Microstructure and Nanomechanical Property of Plasma-Sprayed Nanostructured Yb$_2$SiO$_5$ Environmental Barrier Coatings

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Abstract

In this study, nanostructured Yb$_2$SiO$_5$ coatings were prepared by atmospheric plasma spraying (APS) using nanostructured Yb$_2$SiO$_5$ feedstocks. Conventional Yb$_2$SiO$_5$ coatings were selected for comparison. The microstructure and nanomechanical property of the nanostructured and conventional Yb$_2$SiO$_5$ coatings were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and nanoindentation. Results indicate that the surface of the nanostructured Yb$_2$SiO$_5$ coatings is uniform and denser than the conventional Yb$_2$SiO$_5$ coatings. In addition, Weibull distribution analysis shows that the molten state of the nanostructured Yb$_2$SiO$_5$ coatings present a mono-modal distribution, whereas the conventional Yb$_2$SiO$_5$ coatings show a bi-modal distribution, i.e. molten and unmelted zones. The nanostructured Yb$_2$SiO$_5$ coatings have a higher elastic modulus than the conventional Yb$_2$SiO$_5$ coatings (167.37077 ± 16.88070 GPa versus 153.72856 ± 19.69907 GPa), reflecting their high density.

1. Introduction

To improve the thermal efficiency, thrust-to-weight ratio and inlet temperature of a gas turbine, improvements on thermal barrier coatings have allowed the industry to increase the gas inlet temperatures up to 1500°C [1-3]. Nevertheless, 1500°C exceeds the melting point of Ni-based super-alloys, and new substrate materials should be developed. Non-oxide silicon-based ceramics, such as SiC and Si$_3$N$_4$, show potential applications in high-performance gas turbine engines due to their low density, superior fracture toughness, high strength and reliability in high-temperature environments [4]. However, SiC-based materials are susceptible to corrosion and performance degradation in high-temperature environments, resulting in disaster damage for gas turbine engine [5-9]. Therefore, environmental barrier coatings (EBCs) were developed to protect the SiC substrate and improve the durability of structural components in high-temperature environments [4, 10, 11].

To date, the research on environmental barrier coating systems has focused on Si bond coat, mullite coat and rare-earth silicate topcoat. Ideal EBC materials should have a high melting point, low thermal conductivity, low density, low oxygen diffusion, a favorable chemical stability, thermodynamic compatibility with the substrate and a coefficient of thermal expansion (CTE) near the substrate [12-16]. Rare-earth silicates present potential applications for EBC candidate materials owing to their good oxidation resistance, favorable chemical stability, high melting point and appropriate CTE at elevated temperature. Among these rare-earth silicates, Yb$_2$SiO$_5$ and Yb$_2$Si$_2$O$_7$ are widely investigated as EBC topcoats. Most researchers investigating EBCs have utilised the atmospheric plasma spraying (APS) [17-20], electron beam physical vapor deposition (EB-PVD), and plasma spraying-physical vapor deposition (PS-PVD) methods to fabricate EBCs. APS has great advantages, such as simple preparation process and high productivity, and has become the main preparation method.

Recent studies demonstrated that Yb$_2$SiO$_5$ is a promising EBC material candidate [4, 10, 17]. Yb$_2$SiO$_5$ is a suitable EBC material as a protective layer because of its excellent high-temperature chemical, corrosion
resistance and low CTE. With the development of nano materials science and technology, nano materials have shown unique properties due to some unique effects and consequently become popular in the research and preparation of nanostructured materials [21]. Previous research has established the superior performance of nanostructured coatings over that of conventional structural coatings [22-24]. However, the nanomechanical characterization of nanostructured Yb$_2$SiO$_5$ coatings has not been sufficiently investigated yet.

In this paper, nanostructured and conventional Yb$_2$SiO$_5$ coatings were fabricated by APS. The microstructure and phase composition of nanostructured and conventional Yb$_2$SiO$_5$ coatings were analysed, and their micromechanical properties were investigated by nanoindentation and discussed in detail via fitting the experimental data, including elastic modulus and nanohardness, with probability density function of Weibull distribution.

2. Experiment

2.1 Fabrication of nanostructured Yb$_2$SiO$_5$ EBCs

The nano Yb$_2$SiO$_5$ (n-YbMS) feedstocks (Harbin Institute of Technology, China) have particle sizes between 10 and 90 µm, as shown in Fig. 1(a). For comparison, the micron Yb$_2$SiO$_5$ (m-YbMS) feedstocks (Beijing Sanspurui New Material Co., Ltd) with particle sizes between 10 and 90 µm were selected, as shown in Fig. 1(b). Meanwhile, Si (Hoganas (China) Co., Ltd) and mullite (Harbin Institute of Technology, China) powders were used as bond-coat and intermediate layer materials. SiC (Φ25.4 mm × 5 mm) was used as the substrate. The substrate was cleaned ultrasonically with alcohol for 10 min and then grit-blasted to roughen the surface and enhance the bonding strength. Subsequently, the Si-bond coatings and mullite layer with a thickness of approximately 50 µm and the nanostructured and conventional Yb$_2$SiO$_5$ coatings with a thickness of approximately 150 µm were deposited through APS (Metco), separately. The parameters of APS are listed in Tab. 1.

| Parameters       | Si | n-mullite | n-Yb$_2$SiO$_5$ | m-Yb$_2$SiO$_5$ |
|------------------|----|-----------|----------------|----------------|
| Current (A)      | 420| 450       | 420            | 450            |
| Power (kw)       | 40 | 46        | 43             | 44             |
| Flow rate of Ar (slpm) | 40 | 41        | 38             | 38             |
| Flow rate of H$_2$ (slpm) | 10 | 12        | 12             | 12             |
| Spray distance (mm) | 120| 110       | 120            | 120            |
| Feeding rate (g/min) | 15 | 20        | 20             | 20             |
2.2 Characterization

The microstructure and phase composition of the n-YbMS and m-YbMS feedstocks and the corresponding as-sprayed coatings were characterised by scanning electron microscopy (SEM, JSM-7610FPLUS), transmission electron microscopy (TEM, FEI Tecnai G2 F20) and X-ray diffraction (XRD, Ultima IV, RIGAKU, Japan) using Cu Kα radiation with a scanning rate of 4 °/min at Bragg angle (2θ) between 10 ° and 80 °.

2.3 Nano-indentation test

The nanomechanical properties of the nanostructural and conventional Yb$_2$SiO$_5$ coatings were measured by the nano-indentation method (Bruker Hysitron TI980). The applied load was 98 mN, and the holding time was 10 s. The applied load rate was 196 mN/min. To reduce experimental errors, 22 indents were carried out randomly on the polished cross section of the coatings. The elastic modulus ($E$) and nanohardness ($H$) of the as-sprayed nanostructural and conventional Yb$_2$SiO$_5$ coatings were determined by the nano-indentation method [25].

3. Results And Discussion

3.1. Microstructure of nano Yb$_2$SiO$_5$ feedstocks and micron Yb$_2$SiO$_5$ feedstocks.

Figure 2(a) shows the surface morphology of the n-YbMS feedstocks, and Figure 2(d) shows the surface morphology of the m-YbMS feedstocks. The feedstocks are spherical and smooth, Fig. 1 also shows the flowability of the powders. To observe the internal structure of the feedstocks, the fracture morphology is shown in Figs. 3(a) and 3(b). The microstructure of the n-YbMS feedstocks is denser than that of the m-YbMS feedstocks. Spherical and dense feedstocks with a smooth surface can enhance the flowability of feedstocks and the deposition rate [26, 27]. Moreover, dense feedstocks produce dense coatings with improved wear resistance [26, 28]. On the basis of the heredity of the material tissue, nanostructured feedstocks are denser than conventional feedstocks. Therefore, the coating of the n-YbMS feedstocks is tighter and has better abrasion resistance than that of the m-YbMS feedstocks. Fig. 1(c) is a TEM image of the n-YbMS feedstocks. The grain size is approximately 30–50 nm, showing that the n-YbMS feedstocks are composed of nanostructured agglomerates.

Figure 4 shows the XRD patterns of the n-YbMS and m-YbMS powders. It can be observed that the phase composition is Yb$_2$SiO$_5$ (compared with the X-ray diffraction data of Yb$_2$SiO$_5$ (JCPDS card PDF#40-0386) whether it is n-YbMS powder or m-YbMS powder.

3.2. Microstructure of nanostructured and conventional Yb$_2$SiO$_5$ coatings
The surface morphologies of the nanostructured and conventional Yb$_2$SiO$_5$ coatings are shown in Fig. 5. Some voids and cracks can be observed. The nanostructured Yb$_2$SiO$_5$ coatings have voids and fine cracks, as shown in Fig. 5(a). However, the conventional Yb$_2$SiO$_5$ coatings have more voids and coarse cracks (Fig. 5(b)) than the nanostructured Yb$_2$SiO$_5$ coatings. Therefore, the distribution of cracks in the nanostructured Yb$_2$SiO$_5$ coatings is scattered and narrow, whereas that in the conventional Yb$_2$SiO$_5$ coatings is dense and wide. To observe the internal structure of the coatings, the fracture morphology is shown in Fig. 6(a). Fig. 6(a) shows that the cracks and voids in the nanostructured Yb$_2$SiO$_5$ coatings are fewer and finer than those in the conventional Yb$_2$SiO$_5$ coatings. However, the conventional Yb$_2$SiO$_5$ coatings have more and coarser cracks, including horizontal and vertical cracks, than the nanostructured Yb$_2$SiO$_5$ coatings. These results are consistent with the analysis illustrated in Fig. 5(a) and 5(b). Fig. 6(c) and 6(d) show the Yb$_2$SiO$_5$ topcoats of the partially enlarged detail of the nanostructured and conventional Yb$_2$SiO$_5$ coatings, respectively. Fig. 6(e) is TEM images of nanostructured Yb$_2$SiO$_5$ coatings, it can be seen that there are nano particles in nanostructured Yb$_2$SiO$_5$ coatings.

The XRD patterns of the nanostructured and conventional Yb$_2$SiO$_5$ coatings are shown in Fig. 7. The analysis of the XRD pattern of the coatings reveals that some changes occurred during the spraying process. The phase composition is Yb$_2$SiO$_5$ and Yb$_2$O$_3$ (compared with the XRD data of Yb$_2$SiO$_5$ (JCPDS card PDF#40-0386, 2/a (15)) and Yb$_2$O$_3$ (JCPDS card PDF#65-3173, la-3 (206))). Fig. 7 shows that Yb$_2$O$_3$ crystal planes (222), (400), (411), (134), (422), (440), (622), (136), (444) and (142) are all reflected, indicating that the volatilization of silicon dioxide in plasma spraying leads to the presence of Yb$_2$O$_3$ in the coating. The strength of Yb$_2$O$_3$ crystal plane (222), (400), (440), (622), (136) and (444) in the nanostructured Yb$_2$SiO$_5$ coatings is stronger than that in the conventional Yb$_2$SiO$_5$ coatings, and the nanostructured Yb$_2$SiO$_5$ coatings also show Yb$_2$O$_3$ crystal planes (134) and (136). These findings can be attributed to the easy melting of the nano particles in the nanostructured Yb$_2$SiO$_5$ coatings during the APS preparation process, but the SiO$_2$ volatilization is more serious than that in the conventional Yb$_2$SiO$_5$ coatings.

### 3.3 Mechanical properties of nanostructured and conventional Yb$_2$SiO$_5$ coatings.

Mechanical properties are vital to the evaluation of the durability and reliability of coatings [29]. Micromechanical properties, such as elastic modulus and nanohardness are among the important mechanical properties for coatings [25-27, 30-33]. Fig. 8 shows the typical load–displacement curves derived from the nano-indentation of nanostructured and conventional Yb$_2$SiO$_5$ coatings. According to the load–displacement curves in Fig. 8, the maximum penetration depth of the nanostructured Yb$_2$SiO$_5$ coatings is lower than that of the conventional Yb$_2$SiO$_5$ coatings (853.30 nm vs. 1185.47 nm), and the load-displacement curve distribution of the nanostructured Yb$_2$SiO$_5$ coatings is concentrated and uniform compared with that of the conventional Yb$_2$SiO$_5$ coatings. This finding indicates that the regions in the nanostructured Yb$_2$SiO$_5$ coatings can be considered as melted regions, and those in the conventional
Yb$_2$SiO$_5$ coatings where the penetration depth reaches the maximum (1185.47 nm) can be considered as unmelted or partially melted regions. This result is consistent with the SEM images.

When the feature size of the material is at the nanometer level, the microstructure characteristics of the material will change significantly. The size of the nano-featured structure of the material decreases, and the proportion of each interface (e.g., grain boundary, phase boundary, etc.) in the material increases, resulting in the change of the mechanical properties of the material. The elastic modulus ($E$) and nanohardness ($H$) of coatings can be obtained from each unloading curve, and the data is shown in Table 2. The analysis in Table 2 shows that the nanohardness ($H$) of the nanostructured Yb$_2$SiO$_5$ coatings is not very different from that of the conventional Yb$_2$SiO$_5$ coatings, but the elastic modulus ($E$) of the nanostructured Yb$_2$SiO$_5$ coatings is higher than that of the conventional Yb$_2$SiO$_5$ coatings ($167.37077 \pm 16.88070$ GPa vs. $153.72856 \pm 19.69907$ GPa), indicating that the nanostructure can increase the toughness of the coating [28]. Therefore, to further illustrate the influence of nanostructures on the coatings, statistical analysis should be performed on the measured elastic modulus ($E$) and nanohardness ($H$). Weibull distribution can further describe the difference in the microstructure and performance of different coatings[25, 34].

### Table 2

| Coatings                        | Er(GPa)                  | H(GPa)                  |
|---------------------------------|--------------------------|-------------------------|
| nanostructured Yb$_2$SiO$_5$ coatings | $167.37077 \pm 16.88070$ | $11.21317 \pm 1.52426$ |
| conventional Yb$_2$SiO$_5$ coatings | $153.72856 \pm 19.69907$ | $10.21402 \pm 2.27309$ |

The Weibull distribution can be expressed as following [25]:

\[
p = 1 - \exp\left[-\left(\frac{x}{x_0}\right)^m\right], \tag{1}
\]

\[
p_i = \frac{i - 0.5}{N}, \tag{2}
\]

where $p$ is the cumulative probability density function; $x$ is the value of elastic modulus ($E$) or nanohardness ($H$); $x_0 = 63.2\%$; $m$ is the Weibull modulus; $i = 1, 2, 3, ..., 22$; $N = 22$; $m$ is the dispersion coefficient. Equation (1) is mathematically deformed to obtain Equation (3):

\[
\ln \ln \left(\frac{1}{1-p}\right) = m \ln x - m \ln x_0 \tag{3}
\]

The value of $m$ is calculated by mathematically fitting equation (3). Fig. 9 is a Weibull diagram of the elastic modulus and nanohardness of the cross-sections of the nanostructured and conventional Yb$_2$SiO$_5$
coatings.

As shown in Fig. 9, the nanostructured Yb$_2$SiO$_5$ coatings present a mono-modal distribution, indicating that the nanostructured Yb$_2$SiO$_5$ coatings are evenly distributed. During the spraying process, the nanostructure caused the particles to melt completely, so the nanostructured coatings presented a mono-modal distribution, whereas the conventional Yb$_2$SiO$_5$ coatings showed a bi-modal distribution, that is, the coating appeared both molten and unmelted states during the spraying process. The bi-modal distribution led to uneven coating distribution and increased defects. In Fig. 9(b), $m$ represents the Weibull coefficient. The area with a low $m$ value is the unmelted area of the conventional Yb$_2$SiO$_5$ coatings, and the low $m$ value reflects the large dispersion and fluctuation of the elastic modulus ($E$) and nanohardness ($H$) of the area, also indicating that the conventional Yb$_2$SiO$_5$ coatings have larger holes or wider cracks in the unmelted zone. In Fig. 9(a), $m$ is a single value, indicating that the nanostructured Yb$_2$SiO$_5$ coatings have a single distribution. The nanostructured feedstock have nano-scale grains, and the melting is highly uniform under the same spraying process. Therefore, the nanostructured Yb$_2$SiO$_5$ coatings present a single melting area, and the single distribution makes the coating uniform and dense. The nanostructure feedstocks are composed of raw materials with a nanometre grain size, and the grain size does not influence the hardness properties [35]. Therefore, the nanohardness ($H$) of the nanostructure Yb$_2$SiO$_5$ coatings is not very different from that of the conventional coatings. However, the elastic modulus ($E$) is more sensitive to voids, and the fewer the voids are, the higher the elastic modulus ($E$) is [25, 36-38]. In the previous analysis, the elastic modulus ($E$) of the nanostructured Yb$_2$SiO$_5$ coatings is higher than the conventional Yb$_2$SiO$_5$ coatings, indicating that nanostructured coatings have fewer pores than the conventional coatings. This result is consistent with the SEM images.

| Set   | nanostructured Yb$_2$SiO$_5$ coatings | conventional Yb$_2$SiO$_5$ coatings |
|-------|--------------------------------------|-----------------------------------|
| $m_{E1}$ | 11.17 | 14.35 |
| R-Square | 0.94 | 0.96 |
| $m_{E2}$ | N.A. | 0.49 |
| R-Square | N.A. | 0.55 |
| $m_{H1}$ | 8.50 | 10.32 |
| R-Square | 0.96 | 0.95 |
| $m_{H2}$ | N.A. | 2.89 |
| R-Square | N.A. | 0.87 |
4. Conclusions

In this work, nanostructured and conventional Yb$_2$SiO$_5$ EBCs were prepared by APS. The microstructures of the nanostructured and conventional Yb$_2$SiO$_5$ EBCs were characterized by SEM and XRD. In addition, the mechanical properties of the nanostructured and conventional Yb$_2$SiO$_5$ coatings were systematically investigated. Some conclusions can be gained as follows:

1. The nanostructured Yb$_2$SiO$_5$ coatings have smaller holes and finer cracks than the conventional Yb$_2$SiO$_5$ coatings because the feedstock of the nanostructured Yb$_2$SiO$_5$ coating has nano-scale crystal grains, and the good melting in the spray preparation increases the coating’s density and uniformity.

2. According to the nanoindentation analysis, the nanostructured Yb$_2$SiO$_5$ coatings have a mono-modal distribution, and the conventional Yb$_2$SiO$_5$ coatings have a bi-modal distribution, including both molten and unmelted areas. The mono-modal distribution of the nanostructured Yb$_2$SiO$_5$ coating shows that the surface of the coating is uniform and dense. The double Weibull coefficient ($m$) of the conventional coatings indicates the presence of melting and unmelted zones. The small $m$ value reflects the dispersion of the unmelted zone and obvious coating defects.

3. In the presence of nanostructures in the nanostructured Yb$_2$SiO$_5$ coatings, the nanometre size leads to an increase in the crystal planes of the material, the melting is more complete at high temperatures, and the coating have the fewer voids, increasing the elastic modulus of the nanostructured Yb$_2$SiO$_5$ coatings (167.37077 ± 16.88070 GPa vs. 153.72856 ± 19.69907 GPa).

Declarations

Declaration of Competing Interest

The authors declare no conflict of interest.

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**Figures**

**Figure 1**

Particle size distribution of the feedstock (a) the n-YbMS powder, (b) the m-YbMS powder.

**Figure 2**

SEM images of (a) n-YbMS powder, (b) partial enlarged detail of (a), (c) TEM images of n-YbMS, (d) m-YbMS powder, (e) partial enlarged detail of (d).

**Figure 3**

Cross-sectional morphology SEM images of (a) n-YbMS powder and (b) m-YbMS powder.

**Figure 4**

XRD patterns of n-YbMS powder and m-YbMS powder.

**Figure 5**
Surface morphologies SEM images of (a) nanostructured Yb2SiO5 coatings, (b) conventional Yb2SiO5 coatings.

**Figure 6**

Cross-sectional morphology SEM images of (a) nanostructured Yb2SiO5 coatings, (b) conventional Yb2SiO5 coatings (c) Yb2SiO5 top coats partial enlarged detail of (a), (d) Yb2SiO5 top coats partial enlarged detail of (b), (e) STEM images of nanostructured Yb2SiO5 coatings.

**Figure 7**

XRD patterns of nanostructured and conventional Yb2SiO5 coatings.

**Figure 8**

Load-displacement curves of (a) nanostructured Yb2SiO5 coatings and (b) conventional Yb2SiO5 coatings.

**Figure 9**

Weibull plots of elastic modulus and nanohardness on cross-section for (a) nanostructured Yb2SiO5 and (b) conventional Yb2SiO5 coatings.