Al-modification for PS-PVD 7YSZ TBCs to improve particle erosion and thermal cycle performances

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Abstract: Plasma spray-physical vapor deposition (PS-PVD) as a novel process was used to prepare feather-like columnar thermal barrier coatings (TBCs). This special microstructure shows good strain tolerance and non-line-of-sight (NLOS) deposition, giving great potential application in aero-engine. However, due to serious service environment of aero-engine, particle erosion performance is a weakness for PS-PVD 7YSZ TBCs. As a solution, an Al-modification approach was proposed in this investigation. Through \textit{in-situ} reaction of Al and ZrO\textsubscript{2}, an $\alpha$-Al\textsubscript{2}O\textsubscript{3} overlay can be formed on the surface of 7YSZ columnar coating. The results demonstrate that this approach can improve particle erosion resistance since hardness improvement of Al-modified TBCs. Meanwhile, as another important performance of thermal cycle, it has a better optimization with 350-cycle water-quenching, compared with the as-sprayed TBCs.

Keywords: plasma spray-physical vapor deposition (PS-PVD); thermal barrier coatings (TBCs); Al-modification; particle erosion resistance; thermal cycle performance

1 Introduction

As an advanced technology, the aero-engine has attracted much attention all over the world. Increasing the turbine inlet temperature (TIT) is a direct approach for further enhancing the efficient power and the thrust-weight ratio of the aero-engine [1–4]. High-temperature superalloy components, which were conventionally employed in the aero-engine cannot subject higher turbine inlet temperature due to their relatively low melting point (~1150 °C) [5,6]. Considering this limitation, thermal barrier coatings (TBCs) were proposed to reduce the working temperature of the
high-temperature components. After more than 60 years of research and development, the TBCs prepared by atmospheric plasma spray (APS) and electron beam-physical vapor deposition (EB-PVD) have been widely applied in aero-engines [7,8]. However, the APS process has many disadvantages, such as low strain tolerance and low bond strength [9,10]. The EB-PVD TBCs also have many weaknesses, such as high cost and low efficiency [11,12]. Based on current situation, it is urgent to propose new TBC process for serious requirements of aero-engine [13–15].

The constant progress of the aero-engine TIT determines the development of TBCs. The new deposition technique of plasma spray-physical vapor deposition (PS-PVD) has a different deposition mechanism as compared with the traditional processes [16,17]. The key feature of proposed PS-PVD process is the option of evaporating ceramic powders, which enables the deposition of a feather-like columnar coating through gas–liquid–solid multi-phases [18–22]. The PS-PVD TBCs have many advantages, such as uniform coating on irregularly shaped components, high deposition efficiency, and low thermal conductivity [23,24]. However, the characteristic high porosity (> 15%) of PS-PVD TBCs hurts particle erosion performance [25,26], which is an obstacle for application.

Facing above obstacle, an Al-modification method was proposed for PS-PVD 7YSZ TBCs in this investigation. Through magnetron sputtering, appropriate thickness of Al film was deposited on TBC surface. Subsequently, the Al-deposited TBCs were carried out with vacuum heat treatment, forming Al-modified 7YSZ TBCs. During deposition, Al target (99.99%) was used and the direct current, voltage, and pressure were set as 3 A, 150 V, and $5 \times 10^{-3}$ Pa, respectively. And then, the Al-deposited TBC samples were treated with a certain parameter ($608 \degree C$ for 1 h, 700 $\degree C$ for 1 h, and 980 $\degree C$ for 2 h).

2.2 Plasma flow simulation

The plasma flow of the PS-PVD was equivalent to a quasi-equilibrium continuous medium, and its flow process was described by a Navier–Stokes equation. Therefore, it is accurate to establish the plasma flow model by a computational fluid dynamics (CFD) simulation. The internal and external flow field model of the spray gun was established by ANSYS FLUENT (Ansys 2020 R2, https://www.ansys.com/), which included consideration for the coupling effect of the internal and external flow field. To show the turbulent characteristics of the plasma flow, the shear stress transport & $\kappa-\omega$ two-equation turbulence model was used during the process of the CFD calculations.

2.3 TBC characterization

The microstructures of the as-sprayed and Al-modified 7YSZ TBCs were characterized by field emission-scanning electron microscopy (FE-SEM, 10 kV, Nova-Nono430, FEI) and transmission electron microscopy (TEM, 200 eV, Titan Themis 200, FEI) assisted by focused ion beam (FIB, 450S, FEI) milling. The phase compositions of the as-sprayed and Al-modified TBCs were identified by X-ray diffraction (XRD, 10°–90°, 0.01 (°)/step, scanning speed 0.02 (°)/min, Smart Lab, Rigaku, Japan).
Also, the particle erosion resistances of the TBCs were evaluated by a scratch tester (HH-3000, CAS, China). This is a dead-loaded machine where a separate scratch is made for each applied load. The load was continuously increased from 0 to 80 N with a rate of 100 N/m and a scratch length of 4 mm. The tester was fitted with friction coefficient monitoring equipment which was used as an on-line failure monitor. Besides, the coating hardness was tested by nano indentation instrument (NHT2, Anton Paar) with loading 20 mN (loading time 10 s and unloading time 20 s). The particle erosion performance of both types of TBCs (sample size: \( \Phi 25.4 \text{ mm} \times 5 \text{ mm} \)) was carried out based on the GE standard E50TF121 (impingement angle 20°, distance 101.6 mm, erosion media 240 grit) at room temperature. The thermal cycling properties of the as-sprayed and Al-modified PS-PVD 7YSZ TBCs were analyzed by water-quenching testing. The specimens (sample size: \( \Phi 25.4 \text{ mm} \times 5 \text{ mm} \)) were heated at an evaluated temperature of 1100 °C in a furnace for 10 min followed by direct water-quenching at room temperature for 5 min.

3 Results and discussion

3.1 CFD simulation of PS-PVD TBCs

A typical failure in TBCs can be seen in Fig. 1(a) (Turbo Fan Engine CFM56, Boeing 737). With fast development of aero-engine, the TIT will increase continually, which lead to requirements of high performance TBCs. And the progress of superalloy and TBCs has been summarized in Fig. 1(b). For TBCs, APS, EB-PVD, and PS-PVD are important preparation methods. The APS and EB-PVD TBCs have many advantages and disadvantages, seen in Fig. 1(c). The PS-PVD has a different deposition mechanism as compared with the traditional processes, which can avoid these weaknesses to some extent. The core feature of PS-PVD process is the option of evaporating ceramic powders, obtaining a feather-like columnar coating (Fig. 1(c)).

Inherent weakness of high porosity for PS-PVD TBCs results from its high velocity and high concentration vapor deposition (Fig. 2(a)), which is different from EB-PVD [27]. The high velocity of the plasma flow and the high concentration of vapor particles in the PS-PVD process create a non-line-of-sight (NLOS) deposition, which forms a feather-like columnar coating (Fig. 2(b)) [23]. However, a high concentration leads to a shadow effect created during the nucleation and growth of the vapor particles on the substrate, which is the biggest difference when compared with the EB-PVD process. Additionally, the size of the plasma flame is large, resulting in a large deposition area on the substrate. Figure 2(b) shows that the feather-like columnar 7YSZ
coating has a different-sized gap from the bottom to the top area, where the columns are made of nano-sized grains. A dense coating at the bottom is also a columnar structure, but the gap is very small.

Previous investigations indicate that the promising feather-like columnar microstructure depends on the temperature gradient and the velocity gradient of a plasma flame [27–29]. During the PS-PVD process, the plasma temperature (Fig. 2(c)) and velocity (Fig. 2(d)) distribution were modeled through a computational fluid dynamics (CFD) simulation. The gas flow rate at the inlet of the spray gun was 95 L/min, the boundary pressure of the outflow field was 150 Pa, and a superalloy substrate was set at 1000 mm from the outlet of the spray gun. Figure 2(c) shows that when the plasma gases left the spray gun outlet, the temperature was still as high as 20,000 °C. With an increase in the distance from the gun outlet, the plasma temperature dropped rapidly. The plasma temperature at the position of the substrate decreased to about 1400 °C. As shown in Fig. 2(d), when the plasma left the outlet of the plasma gun, it had a velocity of 7000 m/s. With the increase of the distance from the gun outlet, the plasma velocity dropped rapidly, to 300 m/s when it reached the substrate (1000 mm).

3.2 Solution for particle erosion resistance

It is known that the service environment of TBCs includes high temperature, particle erosion, and CMAS corrosion [1–4]. Due to the high vapor particle concentration, the feather-like columnar 7YSZ coating had a high porosity. This feature led to a low thermal conductivity and high-stress tolerance [30]. However, this microstructure resulted in a negative effect on particle erosion resistance [31]. As a solution to the issue, an Al-modified approach was proposed (Fig. 3(a)) where an Al film was deposited on the feather-like columnar 7YSZ coating. After a vacuum heat treatment, the Al film infiltrated into the columnar coating and reacted with the 7YSZ coating, forming a dense α-Al2O3 overlay. To optimize this process, Al films with different thicknesses (5, 10, and 20 μm) were deposited onto the TBC surface. Figure 3(b) shows a cross-sectional microstructure evolution with the Al film before and
after vacuum heat treatment. The 20 μm Al-modified TBCs were characterized by TEM (Fig. 3(c)), which shows the cross-sectional elemental analysis including HAADF (high angle annular dark field) image and Al, O, and Zr mappings. The experimental results demonstrated that the microstructures of the deposited and Al-modified TBCs are in accordance with the schematic diagram in Fig. 3(a).

The Al-modification process aimed to improve the particle erosion performance. Firstly, to characterize the erosion resistance, the as-sprayed and Al-modified TBCs were compared using scratch testing. Scratch micrographics of different TBCs with different-sized micro-cracks are shown in Figs. 4(a)–4(d), where the various positions P1, P2, and P3 corresponding to the initial position, middle position, and terminal position of the scratch path are presented, respectively. Using a scratch comparison method of the 3D surface profile, the as-sprayed TBCs had a depth of 72 μm, which was deeper than the 5, 10, and 20 μm Al-modified TBCs.
Fig. 4 Scratch test comparison of TBCs. (a) As-sprayed TBCs showing a scratch depth of 72 μm, a profile microstructure, and curves for loading, friction, and the friction coefficient. (b) 5 μm Al-modified TBCs showing a scratch depth of 71 μm, a profile microstructure, and curves for loading, friction, and a friction coefficient. (c) 10 μm Al-modified TBCs showing a scratch depth of 57 μm, a profile microstructure, and curves for loading, friction, and a friction coefficient. (d) 20 μm Al-modified TBCs showing a scratch depth of 56 μm, a profile microstructure, and curves for loading, friction, and friction coefficient.

(71, 57, and 56 μm, respectively). Moreover, the friction forces and friction coefficients of the as-sprayed TBCs had the highest value among the PS-PVD TBCs. Thus, the above phenomenon indicates that the scratch depth was getting shallower with the increasing Al thickness.

Therefore, it can be concluded that the various scratch depth depends on the hardness of the TBCs. The Al-modification process contributed to increasing hardness and it was also expected to improve the particle erosion resistance of PS-PVD TBCs. Through nano indentation testing, the hardness of 20 μm Al-modified TBCs (1650 HV) is greater than the as-sprayed TBCs (999 HV).

The above assumptions have been demonstrated by particle erosion testing, which was based on the testing standard GEAE-E50TF121. The erosion test specimen holder was in accordance with the GE drawing 4013240-525. Particle erosion comparison of the as-sprayed and 20 μm Al-modified TBCs is shown in Figs. 5(a) and 5(b). The weight losses of the as-sprayed and 20 μm Al-modified TBCs are compared in Fig. 5(c) and the corresponding surface images after the particle erosion failure is seen in Fig. 5(d). When the erodent exposure mass of grit reached 60 g, the color of the bond coating was seen. However, the 20 μm Al-modified TBCs lost 90 g of grit before the appearance of the bond coating color. The above results showed that the Al-modified TBCs had a better erosion resistance than the as-sprayed PS-PVD 7YSZ TBCs.

3. 3 Comparison of thermal cycle performance
Apart from the particle erosion resistance, the thermal cycle performance is an important parameter for the characterization of the TBCs representing frequent take-off and landing resistance [30,32]. The images of the as-sprayed and Al-modified PS-PVD with different water-quenching cycles are shown in Fig. 6. The 20 μm Al-modified TBCs showed an optimal thermal cycle performance (Fig. 6(a)) compared to the other APS and EB-PVD TBCs [33–48]. In this work, the as-sprayed and Al-modified TBCs were compared, as shown in Figs. 6(b)–6(i). The first spallation of the as-sprayed TBCs occurred in 162 cycles and after 198 cycles, the TBCs had been completely stripped from the surface of the substrate. With the 5 μm Al-modified TBCs, the first spallation occurred in 100 cycles, and after 115 cycles, the TBCs separated from the substrate. Correspondingly, the 10 μm Al-modified TBCs had first and final spallation occurred in 131 and 162 cycles, respectively.
The above results show that the Al modification process had no positive effect on the thermal cycle performance for the PS-PVD 7YSZ TBCs. However, when the Al thickness increased to 20 µm, the Al-modified TBCs had first spallations occurred in 198 cycles. As the thermal cycle increased to 350 cycles, the TBCs were still not completely stripped from the substrate, and they only showed an increase in the spallation area. And as the Al film continually increased to 30 µm, slight spallation occurred in surface after 131 cycles. However, apparent spallation occurred after 350 cycles. Through comparison, the 20 µm Al-modification was the best process for improving the thermal cycle performance of PS-PVD 7YSZ TBCs.

The results showed that the 20 µm Al-modification was the optimal process for improving the thermal cycle performance of the PS-PVD 7YSZ TBCs. The surface microstructures of the 20 µm Al-modified TBCs are shown in Fig. 7(a). The gap between the cauliflower tops still can be seen and the magnified images indicated that many nanowires had been formed on the surface. Some cauliflower tops were connected by nanowires. The cross-sectional microstructure is shown in Fig. 7(b), where the positions P1, P2, and P3 represent different areas. After the 20 µm Al-modification, a dense overlay was formed on the top columnar coating. In the inner coating, the porous microstructure was still preserved. However, after 350 water-quenching cycles at 1100 °C, many cauliflower tops had been stripped from the surface and the grain size including nanowires had increased (Fig. 7(c)). The cross-sectional microstructures at different positions (P1, P2, and P3) were seen in Fig. 7(d), and were becoming denser than the cross-section in Fig. 7(b). The evolutionary process of the phase composition in the PS-PVD 7YSZ coating from the as-spraying to the Al-modification and to the
Fig. 6 Water-quenching test comparison between the as-sprayed and Al-modified TBCs. (a) Thermal cycle comparison between APS, EB-PVD, and PS-PVD TBCs. (b) Five samples including one as-sprayed sample (left) and four Al-modified samples (right, 5, 10, 20, and 30 μm thick Al film modifications). (c–i) As-sprayed and Al-modified 7YSZ TBCs after 80, 100, 115, 131, 162, 198, 350 cycles.

Fig. 7 Surface evolution of the 20 μm Al-modified TBCs before and after thermal quenching. (a) Surface microstructures before water quenching, showing the nanowires on cauliflower tops. (b) Cross-sectional microstructure before water quenching showing different densities from top to bottom. (c) Surface microstructure after 350 cycles indicating the spallation of the cauliflower tops. (d) Cross-sectional microstructure after 350 cycles showing vertical crack and grain size grown. (e) Phase compositions of the as-sprayed TBCs (M-ZrO₂, T'-ZrO₂, C-ZrO₂), the Al-modified TBCs (new α-Al₂O₃ phase generation) and the Al-modified TBCs (α-Al₂O₃, M-ZrO₂, T'-ZrO₂, C-ZrO₂) after 350 cycles. (f) Corresponding surface grain sizes (21, 35, and 247 nm) of the three types of TBCs. (g) Corresponding stresses (0.4, 0.2, and 0.41 GPa) of the three types of TBCs.
water-quenching state is shown in Fig. 7(e). The XRD patterns indicated that there were three phases M-ZrO2, T’-ZrO2, and C-ZrO2 in the as-sprayed 7YSZ TBCs. After the 20 µm Al-modification, a new phase α-Al2O3 was observed due to the in-situ synthesis between the Al and ZrO2 in the heated vacuum treatment [29,49]. Besides, after 350 water-quenching cycles, the peaks of the M-ZrO2, T’-ZrO2, and C-ZrO2 phases have been analyzed. Based on the analysis of the T’-ZrO2 patterns, the grain sizes of the as-sprayed TBCs, the 20 µm Al-modified TBCs, and the 20 µm Al-modified TBCs after 350 thermal cycles were 21, 35, and 247 nm respectively, seen in Fig. 7(f). However, the α-Al2O3 peaks increased, which means that the degree order of the grain structure has increased. Additionally, based on the analysis of the T’-ZrO2 patterns, the lattice stress of the as-sprayed TBCs, the 20 µm Al-modified TBCs, and the 20 µm Al-modified TBCs after 350 thermal cycles were 0.4, 0.2 and 0.41 GPa, respectively (Fig. 7(g)). This means that the Al-modification process did not add lattice stress to the PS-PVD 7YSZ TBCs.

4 Conclusions

Considering the obstacles of PS-PVD 7YSZ TBCs for aero-engine application, this study sheds light on a new Al-modification solution for particle erosion and thermal cycle. It was found that the Al-modification not only improved the particle erosion resistance but also increased the thermal cycle performance, where the optimal Al film thickness is 20 µm. Moreover, the aspiration is that the PS-PVD 7YSZ TBCs can be rapidly implemented to protect the high-temperature components in an aero-engine. Thus, the hope is that the current study may inspire further attempts to clear the application barriers. The Al-modification is a normal approach. And this technique can be extended to others structural coating such as environmental barrier coating, densified coating, etc.

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