Effect of SiC Whiskers on Properties of SiC Coating for Carbon/carbon Composites

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Abstract: Using SiC whiskers as the reinforcement materials, a kind of whisker-toughened SiC coating (SiCw-toughened SiC coating) was produced on the surface of carbon/carbon (C/C) composites by pack cementation technique. SEM and XRD were adopted to analyze the microstructure and phase composition of as-prepared coating. The thickness and hardness of the as-coated C/C composites were tested by Vickers Sclerometer. The thermal expansion coefficient of the coated C/C composites was analyzed by TMA thermal mechanical analyzer. The oxidation test of SiCw-toughened SiC coated C/C was also carried out in air at 1573K. The results show that SiC whiskers can efficiently decrease the size and frequency of cracks in coating after adding SiC whiskers in SiC coating. The thickness of SiCw-toughened SiC coating is 134μm, while that of SiC coating is up to 164μm. After adding SiCw in SiC coating, the hardness of the coated C/C composites increases, which is 1497kg/mm2. The thermal expansion coefficient of the as-coated C/C composites is 1.55×10-6/K from 532K to 703K and is 1.92×10-6/K from 713K to 873K. While the thermal expansion coefficient of C/C composites coated SiC coating without SiC whiskers is 1.91×10-6/K from 532K to 873K. Moreover, the oxidation protective ability of SiC coating can be improved by the introduction of SiCw. The as-prepared SiCw-toughened SiC coating can protect C/C composites from oxidation at 1573K in air for 21h and the weight loss is 8%, but the weight loss of SiC coating without SiC whiskers has up to 18% after oxidation for the same time.

Keywords: Carbon composites，Coatings，Diffusion

1. Introduction

Carbon/carbon (C/C) composites are widely used as the high temperature materials because of their excellent properties, such as low density, low heat-expansion coefficient and the retention of mechanical properties at the high temperature. Therefore, C/C composites are the ideal materials in the aviation and aerospace industries [1]. However, C/C materials will be oxidized above 773K in oxidation atmosphere，which greatly retards the utilization of C/C composites in high temperature fields [1,2].

Oxidation protective coatings are the effective methods to protect C/C composites from oxidation at high temperature. SiC are the suitable coating materials for C/C composites due to their excellent interface bonding with C/C. SiC coating is usually used as the transition layer on C/C composites [3-5]. However, cracks generally appear in the SiC coating due to the mismatch of the thermal expansion coefficient between SiC coating and C/C composites, which will result in the failure of SiC coating.

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Whiskers are a kind of single crystal materials with high strength and little defect. At present, ceramic matrix composites reinforced by whiskers have been greatly developed [6-8]. According to the toughening mechanism of whiskers including crack bridging, whiskers pull-out and crack deflection, SiC whiskers was used to reinforce the coatings in the present paper which will improve the anti-oxidation property of ceramic coatings.

In the present work, the SiC coating reinforced by SiC whiskers (SiCw) was prepared by pack cementation method. The structure and physical properties play the important role on the oxidation property of coatings. So the structural and physical properties were analyzed. And oxidation protection property of the coating reinforced by SiCw was investigated. The work is useful reference for further study about coatings reinforced by SiC whiskers.

2. Experimental

The specimens (10mm×10mm×10mm) used as substrates were cut from 2D-C/C brake disk with density of 1.8g/cm^3. Before carrying out the coating process, the specimens were hand-polished using 250 grit SiC paper, then cleaned with alcohol and dried in air.

SiCw were bought from Zibo electro-porcelain factory in Shandong province, China. SiCw were dispersed ultrasonically in alcohol solution for 40 minutes. Then the powders composed of Si (300 mesh), graphite (300 mesh) and SiCw were added into the alcohol solution. The solution was mixed by tumbling in a ball mill for 20h and dried at 353K for 4h. The dried powers were mulled. C/C specimens and the mixed powers were placed in a graphite crucible. The crucible was placed into an electric furnace, which was heated in argon atmosphere to 1873K and maintained at that temperature for 2h to form SiC coating reinforced by SiCw. In order to find whether the anti-oxidation property of composites improved or not, we also conducted the SiC coating which was not reinforced by SiCw. Other components and production process for the SiC coating were as same as the SiC coating reinforced by SiCw. The same SiC outer coatings were produced on SiC coating and reinforced SiC coating by pack cementation process of which the detailed operations were as same as that of as-received SiC coating.

The morphology, crystalline structure and thickness of coatings were analyzed using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The Vickers hardness of C/C and the coated C/C were tested by HX-100 sclerometer. TMA thermal mechanical analyzer, with promoting temperature rate 15K/min, was used to measure the thermal expansion coefficient of samples from 532K to 873K.

3. Results and discussion

3.1 Structure of the coatings

Fig. 1a shows the XRD pattern of SiC coating. It shows the coating obtained by pack cementation technique is β-SiC structure. Fig.1b shows the XRD pattern of SiC coating reinforced by SiCw. It shows a new phase of α-SiC was formed in the coating which testifies that SiCw could promote the production of α-SiC phase, but the reason has not been

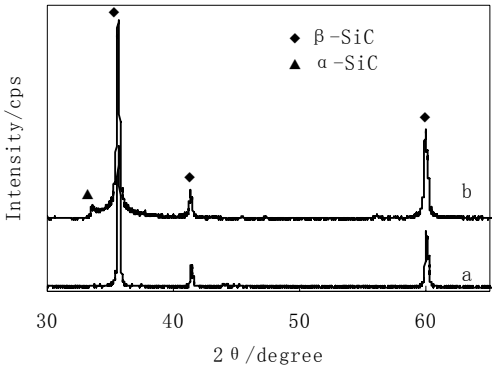
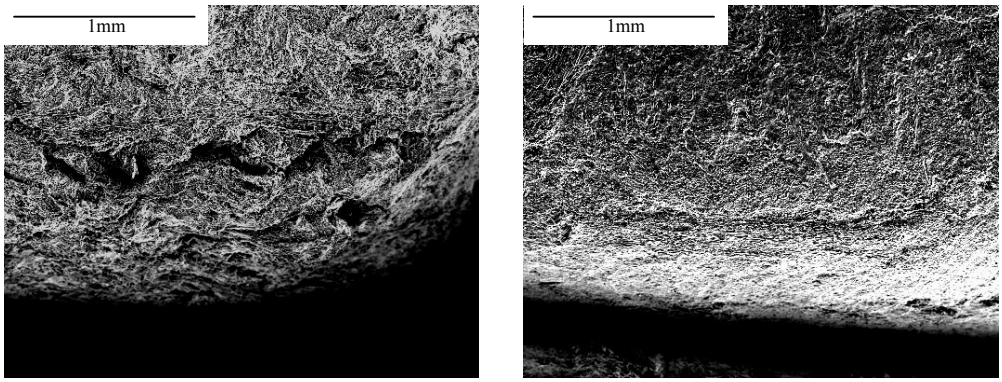


Fig.1 XRD patterns of (a) SiC coating and (b) reinforced SiC coating by SiC whiskers

discovered. Moreover, it is found that the diffractive peaks of β-SiC in Fig. 1b broaden compared with the peaks in Fig. 1a. It is believed that SiCw prevent the SiC crystals from growing up continually. The fine SiC crystal results in the broadening peaks in XRD pattern [9, 10].

The cross section of coatings is the place where the stress focuses on. The cracks usually lie on the cross section. Fig. 2a and 2b show the cross section micrographs of the SiC coating and the reinforced coating respectively. There are cracks at the corner of SiC coating. However the surface of reinforced coating with SiCw is smoother and there are no obvious cracks on it. It is concluded that SiCw would decrease the amount of the cracks and prevents the cracks from enlarging, which is of great benefit to the oxidation protection of coatings.



a b

Fig.2 The cross section of (a) SiC coating and (b) reinforced coating with SiC whiskers

3.2 Physical properties of coatings

Tab. 1 shows the physical properties of C/C composites, C/C composites with SiC coating and C/C composites with SiC coating reinforced by SiCw.

Tab.1 Physical properties of C/C and the coated C/C

	Thickness (um)	**Hardness (kg/mm^2)**	**Thermal expansion coefficient (10^{-6}/K)**
C/C composites		217	1.29
SiC coating on C/C composites	164	1384	1.91
SiC coating reinforced by SiCw on C/C composites	134	1497	1.55 (532-703K) 1.92 (713-873K)

3.2.1 Effects of SiCw on the coating thickness

The thicknesses of coatings strongly affect the anti-oxidation property of coatings. If the coatings were too thin, the continuous coatings would not gained, which will be not benefit to the oxidation protection of coatings. If the coatings were too thick, it is easy for coatings to peel off from substrates. Tab.1 shows that the thickness of the reinforced SiC coating is 134um that is suitable for oxidation protection. The thickness of reinforced coating is less than that of the SiC coating, 164um. It reveals that SiCw block the infiltration of liquid silicon to the C/C matrix,

which make the SiC transition layer thinner. In the SiC coating, it is easier for liquid silicon to enter into C/C deeply and the thicker SiC transition layer is obtained on the surface of C/C.

3.2.2 Effects of SiCw on the hardness of C/C composites

Tab. 1 reveals that the hardness of C/C with SiC coating is far larger than that of C/C, and the hardness of C/C with SiC coating reinforced by SiCw is larger than that of C/C with SiC coating. It testifies that SiCw have the effect of reinforcing SiC coating. SiCw have good chemical compatibility with coating materials. The strong binding power between SiCw and coating material integrates all coating phases into a steady body, which would elevates the anti-distortion ability and will develops the oxidation protection property of coatings.

3.2.3 Effects of SiCw on the thermal expansion coefficient of C/C composites

Tab.1 shows that the thermal expansion coefficient of C/C with SiC coating, 1.91, is higher than that of C/C samples which is 1.29. From 532K to 873K, there is a turning point of the thermal expansion coefficient in the range of testing temperature for the C/C with the reinforced coating. C/C samples with the reinforced coating have a lower thermal expansion coefficient than that of SiC coated. However, from 713K to 873K, the coefficient of C/C samples with the reinforced coating increase to 1.92 which is close to that of C/C samples with SiC coating. At lower temperature, SiCw integrates strongly with SiC coating and the thermal stress within the coating appears weak that come from the different expansion coefficients between the axial and lengthways direction of whiskers, so the expansion is restrained. However, at the higher temperature, the thermal stress increases on account that the coefficients of thermal expansions in different direction of the reinforced coating vary. The conjoint interface between SiCw and the coating is destroyed. The effect of SiCw on the coefficient of thermal expansion of the coating is weakened, that results in the increase of the coefficient of thermal expansion. In order to possess the lower coefficient of thermal expansion at the higher temperature, the amount of SiC whiskers in coatings must be increased to advance the temperature turning-point.

3.3 Oxidation protection property of coatings

To eliminate the effect of cracks at the corner of single-layer coatings on the oxidation protection property, the same outer SiC coatings were produced on SiC coating and reinforced coating by pack cementation method. The results of oxidation test are shown in Fig.3 that reveals that C/C with reinforced inner coating has better anti-oxidation property, of which the weight loss is less than 8% after oxidation for 21h at 1527K.The oxidation curve of C/C with double SiC coating trend to a beeline, of which the weight loss is up to 18% after oxidation for 21h at 1527K. This shows that oxygen has passed through the double SiC coating and reacted with the carbon substrate directly. So, SiC whiskers would be able to retard the generation of big flaws and cracks that pass through coatings. If the amount of SiC whiskers in coatings experimentally altered to a suitable value the oxidation protection property of C/C composites should be excellently increased.

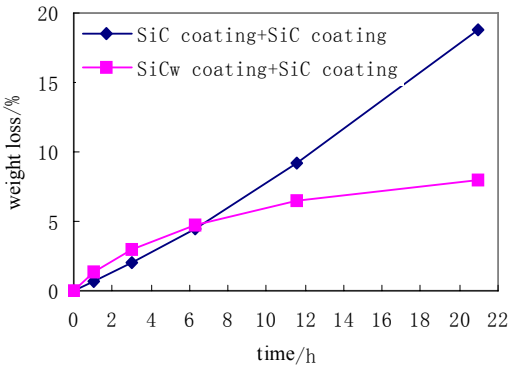


Fig.3 Oxidation curves of C/C composites with double-layer coatings at 1527K

4. Conclusion

The SiC coating reinforced by SiCw on the surface of C/C composite is produced by pack cementation method. SiCw in the SiC coating can decrease the amount of the cracks and prevent the cracks from enlarging. As SiCw block the way of liquid silicon into C/C, the thickness of SiCw coating is thinner than SiC coating. But the hardness of SiCw coating is larger than the SiC coating due to the toughening function of SiCw. In the testing temperature, the values of the coefficient of thermal expansion of SiCw coating have a turning point. The double coating with inner reinforced coating reveals excellent oxidation property.

Acknowledgements

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