Fabrication of MoSi$_2$ coatings on molybdenum and its high-temperature anti-oxidation properties

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Abstract: Industrial spent MoSi$_2$-based materials were used to fabricate oxidation-resistant coatings on molybdenum via slurry painting in air. The microstructure, phase constituent and high-temperature oxidation behaviors of the coatings at 1500 °C were explored. The results show that the bonding layer is generated in the coatings after sintering, which strengthens the metallurgical combination between the coating and the substrate because of the formation of diffusion layers. Rare cracks appear in the coating using pure MoSi$_2$ (PM coating) while the coating using spent MoSi$_2$ (SM coating) is free of cracks due to decreased thermal expansion mismatch. After oxidation, the oxide scale of PM coating possesses large-sized pores while a relatively dense oxide scale is acquired by SM coating. Compared with PM coating, thinner glassy oxide scale with lower mass gain is obtained by SM coating, exhibiting better anti-oxidation properties at 1500 °C.

Key words: recycling; spent MoSi$_2$; slurry painting; anti-oxidation properties; molybdenum substrate

1 Introduction

As one of the most favored refractory metals, molybdenum (Mo) has a high melting temperature (2620 °C), excellent thermal conductivity and high elastic modulus [1−3]. Therefore, it has been widely applied in the key parts of cannon barrels, rocket engines and nuclear reactors [2−4]. However, when temperature reaches 400−750 °C, Mo will be rapidly oxidized to MoO$_3$ in air. The volatilization of MoO$_3$ can cause the rapid invalidation of Mo, which seriously restricts its application at elevated temperature [4].

Fabricating a coating on the surface can effectively extend the lifespan of the substrate without diminishing its properties [5,6]. Lots of coatings, especially silicide coating represented by MoSi$_2$, have been widely researched due to the low density (6.24 g/cm$^3$), high melting point (2030 °C), good thermal conductivity and high working temperature (≤1850 °C) [7,8]. When the temperature is over 1000 °C, a layer of silica can be generated on MoSi$_2$ to prevent further oxidation [9], and consequently MoSi$_2$-based materials are widely used in the high-temperature fields [10].

However, the consumption of MoSi$_2$-based materials increases gradually, and an enormous number of spent MoSi$_2$-based materials are discarded after long-term use in the high-temperature environments, causing environmental pollution and waste of resource. It is worth mentioning that the development of sustainable resources for positive societal, environmental, and...
economic impacts can be effectively driven by achieving increased utilization of wastes [11]. Although the MoSi$_2$-based materials fail after long-term service, there is no change in its major compositions (MoSi$_2$), indicating that spent MoSi$_2$-based materials still possess excellent anti-oxidation properties. In consideration of the advantages of molybdenum disilicide, spent MoSi$_2$-based materials can be recycled as raw materials to fabricate coating on Mo substrate for potential high-temperature protection.

Although lots of coatings are sintered in a vacuum or protective atmosphere (such as argon) to avoid oxidation [12], it is found that the coating preparation can be accomplished in air by fabricating multilayer structures with pressureless sintering, which is easily operated on various shapes and sizes objects [13]. Without the protective atmosphere, cost for the coating sintering can be significantly reduced. The silica layer, which is formed on the surface of the silicide coating during sintering in the air, can inhibit the further diffusion of O$_2$, making tiny amounts of oxidation. Furthermore, slurry painting is a simple method to prepare the coating in the air with relatively low requirements of devices [14]. Therefore, it is meaningful to prepare MoSi$_2$ anti-oxidation coating on Mo substrate by slurry painting.

Herein, spent MoSi$_2$-based materials were used as raw materials, and slurry painting was applied to prepare MoSi$_2$ multilayer coating (SM coating) on Mo substrate. For comparative analysis, pure MoSi$_2$ powders were also used to fabricate the coating (PM coating). After sintering at high temperatures, the microstructural evolution of PM and SM coatings were investigated, and the oxidation behaviors of the multilayer coatings at 1500 °C were studied.

2 Experimental

2.1 Coating fabrication

Molybdenum (12.0 mm in diameter, 2.5 mm in thickness, Qinghe Guantai Metal Material Co., Ltd., China) substrate was polished by 1000 grit SiC abrasive paper to remove the grease and impurities from the surface. Industrial spent MoSi$_2$-based materials (Zhengzhou Juding High Temperature Furnace Technology Co., Ltd., China) were sourced from the electrical furnace that worked at 1000−1700 °C for over 1000 h, and used as coating materials after being crushed into powders. The SEM images and corresponding distribution of the spent MoSi$_2$ powders are illustrated in Figs. 1(a−c). The size of powders roughly ranges from 0 to 30 μm, and they were found to be made up of Si, Mo, Al and O elements in the previous study [15].

Firstly, 5 wt.% bentonite solution (Xinjiang Haotai Huamei Mining Technology Co., Ltd., China), in which the solvent was deionized water, was mixed with Si (99.9% in purity, 5 μm, Beijing Wodetai Technology Co., Ltd., China) and spent MoSi$_2$ powders (mass ratio of 5:1:4) to prepare coating slurry to brush Mo substrate as bonding
layer (Layer I). The main compositions of the bentonite were SiO$_2$ (71.7 wt.%), Al$_2$O$_3$ (16.2 wt.%), Na$_2$O (3.1 wt.%) and CaO (2.4 wt.%). The bonding between the coating and substrate could be enhanced by the diffusion of Si with Mo, which forms a diffusion layer at elevated temperatures. Secondly, spent MoSi$_2$ powders were added into 5 wt.% bentonite solution (mass ratio of 1:1) to brush Layer I as the protecting layer (Layer II) and it acted as the main part of the multilayer coating to protect the Mo substrate. Thirdly, spent MoSi$_2$, Si powders and glass powders (99.5% in purity, 6.0–11.0 μm, Guangzhou Anywhere New Materials Co., Ltd., China) were mixed in 5 wt.% bentonite solution (mass ratio of 3:1:1:4) to brush Layer II as oxygen-barrier layer (Layer III), which was prepared for the formation of liquid SiO$_2$ to inhibit the diffusion of oxygen at elevated temperature. The glass powders mainly consisted of SiO$_2$ (60.48 wt.%) and CaO (25.95 wt.%) by X-ray fluorescence spectrometer (XRF) in Table 1. The glass powders had a low melting temperature (1050 °C) and they were conducive to improving the coating compactness during sintering. Furthermore, SiO$_2$ in the glass powders could enhance the anti-oxidation properties of the coating at high temperatures. For comparison, multilayer MoSi$_2$ coating was also prepared by commercially pure MoSi$_2$ powders (99.0% in purity, Yantai Torch Special High Temperature Ceramics Co., Ltd., China) under the same technological conditions. The SEM images of the pure MoSi$_2$ powders are shown in Figs. 1(d, e), and its size ranged from 0 to 10 μm, as shown in Fig. 1(f).

After being painted, the coating samples were dried at 60 °C for 24 h, and then were placed into Al$_2$O$_3$ crucible. ZrO$_2$ particles (1–3 mm) were put in the bottom of the crucible to prevent the adhesion of coating samples with crucible during high-temperature sintering. The pressureless sintering of the coating samples was carried out in the electrical furnace without a protective atmosphere and the heating rate was 10 °C/min. Before being cooled to room temperature (RT), the coating samples were held for 1 h as the temperature rose to 1100 and 1400 °C, respectively. Corresponding preparation process of the multilayer coatings is provided in Fig. 2.

### 2.2 Oxidation test and characterizations

Coating samples were put into the Al$_2$O$_3$ crucible and the oxidation tests (1500 °C) of PM and SM coatings were performed in the electrical furnace (0–20 h). After an oxidation test, the coating samples were taken out and then cooled to RT. An electronic balance was used to calculate the mass change of the coating samples ($\Delta m_{cs}$):

$$\Delta m_{cs} = (m_a - m_b)/A_{cs}$$

where $m_a$ and $m_b$ are the masses after and before oxidation, respectively, and $A_{cs}$ is the surface area of the coating samples before oxidation test.

Scanning electron microscopy (SEM, JEOL JSM–IT300LV) with energy dispersive spectroscopy (EDS) was used to observe microstructure of the powders and coatings, and the phase composition of PM and SM coatings was detected by X-ray diffraction (XRD, Panalytical Empyrean). The chemical composition of the glass powders was tested by X-ray fluorescence spectrometer (XRF, Table 1 Chemical composition of glass powders by XRF (wt.%))

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</tr>
<tr>
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Fig. 2 Schematic illustration of preparation process for multilayer coating by slurry painting
Thermo Fisher 9900). Transmission electron microscopy (TEM, JEM−3010) was used to analyze the micromorphology of spent MoSi2 powders.

3 Results and discussion

3.1 Phase and microstructure

Figure 3 shows the XRD patterns of pure MoSi2 and spent MoSi2 powders. MoSi2 has high intensity in the XRD patterns of the pure and spent MoSi2 powders, and Mo5Si3 phase with relatively weak intensity can be observed in the spent powders since Mo5Si3 and SiO2 phases are generated in the MoSi2-based materials after long-term service at elevated temperature. Based on our previous research [15], the spent powders consist of Mo (60.17 wt.%) and Si (33.62 wt.%) with rare amounts of O (3.09 wt.%), Al (1.76 wt.%), Ca (0.11 wt.%), etc. However, owing to the low content and amorphous state of the SiO2 phase, it is difficult to be detected in the XRD patterns.

TEM image and high-resolution morphology of spent MoSi2 powders are represented in Fig. 4. The interplanar spacings of the (002) and (103) planes of MoSi2 are approximately 3.9231 and 2.0264 Å (Fig. 4(b)), respectively, after comparing with the standard XRD PDF card of No. 41-0612 (MoSi2). In addition, a layer of amorphous phase (aluminosilicate) is observed to surround MoSi2 particles. Bentonite (aluminosilicate) is added into MoSi2 as the sintering additive during the preparation of MoSi2-based materials [10]. However, no peak of bentonite is observed in the XRD patterns. The content of bentonite is too low (about 10 wt.%) to be detected in spent MoSi2. Furthermore, the absorption coefficient of XRD is increased with increasing atomic number, the atomic number of aluminosilicates is less than the Mo−Si phase, and thus notably weak intensity is acquired by bentonite [16].

After sintering, the XRD patterns of PM and SM coatings are provided in Fig. 5. As shown, MoSi2, Mo5Si3, SiO2 and CaMoO4 phases appear in the two coatings. MoSi2 phase is oxidized to MoO3 and SiO2 phases during the heating period of the sintering [17]:

$$2\text{MoSi}_2 + 7\text{O}_2 = 2\text{MoO}_3 + 4\text{SiO}_2$$  \hspace{1cm} (2)

When the sintering temperature remains unchanged at 1400 °C, the thickness of the silica layer is increased as time prolongs. The low oxygen partial pressure between MoSi2 and SiO2 layer inhibits the formation of MoO3, causing MoSi2 to be oxidized to SiO2 and Mo5Si3 [18]:

$$5\text{MoSi}_2 + 7\text{O}_2 = 2\text{Mo}_3\text{Si}_3 + \text{SiO}_2$$  \hspace{1cm} (3)

In addition, the glass powders and Si also provide SiO2 phases during oxidation. However,
MoO$_3$ is not found in XRD patterns since it is in the volatile state above 750 °C. Furthermore, reaction can occur between MoO$_3$ and CaO, making the formation of CaMoO$_4$ [19]:

$$\text{CaO} + \text{MoO}_3 = \text{CaMoO}_4 \quad (4)$$

After sintering, the surface morphologies of PM and SM coatings are represented in Fig. 6. The surfaces of PM and SM coatings are both made up of gray and white phases, which are detected to be SiO$_2$ and CaMoO$_4$ by EDS in Table 2, respectively, which is consistent with the XRD patterns (Fig. 5). No obvious pores are found on the surface of PM and SM coatings and this is mainly attributed to the formation of CaMoO$_4$ phase. At elevated temperatures, pores are generated in the coating by the volatilization of MoO$_3$ based on Eq. (2), and CaMoO$_4$, which is formed by the reaction of MoO$_3$ with CaO in the glass powders, can effectively reduce the content of volatile MoO$_3$ that penetrates into the oxide scale. According to Refs. [20,21], CaMoO$_4$ possesses excellent chemical stability, and thus it is hard to be eroded by oxygen. In addition, CaMoO$_4$ possesses a lower melting temperature (950 °C) than the sintering temperature, and it is in liquid phases at elevated temperature, which effectively promotes the densification sintering. It is also found that the amount of CaMoO$_4$ phases in SM coating is larger than that of PM coating (Figs. 6(a, b)), which is attributed to the CaO in spent MoSi$_2$ powders. However, obvious cracks can be detected on the surface of PM coating after sintering. During the cooling process, thermal stress in the coating causes the generation of cracks since a mismatch of thermal expansion coefficient (CTE) exists between MoSi$_2$ (8.1×10$^{-6}$ K$^{-1}$) and SiO$_2$ (0.6×10$^{-6}$ K$^{-1}$) [21]. By contrast, no cracks are observed in SM coating, and the crack-free morphologies can be attributed to the existence of bentonite and SiO$_2$ phases in the spent MoSi$_2$ powders. Many reports [22–24] claim that the addition of the phases that possess low CTE can adjust the CTE value of the composites, and the content and CTE of the components in the composites controls the general CTE [25], which is given in Eq. (5):

$$\alpha_{co} = \sum_{k=1}^{n} \alpha_k w_k \quad (5)$$

where $\alpha_{co}$ and $\alpha_k$ are the CTE values of the composite coating and component $k$, respectively,
$w_k$ is the mass fraction of the component $k$, and $n$ is the component number. Bentonite ($1.0 \times 10^{-6} \text{ K}^{-1}$) has low CTE [26], and the CTE value of spent MoSi$_2$ is calculated to be $7.39 \times 10^{-6} \text{ K}^{-1}$ by Eq. (5), exhibiting that the addition of bentonite effectively reduces the CTE mismatch between the coating and oxide scale. In addition, bentonite is in the liquid phase during sintering due to its low melting temperature (1340 °C), which benefits the acceleration of the redistribution of coating powders to promote the densification of powders during sintering [27].

Figure 7 shows cross-sectional morphologies of PM and SM coatings after sintering. As shown in Figs. 7(a, e), the coatings are divided into three layers (I, II and III) after sintering, and no

![Fig. 7 Cross-sectional morphologies of PM (a–d) and SM (e–h) coatings after sintering](image-url)
Table 3 Contents of Mo, Si, O and Ca by EDS in Fig. 7 (wt.%)

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<th>Si</th>
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<th>Ca</th>
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<tr>
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<td>50.64</td>
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debonding and spalling exist between the layers, indicating that the layers in the coatings are closely bonded. Furthermore, no oxidation of Mo substrate is found in PM and SM coatings since no gaps caused by the oxidation of Mo are detected between the coating and substrate, illustrating that the substrate keeps unoxidized morphologies during the short-term sintering in air. Layer I is the bonding layer (Figs. 7(a, b)) and mainly made up of MoSi₂ and small amounts of Si. The formation of Mo5Si3 and MoSi 2 diffusion layers can be also found between the substrate and coating. Layer II is the protecting layer and its major composition is MoSi₂. Layer III is the oxygen-barrier layer and consists of SiO₂ and CaMoO₄ phases. However, a slight longitudinal crack appears in the Layer II and Layer III of the PM coating due to the CTE mismatch between the MoSi₂ coating and oxygen-barrier layer. The cracks may provide diffusion path for O₂, which will decrease the oxidation resistance of the coating. Although the bonding layer and protecting layer are relatively dense without any large-sized cracks, pores can be also observed in the oxide scale of the PM coating. During high-temperature sintering in air, Reaction (2) occurs in the MoSi₂-based coating, and MoSi₂ is oxidized to volatile MoO₃. Layer III is close to the oxygen and it first undergoes violent oxidation in air to form SiO₂ oxide scale to block the O₂ diffusion, but pores are also generated by the volatilization of MoO₃. By comparison, no direct contact exists between Layer I–II and the air, and the oxide scale in Layer III also provides corresponding protective effect to inhibit the oxidation. Therefore, rare oxidation reactions happen in the protecting layer and bonding layer, leading to relatively fewer pores found in the two layers.

In comparison with PM coating, although SM coating (Fig. 7(h)) possesses rare amounts of pores in Layer III (oxide scale), it exhibits relatively crack-free coating morphologies after high-temperature sintering. Furthermore, MoSi₂ and Mo₅Si₃ diffusion layers are found between the coating and Mo substrate without debonding or gaps, showing good metallurgical bonding after sintering. At high temperatures, Si has high activity and it diffuses into the Mo substrate to form MoSi₂ layer. In addition, Si atoms in the MoSi₂ layer also diffuse into Mo, which causes the generation of Mo₅Si₃ diffusion layer. It is shown that the diffusion layer can strengthen the bonding via improving the metallurgical bonding between coating and substrate [28] since the mutual diffusion between atoms can promote the formation of a firmly metallurgical interface between coating and substrate at high temperature. Therefore, the formation of the diffusion layers generated by the mutual diffusion of Si and Mo plays an important role in the metallurgical bonding of the coating and substrate, which effectively prevents the spalling and debonding of the coating, and thus the bonding between coating and substrate is strengthened.

3.2 Oxidation behavior of PM and SM coatings at 1500 °C

The XRD patterns of PM and SM coatings after oxidation of 20 h at 1500 °C are shown in Fig. 8. Small amounts of MoSi₂ phase can be still detected in the surface of PM coating after oxidation while only SiO₂ and CaMoO₄ phases
exist in the surface of SM coating. Compared with the coatings before oxidation, the amounts of MoSi$_2$ phases sharply decrease since MoSi$_2$ phases in the coating surface react with O$_2$ to form SiO$_2$ during oxidation at elevated temperature by Eq. (3) [19].

Figure 9 presents the surface morphologies of PM and SM coatings after oxidation of 20 h at 1500 °C. According to Figs. 9(a, b) and Table 4, the gray oxide scale (SiO$_2$) of PM coating is smooth with some white phase (CaMoO$_4$) occupying on the surface, indicating that large amounts of SiO$_2$ are generated during oxidation. As reported [29], silica can improve the anti-oxidation properties of the coating because softened SiO$_2$ can fill the micro-cracks or pores in a high-temperature environment, which blocks the diffusion path of oxygen. Furthermore, SiO$_2$ possesses a low oxygen diffusion coefficient ($4.4 \times 10^{-15}$ m$^2$/s) at high temperature [30] and it serves as the oxygen barrier to inhibit further oxidation in the coating. Thus, no obvious cracks can be found in the PM coating after long-term oxidation due to the self-healing effect of silica. However, some large-sized pores (about 20 μm), which are caused by volatile MoO$_3$ during high-temperature oxidation, are observed on the surface of the oxide scale of PM coating, and they are beneficial to the high-temperature diffusion of O$_2$. As shown in Figs. 9(c, d), although some pores are found in the silica oxide scale of SM coating, the size of the pores is much smaller (about 6 μm), and the oxide scale is dense and complete with even distribution of white CaMoO$_4$ phase on the surface. Thus, SM coating shows improved oxidation resistance than PM coating.

The mass gains of PM and SM coatings during oxidation at 1500 °C for 0–20 h are shown in Fig. 10(a). Although rapid mass gains of the two coatings occur during initial oxidation (0–5 h), the rate of the mass gain decreases as the oxidation time prolongs (5–20 h). After oxidation of 20 h, the mass gains of PM and SM coatings reach the maximum values of 9.21 and 6.28 mg/cm$^2$, and the oxidation rates decrease to the minimum values of 0.46 and 0.31 mg/(h·cm$^2$), respectively. According to Fig. 11, which presents the standard Gibbs free energy change of Reactions (2)–(4) from 0 to 2000 °C, Reactions (2) and (3) have relatively low free energy among the oxidation reactions, exhibiting that SiO$_2$ is preferentially formed by the oxidation of MoSi$_2$. During the rapid mass change stage (0–5 h), or Stage I, the formation of SiO$_2$ causes the rapid mass gain of the coating. When the oxidation time increases (5–20 h, Stage II), the reaction rate of MoSi$_2$ with O$_2$ decreases because of

![Fig. 9 Surface morphologies of PM (a, b) and SM (c, d) coatings after oxidation at 1500 °C for 20 h](image)
Table 4 Contents of Mo, Si, O and Ca by EDS in Fig. 9 (wt.%)

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Fig. 10 Mass gain (a) and corresponding fitting curves of oxidation time versus square of mass gain (b) of PM and SM coatings after oxidation at 1500 °C for 0−20 h

Fig. 11 Standard Gibbs free energy change of Reactions (2)−(4) from 0 to 2000 °C

respectively. Compared with PM coating, SM coating exhibits lower mass gain and oxidation rate constant at 1500 °C because of the relatively crack-free and dense morphologies after sintering, which is beneficial to inhibiting the high-temperature diffusion of O₂. Therefore, SM coating shows better anti-oxidation properties.

Cross-sectional morphologies of PM and SM coatings after oxidation at 1500 °C for 20 h are shown in Fig. 12. In Figs. 12(a−c), PM coating still maintains layered structure after 20 h oxidation. Layer I is mainly made up of MoSi₂, and Layer II consists of MoSi₂ and SiO₂ that are formed during oxidation. Layer III is the oxide scale (541 μm), the composition of which is SiO₂ and CaMoO₄ phases, and some pores are found in the oxide scale as well. After oxidation of 20 h at 1500 °C, an obvious diffusion layer is detected in the PM coating. According to the EDS analysis (Fig. 5), the diffusion layer mainly consists of the MoSi₂ layer and Mo₅Si₃ layer because Si continuously reacts with Mo at elevated temperatures. The diffusion coefficient of Si and reaction time influence the diffusion layer thickness in high-temperature environment. Compared with the sintering process, the temperature of the oxidation is higher and it makes the diffusion coefficient of Si increase. Furthermore, the increasing oxidation time causes the thickening of the diffusion layer as well. As shown in Figs. 12(d−f), layered structure can be also observed in the SM coating, which has similar chemical composition in Layers I, II and III of PM coating. Although some pores exist in the oxide scale, no debonding or obvious cracks can be
detected after oxidation of 20 h. Compared with PM coating, SM coating has relatively crack-free and dense morphologies with a thinner oxide scale (423 μm), showing less consumption of the coating with improved anti-oxidation properties.

Figure 13 represents the schematic diagram of the sintering and oxidation mechanism of SM coating on Mo substrate. During sintering, large numbers of Si atoms in Layer I diffuse into Mo substrate to form the MoSi₂ layer. As the sintering time increases, Si atoms diffuse into Mo to form diffusion layers and the bonding layer is formed to enhance the metallurgical bonding between the coating and substrate. Layer II, which possesses the highest content of the MoSi₂ phase, is the major structure of the coating system and it acts as the protecting layer to safeguard the Mo substrate. These MoSi₂ phases are covered by bentonite with low melting temperature and good oxidation resistance, which is conducive to improve the compactness of the coating and anti-oxidation properties during sintering. Layer III plays the most important role in preventing the diffusion of oxygen. During sintering, the MoSi₂ and Si in the coating are oxidized to SiO₂, which is the main phase of the oxygen-barrier layer. The glass powders also provide plenty of SiO₂ phases for Layer III. SiO₂ has a low oxygen diffusion coefficient and it can block the oxygen diffusion channel to improve the oxidation resistance of the coating. In addition, the CaO phase in the glass powders reacts with volatile MoO₃ to form CaMoO₄ that has good chemical stability, inhibiting the formation of pores in the coating. As the oxidation time prolongs and the temperature rises (1500 °C), the Mo₅Si₃ diffusion layer is thickened by the diffusion of Si. Furthermore, the thickness of the oxide scale increases due to the oxidation of the MoSi₂ phase in Layer III. After high-temperature oxidation, except for some pores in the oxide scale, no obvious defects such as debonding and spalling can be detected in Layers I, II and III, exhibiting that SM coating possesses good anti-oxidation properties at elevated temperature.

Table 5 Contents of Mo and Si by EDS in Fig. 12 (wt.%)

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<td>14.59</td>
</tr>
<tr>
<td>3</td>
<td>64.78</td>
<td>35.22</td>
</tr>
<tr>
<td>4</td>
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<td>29.04</td>
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</tbody>
</table>

Fig. 12 Cross-sectional morphologies of PM (a−c) and SM (d−f) coatings after oxidation at 1500 °C for 20 h
4 Conclusions

(1) Spent MoSi$_2$ is successfully recycled to prepare the multilayer MoSi$_2$-based anti-oxidation coating on Mo substrates by slurry painting. MoSi$_2$, Mo$_5$Si$_3$, SiO$_2$ and CaMoO$_4$ phases are detected in PM and SM coatings after sintering, and the formation of pores caused by volatile MoO$_3$ is inhibited by the reaction of MoO$_3$ with CaO in the glass powders. Obvious cracks are formed in the PM coating after short-term sintering while the SM coating is crack-free and complete without debonding or gaps.

(2) Although no obvious cracks are detected in the surface of PM coating after oxidation of 20 h, large-sized pores can be still observed in the silica layer while a relatively dense and continuous silica layer with rare amounts of pores is generated on SM coating. These large-sized pores provide a diffusion path for oxygen to react with MoSi$_2$, causing rapid mass gain of PM coating. After oxidation of 20 h at 1500 °C, the mass gain of SM coating is only 6.28 mg/cm$^2$, which is lower than that of PM coating (9.21 mg/cm$^2$). MoSi$_2$ and Mo$_5$Si$_3$ diffusion layers are generated in SM coatings during oxidation and the oxide scale of SM coating is thinner than PM coating, exhibiting less coating consumption with better anti-oxidation properties at high temperatures.

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钼基体表面二硅化钼涂层的制备及其高温抗氧化性能

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摘要: 以工业废弃硅化钼棒为原料, 采用浆料涂刷法在钼基体表面制备 MoSi2 基抗氧化涂层。研究涂层的显微组织、相组成及在 1500 ℃的高温氧化行为。结果表明, 以废弃硅化钼棒和纯 MoSi2 粉末为原料制备的涂层和基体之间形成明显的连接层。纯 MoSi2 制备的涂层(PM)在烧结后出现少量裂纹, 而以废弃硅钼棒为原料制备的涂层(SM)在烧结后没有裂纹。1500 ℃氧化后, PM 涂层的氧化膜存在较大尺寸的孔隙, 而 SM 涂层表面氧化膜相对更完整和致密, 且在氧化过程中质量增加更少, 表现出更好的抗氧化性能。

关键词: 再利用; 废弃硅化钼棒; 浆料涂刷; 抗氧化性能; 钼基体

(Edited by Bing YANG)