

Formation mechanism and oxidation behavior of MoSi₂–SiC protective coating prepared by chemical vapor infiltration/reaction

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Abstract: In order to protect C/C composites from oxidation, SiC–MoSi₂ composite coating was synthesized by chemical vapor infiltration /reaction (CVI/CVR) technology. A porous Mo layer was prefabricated on SiC coated C/C composites, and then MoSi₂ and SiC were subsequently prepared in a CVI /CVR process using methyltrichlorosilane (MTS) as precursor. The deposition and reaction mechanism of the MoSi₂–SiC composite coating was investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The oxidation behavior of SiC–MoSi₂ coated specimens was tested. The results show that the porous Mo layer can be densified with SiC phase decomposed from MTS, and transformed into SiC–MoSi₂ by reacting with MTS as well. A dense composite coating was prepared with optimized deposition parameters. The coated specimen exhibits a good oxidation resistance with a little mass loss of 1.25% after oxidation at 1500 °C for 80 h.

Key words: MoSi₂–SiC coating; deposition temperature; initial partial pressure of MTS; oxidation resistance

1 Introduction

In the past decades, carbon/carbon (C/C) composites have been paid much attention for their illustrious properties such as light weight, high toughness and modulus above 2000 °C, good thermal shock resistance, and low coefficient of thermal expansion (CTE) [1,2]. However, the poor oxidation resistance of C/C composites has seriously hindered their development in the field of high temperature [2]. To improve the anti-oxidation performance of C/C composites, a protective coating is applied to the surface of C/C composites [3,4].

MoSi₂ is known as a potential candidate for protective coating material due to its outstanding performance in resistance to oxidation and corrosion up to 1800 °C [5]. But there are several detrimental habits of MoSi₂ coating including the low ductility at ambient temperature, the pest oxidation in the intermediate temperature range, the poor strength at high temperature and CTE mismatch between MoSi₂ coating ($8 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$) and C/C substrate ($1 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$) [6,7]. To remedy the

above drawbacks of pure MoSi₂, introducing a second phase (in the form of particles, fibers or whiskers) to prepare a composite coating is one of the most effective methods. SiC has been considered an appropriate second phase material because of its thermodynamical and chemical stability with MoSi₂, lower CTE ($4.5 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$), and good oxidation resistance [8,9].

Although many studies have been carried out about the fabrication of MoSi₂–SiC composites [10,11], there was just a little research about producing MoSi₂–SiC composite coating. YOON et al [12,13] fabricated a MoSi₂–SiC nanocomposite coating by carbonizing and then silicizing the Mo substrate in a chemical vapor deposition (CVD) process. ZENG et al [8] fabricated a MoSi₂–SiC composite coating by pack cementation, which could well protect C/C composites from oxidation up to 1600 °C [8]. WANG et al [14] fabricated a SiC_n–MoSi₂ coating on C/C composite surface by hydrothermal electrophoretic deposition process.

As an extension of CVD, chemical vapor infiltration (CVI) can uniformly densify a porous preform at low temperatures by infiltrating and depositing coatings within the porous preform [15,16]. Meanwhile, the in

situ formation of the second phase in a matrix can generate a stable interface between them, and improve the thermodynamical and chemical stability of composites [17,18]. Combining the above technologies, chemical vapor infiltration /reaction (CVI /CVR) was employed to synthesize a MoSi_2 -SiC composite coating to protect C/C composites from oxidation in this work.

A porous Mo layer was preformed on the SiC coated C/C composites by a slurry method, and then MoSi_2 -SiC composite coating was prepared with the deposition of SiC and the simultaneous reaction with Mo during CVI /CVR process using methyltrichlorosilane (MTS) as precursor. The deposition temperature and initial partial pressure of MTS were investigated and optimized. The isothermal oxidation resistance of the as-coated samples at 1500 °C was explored.

2 Experimental

2D-C/C composites, as substrate materials, were cut into small specimens with dimensions of 10 mm×10 mm×10 mm. The small specimens were hand-polished with 400 grit SiC sandpaper and cleaned with ethanol. The cleaned specimens were lastly dried in a oven at 90 °C before the next step.

A Mo slurry made up of Mo powders and adhesive phenolic resin was brushed on the surface of specimens. The obtained specimens were treated in a vacuum environment at 1000 °C for 2 h. The thickness of the porous Mo layer was dependent on the viscosity of the slurry and brush times. The vacuum heat treatment was carried out in an electric furnace.

CVI /CVR process, using methyltrichlorosilane (MTS, CH_3SiCl_3) /hydrogen (H_2) as reactant species, was also performed in the electric furnace. The specimens were hung inside the reaction chamber with Mo wires. The liquid MTS precursor was transported into the reaction chamber using a bubbling method with a carrier gas of H_2 . The mass flow of MTS was manipulated by the flow rate of carrier H_2 . The flow rate of the dilute H_2 was fixed at 1200 mL/min, and the flow rate of dilute Ar was 300 mL/min. The deposition temperature, time and pressure were in the range of 1100–1250 °C, 5–10 h and 5–10 kPa, respectively.

The isothermal oxidation test was performed in the electrical furnace in air. An inner SiC coating was pre-coated on the specimens by pack cementation method [19], and then an outer MoSi_2 -SiC composite coating was prepared through a CVI /CVR process. During the oxidation test, the coated specimens were firstly put in the furnace at 1500 °C and endured for a certain time. After that they were got out of the furnace immediately and then cooled down quickly to room temperature. The mass change of the specimens was

examined with an electronic balance (sensitivity: 0.1 mg). When the specimens were put into the furnace again, another oxidation cycle was begun. Therefore, the thermal shock performance of the specimens was evaluated during the isothermal oxidation test.

The crystal structure and morphology of the MoSi_2 -SiC composite coating before and after oxidation were analyzed by X-ray diffraction (XRD, X'Pert Pro MPD) and scanning electron microscopy (SEM, Tescan VEGA TS5136XM), respectively.

3 Results and discussion

3.1 Characteristics of porous Mo layer

Figure 1 shows the phase composition (Fig. 1(a)) and the SEM image (Fig. 1(b)) of the pre-coated Mo layer after vacuum heat treatment. It can be seen that the layer consists of pure Mo phase in Fig. 1(a), and the Mo particles are connected with each other to form a porous structure with uniform distribution of pores shown in Fig. 1(b).

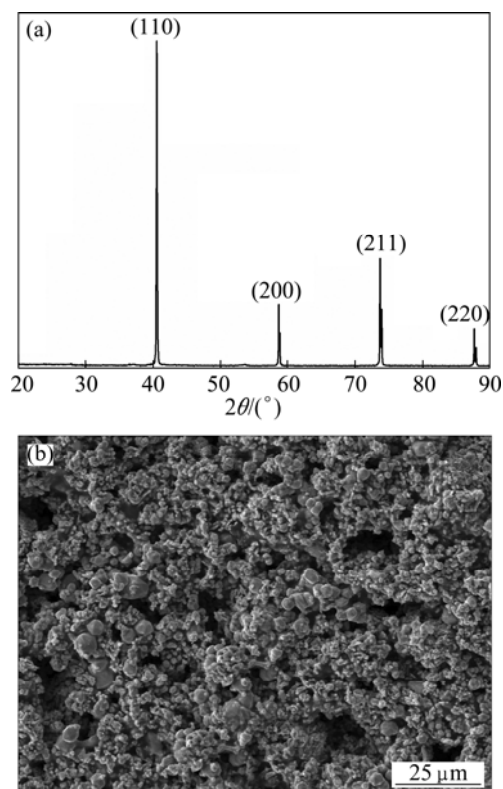


Fig. 1 XRD spectrum (a) and SEM image (b) of surface of coated samples

However, the pore geometry is irregular and a wide pore size distribution is ranging from 5 to 25 μm , which resulted from the unhomogeneity of the Mo powders and the phenolic resin adhesive in the brushing process. During the vacuum heat treatment, the phenolic resin combining the Mo powders pyrolyzed and released

rapidly large amounts of CO, CH₄, H₂O and H₂ [20]. The gaseous products were pumped out of the furnace quickly, which left abundant pores in the Mo layer.

3.2 Effects of deposition parameters on MoSi₂–SiC composite coating

In the CVI/CVR process, the initial partial pressures of MTS (in terms of $\alpha = p_{\text{H}_2}/p_{\text{MTS}}$) and the deposition temperature had a crucial influence on the phase composition and microstructure of deposits. Therefore, the effects of α and temperature were investigated in the next section.

3.2.1 Effect of $\alpha = p_{\text{H}_2}/p_{\text{MTS}}$

XRD spectra of obtained coatings, deposited at 1100 °C for 5 h with different values of α , are shown in Fig. 2. It can be seen that there are the most intense Mo peaks in all XRD spectra because of the short deposition time leaving a lot of unreacted Mo. For $\alpha=15$, the coating is composed of Mo, Mo₅Si₃, Mo₃Si and Mo₂C, and it is composed of Mo, Mo₅Si₃, Mo₃Si, Mo₂C and SiC for $\alpha=20$. When α rises to 30, Mo₃Si and Mo₂C phases disappear with the formation of MoSi₂.

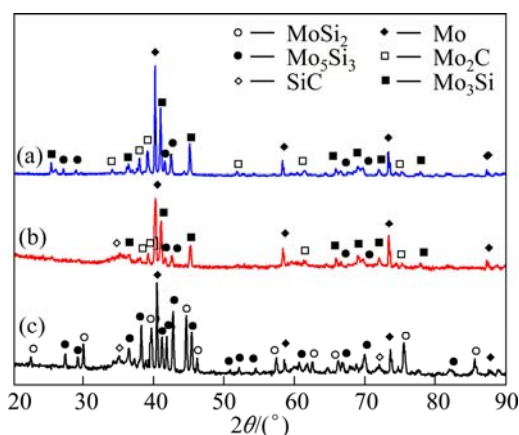


Fig. 2 XRD spectra of coatings deposited at 1100 °C for 5 h: (a) $\alpha=15$; (b) $\alpha=20$; (c) $\alpha=30$

The deposition of SiC in the CVI/CVR process is originated by a decomposition of MTS at hydrogen ambient. Actually, MTS was decomposed into —CH_3 and —SiCl_3 radicals by breaking the Si—C bond first, and then to generate the following reactive species: CH₄, C₂H₂, C₂H₄, C₂H₆, SiHCl₂, SiCl₂, SiCl₄, and so on [21–23]. A series of complicated reactions took place among the above gaseous species and hydrogen to form solid SiC, which can be summarized a combination of reactive C and Si (see Eq. (1)). In addition, hydrogen can suppress the formation of carbon from the carbon groups and promote the formation of silicon from the silicon groups (SiHCl₂, SiCl₂, SiCl₄, etc.) [23]. Therefore, a small value of α would lead to an excessive carbon, and a large one would lead to an excessive silicon.

The pre-coated Mo may react with active C and Si atoms and transform into Mo₃Si, Mo₅Si₃, MoSi₂ or Mo₂C described by Eqs. (2)–(5). Based on the online database of facility for the analysis of chemical thermodynamics (FACT) [24], the Gibbs free energy changes (ΔG) of Eqs. (1)–(5) from 750 to 1300 °C are calculated and indicated in Fig. 3. ΔG values of all equations are negative, which means that they can occur spontaneously at the temperature of 750–1300 °C. Furthermore, the more negative values of ΔG represent the thermodynamically most favorable reactions among Eqs. (1)–(5). That is to say, Mo₅Si₃, MoSi₂ and SiC may be the most stable products among all reaction products.

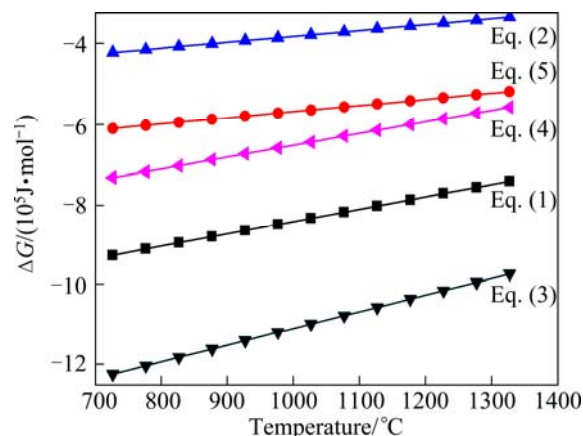
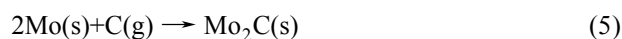
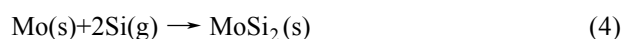
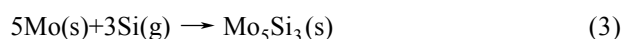
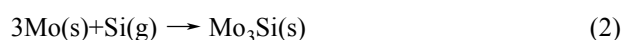
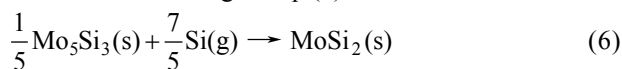


Fig. 3 Variation of Gibbs free energy of each reaction at different temperatures

Although Mo₅Si₃ would probably be formed preferentially on Mo surface from Fig. 3, the further reaction of Mo with C and Si must obey a competitive mechanism from the point of view of the reaction and diffusion kinetics. When $\alpha=15$ or 20, reactive C prevents the generation of MoSi₂ and diffuses through Mo₅Si₃ to form Mo₂C. On the other hand, Si may be obstructed by Mo₅Si₃ to diffuse extensively into the Mo. The nucleation and formation of SiC from the gas phase are impeded by the affinity of Mo with C and Si. Besides, the thermodynamic incompatibility between SiC and Mo, similar to the SiC/Mo diffusion couple, leads to the littleness or absence of SiC [25–27]. For $\alpha=30$, continuous supply of a large amount of Si surmounts the obstacles of diffusion into the Mo and inhibits the adverse effect of C. Along with the successive formation

and diffusion of abundant silicon in the CVI /CVR process, Mo_5Si_3 is stabilized and prompted to transform into MoSi_2 according to Eq. (6):



When adequate molybdenum silicides presented on the Mo surface, SiC can be nucleated and formed near $\text{Mo}_5\text{Si}_3/\text{MoSi}_2$. A composite coating can be produced finally with the further reaction and siliconization of Mo, as well as the deposition and diffusion of SiC. For a higher value of α , the supersaturation of MTS in the deposition region may be reduced too much to furnish the reaction and deposition efficiency.

3.2.2 Effects of deposition temperature

Figure 4 illustrates the XRD spectra of coatings deposited for 10 h with $\alpha=30$ at different temperatures. It can be found that the composite coating is composed of Mo, Mo_5Si_3 , MoSi_2 and SiC when deposited at 1100 °C. The other coatings deposited at 1150, 1200 and 1250 °C consist all of MoSi_2 and SiC phases with a different ratio between the intensities of MoSi_2 peaks and SiC peaks.

As well known, the reaction speed is influenced greatly by temperature according to Arrhenius law. There are still a lot of unreacted Mo, some molybdenum silicides and a minor SiC phase at 1100 °C. When temperature was increased from 1150 °C to 1250 °C, the reaction of pre-coated Mo and deposition of SiC are accelerated exponentially with the increasing temperature.

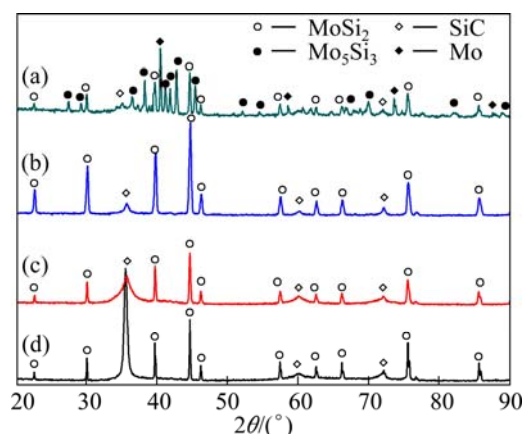


Fig. 4 XRD spectra of coatings deposited for 5 h with $\alpha=30$ at different temperatures: (a) 1100 °C; (b) 1150 °C; (c) 1200 °C; (d) 1250 °C

The backscatter electron (BSE) images of the cross-section of SiC– MoSi_2 composite coatings deposited at different temperatures are shown in Fig. 5. From Fig. 5(a), the obtained coating peeled off the substrate after CVI /CVR process and has a porous structure with white and grey phases. Based on the above XRD results, it can be concluded that the main part of white phase is composed of the residual Mo and some molybdenum silicides, and the minor part of grey phase is composed of SiC. When deposition temperature was increased to 1150 °C and 1200 °C, the coatings have a

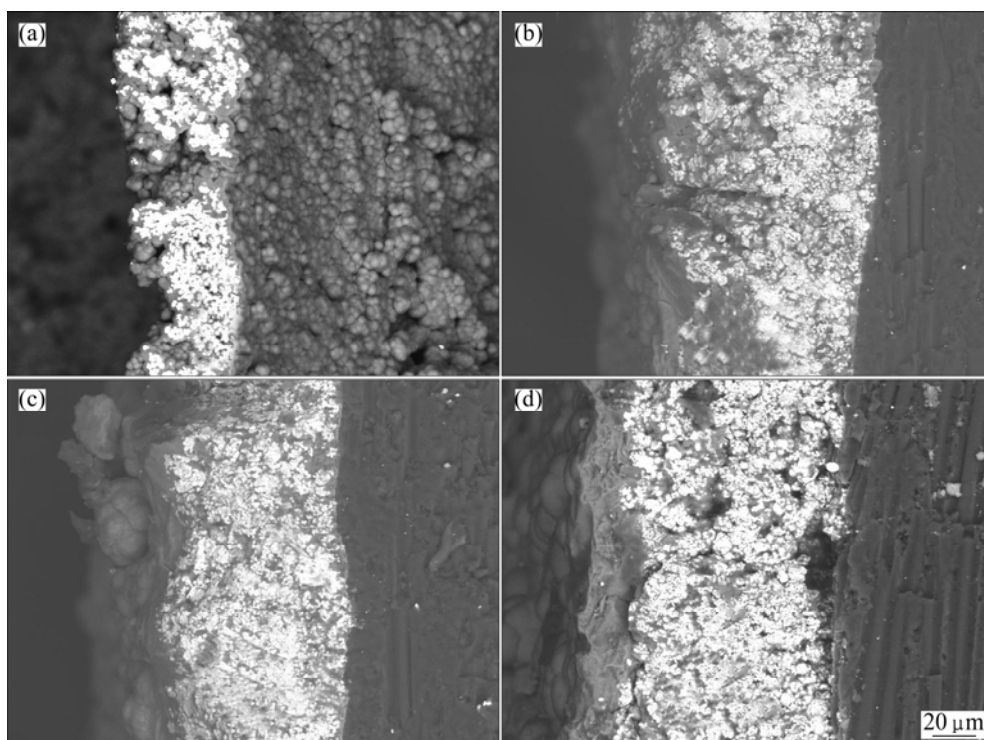


Fig. 5 BSE images of cross-section of MoSi_2 –SiC composite coatings at different temperatures: (a) 1100 °C; (b) 1150 °C; (c) 1200 °C; (d) 1250 °C

dense structure with white MoSi_2 particles dispersed in the successive grey SiC phase from Fig. 5(b), Fig. 5(c) and the relevant XRD spectra. From Fig. 5(d) the outer part of the coating is also composed of white MoSi_2 phase and grey SiC phase. There are still some pores and big cavities at the bottom part of the coating, which implies a porous structure and poor properties of the coating deposited at 1250 °C.

The deposition and infiltration of SiC in CVI process are controlled by not only reaction mechanism but also diffusion mechanism [15,16,23,28]. The simultaneous reaction with pre-coated Mo increases the complexity of the whole system of CVI /CVR process. Nevertheless, the preparation of MoSi_2 – SiC composite coating includes the following steps.

1) MTS was transported from the main gas flow to the inner surface of pores inside the porous pre-coated Mo layer.

2) Reactive radicals were decomposed from MTS and reacted with Mo and themselves to form molybdenum silicides and SiC .

3) By-products were desorbed from the surface to the main gas flow and then discharged into the atmosphere through a vacuum pump.

At low temperatures, such as 1100 °C, the reaction among all reactive groups is slower than the diffusion of the reactants from MTS [15]. As a result, the pre-coated Mo seldom transits to molybdenum silicides and the deposition of SiC is too little to fill the pores, which causes the obtained coating to peel easily off the substrate. However, the whole process of preparation is dominated by the reaction of Mo and the deposition of SiC at high temperatures (i.e. 1250 °C) although the diffusion of the reactants is promoted [15]. SiC deposits preferentially on the external surface of the pre-coated layer, instead of the inner surface of pores inside the layer. The formation of bottleneck pores or sealed pores terminates the further infiltration into these pores, and some big cavities are left.

For the deposition temperatures of 1150 °C and 1200 °C, the relative balance of reaction and diffusion results in a synchronous formation of molybdenum silicides with the infiltration of SiC into pores of the porous Mo layer. The abundant pores, served as conductive passage for the infiltration of SiC , enable Mo to enlarge the contact area and promote the reaction with the reactive groups. Accordingly, a dense MoSi_2 – SiC composite coating is obtained efficiently, and integrated with the substrate by means of the continuous CVD- SiC phase.

3.3 Oxidation behavior of coated specimens

Figure 6 indicates the XRD spectra of the coated specimens before and after the isothermal oxidation at

1500 °C for 80 h. It can be seen that there is a major phase of SiC in the coating both before and after the oxidation, and the MoSi_2 peak is quite weak on the contrary. In addition, a new SiO_2 phase is formed after the oxidation of SiC and MoSi_2 from Fig. 6(b). Because the specimens are hung in the alumina tube with Mo wires during the CVI /CVR process, there are some preparation defects at the tied position in the MoSi_2 – SiC composite coating. A further deposition of pure SiC on the composite coating has to be performed to seal the defects so that MoSi_2 phase is thoroughly covered with SiC .

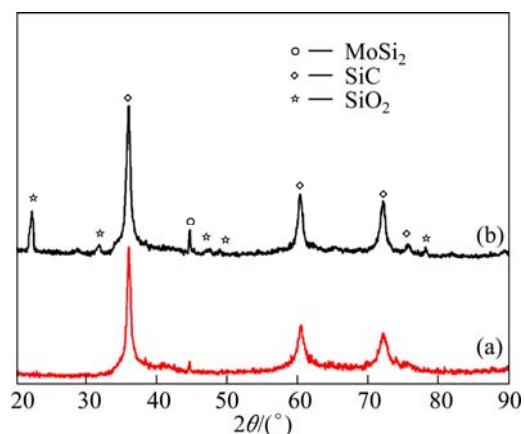


Fig. 6 XRD spectra of coated samples before (a) and after (b) oxidation test at 1500 °C for 80 h

SEM image of the surface and BSE image of the cross-section of the as-coated specimen are respectively shown in Fig. 7(a) and Fig. 7(b) after oxidation at 1500 °C for 80 h. As shown in Fig. 7(a), there is a smooth silica glass film on the surface of the equiaxed grains of SiC because of the oxidation of SiC . Some microcracks, bubbles and pinholes are found to distribute over the surface of the silica film, resulting from the residual thermal stress in the glass film and the release of gaseous oxides during oxidation test. From Fig. 7(b), a penetrable crack passes through the whole coating and reaches the C/C composites. The oxygen can diffuse into the protective coating through these cracks and react with the C/C composites, inducing the oxidation and a mass loss of the C/C matrix.

Figure 8 shows the mass loss curve of the coated specimens as a function of oxidation time. It can be seen that the MoSi_2 – SiC composite coating can protect the SiC coated C/C specimens from oxidation at 1500 °C for 80 h with a mass loss of 1.25%. The mass loss sharply increases to 3.8% with the further oxidation.

In the early oxidation stage, the composite coating reacts with oxygen and generates silica glass film on the surface. The glass film can slow down the reaction of

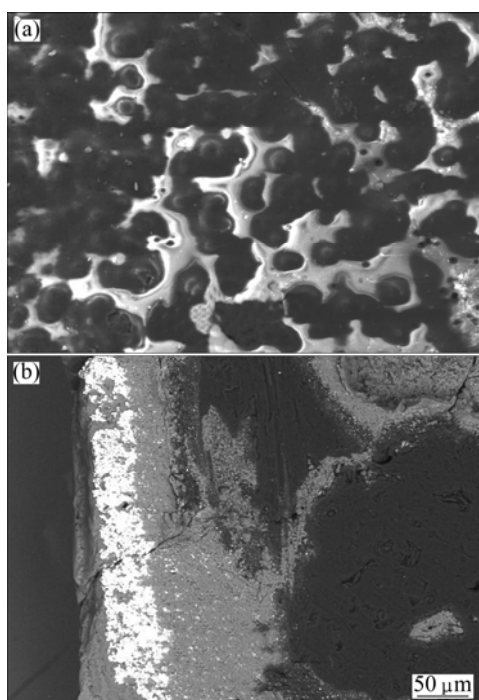


Fig. 7 SEM images of surface (a) and cross-section (b) of coated samples after oxidation at 1500 °C for 80 h

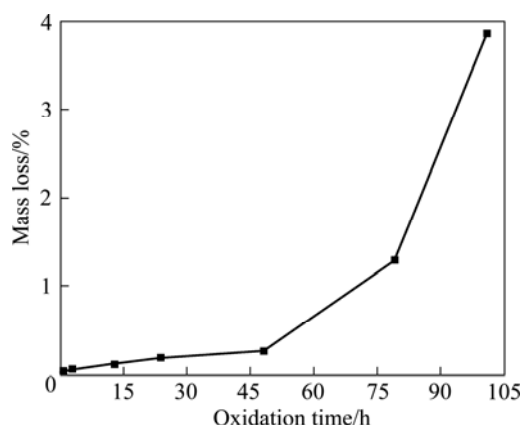


Fig. 8 Oxidation curve of coated specimens

MoSi₂–SiC composite coating with oxygen owing to its low oxygen permeation rate and good viscosity at high temperatures. As a result the coated specimens possess a good oxidation resistance with a minor mass loss. Nevertheless, the defects and the penetrable cracks resulting from the residual thermal stress provide passages for oxygen to diffuse into the protective coating and react with C/C composites. A considerable mass loss of the specimens can lead to a rapid failure of C/C composites in real applications.

4 Conclusions

1) A MoSi₂–SiC composite coating can be prepared by a combination of slurry and CVI /CVR technologies. The compact coating was achieved under the conditions

of the ratio of initial pressure $\alpha=30$ and deposition temperature of 1150–1200 °C.

2) In the CVI/CVR process SiC deposited from MTS can fill the pores in the pre-coated Mo layer, and the Mo layer can react with MTS to form MoSi₂ at the same time. As a result, a dense structure is composed of a disperse MoSi₂ phase and a continuous SiC phase.

3) The MoSi₂–SiC composite coating can protect SiC coated C/C composites against oxidation at 1500 °C for 80 h with a mass loss of 1.25%.

4) It is necessary to pay more attention to improving the uniform distribution of the pre-coated Mo layer in order to optimize the preparation of MoSi₂–SiC composite coating.

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化学气相浸渗/反应法制备 MoSi_2 -SiC 涂层的合成机理及其氧化性能

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摘 要: 采用化学气相浸渗/反应法(CVI/CVR)在 C/C 复合材料表面制备 MoSi_2 -SiC 防氧化涂层。首先, 制备多孔金属 Mo 层, 然后以甲基三氯硅烷(MTS)为前驱体, 利用 CVI/CVR 技术制备 MoSi_2 -SiC 复合涂层。采用 XRD、SEM 技术研究 MoSi_2 -SiC 复合涂层的制备机理, 并考察涂层样品的抗氧化性能。结果表明, MTS 分解的 SiC 填充了多孔金属 Mo 层中的孔隙, 并与金属 Mo 发生反应, 制备出致密的 MoSi_2 -SiC 复合涂层; 在优化工艺条件下所得到的涂层样品在 1500 °C 空气中氧化 80 h 后质量损失仅为 1.25%。

关键词: MoSi_2 -SiC 涂层; 沉积温度; MTS 原始分压; 氧化性能

(Edited by Xiang-qun LI)