**Dependence of scale thickness on the breaking behavior of the initial oxide on
plasma spray bond coat surface during vacuum pre-treatment**

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Abstract

The thermally grown oxide (TGO) on the thermal spray bond coat surface was one of the most important factors which would influence the lifetime of thermal barrier coatings (TBCs). Pre-diffusion treatment (high temperature vacuum treatment) plays an important role in the growth of the TGO. Results show that the initial thin oxide scale, formed during deposition process, on the as-sprayed bond coating surface has broken and shrunk to discontinuous oxide particles through the elements diffusion during the pre-diffusion. Two kinds of bond coats with different initial oxide scale thicknesses were subjected to the same pre-diffusion. The two pre-diffused bond coats show different results of the average distance between the individual oxide particles. In this study, a three dimensional model with thermal grooving theory was developed to explore the essential condition for the scale breaking and explain the differences of these results. This research can provide reference for the preparation optimization and pre-treatment optimization of bond coat towards high performance TBCs.

Keywords: vacuum pre-treatment, bond coat, thermally grown oxide, oxide scale breaking, 3D model

1. Introduction

In recent years, due to the demand for more efficient gas turbines, gas turbine inlet temperatures have increased [1-5]. Thermal barrier coatings (TBCs) have been developed in order to protect the hot section parts (superalloy) in gas turbines against high temperature oxidation and hot corrosion in the aggressive environment [2, 6-9]. Thermal barrier coatings generally consist of an Y_2O_3 stabilized ZrO_2 (YSZ) top coating (TC), a MCrAlY bond coat (BC) on the surfaces of components made of superalloys, and a layer of thermally grown oxide (TGO) formed at the interface between the bond coat and YSZ top coating during high temperature exposure [6, 10-21]. The ceramic top coating is designed to provide thermal insulation, while the bond coat prevents the superalloy substrate from high temperature oxidation by forming the protective oxide scale (TGO). Therefore, TBCs actually is the multi-layers series system composed of the top coating, TGO, and bond coat. The degeneration of any layer will result in the failure of the service properties of the whole TBCs.

It has been reported that TGO was one of the most important factors which would influence the lifetime of TBCs [6, 10, 11, 15, 22]. The TGO growth is the main source of strain which will drive the delamination/spallation of the YSZ top coating [17, 23-25]. With the TGO thickness increasing during high temperature exposure or thermal cyclic condition, the complex strain formed in the TGO will produce much stress in near TGO region, especially in the top coating near the TGO [22, 26, 27]. The residual stress increases as the TGO grows during thermal exposure, which will accelerate the spallation of the YSZ top coating [4, 6, 15, 27-30]. When the TGO thickness reaches a critical thickness, failure of TBCs will occur quickly [6, 23, 27, 31]. It has been reported that the failure of TBCs upon thermal cycling generally initiates and propagates within the TGO, along the interface between TGO and bond coat or near the TC/TGO interface [6, 10, 11]. Therefore, reducing TGO

growth rate is of great importance for prolonging TBCs lifetime. A stable, continuous, slow-growing, and highly adherent TGO at the interface between the YSZ top coating and bond coat is optimal, which can better protect the substrate against high temperature oxidation [6, 15, 32]. TGO mainly consists of α -Al₂O₃ and mixed oxides (Cr₂O₃, NiO, and (Ni,Co)(Cr,Al)₂O₄ spinel) [6, 33]. The short supply of Al in bond coat for oxidation is one of the main reasons for the formation of the mixed oxides [33-36]. It was reported that the mixed oxides fast growth, accompanied with large volume expansion, will result in the failure of TBCs by causing the cracks in the top coating [37-43]. It has been found that the thermal cyclic lifetime of TBCs is inversely proportional to the coverage ratio of the mixed oxides, and the high coverage of the mixed oxide leads to the early failure of TBCs [41]. Therefore, suppressing the generation and reducing the content of the mixed oxides can significantly improve the lifetime of TBCs. In this regard, a TGO layer made up of pure α -Al₂O₃ on the bond coat surface is preferred due to its low diffusion coefficient of both oxygen ions and metal ions, high chemical stability and thermal stability during thermal exposure [15, 44-50]. From the above it can be seen that lowering the TGO growth rate and reducing the mixed oxides content play an important role in prolonging the TBCs lifetime.

The pre-oxidation has been generally regarded as the initial stage of TGO generation in all the relevant papers. It has been reported that an appropriate pre-treatment of the bond coat could effectively lower TGO growth rate and reduce the mixed oxides content [51-57]. Therefore, much attention was paid to the study of pre-treatment for controlling TGO growth behavior. It has been reported that the proper pre-oxidation of the bond coat under a low oxygen partial pressure before the deposition of the YSZ top coating is an effective way to suppress the TGO growth and reduce mixed oxides content due to the formation of a thin and continuous α -Al₂O₃ TGO layer at the interface

between the top coating and bond coat in the protective atmosphere [52-56]. Furthermore, it was reported that the pre-annealing under a very low oxygen partial pressure (pre-diffusion, high temperature vacuum treatment) before a pre-oxidation in a relatively higher oxygen partial pressure can benefit the lifetime of TBCs compared to samples subjected directly to the pre-oxidation treatment [52, 57]. However, what happened to the TGO structure during pre-diffusion and the mechanism of the influence of pre-diffusion on TGO growth behavior have not been reported. It should be noted that there is a very thin oxide scale (TGO), formed during the deposition process due to the spraying particles' high temperature and the certain oxygen partial pressure of the spraying atmosphere. TGO has actually formed on the as-sprayed bond coat surface during the deposition process of the bond coat, which has not been noticed in the other papers. Therefore, the pre-treatment process of the thermal spray bond coat is not the process that new oxide scale forms on the surface of the fresh metallic bond coat, but the process that the pre-existing oxide scale evolves. However, this important matter has been ignored during the preterit researches of the oxidation behavior of the thermal spray bond coat. Therefore, the study on the evolution of the initial oxide scale structure during the pre-diffusion process is the important theoretical foundation for understanding and evaluating the lifetime service of TBCs.

In this research, the bond coats, with different initial oxide scale thicknesses, deposited with the plasma spray equipment were subjected to the same pre-diffusion. The particular microstructure of the TGO formed on the pre-diffused bond coat surface was examined, and the formation mechanism of the TGO was explained by a 3D model with thermal grooving theory according to thermodynamics.

2. Experimental procedure

The 150-200 μm thick bond coats were deposited by plasma spray using CoNiCrAlY powder in a

spherical shape (Amdry 9951, Sulzer-Metco, USA). The spray powder had a mean particle size of $\sim 25.8 \mu\text{m}$ ($d_{10}=12.9 \mu\text{m}$, $d_{90}=38.6 \mu\text{m}$). Nickel-based superalloy Inconel 738 ($\phi 25.4 \text{ mm} \times 3 \text{ mm}$) was used as a substrate.

The bond coat was divided into two groups with different initial oxide scale thicknesses, i.e. 17 nm and 60 nm. The bond coat with 17 nm initial scale was prepared by low pressure plasma spray (LPPS) with the oxygen partial pressure of 1.7 Pa in chamber. The bond coat with 60 nm initial scale was prepared by protective atmosphere plasma spray with the oxygen partial pressure of 3000 Pa, which is much lower than oxygen partial pressure of 20000 Pa for conventional atmosphere plasma spray. The detailed spray torch parameters are shown in Table 1.

Table 1

Spraying parameters

Parameters	Value
Arc current (A)	620
Arc voltage (V)	72
Plasma gas (Ar) flow ($\text{L} \cdot \text{min}^{-1}$)	40
Plasma gas (H_2) flow ($\text{L} \cdot \text{min}^{-1}$)	8
Powder feeding gas (Ar) flow ($\text{L} \cdot \text{min}^{-1}$)	2
Spray distance (mm)	160

All the as-sprayed bond coats were subjected to the pre-diffusion treatment (1080°C for 3 h, $p\text{O}_2 < 0.002 \text{ Pa}$) in a vacuum heat treatment furnace. The pre-diffusion can make elements have a

comparatively homogeneous distribution in the bond coat, and make the splats have a better bonding between each other [58].

The oxygen contents of the spray powder and the coatings were determined with oxygen determinator (Ro-316, LECO, USA). Field emission scanning electron microscope (SEM, MIRA 3 LMH, TESCAN, Czech Republic) equipped with the energy dispersive spectrometer (EDS, Aztec EDS + EBSD, Oxford Instruments, United Kingdom) was employed to characterize the microstructure of the bond coat. A layer of Ni on the bond coat surface was deposited by the Ni-electroless plating to protect the TGO scale from spalling off during the cutting and polishing of the samples.

3. Results and discussion

3.1. Microstructure of the as-sprayed bond coat

Fig. 1 shows the as-sprayed bond-coating microstructures deposited at the oxygen partial pressures of about 1.7 Pa and 3000 Pa, respectively. From these pictures, it can be seen that the two coatings have basically similar rough surface morphologies due to the random stacking of the splats [59]. On the coating surface, there are many convexes (dozens of micrometers) which are comparable to the feedstock powder. It has been reported that the convexes can be regarded as resulting from the partially melted particles [60-62]. The convexes contribute to the surface roughness of the as-sprayed bond coat according to Fig. 1. Fig. 1 reveals that the bond coatings have the typical layer structures. From the cross structures, it can be seen that some splats (layers) incompletely bond with the ones next to them, and the fine gray lines in the coatings are the incompletely unbound interfaces. It is worth to note that there is a very thin oxide scale, formed during the deposition process due to the spraying particles' high temperature and the certain oxygen partial pressure in the chamber. According to the plasma spray

characteristics, the splat was derived from the deposition of the melted particle. The melted particles, sprayed onto the deposited surface, solidified rapidly (about $10^6 \sim 10^8$ °C/s), and the melted particles needed about no more than twenty or thirty microseconds for solidification [63-65]. The time (about 0.1 s) between two spraying melted particles impacted onto the deposited surface is much larger than the solidification time of the melted particles [66]. Therefore, the splat had sufficient time for cooling, and it can be considered that the deposition behavior of every spraying particle is independent. There exists thin oxide scale with uniform thickness on the surface of every splat. The thin oxide scale thickness on the surface of the two as-sprayed bond coats can be obtained through the calculation with the oxygen contents of the two coatings. The oxygen content of the bond coat deposited at the lower oxygen partial pressure is 0.86 ± 0.30 wt.%, while the value of the other bond coat is 3.1 ± 1.3 wt.%. Therefore, the calculation results is that the thickness of the initial thin oxide scale is about 17 nm for the bond coat deposited at the oxygen partial pressure of 1.7 Pa, while the value of the other bond coat is about 60 nm.

Fig. 1. Microstructures of as-sprayed bond coats deposited at the oxygen partial pressure of about 1.7 Pa (a) and 3000 Pa (b), respectively. The insets in (a) and (b) are surface morphologies of the corresponding bond coat.

3.2. Microstructure of pre-diffused bond coat deriving from the as-sprayed bond coat with 17 nm initial oxide scale

Fig. 2 shows the surface morphology of the pre-diffused bond coat deriving from the as-sprayed bond coat with the initial oxide scale thickness of 17 nm on the coating surface. The pre-diffused bond coat surface presents two discontinuous phases with different contrasts. Fig. 3 shows the EDS element energy spectrum mappings of the pre-diffused bond coating surface. Results reveal that isolated oxide particles exist on the pre-diffused bond coat surface. Further, the EDS results (Fig. 4) of the points marked in Fig. 3 show that the dark zone (oxide) on the pre-diffused bond coat surface is Al_2O_3 , and the light zone, which has emerged in many places on the surface, is the bared metallic bond coating. Therefore, it can be known that there exists discontinuous Al_2O_3 scale on the pre-diffused bond coat deriving from the as-sprayed bond coat with 17 nm initial oxide scale on the coating surface. In order to further reveal the oxide structure on the pre-diffused bond coat surface, the cross sectional

microstructure (Fig. 5) of the pre-diffused bond coat was observed carefully. From Fig. 5, it can be known that the oxide scale on the pre-diffused bond coat surface sure is discontinuous and the bared metallic bond coating has emerged. The above results indicate that the initial thin oxide scale on the bond coating surface has broken and shrunk to the isolated oxide particles (namely, the discontinuous oxide mentioned above), covering the bond coating surface, and the bared metallic bond coating surface has emerged in some places on the pre-diffused coating surface. This is due to the surface tension of the oxide scale and the elements diffusion during the pre-diffusion. Meanwhile, it can be found that the incompletely unbound interfaces (the gray lines) in the as-sprayed bond coat with the 17 nm initial oxide scale (Fig. 1a) have been broken into some small sections in the pre-diffused ones (Fig. 5) during the pre-diffusion due to elements diffusion, which made the splats have a better bonding between each other.

Fig. 2. Surface morphology of the pre-diffused bond coat deriving from the as-sprayed bond coat with the initial oxide scale thickness of 17 nm on the coating surface.

Fig. 3. The EDS element mappings of the surface on the pre-diffused bond coat deriving from the as-sprayed bond coat with the initial oxide scale thickness of 17 nm on the coating surface.

Fig. 4. EDS results of the points marked in Fig. 3. (a) Point 1, (b) point 2.

Fig. 5. Cross sectional microstructure of the pre-diffused bond coat deriving from the as-sprayed bond coat with the initial oxide scale thickness of 17 nm on the coating surface.

3.3. Microstructure of pre-diffused bond coat deriving from the as-sprayed bond coat with 60 nm initial oxide scale

Fig. 6 shows the surface morphology of the pre-diffused bond coat deriving from the as-sprayed bond coat with the initial oxide scale thickness of 60 nm on the coating surface. From Figs. 2 and 6, it can be seen that the surface morphologies of the two coatings with pre-diffusion are generally similar. There are also two discontinuous phases with different contrasts on the surface of the pre-diffused

coating in Fig. 6b. The EDS results (Fig. 7) of the points marked in Fig. 6b and the cross structure (Fig. 8) of the pre-diffused bond coat clearly reveal that the oxide scale on the pre-diffused bond coat surface is discontinuous Al_2O_3 . Therefore, the initial oxide scale with a thickness of 60 nm, on the surface of these pre-diffused bond coating (Figs. 6 and 8), also has broken and shrunk to the isolated oxide particles, covering the bond coating surface. However, it can be intuitively found that the coverage of the discontinuous oxide scale on the pre-diffused coating surface in Fig. 6 is bigger than on the coating surface in Fig. 2. The cross structures (Figs. 5 and 8) of the two bond coatings can more directly reflect the oxide scale differences between the two pre-diffused bond coatings. It can be seen that the average distance between each isolated oxide particles on the pre-diffused bond coat shown in Fig. 5 ($0.61 \pm 0.33 \mu\text{m}$) is much bigger than in Fig. 8 ($0.17 \pm 0.12 \mu\text{m}$), which must be resulted from the difference between the initial oxide scale thicknesses of the two coatings. It can be found that the gray lines in the as-sprayed bond coat with the 60 nm initial oxide scale (Fig. 1b) also have been broken into some small sections in the pre-diffused ones (Fig. 8) during the pre-diffusion.

Fig. 6. Surface morphology of the pre-diffused bond coat deriving from the as-sprayed bond coat with the initial oxide scale thickness of 60 nm on the coating surface.

Fig. 7. EDS results of the points marked in Fig. 6b. (a) Point 3, (b) point 4.

Fig. 8. Cross sectional microstructure of the pre-diffused bond coat deriving from the as-sprayed bond coat with the initial oxide scale thickness of 60 nm on the coating surface.

3.4. Thermal grooving and breaking behavior of oxide scale

As discussed in the above text, the very thin oxide scale on the as-sprayed bond coat surface has broken and shrunk to isolated oxide particles during the pre-diffusion. To further understand the mechanism of the differences of the oxide structures between the two pre-diffused bond coatings, especially the influence of the initial oxide scale thickness on the average distance between the each isolated oxide particle, the breaking of the initial thin oxide scale on the two as-sprayed bond coating surfaces should be deep discussed. It is obvious that the oxide scale breaking is more difficult with the oxide scale thickness increasing. To better understand the influence of the oxide scale thickness on the

oxide scale breaking, a three-dimensional model (Fig. 9) was proposed to obtain the thickness limit for oxide scale breaking. Thermal grooving of grain boundaries of the oxide scale is the main reason for the breaking of oxide scale. The computational process was made with the thermodynamics. To simplify the model, the oxide scale was assumed to be isotropic, which permits the free oxide surface to be flat or spherical morphology. It is assumed that the oxide scale thickness is large enough, and the oxide grains are regular hexagonal prisms (Fig. 9). It is obvious that the three grains' junction will form the biggest depth (H) after the grain boundary thermal grooving with enough time according to the thermodynamics (Fig. 9). Therefore, the influence of thermal grooving growth of grain boundary at the three oxide grains' junction on the oxide scale breaking evolution was studied in this paper.

One oxide grain on the bond coat surface was selected for study object. Its original structure state before thermal grooving and equilibrium structure state after enough long time elements diffusion during high temperature vacuum heat treatment are shown in Fig. 11b. According to thermodynamics, the upper part of the regular hexagonal prism oxide grain will eventually evolve into a part of a hemisphere after thermal grooving and enough long time elements diffusion during high temperature vacuum treatment (Fig. 9). After the grain boundary thermal grooving with enough long time, the biggest depth (H) at the three grains' junction can be formed. Based on the mass conversation rule and geometrical relationship, H and the side length (b) of the regular hexagon have the relationship expressed by the following equation,

$$6 \times \frac{1}{2} \times b \times b \sin 60^\circ \times H = \frac{1}{2} \times \frac{4}{3} \times \pi b^3 - 6 \times \pi \left(b - b \sin 60^\circ \right)^2 \left(b - \frac{b - b \sin 60^\circ}{3} \right) \quad (1)$$

From the above equation, the H can be expressed by the following equation,

$$H = \frac{27\sqrt{3} - 40}{18\sqrt{3}} \pi b \approx 0.68b \quad (2)$$

Based on Eq. 2 and geometrical relationship shown in Fig. 11b, the biggest depth (h) at the boundary

of two grains can be obtained as the following equation,

$$h = H - \frac{b}{2} = 0.18b \quad (3)$$

The Eqs. 2 and 3 can reveal the relationship between the oxide grain size in in-plane direction and the depth of the thermal groovings of oxide scale grain boundary. The two equations show that the oxide scale breaking is more likely to happen at the three grains' junction. It is indicated that the grain size in in-plane direction and the oxide scale thickness play an important role in the oxide scale breaking process. Figs. 9b and 9c show the schematic diagrams of the grain structure evolution of the thinner oxide scale and the thicker one, respectively. The two bond coats were subjected to the same pre-diffusion. The diffusion state shown in Fig. 9 is the oxide grain structure state when the pre-diffusion proceeded at the certain moment. It can be found that the oxide scale breaking can happen when the thickness of the oxide scale is sufficiently small compared to the oxide in-plane size. The results reveal that the thinner oxide scale (Fig. 9b) on the pre-diffused coating surface broke earlier than the thicker one (Fig. 9c) during the same pre-diffusion, which would lead to the oxide particles resulted from the former oxide scale have longer time for shrink. Therefore, the average distance between each oxide particles resulted from the thinner oxide scale would be bigger than the from the thicker oxide scale. The results lead to a further understanding of the TGO growth mechanism, which can provide reference for the preparation optimization and pre-treatment optimization of bond coat towards high performance TBCs.

Fig. 9. 3D model of the oxide scale breaking, charged by thermal grooving, on the bond coating surface.

(a) Schematic diagram of the oxide scale breaking, (b) schematic diagram of the grain structure evolution of the thinner oxide scale, (c) schematic diagram of the grain structure evolution of the thicker oxide scale.

4. Conclusions

The initial thin oxide scale on the as-sprayed bond coat has broken and shrunk to discontinuous oxide particles during the pre-diffusion treatment. The average distance between each other of the oxide particles resulted from the thinner oxide scale on the pre-diffused surface is bigger than from the thicker oxide scale. In order to further understand the oxide scale breaking during the pre-diffusion, a 3D model was developed to explore the essential condition for the scale breaking. Results show that the grain size in in-plane direction and the oxide scale thickness play an important role in the oxide scale breaking process. The bigger the grain size in in-plane direction and the thinner the oxide scale, the easier the oxide scale breaks. The results reveal that the thinner oxide scale broke earlier than the

thicker one during the pre-diffusion treatment, which would lead to the oxide particles resulted from the former oxide scale have longer time for shrink. Therefore, the average distance between each oxide particles resulted from the thinner oxide scale would be bigger than the from the thicker oxide scale. The results lead to a further understanding of the TGO growth mechanism, which can provide reference for the preparation optimization and pre-treatment optimization of bond coat towards high performance TBCs.

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