Investigation of microstructure changes in Al_2O_3 –YSZ coatings and YSZ coatings and their effect on thermal cycle life

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Abstract: Yttria-stabilized zirconia (YSZ) coatings and Al_2O_3 –YSZ coatings were prepared by atmospheric plasma spraying (APS). Their microstructural changes during thermal cycling were investigated via scanning electron microscopy (SEM) equipped with electron backscatter diffraction (EBSD) and X-ray diffraction (XRD). It was found that the microstructure and microstructure changes of the two coatings were different, including crystallinity, grain orientation, phase, and phase transition. These differences are closely related to the thermal cycle life of the coatings. There is a relationship between crystallinity and crack size. Changes in grain orientation are related to microscopic strain and cracks. Phase transition is the direct cause of coating failure. In this study, the relationship between the changes in the coating microstructure and the thermal cycle life is discussed in detail. The failure mechanism of the coating was comprehensively analyzed from a microscopic perspective.

Keywords: thermal barrier coatings (TBCs); thermal cycling; microstructure change; microscopic strain; failure mechanism

1 Introduction

Thermal barrier coatings (TBCs) are widely used in hot-end parts such as gas turbine blades in the aerospace field to provide protection for superalloys so that they can work at higher intake temperatures. The ultimate goal of TBCs is to improve the thrust of the aero-engine and the thermal efficiency of the fuel [1–4]. However, turbine inlet temperature requirements are

continuously increasing with continuous development in the aerospace field. The performances of thermal barrier coatings, such as thermal conductivity and thermal stability, need to be further improved in order to meet the higher application temperature and more serve working condition [5–7]. Therefore, in recent years, numerous studies have focused on developing new TBC systems with lower thermal conductivity, including doping other metal oxides such as Sc_2O_3 , Al_2O_3 , CeO_2 , SnO_2 , La_2O_3 , Gd_2O_3 , and Yb_2O_3 into yttria-stabilized zirconia (YSZ) coatings [8]. Among these metal oxide doped materials, Al_2O_3 –YSZ coating

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is expected to become a candidate material for TBCs owing to its excellent performance. Al_2O_3 –YSZ coatings prepared by Tarasi *et al.* [9] and Song *et al.* [10] had low thermal conductivities of 0.99 and 0.91 W/(m·K) at room temperature, respectively, which were lower than that of YSZ coatings. Yu *et al.* [11] prepared nanostructured 13 wt% Al_2O_3 –8 wt% Y_2O_3 – ZrO_2 (13AlYSZ) coatings by atmospheric plasma spraying (APS). The addition of nano- Al_2O_3 effectively restrained the grain growth of the zirconia phase and improved the thermal stability of the coating at high temperatures. Yin *et al.* [12] studied the corrosion resistance and mechanical properties of Al_2O_3 -modified YSZ coatings. It was found that Al_2O_3 doping could prevent CMAS corrosion due to the formation of anorthite.

It has been shown that Al_2O_3 –YSZ coating has better thermal insulation performance, but its thermal cycle life is relatively worse compared with YSZ coatings. There was also a report that the thermal cycle life of Al_2O_3 –YSZ coatings was even comparable with their counterpart of YSZ coatings by applying the double-layer coating design [13]. So, if we can know well about the factors to influence the thermal cycle life of Al_2O_3 –YSZ coatings, it would be helpful to meet the requirement of next generation thermal barrier coatings. However, current research on the thermal cycle life of TBCs is mainly focused on macroscopic stress, concentrating on the thermal expansion coefficient, or residual stress [14–16]. Xu *et al.* [17] established the relationship between the thermal expansion coefficient and the Al_2O_3 doping amount. They found that the thermal expansion coefficient showed a downward trend with an increasing amount of Al_2O_3 doping. Song *et al.* [18] found that the change in thermal expansion coefficient caused by Al_2O_3 doping could be attributed to the change in lattice constant, which led to a change in the bonding strength of Al_2O_3 -doped t-YSZ. Wang *et al.* [19] attributed delamination failure of the coating to the large stress induced by the appearance of the thermally grown oxide. However, few studies have explored the effect of the microstructure and its changes on the thermal cycle life of the coatings from a microscopic point of view. Yang *et al.* [20] explored the effect of nanostructure content on thermal shock resistance of the coatings. When the nanostructure content was medium, the coatings showed best thermal cycle life. Tarasi *et al.* [9] found that the amorphous phase impaired the mechanical properties of the coatings. Loganathan and Gandhi [21] studied the

relationship between the fracture toughness and microstructural changes such as increasing cubic phase fraction and residual stresses. Fan *et al.* [22] mentioned the influence of grain size on thermal shock resistance. They found that small grain size had the positive effect on thermal cycle life. This was thought to have resulted from an increase in the number of grain boundaries that restricted the grain motion during deformation. However the relationships among crystallinity, grain morphology, phase transition, and thermal cycling life were often ignored, and grain deformation resulting in microscopic stress during thermal cycling was seldom characterized. According to finite element simulation, the magnitude of the numerical change in the microscopic stress was at the megapascal level, and it could even reach the gigapascal level in some cases [23–25]. Therefore, the influence of microscopic stress on the thermal cycle life of the coating cannot be ignored.

In this study, YSZ and the single-layer Al_2O_3 –YSZ coatings were prepared by APS technology in order to simplify the experiment. The same position was analyzed by the scanning electron microscopy (SEM) equipped with the electron backscatter diffraction (EBSD) to determine the microstructural changes occurring during thermal cycling. By comparing the microstructure of the two coatings and the changes in microstructure during thermal cycling, the failure mechanism of Al_2O_3 –YSZ coatings was analyzed by microscopic strain and phase transition.

2 Experimental

2.1 Materials and coating preparation

In this experiment, the TBC system included a metal substrate, a metal bonding layer, and a ceramic layer. A Ni-based superalloy was selected as the metal substrate, and cuboid samples with the dimensions of 20 mm×10 mm×2 mm were prepared. A bonding layer with a thickness of 100 μm was prepared by the vacuum plasma spraying (VPS-Sulzer Metco AG, Switzerland), which was used as a bonding layer and could also reduce the thermal stress due to the mismatching of thermal expansion between the ceramic layer and the substrate. The chemical composition of the bonding layer powders is listed in Table 1. Commercial YSZ powder (7.5 wt% Y_2O_3) and Al_2O_3 –YSZ powder (10 wt% Al_2O_3 , 7.5 wt% Y_2O_3) were used to prepare the

Table 1 Chemical composition of NiCrCoAlY powder in bond coat (wt%)

Element	Co	Ni	Cr	Al	Y
Content	Bal.	29.0–35.0	29.0–35.0	5.0–11.0	0.1–0.8

Table 2 Parameters of plasma spraying

Parameter	Bond coat	Ceramic topcoat
Spraying process	VPS	APS
Current (A)	720	600
Ar (L/min)	50	30
H ₂ (L/min)	9	10
Spray distance (mm)	275	120
Powder feed rate (r/prm)	25	28

ceramic layer with a thickness of 200 μm by the APS (Sulzer Metco AG, Switzerland). Table 2 lists the spraying parameters used.

2.2 Thermal cycling test

The purpose of the thermal cycling experiments was to understand the changes in coating microstructure that occurred with temperature changes. First, the two samples were placed in a tube furnace at 950 $^{\circ}\text{C}$ for 15 min, and then they were removed and placed in air

for 25 min to cool the samples to near room temperature [26]. The two samples were characterized after different numbers of thermal cycles (0, 5, 10, 15, 20, 25, 30, and 40).

2.3 Ex-situ characterization and calculation of misorientation

For the thermal cycling experiments of YSZ and Al_2O_3 –YSZ coatings, one area was selected in the two coatings' cross-sections for *ex-situ* characterization. The microstructure of the coating was observed using a field emission scanning electron microscope (Magellan 400, FEI, USA) equipped with an EBSD probe (INCA SERIES, Oxford Instrument, UK). While obtaining the morphology information, the orientation information of each grain was also included, which was expressed in the form of three Euler angles. In this experiment, the orientation change of some randomly selected grains during thermal cycling was calculated, and the detailed process is as follows. First, two groups of Euler angles before and after thermal cycling of the same grain were transformed into an orientation matrix. The transition formulas of the Euler angle (ϕ_1 , Φ , ϕ_2) and orientation matrix are as follows:

$$\begin{vmatrix} \cos\phi_1\cos\phi_2 - \sin\phi_1\sin\phi_2\cos\Phi & \sin\phi_1\cos\phi_2 + \cos\phi_1\sin\phi_2\cos\Phi & \sin\phi_2\sin\Phi \\ -\cos\phi_1\sin\phi_2 - \sin\phi_1\cos\phi_2\cos\Phi & -\sin\phi_1\sin\phi_2 + \cos\phi_1\cos\phi_2\cos\Phi & \cos\phi_2\sin\Phi \\ \sin\phi_1\sin\Phi & -\cos\phi_1\sin\Phi & \cos\Phi \end{vmatrix}$$

The relationship between $\mathbf{M}_1$ and $\mathbf{M}_2$ can be defined as follows: $\mathbf{M}_2 = \mathbf{M}_{1 \rightarrow 2} \cdot \mathbf{M}_1$ or $\mathbf{M}_{1 \rightarrow 2} = \mathbf{M}_2^{-1} \cdot \mathbf{M}_1$. $\mathbf{M}_{1 \rightarrow 2}$ is a disorientation matrix. $\mathbf{M}_2^{-1}$ is the inverse matrix of $\mathbf{M}_2$. In this work, matrix $\mathbf{M}_{1 \rightarrow 2}$ was converted to the rotation axis and rotation angle. The rotation angle was used as the criterion to evaluate the degree of grain orientation change. The formula for calculating the rotation angle is as follows:

$$\theta = \arccos[0.5 \times (\mathbf{M}_{1 \rightarrow 2} 11 + \mathbf{M}_{1 \rightarrow 2} 22 + \mathbf{M}_{1 \rightarrow 2} 33 - 1)] \quad (1)$$

The phase composition and microscopic strain of the coating at different thermal cycles were characterized using a two-dimensional micro-focal spot X-ray diffractometer (XRD; D8 ADVANCE, Bruker, Germany). When the microscopic stress occurs in the coating, the peaks in the XRD patterns will be broadened. Therefore, the microscopic strain of the coatings can be calculated after the refinement of the XRD patterns.

3 Experimental results

3.1 Microstructure of the as-sprayed YSZ and Al_2O_3 –YSZ coatings before thermal cycling

The XRD patterns of YSZ and Al_2O_3 –YSZ coatings before thermal cycling are shown in Fig. 1. At high magnification of the XRD patterns, it can be seen that there is a unique peak from the tetragonal phase in YSZ coating at approximately 43° when the two patterns are compared. This peak appears only when the content of the tetragonal phase is high. The tetragonal phase can also be characterized by the splitting of multiple peaks from those of the cubic phase. Therefore, YSZ coatings are mainly composed of a tetragonal phase and a small amount of cubic phase, while Al_2O_3 –YSZ coatings consist of cubic phase. According to the peak intensity given by Jade and the approach reported by Yang *et al.* [27], the cubic phase content of Al_2O_3 –YSZ

Fig. 1 XRD patterns of the as-sprayed YSZ and Al_2O_3 -YSZ coatings.

coatings is close to 100%, and that of YSZ coating is only about 5%. It was found that there is no α - Al_2O_3 phase, which can be explained by a solid solution of some Al atoms in ZrO_2 during plasma spraying [11] and aggregation of some Al atoms at grain boundaries to form amorphous phase [18].

Figure 2 shows the backscattered electron image of the microstructure of the sprayed YSZ and Al_2O_3 -YSZ coatings. It can be seen that there are significant differences in microstructure between the two coatings. SEM images of the two coatings were analyzed by image processing software Image J. The original three-channel and three-color RGB images are linearly converted into a single-channel 8-bit grayscale image. After setting the correct scale, the crack size is calculated statistically. The average crack size of Al_2O_3 -YSZ coatings was 26 μm and that of YSZ coatings was 12 μm . The principal stress at the crack tip increases with an increase in crack size [28]. According to Griffith's theory of microcracks, the strength of

materials does not depend on the number of cracks but on the size of cracks. Once a crack exceeds the critical size, the crack will propagate and connect rapidly, which has an adverse effect on the thermal cycle life of the coating. An analysis of the experimental data shows that there is a close relationship between crack size and crystallinity.

By calculating the crystallinity of the two coatings, it was found that the crystallinity of YSZ coatings is very high, close to 100%, while the crystallinity of Al_2O_3 -YSZ coatings is 78.56%. As shown in Fig. 3, the crystallinity in different regions of the coating is different. The EBSD scanning and backscattered electron images of Al_2O_3 -YSZ coatings in the same area were compared. It was found that the size of the larger cracks in the EBSD scan is significantly larger than that in the backscattered electron image. This phenomenon indicates that an amorphous phase exists near the crack. And the grain size gradually decreases along the direction of the arrow, which means that there is a gradual transition to the amorphous phase near the crack. For the coatings with poor crystallinity, microscopic stress concentration and tensile stress will occur [29], which means that microscopic stress of Al_2O_3 -YSZ coatings near the crack is greater than that of YSZ coatings, explaining why the average crack size in Al_2O_3 -YSZ coatings is larger, which leads to easier propagation of cracks. So, poor crystallinity is one of the main reasons for the formation of larger cracks, which is not conducive to the thermal cycle life of Al_2O_3 -YSZ coatings.

3.2 Effect of grain orientation variation and grain morphology on thermal cycle life

3.2.1 Grain orientation variation

Figure 4 shows the backscattered electron images of

Fig. 2 SEM images from cross-sections of (a) YSZ coatings and (b) Al_2O_3 -YSZ coatings.

Fig. 3 (a) SEM image and (b) EBSD image of Al_2O_3 -YSZ coatings.

Fig. 4 SEM images of polished cross-sections of different coatings before and after thermal cycles: (a, b, d, e) before thermal cycles and (c, f) after five thermal cycles.

YSZ and Al_2O_3 -YSZ coatings before and after five thermal cycles. After five thermal cycles, there is no obvious change in the microstructure of YSZ coatings, but there is obvious crack propagation and connection in Al_2O_3 -YSZ coatings. This experiment was conducted from a microscopic perspective. The experimental data show that the difference between the two coatings is not only related to a larger crack size, but also closely related to the microscopic stress.

Approximately ten grains were randomly selected from the EBSD scans of YSZ and Al_2O_3 -YSZ coatings. As shown in Figs. 5(a) and 5(b), the disorientation of these ten grains compared with the previous thermal cycles was calculated based on the Euler angle data provided by the software. It was found that the change in degree of orientation of each grain in the two kinds of coatings showed the same trend during the thermal

cycles. This is because the grains need to coordinate with the surrounding grains in the process of micro-strain and maintain stress/strain coordination among the various parts [30]. During five thermal cycles, the average change in grain orientation of YSZ coatings was 1.49° , and that of the Al_2O_3 -YSZ coatings was 5.42° . Comparing the grain orientation change trend diagram of Fig. 5(b) with the micro-strain diagram of Fig. 5(c), it can be seen that the degree of grain orientation change is positively correlated with the micro-strain. This indicates that the degree of change in grain orientation is representative. Therefore, during five thermal cycles, Al_2O_3 -YSZ coatings have a large micro-strain, and the resulting micro-stress is of the order of magnitude of megapascal. A larger micro-strain promotes crack propagation and connection, which is unfavorable to the thermal cycle life of Al_2O_3 -YSZ coatings.

Fig. 5 Grain orientation variation trend of (a) YSZ coating and (b) Al_2O_3 -YSZ coating during thermal cycles; (c) microscopic strain of Al_2O_3 -YSZ coating during thermal cycles.

3.2.2 Grain morphology

On closer analysis of the propagation cracks, it was found that different grain shapes have different effects on crack propagation. In this experiment, two regions (sites 1 and 2) near the crack were selected and characterized by EBSD, as shown in Fig. 6. Compared to Fig. 4(e), it can be seen that a new crack appears in the equiaxed grain region. From the different grain orientations on both sides of the crack, it can be observed that the propagation mode of the crack in the equiaxed grain region is intergranular. The crack in the columnar region propagates further based on the original crack. The same grain orientation on both sides of the crack indicates that the crack propagates in transgranular mode in the columnar grain region. From Figs. 4(f) and 6(c), it can be seen that the transgranular crack separates into two cracks during propagation, which eases the stress concentration at the crack tip [31]. In addition, transgranular crack propagation needs

to consume more fracture energy, which means that transgranular cracks in columnar grains are more difficult to propagate. In contrast, intergranular cracks are more likely to occur when the energy at the grain boundary is high and the atoms are in an unstable state. This is consistent with the fact that transgranular cracks in the columnar grains only occur as the propagation of an original crack, while new cracks appear at equiaxed grains. In general, the existence of columnar grains blocks crack propagation to a certain extent.

The reason for different crack propagation forms in different regions is that the microscopic stress concentration regions of equiaxed and columnar grains are different after the occurrence of micro-strain. As shown in Fig. 6(g), the microscopic stress concentration at the equiaxed grain exists at the grain boundary, which affects the bonding of the grain boundary at the equiaxed grains. From a comparison of Figs. 7(a) and 7(b), it is obvious that the equiaxed grains exhibit high

Fig. 6 (a) SEM image of Al_2O_3 -YSZ coating; two areas selected near the crack: (b) site 1 and (e) site 2; (c, f) EBSD images and (d, g) local misorientation distribution images of sites 1 and 2, respectively.

Fig. 7 SEM images of equiaxed grains in Al_2O_3 –YSZ coatings (a) before and (b) after thermal cycles.

and low fluctuations, and there are voids at the grain boundary, especially at the junction of multiple grains. This is consistent with the fact that the crack propagates along equiaxed grains. However, as shown in Fig. 6(d), there is a high probability of stress concentration within the grain after micro-strain of the columnar grains occurs. There is also high energy inside the columnar grains, so the strength difference between the grain boundaries and grains decreases, and the cracks propagate through the columnar grains. In addition, the cracks in the columnar grains are horizontal cracks, which increase the propagation path of vertical cracks and block the penetration of the cracks to a certain extent.

So, a larger micro-strain generates a larger micro-stress, which makes it easier for the cracks to expand and connect, and is not conducive to the thermal cycle life of Al_2O_3 –YSZ coatings. Compared with equiaxed grains, columnar grains play a role in blocking the penetration of cracks to a certain extent.

3.3 Effect of phase and phase transition on thermal cycle life

Figure 8 shows the macroscopic morphologies of Al_2O_3 –YSZ coatings during thermal cycling. Although crack connection and propagation occurred during five thermal cycles, the coatings did not peel off. The most serious spalling occurred after 5–10 thermal cycles. As shown in Fig. 9, the unique minuscule peak appears at approximately 43° and the peak splitting occurs at approximately 73° after 10 thermal cycles. Therefore, it can be seen from the XRD analysis that Al_2O_3 –YSZ coatings are transformed from the cubic phase to the tetragonal phase during these 5–10 thermal cycles. The crystal transition from c-ZrO_2 to t-ZrO_2 is a displacement transition without interatomic diffusion. There is a 2%

Fig. 8 Images of Al_2O_3 –YSZ coatings during thermal cycling.

Fig. 9 XRD patterns of Al_2O_3 –YSZ coatings after 5 and 10 thermal cycles.

volume shrinkage in the transition from the cubic to the tetragonal zirconia phase [32]. Phase transition coupled with the existing cracks lead to the most serious spalling of Al_2O_3 –YSZ coatings during this period.

In addition, a change in grain orientation during thermal cycling of Al_2O_3 –YSZ coatings is observed in Fig. 5(b). It can be seen that 10 thermal cycles mark a significant change. From the 10th thermal cycle onward, the degree of change in the grain orientation of Al_2O_3 –YSZ coatings is reduced to approximately 1.5° , which is similar to that of YSZ coatings. Correspondingly,

Fig. 10 XRD patterns of the as-sprayed YSZ coatings and YSZ coatings after 40 thermal cycles.

the micro-strain of Al_2O_3 –YSZ coatings after 10 thermal cycles is also small. The important reason for the difference in the micro-strain before and after thermal cycling is that the toughness and strength of the zirconia tetragonal phase are better than those of the cubic phase [33]. Therefore, it is more difficult for micro-strain of the grains to occur, and the micro-stress generated is relatively small. It can also be seen in Fig. 8 that the exfoliation rate of the coatings slows down after 10 thermal cycles. This confirms that micro-stress has an important influence on the thermal cycle life of the coatings.

It can be seen that the small changes in grain orientation of YSZ coatings are due to not only a difference in thermal expansion coefficient, which leads to a difference in thermal stress, but also the main phase of YSZ coatings being the tetragonal phase from beginning to end as shown in Fig. 10.

4 Conclusions

In this study, two types of TBCs, YSZ coatings, and Al_2O_3 –YSZ coatings with worse thermal cycle life were prepared. The changes of these two coatings in the microstructure before and during thermal treatment were compared. The entire failure process and factors influencing the thermal cycle life of the coatings during thermal cycles were comprehensively analyzed. The results are as follows:

1) The large crack size is attributed to the appearance of an amorphous phase, which is more likely to expand during thermal cycling. Therefore, low crystallinity is

not conducive to the thermal cycle life of Al_2O_3 –YSZ coatings.

2) The micro-stress caused by the large micro-strain of Al_2O_3 –YSZ coatings during thermal cycling promotes crack propagation and connection. And the grain morphology affects the microscopic stress concentration area, thus affecting crack propagation.

3) A transition from the cubic phase to the tetragonal phase, combined with connected cracks, is the direct cause of spalling failure of Al_2O_3 –YSZ coatings. However, tetragonal zirconia has less micro-strain in thermal cycling, which can slow down spalling of Al_2O_3 –YSZ coatings.

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