

# Study on the Preparation of SiC Coating by Chemical Vapor Phase and the Mechanical Properties of Ceramic Matrix Composites

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In view of the oxidization-prone feature of C/C composites at high temperature, the effect of SiC ceramic coating on the structure and performance of the composites and the oxidation resistance of new nano-carbon particles/ silicon carbide ceramics are explored. The results show that the chemical vapor phase method applied in preparing SiC coated C/C composites can produce good coatings, which is large in coating thickness, uniform in distribution and good in combination with the matrix. The average bending strength of the SiC coated material is 40.4% higher than that of the original material, and the average maximum fracture displacement is increased by 9.26%. The vapor permeation and the enhanced interaction between SiC particles during the preparation of the SiC coating lead to the improvement of strength of the coated samples. After the coating, the fracture mode of the original material does not change fundamentally with the phenomena including interlayer debonding and fiber removal still occurring at the time of fracture, showing good damage resistance and pseudoplastic fracture characteristics. The oxidation process of the new Cp/SiC composite ceramics at 400-700°C is mainly controlled by C-O<sub>2</sub> reaction, where the apparent porosity of Cp/SiC increases and the bending strength decreases. At 700-1100 °C, the SiC on the surface of the material is oxidized into SiO<sub>2</sub> protective film with the apparent porosity decreased and the bending strength increased. The content of nano-carbon is the key factor affecting the strength and the complexity of oxidation of Cp/SiC composite ceramics. The SiC composite ceramics with 15% of nano-carbon has excellent oxidation resistance.

## 1. Introduction

Ceramic matrix composite is a new kind of high-temperature structural material equipped with the advantages of low density, good corrosion resistance and high strength (Fan et al., 2016; Trinh and Samhaber, 2016; Rapagnà and Spinelli, 2016). It not only overcomes the high brittleness of the traditional ceramic but also effectively avoids the layering-prone and damage-prone deficiencies as well as the poor impact resistance of laminates composite materials, which has enabled it to gradually replace the high-temperature alloy as a widely-used light high-temperature material (Vignoles et al., 2009; Li et al, 2006; Fu et al, 2006; Li et al., 2015). In recent years, researchers have gradually found a new kind of ceramic matrix composite materials on the basis of SiC. The high-temperature resistance and corrosion resistance of such materials are greatly improved than the original ceramic matrix composite materials, which includes the nano-carbon particles/silicon carbide ceramic composite material (Cp/SiC) (Rodríguez-Rojas et al, 2012; Christin, 2010). It has been a popular topic of the relevant research to prepare antioxidant coatings with SiC ceramics and thus to improve the antioxidant capacity of carbon materials (Xin et al, 2010; Jian et al; Salman et al, 2015; Fu et al., 2005).

The current preparation of SiC ceramic antioxidant coating is mainly conducted by the chemical vapor deposition and brushing reaction method (Naslain, 2004; Wei et al, 2007; Kang et al, 2013). The chemical vapor deposition (CVD) is characterized by the advantages of wide application, low mechanical damage to fiber and low temperature in manufacturing, having become the preferred method for preparing antioxidant coatings (Li et al, 2000; Huang et al, 2004; Zhao et al, 2007; Friedrich et al., 2002).

Aimed at the oxidization-prone feature of C/C composites at high temperature, this paper discusses the influence of preparing SiC ceramic coating on the structure and properties of the composites and analyzes the oxidation resistance of the new nano-carbon particles / silicon carbide ceramics, the conclusion of which can provide theoretical reference for the preparation of coating materials.

## 2. The influence of SiC ceramic matrix coating on mechanical properties of C/C composites

### 2.1 Preparation and performance test of the sample

The C/C composites adopt the needled laminated carbon felt which is mainly prepared by cyclic overlap of non-woven cloth and fiber web from different angles. The C/C composites are processed with the chemical vapor deposition with a deposition temperature of 900-1100 °C, an impregnation temperature of 200 °C, a pressure of 1.7MPa and a carbonization temperature of 1000 °C. The density of the processed C/C composites is measured to be 1.84g/cm<sup>3</sup>. After polishing, ultrasonic cleaning and drying, the samples are placed in a high temperature furnace containing mixed steam of Si, SiO and SiO<sub>2</sub> to produce SiC coating. The bending strength of the material is tested by a mechanical testing machine, and the performance of C/C composite surface coating is analyzed by an X-ray diffractometer.

### 2.2 Analysis of test results

Figure 1 shows the XRD pattern where the SiC coating is applied to the C/C composite by chemical vapor phase. It can be seen that only the diffraction peak of SiC exists in the figure, proving that the coating is mainly composed of SiC phase. The SiC coating is mainly composed of fine grains arranged closely, effectively inhibiting the stress concentration on the coating which may lead to cracks and improving the mechanical properties of C/C composites.

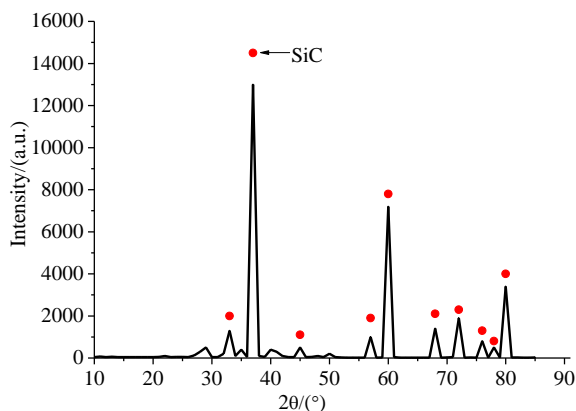


Figure 1: XRD pattern of the C/C composites with SiC coating

The comparison of mechanical properties between the original C/C composite and the SiC-coated composite is shown in Table 1. As can be seen from the table, the average bending strength of the uncoated C/C composites is 175.2 MPa, and that of the SiC-coated composites is 246 MPa, indicating an improvement by 40.4 %. The average fracture displacement of the original C/C composites is 1.08mm, and that of the composites after coating is 1.18mm, showing a promotion by 9.26%.

Table 1: Flexural strength and maximum fracture displacement of C/C composites with and without SiC coating

Sample	Without SiC coating			With SiC coating		
	1	2	3	1	2	3
Flexural strength/MPa	180.5	175.9	169.2	246.2	255.1	236.7
Maximum fracture displacement/mm	0.91	1.46	0.86	1.32	1.18	1.03

Figure 2 shows the relation curve between the fracture load and the displacement before and after applying the coating to the C/C composite material. As can be seen from the figure, when the displacement is 0.5mm, the fracture load reaches the maximum of 530N. With the further increase of the displacement, the fracture load is still kept at a high level with a gradual oscillatory decline trend, indicating that the material has the

typical pseudoplastic fracture properties. After applying the SiC coating, the fracture-displacement curve of the material changes obviously. When the displacement is 0.9mm, the fracture load reaches the maximum value; and then the curve shows different degree of deflection similar to “a drop platform”, which verifies that the coated material demonstrates better damage resistance and pseudoplastic fracture characteristics.

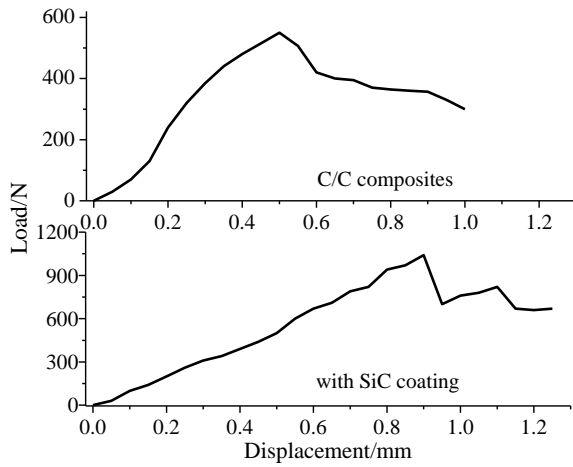


Figure 2: Load-displacement curves of C/C composites and SiC coating

### 3. The mechanical property analysis of nano-carbon particles/silicon carbide ceramic matrix composites

In recent years, the researchers have gradually discovered a new ceramic matrix composite material on the basis of SiC, which is greatly improved in terms of its high-temperature resistance and corrosion resistance compared with the original ceramic matrix composite materials. One kind of it is the nano-carbon particles / silicon carbide ceramic composites (Cp/SiC), and the following is the analysis of the mechanical properties of Cp/SiC materials.

To analyze the mechanical properties of Cp/SiC materials more clearly, the original SiC material is compared with the SiC ceramic matrix materials containing 15% and 25% of nano-carbon, respectively. Figure 3 shows the mass change of the three kinds of materials at different temperatures. As can be seen from the figure, the mass of the SiC sample without nano-carbon is almost constant below 900 °C and presents a rapid increase when the temperature exceeds 900 °C; when the temperature reaches 1100 °C, the mass is increased by 11.7%. For the SiC materials with 15% and 25% of nano-carbon, the variation trend of their mass is basically the same as the temperature increases, i.e. the mass decreases first and then increases. The mass loss at the temperature between 600-750 °C is because of the oxidation; and when the temperature exceeds 750 °C the SiC surface is also oxidized to form the amorphous SiO<sub>2</sub>, leading to the increase of the mass of the sample.

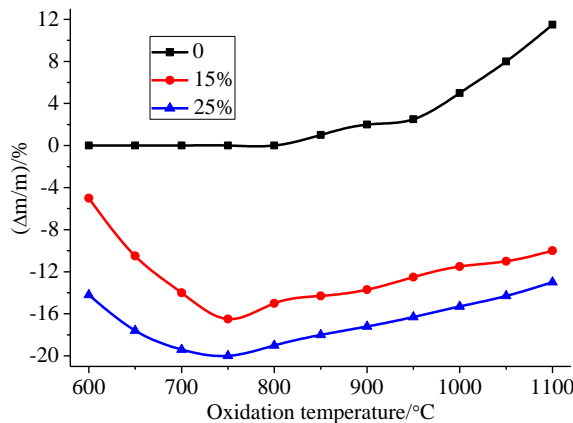


Figure 3: Relation curve between Mass change rate and oxidation temperatures

Figure 4 shows the relation curve between the mass change rate and the oxidation time of the SiC sample and the samples with 15% and 25% of nano-carbon. The figure shows that the mass change rate of the ordinary SiC sample remains basically 0 in the first 2 hours, increases rapidly during 2-6hours and increases slowly after 6 hours. With the increase of the oxidation time, the SiC on the surface of the sample is oxidized into SiO<sub>2</sub> and gradually crystallized into cristobalite, forming the protective layer to prevent the oxidization of the internal part, which explains why the mass of the sample remains steady after 6 hours. The mass of the samples with 15% and 25% of nano-carbon decreases with the increase of the oxidation time because the nano-carbon is easy to be oxidized by SiC.

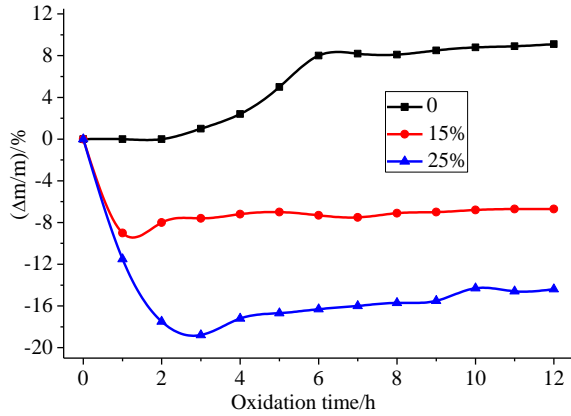


Figure 4: Relation curve between mass change rate and oxidation time

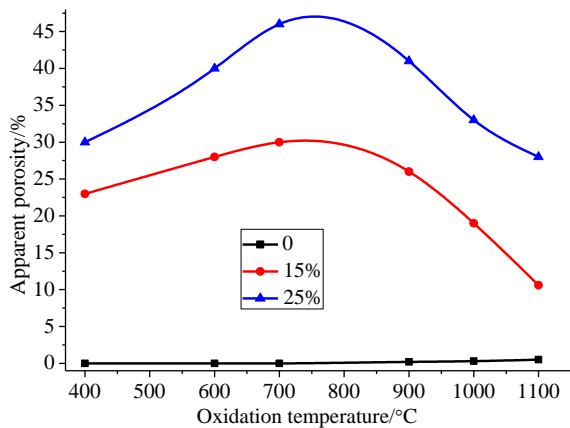


Figure 5: Relation curve between apparent porosities and oxidation temperatures

The apparent porosity ( $P_a$ ) of the ceramic matrix composite material can be expressed as

$$P_a = \frac{M_3 - M_1}{M_3 - M_2} \quad (1)$$

$M_1$ ,  $M_2$  and  $M_3$  represent the mass of the samples in the air under dry conditions, saturated conditions and saturated conditions, respectively. The relationship between  $P_a$  and oxidation temperature after 10-hour oxidation of the three samples at 400-1100°C is calculated according to Equation 1, as is shown in Figure 5. The figure shows that the apparent porosity of the original SiC sample remains basically unchanged after oxidation. The apparent porosity of SiC samples with 15% and 25% of nano-carbon increases first and then decreases. The samples are susceptible to oxidation because of the presence of nano-carbon at 400-700 °C, leading to the increase of the apparent porosity; when the temperature is further increased, the formed SiO<sub>2</sub> protective film fills the pores in the sample and improves the surface defects of the material, thus leading to the decrease of the apparent porosity.

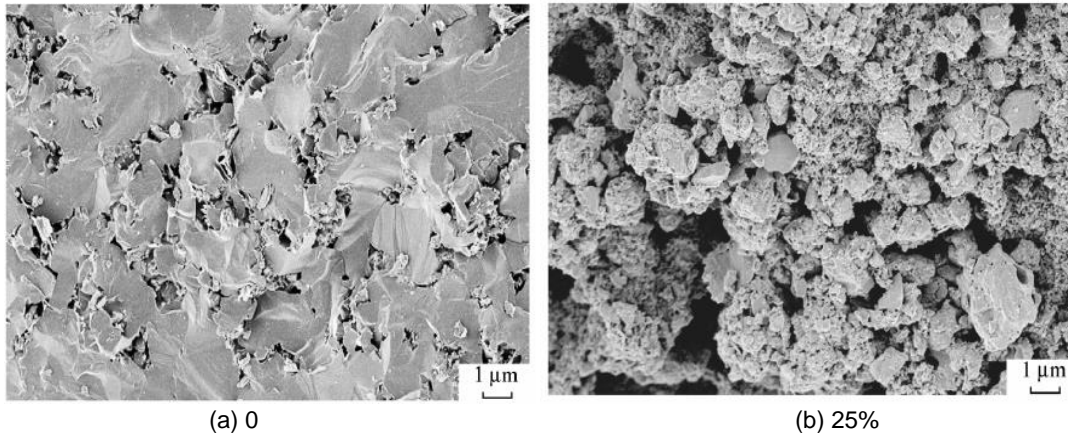


Figure 6: SEM images of Cp/SiC composite with 0 and 25% carbon contents

The difference between samples with different Cp/SiC contents can be better observed by SEM technique. Figure 6 is the SEM images of the original SiC sample and the SiC sample with 25% of nano-carbon. As can be seen from the figure, the ceramic structure in the original SiC sample is very compact and the apparent porosity is basically zero; and the nano-carbon particles in the SiC sample with 25% of nano-carbon are uniformly distributed around the original SiC crystal, which forms the new material Cp/SiC composite ceramics. The nano-carbon is easy to form granular aggregates and thus form the transport network cavity on the sample surface, resulting in the significant reduction of the strength of the ceramic matrix composite materials. Figure 5 shows that the apparent porosity is significantly increased when the temperature exceeds 900 °C..

#### 4. Conclusion

Aimed at the oxidization-prone feature of C/C composites at high temperature, this paper discusses the influence of preparing SiC ceramic coating on the structure and properties of the material and analyzes the oxidation resistance of the new nano-carbon particles / silicon carbide ceramics. The research conclusions are as follows:

- (1) The chemical vapor phase method applied in preparing SiC coated C/C composites can produce good SiC coating, which is large in coating thickness, uniform in distribution and good in combination with the matrix. The average bending strength after applying SiC coating is 40.4% higher than that of the original material, and the average maximum fracture displacement is increased by 9.26%. The vapor permeation and the enhanced interaction between SiC particles during the preparation of the SiC coating lead to the improvement of strength of the coated samples. After the coating, the fracture mode of the original material does not change fundamentally with the phenomena including interlayer debonding and fiber removal still occurring at the time of fracture, showing good damage resistance and pseudoplastic fracture characteristics.
- (2) The oxidation process of the new Cp/SiC composite ceramics at 400-700°C is mainly controlled by C-O<sub>2</sub> reaction, where the apparent porosity of Cp/SiC increases and the bending strength decreases. At 700-1100 °C, the SiC on the surface of the material is oxidized into SiO<sub>2</sub> protective film with the apparent porosity decreased and the bending strength increased. The content of nano-carbon is the key factor affecting the strength and the complexity of oxidation of Cp/SiC composite ceramics. The SiC composite ceramics with 15% of nano-carbon has excellent oxidation resistance.

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