparations of SiC *via* CVD and Yb₂Si₂O₇ by Sol-Gel. The density and open porosity of SiC/SiC mini-composites are 2.61 g/cm³ and 13.90%, whereas SiC/SiC-Yb₂Si₂O₇ mini-composites are 2.52 g/cm³ and 14.12%, respectively. The content of Yb₂Si₂O₇ is about 5.9% of the total content of SiC matrix. Some Yb₂Si₂O₇ concentrates in the matrix, particularly in the bridge region between the fibers, resulting in the

formation of SiC-Yb₂Si₂O₇ complex phase matrix. In comparison to SiC/SiC mini-composites, the internal pores of SiC/SiC-Yb₂Si₂O₇ mini-composites increase, predominantly concentrated within the Yb₂Si₂O₇ layer enrichment. This might be attributed to the introduction of Yb₂Si₂O₇ blocking the channel of reaction gas into the fiber bundle during CVD process. This conclusion is also supported by the findings of Wang *et al.*^[14], who prepared Y₂O₃-modified SiC/SiC composites *via* CVI. The results indicate that an increase in the open porosity of the composites could lead to a decrease in their mechanical strength.

The tensile tests of SiC/SiC mini-composites and SiC/SiC-Yb₂Si₂O₇ mini-composites were conducted at room temperature. As shown in Fig. 3, the force-displacement curves for both mini-composites exhibit non-linear behavior. The tensile fracture morphologies are illustrated in Fig. 4. Compared with Fig. 4(a, c), it's

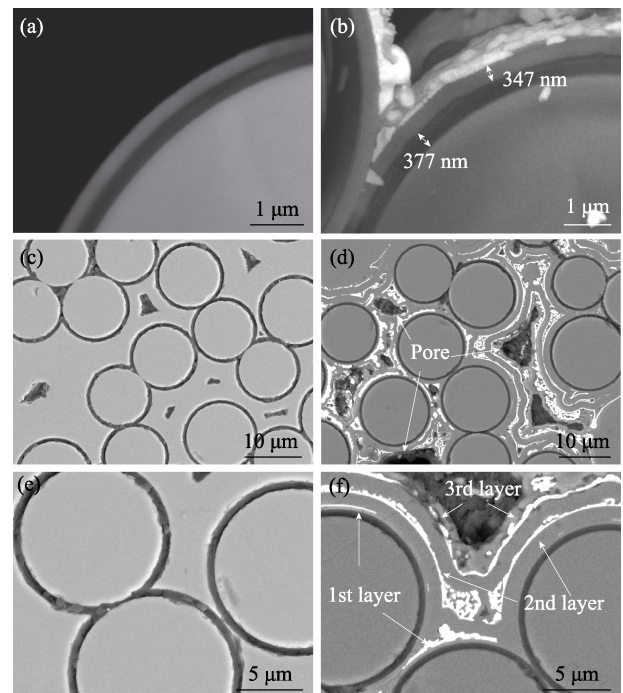


Fig. 2 SEM images of cross-sections of (a, b) coated fibers, (c, e) SiC/SiC, and (d, f) SiC/SiC-Yb₂Si₂O₇ mini-composites

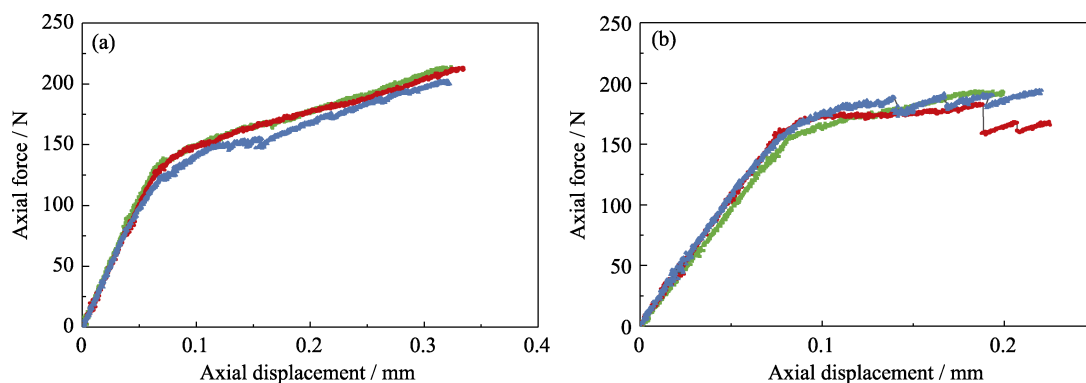


Fig. 3 Tensile force-displacement curves of (a) SiC/SiC and (b) SiC/SiC-Yb₂Si₂O₇ mini-composites

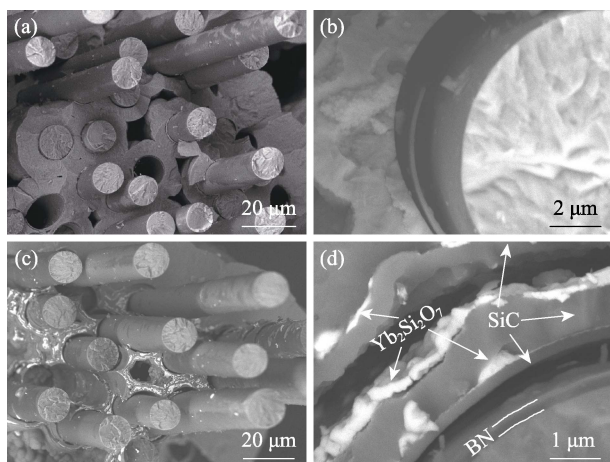


Fig. 4 Fracture morphologies of (a, b) SiC/SiC and (c, d) SiC/SiC-Yb₂Si₂O₇ mini-composites

evident that both mini-composites exhibit distinct non-brittle fracture characteristics. Notable fiber pullout is observed, which is unimpeded by the modification of Yb₂Si₂O₇ regarding the length of fiber pullout. Additionally, interface debonding is exclusively observed at BN interface in SiC/SiC mini-composites (Fig. 4(b)), while it occurs at both BN and Yb₂Si₂O₇-SiC interfaces in SiC/SiC-Yb₂Si₂O₇ mini-composites (Fig. 4(d)). Boakye *et al.*^[12] reported that debonding at the rare earth silicate and SiC interface in SiC/Y₂Si₂O₇/SiC mini-composites was attributed to the weak bond strength between the rare earth silicate and SiC. The fracture morphologies indicate that the addition of Yb₂Si₂O₇ enhances the energy dissipation path of SiC/SiC mini-composites, thereby contributing to the toughening of the material.

SiC/SiC and SiC/SiC-Yb₂Si₂O₇ mini-composites underwent oxidation in air at 1200 and 1400 °C for 50 h, and the oxidation kinetics was analyzed as depicted in Fig. 5. The results reveal that the weights of both

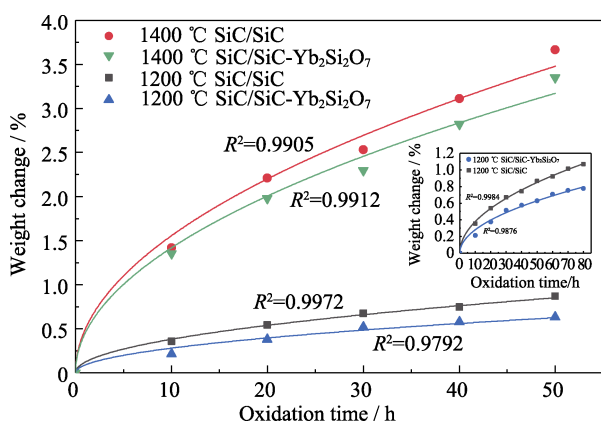


Fig. 5 Weight change of SiC/SiC and SiC/SiC-Yb₂Si₂O₇ mini-composites during oxidation at 1200 and 1400 °C for 50 h with inset showing the weight change during oxidation at 1200 °C for 80 h

materials increase with the extended oxidation time, whereas the weight gain rates gradually decrease. The weight change during oxidation at 1400 °C is significantly greater than that at 1200 °C, which indicates that oxidation is intensified with rising temperature. The weight change curves are fitted to the parabolic power law expressed in Eq. (1). The fitting results exhibit high R^2 (>0.97), and the inset demonstrates that a similar R^2 is sustained even after 80 h of oxidation at 1200 °C. This suggests that high accuracy of the fitting results, and the oxidation process of the two mini-composites is governed by a diffusion mechanism. Compared to SiC/SiC mini-composites, the weight gain rate of SiC/SiC-Yb₂Si₂O₇ mini-composites displays a decreasing trend. This phenomenon may be attributed to the presence of Yb₂Si₂O₇ phase, which has a low oxygen diffusion coefficient, thus reducing the overall oxygen diffusion rate.

The tensile properties of different composites after oxidation at 1200 and 1400 °C were assessed. Tensile fracture morphologies of SiC/SiC mini-composites in Fig. 6(a, c, e) indicate that the fiber pullouts from the matrix are significantly shorter than those in the as-manufactured samples. The tensile fracture in SiC/SiC mini-composites displays a visible flat area. As shown in Fig. 6(c), pores are present near the BN interface within the flat region. These pores may be caused by the volatilization of gas generated during the oxidation of BN, leading to interface embrittlement and observed flat fractures. The lengths of fiber pullouts in SiC/SiC-Yb₂Si₂O₇ mini-composites are longer than those in SiC/SiC, with the pullout of SiC-Yb₂Si₂O₇ interface still observable. Results obtained by energy dispersive spectrometer (EDS) show that the oxygen atom ratio at BN interface in SiC/SiC-Yb₂Si₂O₇ mini-composites is lower than that in SiC/SiC mini-composites.

Table 1 summarizes the changes in tensile strength of mini-composites before and after oxidation. The room temperature tensile strengths of SiC/SiC and SiC/SiC-Yb₂Si₂O₇ mini-composites are 506 and 484 MPa, respectively, which are relatively high strength values compared with the existing literature reports^[17-18]. Following oxidation for 50 h, the strengths of the mini-composites decrease, and SiC/SiC-Yb₂Si₂O₇ mini-composites maintain retention rates of 88% and 77% after oxidation at 1200 and 1400 °C, respectively, whereas SiC/SiC mini-composites retain 77% and 69%. The improved strength retention rate in SiC/SiC-Yb₂Si₂O₇ mini-composites is attributed to the reduced oxygen content at BN interface.

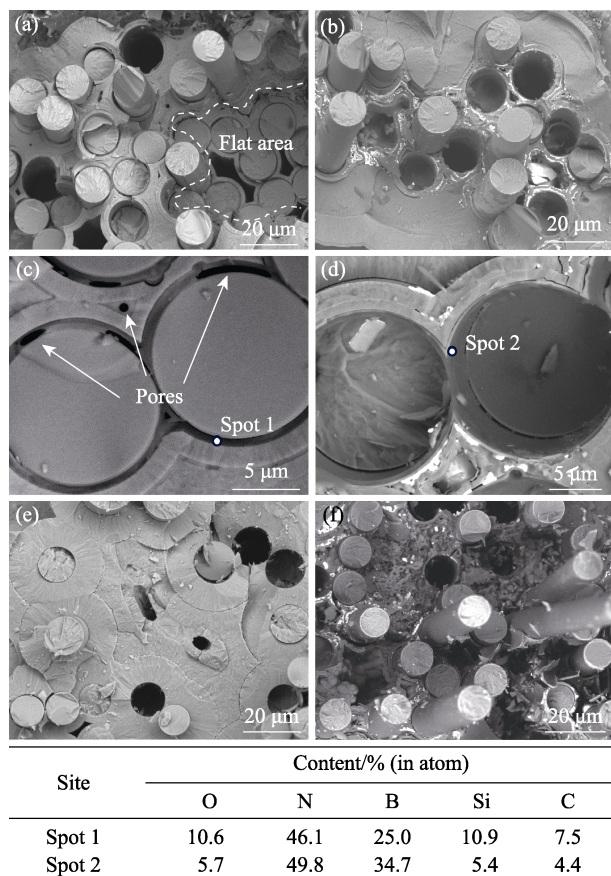


Fig. 6 Tensile fracture morphologies of (a, c, e) SiC/SiC, and (b, d, f) SiC/SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$ mini-composites after oxidation at (a-d) 1200 °C with EDS results (table at the bottom), and (e, f) at 1400 °C, respectively

Table 1 Tensile properties of mini-composites

Material	Tensile strength/MPa		
	As-prepared	Oxidation at 1200 °C	Oxidation at 1400 °C
SiC/SiC	(506±56)	(391±69)	(351±56)
SiC/SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$	(484±83)	(425±21)	(374±33)

3 Conclusions

SiC/SiC mini-composites modified by $\text{Yb}_2\text{Si}_2\text{O}_7$ were prepared successfully. $\text{Yb}_2\text{Si}_2\text{O}_7$, introduced via Sol-Gel, exhibits a partial lamellar distribution in matrix. Interlayer debonding occurs at $\text{Yb}_2\text{Si}_2\text{O}_7$ -SiC interface, contributing to material toughening. During the oxidation process in static air at 1200 and 1400 °C for 50 h, the weight change of the material follows the parabolic power law, indicating that the oxidation is predominantly governed by the diffusion mechanism. The incorporation of the $\text{Yb}_2\text{Si}_2\text{O}_7$, which has a low oxygen diffusion coefficient, decreases the weight change rate of the composite during oxidation. After oxidation, the fracture morphology of SiC/SiC

mini-composites displays a flat fracture, and SiC/SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$ mini-composite maintains a significant fiber pullout. Both interface debonding and fiber pullout are observable at $\text{Yb}_2\text{Si}_2\text{O}_7$ -SiC interface. Moreover, EDS results reveal that the interface oxygen content at the fracture surface of SiC/SiC mini-composites is higher than that of SiC/SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$ mini-composites. After being exposed to oxidation in air at 1200 and 1400 °C for 50 h, the tensile strength retention rates of SiC/SiC- $\text{Yb}_2\text{Si}_2\text{O}_7$ mini-composites are 88% and 77%, respectively. The findings suggest that modifying by $\text{Yb}_2\text{Si}_2\text{O}_7$ can enhance the oxidation resistance of the composite.

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Yb₂Si₂O₇ 改性 SiC/SiC 复合材料的氧化行为研究

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摘要: 碳化硅纤维增强碳化硅陶瓷基(SiC/SiC)复合材料具有低密度、耐高温、高强度等优异性能, 在热端部件结构材料领域具有广泛应用前景。本研究通过溶胶-凝胶法在 SiC 纤维束内引入 Yb₂Si₂O₇ 抗氧化相, 并结合化学气相沉积(CVD)法沉积 SiC 基体, 制备了 Yb₂Si₂O₇ 改性 SiC/SiC(SiC/SiC-Yb₂Si₂O₇)复合材料, 讨论了 Yb₂Si₂O₇ 对 SiC/SiC 复合材料微观结构、力学性能及氧化行为的影响。结果表明, SiC/SiC 复合材料经过 1200 和 1400 °C 空气氧化 50 h 后的强度保持率分别为 77%和 69%, 拉伸断口处观察到纤维基体平齐断裂。溶胶-凝胶法引入的 Yb₂Si₂O₇ 在基体中部分呈层状分布, 层状 Yb₂Si₂O₇ 相有利于材料的增韧, 拉伸断口处出现层间脱黏现象, 扩展了 SiC/SiC 复合材料能量耗散机制。SiC/SiC-Yb₂Si₂O₇ 复合材料的室温拉伸强度为 484 MPa, 在 1200 和 1400 °C 空气中氧化 50 h 后的剩余强度分别为 425 和 374 MPa, 强度保持率分别为 88%和 77%, 仍表现出典型的非脆性断裂特征。SiC/SiC 复合材料断口处界面氧含量大于 SiC/SiC-Yb₂Si₂O₇ 复合材料, 说明引入 Yb₂Si₂O₇ 缓解了氧向界面内的扩散, 改善了 SiC/SiC-Yb₂Si₂O₇ 复合材料的抗氧化性能。

关键词: SiC/SiC 复合材料; 基体改性; Yb₂Si₂O₇; 氧化行为

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