Si/SiC Optical Coatings for C/SiC Composites via Gel-casting and Gas Silicon

Infiltration: Effects of Carbon Black Content

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Abstract: The dense Si/SiC coatings are fabricated by means of gel-casting (GC) and gas silicon infiltration (GSI) for the first time, using carbon black and α -SiC as raw materials. The influences of the carbon black content on the properties of the coating samples have been investigated. Results show that, with the increase of the carbon black content, the viscosity of the slurry increase dramatically, the density and the porosity are closer to theoretical results, leading to the higher density after GSI. And the β -SiC content increases as well as the Si content decreases, resulting in the increase of the mechanical and the thermal matching properties. It is also worth noting that the roughness of the coating of $\phi 160\text{mm}$ comes to 0.57nm after polished. The study highlights that the combined application of the GC and the GSI processes can produce high-quality Si/SiC coatings that are suitable in the space optical applications.

Key words: SiC; coatings; optical applications; C/SiC composites; gel-casting; gas silicon infiltration

1 Introduction

Space mirror, as an important part of optical systems, plays a crucial role in collecting information and has drawn intensive attentions around the world. Among several candidate materials used in this field, short carbon fiber reinforced silicon

carbide (C/SiC) composites have been considered in view of their outstanding performance, including the low specific density (2.65g/cm^3), low coefficient of thermal expansion (CTE: $2\times 10^{-6}\cdot\text{K}^{-1}$ at room temperature and near zero below 100 K), high thermal conductivity ($\sim 125\text{W/mK}$), high stiffness (220GPa) and bending strength (160MPa), very high chemical corrosion resistance, etc ^[1-4]. However, the short carbon fiber could be oxidized during the optical process, resulting in the roughness of the C/SiC composites ($\sim 60\text{nm}$) cannot meet the demand of high optical performances. Therefore it is common to prepare a layer of optical coating which can be polished to as low as 1 nm on the surface of the composites to solve this problem.

Reportedly, the CVD-SiC coating is one of the most promising materials for the coating of C/SiC composites, because it is the only material which has been found which the surface roughness can be polished to around 0.3nm ^[5], however, the application of CVD is limited because of the high cost and long cycles of such method and the thermal mismatch between coatings and the basement ^[6]. By contrast, slurry method and tape-casting method have the advantages of easy operation and low cost, but the coatings formed by both methods cannot be polished to less than 1 nm as reported by Krödel and Song^[1, 7]. At present, the preparation of optical coatings with high performance as well as low-cost is still a challenge.

In this study, a combination of the gel-casting method and the GSI method (GC-GSI) is selected to prepare the Si/SiC optical coatings on the surface of C/SiC composites. As for the fabrication of the preform of coatings, the gel-casting method is selected considering its advantages such as near-net-shape forming, high strength,

high quality, and high density, the gel-casting process has been successfully applied during the preparation of the high-performance ceramics, but such process is select to prepare the coatings has not been reported yet^[8-13]. Meanwhile, the gas silicon infiltration (GSI) method which serves as sintering process of the C/SiC composites is also used in the preparation of the coating, due to its short preparation period, low sintering temperature, minimum sintering shrinkage and low cost^[14, 15]. In our previous works^[16], the process optimization of GC-GSI have already been carried out. In this study, effects of the carbon black content in raw materials on the properties of the final product have been investigated.

2 Experimental

The flowchart of the GC-GSI process is shown in Fig.1.

Fig.1 Flowchart of the GC-GSI process

2.1 Preparation of C/C green body

As for the preparation of the C/C green body, firstly, the randomly oriented milled short carbon fibers and the binder phenolic aldehyde (mass ratio: 1/1) were put into the mold and the C/phenolic resin green body was prepared by the pressing

molding method. Secondly, the preform was infiltrated by the solution consisting of the coal tar pitch and the ethanol (mass ratio: 9/5). Finally, after the pyrolysis process, the phenolic resin turned to C and the C/C green body was fabricated, the pyrolysis temperature was 1100°C and the heating rate was 5°C/min.

The C/C green body was cut to a cylindrical shape of 160mm in diameter and 20mm in height. One side of the body was machined to honeycomb structures for the demand of light weight and another side was used to carry the optical coatings.

2.2 Gel-casting process

The pre-coating was fabricated by gel-casting^[16]. Firstly, AM and MBAM were dissolved in deionized water to form a solution, AM and MABM took 25wt.% and 2wt.% in water respectively. Secondly, the carbon black and SiC powders (a mixture of 43μm and 3.8μm, mass ratio: 3/2) were added to the above solution to form slurry, with the addition of PVP (polyvinyl pyrrolidone) and TMAH (Tetramethylammonium hydroxide, 0.6wt% in slurry) as the dispersants of C and SiC respectively. The carbon black takes 10wt.%, 15wt.% and 20wt.% in solid powders. Subsequently, the above slurry is subjected to 8h ball-mixing and 30min vacuum-degassing, AIBA (2,2'-Azobis (2-methylpropionamidine) dihydrochloride, 20μl/g in slurry.) was added to the slurry. Afterwards, the slurry was poured on the surface of the C/C green body, the gelation reaction took place in 70°C for 50mins. The thickness of the coating was 1.5mm. Finally, the coated samples were dried to constant weight at room temperature. The basic properties of the solid powders are listed in Tab.1.

Tab.1 Properties for the raw materials of solid powders

	ρ (g/cm ³)	M (g/mol)	V_m (cm ³ /mol)
SiC	3.21	40.10	12.49
C	1.86	12.01	5.72
Si	2.33	28.09	12.06

2.3 Gas silicon infiltration process

The coated samples were prepared by the GSI method. The silicon powders were filled in the bottom of the furnace while the above-obtained green body was hinging in the middle. The furnace was heated up to 1500°C in 5h in the argon atmosphere to turn the solid Si to liquid phase. Subsequently the furnace was fired to 1700°C in 1h in vacuum and dwelled at this temperature for 2h, during which the liquid Si turned to gaseous phase and the silicon-carbon reaction took place.

2.4 Samples characterization

The viscosity of the slurries was tested by the rotary viscometer (NDJ-8S), the viscosity was collected under the rotor speed of 1.5, 3, 6, 12, 30, 60 r/min.

The density (ρ_v) and porosity (P) of samples were calculated according to the Archimedes displacement method by Eq.1 and Eq.2, respectively, where ρ_0 represents the density of water under the experimental condition, m_1 is the dry mass of samples, m_2 and m_3 correspond to the saturated mass under water and wet mass in air samples immersed in boiled water for about 30 min, respectively.

$$\rho_v = \frac{m_1 \rho_0}{m_3 - m_2} \quad (\text{Eq.1})$$

$$P = \frac{m_3 - m_1}{m_3 - m_2} \times 100\% \quad (\text{Eq.2})$$

The microstructures of the specimens before and after sintering were examined by the scanning electron microscopy (SEM, FEI Quanta-200).

The phase and composition of the samples were identified by the X-ray diffraction (XRD, D8 Advance), with the Cu- α radiation. The samples were scanned from 10 to 80°, with 0.01° as the step length and holding 1 s at each step.

The mechanical properties are evaluated by the three-point bending test. The samples were cut and polished to 40 mm in length, 4 mm in width and 3 mm in thickness. The length and width directions were parallel to warp and weft directions, respectively. During the three-point bending test, the ratio of span to height was kept as 15 and the crosshead speed was 0.5 mm/min. The flexural strength σ_b and elastic modulus E_b are calculated according to Eq.3 and Eq.4 respectively.

$$\sigma_b = \frac{3PL}{2bh^2} \quad (\text{Eq.3})$$

$$E_b = \frac{L^3}{4bh^3} \cdot \frac{\Delta P}{\Delta f} \quad (\text{Eq.4})$$

Where P is the maximum load, L is the span, b is the specimen width, h is the specimen thickness, and $\Delta P/\Delta f$ is the slope of the linear portion of the load-displacement curve.

The bonding strength was evaluated by Eq.5 according to the standard test method of ASTM D905-89^[17], the where τ is the shear strength of the coated samples, F is the maximum load, A is the contact area of the coating and the composites.

$$\tau = \frac{F}{A} \quad (\text{Eq.5})$$

The thermal expansion coefficient of the coatings and the composites was tested

by the push rod dilatometer (Netzsch DIL 402C/E), the samples were cut to 25 mm in length, 5 mm in width and 5 mm in thickness, and the CTE (α) of the samples were calculated by Eq.6:

$$\alpha = C_0 \cdot \frac{\Delta L_\alpha}{L_r} + C_1 \quad (\text{Eq.6})$$

where ΔL_α is the length variation from the minimum temperature to the required temperature, L_r is the length of the samples at room temperature, C_0 and C_1 refer to the correction factors of the dilatometer. The temperature ranged from -100 °C to 400 °C and the heating rate was 5 °C/min.

3 Results and discussion

3.1 Preparation of high solid content slurry

Fig.2(a) exhibits the effect of the dispersant PVP content on the viscosity of the slurry, where the carbon black content is kept constant as 15 wt.% and the solid content of the slurry is 60 vol.%. As shown, the carbon black can hardly be dispersed in the water without the presence of the dispersant PVP, which can be reflected by the extremely large viscosity value. With the addition of the dispersant PVP, the viscosity value drops dramatically, suggesting the PVP is a good dispersant of carbon black in an aqueous suspension. The viscosity of the slurry reaches the lowest value at the PVP content of 1.5 mg/m², the specific surface area of the carbon black used in this study is 35m²/g, beyond which the viscosity tends to increase gently, probably attributing to the overlapping of polymer chains.

Fig.2(b) shows the effect of the carbon black content on the viscosity of the slurry. As shown, the viscosity increases dramatically with the increase of the carbon black

content, which can be attributed to the following reasons: Firstly, the addition of the carbon black in the water brings obvious agglomeration between the powders, with the increase of the carbon black content, the interaction become more violent. Secondly, the adsorbed water on the surface of the carbon black increases with the addition of the carbon black, which leads to the decrease of the free water in the slurry. Thirdly, the addition of the dispersant PVP increases with the increase of the carbon black content, and the effect of the overlapping of PVP chains become more obvious. In order to get an even smooth coating, the slurry with low viscosity is needed, therefore, the 20wt.% one may not be chosen to prepare the coating.

Fig.2 Effects of PVP content (a) and carbon black content (b) on the viscosities of the slurries

3.2 Composition and structure of the coatings

The component and structure of the coating after GSI are affected by the density and porosity of pre-coating, while the density and porosity are influenced by the pre-coating composition. Suppose that the carbon black content in the pre-coating is m_C , the SiC content is $1-m_C$, considering that polymer takes a tiny percentage in green body. There is:

$$V = V_\alpha + V_C + V_P = \frac{1-m_C}{\rho_{SiC}} + \frac{m_C}{\rho_C} + V_P \quad (\text{Eq.7})$$

Where V is the volume of the pre-coating, V_p is the porosity. After GSI process, the sample consists of α -SiC, β -SiC and Si, imaging that the volume do not change before and after sintering, which can be expressed as following:

$$V = V_{\alpha} + V_{\beta} + v_{Si} \cdot V = V_{\alpha} + \frac{m_C \cdot \frac{M_{SiC}}{M_C}}{\rho_{SiC}} + v_{Si} \cdot V \quad (\text{Eq.8})$$

Where v_{Si} refers to the residual silicon content. The density (ρ) and the porosity (P) of green body are developed together with Eq.7 - Eq.8:

$$\rho = \frac{\rho_{SiC} \cdot (1 - v_{Si})}{1 + \frac{M_{SiC} - M_C}{M_C} \cdot m_C} \quad (\text{Eq.9})$$

$$P = \frac{V_p}{V} = 1 - \frac{(1 - v_{Si}) \cdot (1 + \frac{\rho_{SiC} - \rho_C}{\rho_C} \cdot m_C)}{1 + \frac{M_{SiC} - M_{Si}}{M_C} \cdot m_C} \quad (\text{Eq.10})$$

The unreacted residual silicon can cause the decrease of the mechanical and thermal properties, which is apparently satisfied with the general requirement. When $v_{Si}=0$, the values got from Eq.9 and Eq.10 are called theoretical density and theoretical porosity respectively, the final product consists of only SiC. The theoretical and actual value of the density and the porosity are listed in Tab.2.

Tab.2 Density and porosity of the green bodies with different carbon black contents

Carbon black content	Theoretical density (g/cm ³)	Actual density (g/cm ³)	Theoretical porosity	Actual porosity
10wt.%	2.602	1.929	13.07%	27.95%
15wt.%	2.376	1.997	17.97%	23.32%
20wt.%	2.187	2.043	21.98%	22.57%

As shown, with the increase of the carbon black content, the density of the pre-coatings increases as well as the decrease of the porosity, which illustrates that the carbon black content can cause a certain effect on the structures of the coatings: With the increase of the carbon black content, the solid content of the slurry increases as the rest of the variables are kept constant, so that the spacing of the particles decreases, leading to the increase of the density. The pores caused by the loss of the free water during the drying procedure decrease for the same reason. It is also worth noting that the actual value is closer to theoretical results with the increase of the carbon black content, leading to the lower Si/SiC ratio after sintering, which are confirmed in the XRD patterns in Fig.3.

Fig.3 XRD patterns of the coatings and the composite after the GSI process (a) C/SiC (b) 10 wt.% (c) 15 wt.% (d) 20wt.%

As for the coatings (Fig.3(b)-(d)), there are three phases present in the final product, referring to β -SiC, α -SiC and cubic silicon respectively. While the β -SiC is the reaction product of the GSI process, the α -SiC originates from the raw materials and the cubic silicon is the unreacted residual silicon during the GSI process.

Meanwhile, two phases are found in C/SiC composites (Fig.3(a)), referring cubic silicon and β -SiC, respectively. There are no detectable peaks that can be assigned to carbon, suggesting almost all of carbon black takes reaction in the GSI process. The ratio of α -SiC/ β -SiC (X_α/X_β) can be calculated according to the K value method (Eq.11)

$$\frac{X_\alpha}{X_\beta} = \frac{I_\alpha \cdot K_\beta}{I_\beta \cdot K_\alpha} \quad (\text{Eq.11})$$

Where I is the diffraction intensity gotten from XRD pattern, K refers to the K value of the samples where Al_2O_3 is used as the reference ($K_\alpha=1.600$, $K_\beta=1.811$), since both the first and second strongest diffraction peaks of α -SiC and β -SiC coincide in the XRD pattern, the third strongest peaks (α -SiC of 34.18° and β -SiC of 71.78°) are used to calculate the X_α/X_β value.

The coating samples are soaked in a mixture of HF and HNO_3 (mass ratio: 1/2) for 48 hours, and the following two reactions occur:

The volume fraction of the unreacted residual silicon can be obtained after corrosion by Eq.15: where W_1 and W_2 refer to the mass of the samples before and after soaking, ρ_v is the density of the coating samples, ρ_{Si} is the density of the silicon.

$$F_{\text{Si}} = \frac{(W_1 - W_2)\rho_v}{W_1\rho_{\text{Si}}} \quad (\text{Eq.15})$$

Combing with the above-mentioned computation from the XRD pattern, the volume fraction of the coating samples are listed in Tab.3.

Tab.3 Density and composition of the coatings with different carbon black contents after GSI

Carbon black content	Density (g/cm ³)	Volume Fraction		
		α -SiC	β -SiC	Si
10wt.%	3.021	57.52%	21.30%	21.18%
15wt.%	3.078	56.19%	33.05%	10.75%
20wt.%	3.092	51.98%	43.32%	4.71%

After GSI process, part of the pores in green body is filled by the volume expansion of the Si-C reaction, the rest are filled by the unreacted silicon. When the carbon black content is low, the porosity of the pre-coating is much higher than the theoretical value, the volume fraction of the residual silicon tends to be relatively high and the density of the coating tends to be low. On the contrary, with an increase of the carbon black content, although the quantity is not enough to reach the theoretical state, the density increases obviously whilst the residual silicon decreases continuously. The above explanations are further supported by the structure of the coatings.

Fig.4 Metallography photos of the coatings (a) 10 wt.% (b) 15 wt.% (c) 20wt.%

Fig.4 exhibits the preliminary-polished coatings in the metallurgical microscope, the gray areas refer to SiC while the white areas refer to Si. As seen, large aggregates of silicon are found in the sample with the 10wt.% carbon black content (Fig.4(a)), Si can be considered as the continuous phase in this case. It is well known that the

mechanical and thermal properties are mainly decided by the property of the continuous phase. Gradually, when the carbon black content is 15wt.% (Fig.4(b)), the distribution of silicon reduces obviously. Unlike the phenomenon above, the silicon phase is dispersive and takes little percentage in 20wt.% samples, the SiC becomes the continuous phase instead.

3.3 Mechanical and thermal properties of the coating samples

Tab.4 lists the flexural strength, the elastic modulus and the shear strength of the coating samples, the mechanical properties increase with the carbon black content, which is determined by the capacity of the continuous phase. The change of the mechanical properties is closely related to the structures. As to study the fracture mechanism, the fractured samples are soaked in the mixed-aid, and then the cross-sections of the coatings part after corrosion are shown in Fig.5.

Tab.4 Mechanical properties of the samples with different carbon black contents

Carbon black content	Flextural strength (MPa)	Elastic modulus (GPa)	Shear strength (MPa)
10wt.%	205.99±4.66	214.52±2.34	24.9±1.01
15wt.%	234.12±2.76	220.21±2.55	25.8±0.87
20wt.%	235.30±2.88	229.28±1.50	26.2±0.94

Fig.5 SEM photos of the coatings after corrosion (a) 10 wt.% (b) 15 wt.% (c) 20wt.%

From Fig.5, all the samples show typical brittle fracture mode, the difference is that the fracture morphology changes with carbon black content. A mass of holes caused by silicon corrosion are found in the section of 10wt.% carbon content (Fig.5(a)), silicon served as the continuous phase bears the load in this case. It is worth noting that the SiC grains remains intact after the bending test, indicating that the crack propagation form is the intergranular fracture, the bending strength is relatively low. As for the 15wt.% sample (Fig.5(b)), the porosity caused by silicon corrosion is much lower than the former one, the SiC grains overlap with each other and the fracture mode is called grain bridging, the ability of exterior force resistance is higher accordingly. When the carbon black content is 20wt.% (Fig.5(c)), only few holes distribute in the sample after corrosion, the cross-section is relatively flat, the cracks transfer through the crystal and the fracture mode is transgranular fracture, the sample shows the highest bending strength.

As for multiphase materials, the elastic modulus is additive, as is shown in the following:

$$E = E_{SiC} \cdot v_{SiC} + E_{Si} \cdot v_{Si} \quad (\text{Eq.16})$$

From the equation, the elastic modulus is determined by the volume proportion of Si and SiC. As known, the elastic modulus of the SiC is higher than the silicon. With the increase of the carbon black content, the continuous phase changes from Si to SiC gradually, and the SiC/Si ratio got from Tab.4 increases after sintering, leading to the increase of the elastic modulus.

The shear strength is used to evaluate the binding property of the coating and the

sample. With the All the coatings show good bind properties, and the shear strength of all the samples are over 24MPa, approaching to the shear strength of the C/SiC composite itself (28.4 MPa), suggesting that the coating is strongly jointed with the composite. The calculations are also supported by the microstructure of the coated composite, the morphology of the sample with 15wt.% carbon content are shown in Fig.6.

Fig.6 Cross-section of the coated composite

There is no clear interface between the coating and the composite, the thickness of the binding region is over 20 μm . The phenomenon above can be explained in the following reason: During the process of the casting, some of the slurries are infiltrated into the C/C green body and a pinned layer forms when the gelation reaction occurs. While during the GSI process, both the carbon in the matrix of the C/SiC composites and the carbon black in the coating react with the gas silicon to form $\beta\text{-SiC}$, the reaction layer can be considered as $\beta\text{-SiC}$ and residual Si, which is similar to the matrix of the C/SiC composites. Hence, the bonding strength between the coating and the composite is extremely high ^[18].

In order to keep in service for longer cycles in the thermal shock environment, the thermal expansion coefficient (CTE) of the optical coating need to be close to the

matrix. The CTE of the Si/SiC coatings and the C/SiC composites are presented in Fig.7.

Fig.7 CTE of the coating samples and the composites

As shown, during temperature range of -100°C to 400°C, the coatings with higher carbon black provide a better fit for the C/SiC composites, this can be accounted for as follows: As for multiphase materials, the CTE (α_s) of the samples comply the following empirical formula:

$$\alpha_s = \frac{\sum_i \alpha_i K_i F_i / \rho_i}{\sum_i F_i / \rho_i} \quad (\text{Eq.17})$$

Where α_i , K_i , F_i and ρ_i refer to the thermal expansion coefficient, bulk modulus, volume fraction and bulk density of each phase respectively. According to Eq.17, the CTE of the multiphase material is related to the volume fraction of each phase. The CTE of α -SiC, β -SiC and Si are $5.12 \times 10^{-6} \cdot \text{K}^{-1}$, $3.80 \times 10^{-6} \cdot \text{K}^{-1}$ and $2.5 \times 10^{-6} \cdot \text{K}^{-1}$ respectively. As is seen in Fig.7, the CTE of the coating with the least carbon black content shows the most obvious division with the C/SiC composites, owing to its highest residual silicon in the coating. Meanwhile, the coatings with higher carbon

black content show better matching abilities with the composites, attributing to the higher β -SiC content and lower Si, and the coating sample with 15wt.% carbon black content show the most closely matching property with the composite.

3.4 Properties of the final Si/SiC optical coating

Considering the factors discussed above, the carbon black content of 15 wt.% is selected in the current study and the final product is produced via gel-casting and gas silicon infiltration. Fig.8 (a)-(c) are the pictures of the C/C green body, the pre-coating produced by the gel-casting condition and the sintered samples after the GSI process, respectively. As seen, these samples made from such optimized conditions are rather dense, free from cracks, pores or holes.

Fig.8 Photos of the final product (a) C/C green body (b) after GC (c) after GSI

The surface of the coated composite is grade polished. At the beginning, the metallographic sandpaper is used to remove the scratches caused by the grinder; secondly, a wheel-shiner machine may be used to re-shine the rest infected area; finally, the coating is ultra-fine polished by the diamond abrading agent ^[19]. The final mirror is shown in Fig.9. It can be seen that an extremely smooth mirror surface is obtained without any cracks or holes. The surface data of the mirror is also tested which are depicted in Fig.9. The roughness (Ra) of the made mirror by GC-GSI process is 0.57 nm, lower than those from other pre-coating preparation methods such

as the slurry method or the tape casting method ^[20], indicating that Si/SiC coating with and extremely smooth surface is successfully fabricated by a combination of the gel casting and GSI techniques.

Fig.9 Mirror after polishing and its surface data

4 Conclusions

In order to prepare an optical coating with high performance and low-cost, the current study employs a combination of the gel-casting and GSI techniques to produce a dense Si/SiC coating for the C/SiC composite. Effects of the carbon black content on the properties of the coating have been investigated. The major conclusions are as follows.

(1) The pre-coating is fabricated by the gel-casting process. With the increase of the carbon black content, the viscosity of the slurry increase dramatically, and the density and the porosity are closer to theoretical results, leading to the higher density of the final product.

(2) After GSI process, with the increase of the carbon black content, the β -SiC content increases and the Si content decreases, the continuous phase change from Si to SiC, the fracture mode change from intergranular fracture, grain bridging to

transgranular fracture, resulting in the increase of the mechanical and the thermal matching properties. The shear strength of both the samples is approaching to that of the composite itself.

(3) The roughness of the mirror after polished reaches 0.57 nm, lower than those from other methods. The results suggest that a Si/SiC coating with very high performance has been successfully fabricated by a combination of the gel-casting and GSI techniques.

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