



Thermal Stability of PS-PVD YSZ Coatings with Typical Dense Layered and Columnar Structures

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Abstract: Yttria-stabilized zirconia (YSZ) coatings with typical pyramid columnar and dense layered structure were prepared by plasma spray-physical vapor deposition (PS-PVD). The evolution behavior of microstructure and crystallography of the coatings before and after thermal aging treatment were observed by electron backscatter diffraction (EBSD) and field emission scanning electron microscopy (FE-SEM). Results showed that the as-deposited coatings exhibited many types of structures and were mainly composed of a nonequilibrium tetragonal (t'-ZrO₂) phase. With the prolonging of thermal exposure time, the initial nonequilibrium tetragonal phase of YSZ coatings gradually transformed into a monoclinic (m-ZrO₂) phase. During the process of stationary deposition, at a proper spraying distance, each column exhibited a certain preferred orientation, but the ceramic topcoat did not exhibit distinct preferred orientation statistically.

Keywords: plasma spray-physical vapor deposition; electron backscatter diffraction; thermal barrier coatings

1. Introduction

The deposition of thermal barrier coatings (TBCs) on the surface of hot-section end components of engines can improve the inlet temperature of gas, thus improving the efficiency and extending the lifetime of heat engines [1–3]. At the moment, the two main typical technologies used for thermal barrier coatings deposition are air plasma spraying (APS) and electron beam-physical vapor deposition (EB-PVD) [4–6]. Plasma spray-physical vapor deposition (PS-PVD) process is a novel technique that combines high deposition rates and cost-efficiency of APS and columnar structure of EB-PVD coatings, so that PS-PVD has received widespread attention in the manufacture of some advanced turbine TBCs [7–10].

Due to the use of low working pressure and high power plasma spray gun, the characteristics of PS-PVD plasma plume have changed greatly. The length of plasma plume is more than 2 m and the diameter is 200–400 mm. The highest temperature of plasma plume is on the axis of plasma flame. Although some powder particles are not injected into the center of the flame, the temperature of plasma flame is much higher than melting point of ZrO_2 , so the powder can be vaporized well. This is not only conducive to the full heating of the spray particles, but also can flow through the shadow area of the twin vanes like components with complex shape along with the plasma jet, so as to realize the rapid, uniform and non-line of sight deposition of the coating on the surface of the



components with complex geometry [11–13]. The agglomerated and loose powders are dispersed immediately after entering the plasma flame, heated and vaporized to form a coating along with the flame flow [14]. The higher the plasma flame temperature, the more significant influence on the melting and gasification of the powder. The energy absorbed by the powder and the shape of the coating are largely affected by the composition of the plasma gas and the characteristics of the powder [15]. The powder has different states at different positions of the plasma flame, forming different deposition units, including melt droplets, clusters and vapor. The microstructure of the coating is closely related to the deposition unit, and controllable deposition of different deposition units can be achieved through

adjustment [16,17]. By adjusting the spraying distance, dense layered, columnar and mixed structure coatings can be prepared by PS-PVD [14,18–20]. The thermal cycle life of thermal barrier coatings with columnar structure prepared by PS-PVD is more than twice that of conventional APS coatings [21–23]. In addition, the characteristics of high deposition rate and non-line of sight deposition makes PS-PVD the most promising alternative process for columnar TBCs.

Due to the high temperature environment of service behavior, thermal stability is the key index to evaluate the performance of thermal barrier coatings. In order to study the high temperature stability of thermal barrier coatings, it is necessary to further study the deposition and grain growth mechanism of the PS-PVD coating, and to summarize the systematic changes in the coating deposition characteristics, microstructure and performance. The columnar structure coating of PS-PVD for engineering application is deposited by moving orientation, and the columnar structure is mixed structure with imperfect columnar crystals [24]. It is incomplete to explore the evolution law of this kind of columnar structure coating under high temperature service. Therefore, two kinds of typical extreme structure coatings were prepared by PS-PVD technology, including dense layered and pyramid columnar coatings. By studying the evolution law of microstructure and crystal texture of two typical coatings at high temperature, the experimental basis is provided for exploring the relationship between deposition characteristics and high temperature stability of thermal barrier coatings, and finally providing experimental basis for engineering application of PS-PVD TBCs.

2. Experimental Procedures

2.1. Preparation and Thermal Aging Treatment of PS-PVD Coatings

YSZ coatings were deposited on graphite substrates by PS-PVD process. The spraying material is ZrO_2 -(7.5 wt.%) Y₂O₃ agglomerated powder (Metco 6700, Oerlikon Metco, Wohlen, Aargau, Switzerland) with the particle size of 5–20 µm. PS-PVD spraying equipment (Oerlikon Metco, Wohlen, Aargau, Switzerland) was adopted system equipped with O3CP spray gun, 60CD powder feeder, ABB manipulator and vacuum components produced by Oerlikon Metco. Two groups of different spraying parameters were selected for comparison under the power of 120 kW (test A) and 130 kW (test B), and the specific parameters are displayed in Table 1.

Items	Test A	Test B
Plasma gas flow rate (slpm)	Ar: 35, He: 60	Ar: 35, He: 60
Power (kW)	120	130
Current (A)	2600	2700
Feed rate (g/min)	5	5
Spray distance (mm)	500	700
Oxygen flow rate (splm)	2	2

Table 1. Spray parameters for YSZ TBCs prepared by PS-PVD.

The powder was put into the powder feeder and the temperature was maintained at 60 $^{\circ}$ C, so as to prevent the moisture absorption of the powder and affecting the flowability of powder. When spraying on the surface of graphite substrate, the spray gun shall be aligned with the center of the test piece to maintain a certain spraying distance, and the gun and the sample table shall be relatively

stationary for 100–200 s. The free-standing coatings can be peeled off from the surface of graphite substrate after spraying.

The dense layered coating and pyramid columnar coating were selected to study the high temperature thermal stability of PS-PVD coatings. The dense layered coating in test A and pyramid columnar coating in test B were named YSZ-1 and YSZ-2. A high-temperature box furnace (HTK 20/17, ThermConcept, Bremen, Germany) was used to isothermally heat treat on free-standing YSZ coatings at 1550 °C for 20–100 h, respectively. Free-standing YSZ TBCs samples with two typical microstructures were put in the high-temperature box furnace and heated to the target temperature at a rate of about 10 °C /min with holding for the selected time. The samples were then cooled at a rate of about 15 °C/min to the room temperature in the furnace.

2.2. Characterization of the Coatings

In order to observe the cross-section microstructure of the substrate-free coating, the polished cross-section samples of the two coatings were prepared by the following process: the samples were cut at a feed rate of 1.2 mm/min after hot setting by conductive resin, then polished by multi-stage diamond grinding, ultrasonic cleaning and dried by compressed air. The carbon film was evaporated and used for electron microscopy analysis. The surface and fracture section of the coating were observed by field emission scanning electron microscopy (FE-SEM, Carl Zeiss, Jena, Thuringia, Germany).

The X-ray diffractometer (D/max 2550 V, Rigaku, Akishima-shi, Tokyo, Japan) was used to detect the phase composition of the as-sprayed and isothermally heated coating samples. The XRD operated in the reflection mode with Cu-K α radiation (40 kV, 100 mA) and step scan mode with the step rate of 0.02° (2 θ)/s. The crystal texture of the coatings was analyzed by EBSD (Nordlysmax2, Oxford, Abindon, Oxfordshire, UK). Under the action of 20 kV accelerating voltage and 30 nA probe current, the step size is 20–100 nm.

The samples used for EBSD observation samples need high quality polished section. The mechanically polished metallic substrate section samples were polished by argon ion polishing instrument (EM tic 3x, Leica, Weztlar, Hesse-Darmstadt, Germany). The polished samples were deposited with a vacuum coater (EM ace200, Leica, Weztlar, Hesse-Darmstadt, Germany) to deposit a 5 nm carbon film for EBSD observation.

3. Results and Discussion

3.1. Microstructure

Figure 1 shows the microstructure of two typical types of as-sprayed PS-PVD TBCs with the fracture section and surface. Figure 1a,c shows that the dense layered coating was mainly formed by spreading and accumulating completely melted droplets. The YSZ material reaching the surface of the substrate mainly existed in the form of a liquid phase, and contained a small amount of vapored-phase clusters and solid particles. When the droplets hit the surface of the substrate at high speed, they will spread out, form a thin disk and solidify to form a dense coating. At the same time, a small amount of solid particles was deposited on the surface of the substrate, resulting in holes and defects in the coating. A small amount of vapor clusters will condense on the surface of the layered structure or fill the gap between the splats, which is conducive to improving the density of the coating [8].

As shown in Figure 1b,d, the surface morphology of coating YSZ-2 exhibited pyramid structure. The coating consisted of columnar structure with different sizes similar to that of EB-PVD coatings, and there were gaps between the columns through the thickness of the entire ceramic layer. At the initial stage of coating deposition, a dense layer formed by the accumulation of fine grains formed by heterogeneous nucleation. With the increase of thickness, the columnar structure grew vertically to the substrate. Each column was not monocrystalline structure, but a polycrystal containing a variety of smaller grains [10]. The gap between columns came from the condensation of gas phase and the macro shadow caused by the uneven columns, which made the columns growing inside the coating compete

with each other, some of the small columns stop growing, and eventually formed a structure in which large and small columns were stacked alternately. The working pressure of PS-PVD was lower, and the plasma flame formed was longer than that under normal pressure. The formation mechanism of the coating was relatively complex, which was mainly due to the island growth mode in the vapor deposition process, and finally forming the pyramid agglomerated columnar structure coating [25]. The thermal conductivity of the columnar YSZ coating produced by the PS-PVD at 1200 °C was about 1.155 W/mK, which is lower than the thermal conductivity (~2.2 W/mK, 1200 °C) of the coating with dense layered structure [26]. These differences may be mainly related to the morphology and porosity of the two coatings studied. Obviously, the columnar coating exhibited the highest porosity and defects, which will enhance the phonon scattering and reduce the average free path of phonons, resulting in low thermal conductivity. It is generally believed that the oxidation resistance of dense layered coating was better than that of columnar structure coating, which is mainly due to the fact that the columnar structure coating had many pores and the layered coating was denser. The pre-oxidized TGO layer had a denser structure, and aluminum oxide can effectively isolate the diffusion of oxygen to the bond coat [27,28]. Therefore, the YSZ coating of PS-PVD TBC improved the oxidation resistance of the coating by pre-oxidizing the bond coat. The columnar structure improved the strain tolerance of the coating and can be opened and closed during the cool and heat cycles, releasing the thermal mismatch stress between the substrate/bond-coat and the ceramic layer. In addition, the columnar gap can effectively inhibit the crack growth and improve service lifetime and reliability of coating [29–31].



Figure 1. The fracture section (**a**,**b**) and surface (**c**,**d**) of the as-sprayed TBCs prepared by PS-PVD: (**a**,**c**) YSZ-1; (**b**,**d**) YSZ-2.

Figure 2a–f shows the fracture section morphology of the dense coating before and after high temperature aging treatment. It can be observed that the grain size of the as-sprayed dense layered coating was very small, and grew obviously with the extension of heat treatment duration. The fracture section of the coating YSZ-2 before and after high temperature aging treatment is shown in Figure 3. Figure 3a shows the local enlarged morphology of the as-sprayed coating. It can be seen that the surface of columns presented feathery structure, and the columnar surface was deposited with micro-nano scale small grains. There was a large number of interfaces between the grains, which hinder the heat transfer of "phonon". Figure 3b–f show the natural fracture section morphology after 20–100 h at 1550 °C, respectively. As can be seen from the figure that compared with the as-sprayed coating,

the columnar structure became smooth after high temperature aging, the feather like structure of columnar coating disappeared, and some micro-nano grains of feather like structure also disappeared due to high temperature sintering and melting. From the surface of columns, with the increase of heat-treatment time, the number of pores in the coating decreased significantly, the grains gradually changed into equiaxed crystalline grains and the average grain size increased. When the average size increased, some grains would shrink or disappear. The driving force is the energy difference between the products of fine and large grains, which is caused by the decrease of the area of grain boundary and the total interface energy. The interface energy is related to the interface between grains. The driving force and growth rate of grain are inversely proportional to the grain size. Large grains tend to grow and fine grains tend to disappear.



Figure 2. SEM images of fracture section of YSZ-1 coatings before and after thermal aging treatment in air at 1550 °C as a function of time: (**a**) as-sprayed; (**b**) 20 h; (**c**) 40 h; (**d**) 60 h; (**e**) 80 h; (**f**) 100 h.



Figure 3. SEM images of fracture section of YSZ-2 coatings before and after thermal aging treatment in air at 1550 °C as a function of time: (**a**) as-sprayed; (**b**) 20 h; (**c**) 40 h; (**d**) 60 h; (**e**) 80 h; (**f**) 100 h.

3.2. Phase Transformation

Figure 4 shows the XRD patterns of PS-PVD YSZ coatings. The results show that the XRD spectrum of the as-sprayed coating YSZ-2 was consistent with the characteristic peak of t'-ZrO₂ of standard PDF card, so the as-sprayed coating consisted of non-equilibrium t'-ZrO₂ phase. This is due to that in the process of coating deposition, the vaporized YSZ atoms collided with the surface of graphite to form a rapid heterogeneous nucleation deposition, the cooling rate was too fast, and the high temperature tetragonal phase was too late to have a diffusion phase transformation and remain, forming t'-ZrO₂ structure. The results indicate that the main phase of the as-deposited coating YSZ-1 was t'-ZrO₂, a small amount of m-ZrO₂ also existed in the as-deposited YSZ coating. This is because there were some unevaporated or unmelted raw material powder particles directly incorporating and solidifying in the coating. Phase of the coatings was also analyzed by EBSD and the results are depicted in Table 2 and Figures 5–8. The major phases found in the coating was dominated by tetragonal phase, including tetragonal (82.3%) and monoclinic. The coating YSZ-2 contained 97.9% of tetragonal phase. After heat treatment at 1550 °C for 100 h, the tetragonal phase of the coating YSZ-1 and YSZ-2 decreased from 82.3% to 74.8% and 97.9% to 78.5%, respectively.



Figure 4. XRD patterns of the as-sprayed coatings YSZ-1 and YSZ-2.



Figure 5. EBSD images of TBC YSZ-1: (**a**) quality maps; (**b**) orientation maps; (**c**) phase maps; The orientations are defined by tetragonal lattice symmetry.

Tetragonal Phase
82.3%
97.9%
74.8%
78.5%

Table 2. Phase content of YSZ coatings prepared by PS-PVD.



Figure 6. EBSD images of TBC YSZ-2: (**a**) quality maps; (**b**) orientation maps; (**c**) phase maps; The orientations are defined by tetragonal lattice symmetry.



Figure 7. EBSD images of TBC YSZ-1 after heat treatment at 1550 °C/100 h: (**a**) quality maps; (**b**) orientation maps; (**c**) phase maps; The orientations are defined by tetragonal lattice symmetry.

The t'-ZrO₂ phase of the coatings is stable at room temperature. Only at high temperature, the phase is first transformed into c-ZrO₂ phase with high Y_2O_3 content and tetragonal with low Y_2O_3 content. At high temperature, the phase transformation from tetragonal to monoclinic phase occurs with the decrease of temperature, so as the coating cracks and falls off with the huge expansion of lattice. The YSZ coating of t' phase has a higher thermal service lifetime. The content of monoclinic in

the coating is less than 10 mol.%, which is a good way for prolonging the life of the coating. It can improve the fracture toughness of the coating, because when a small amount of tetragonal crystal transforms into monoclinic crystal, the lattice volume will expand and produce the compress stress in the coating during cooling. At the same time, micro-cracks network are produced in the coating due to phase transformation, which improve the thermal shock resistance of the coating. If the monoclinic content in the coating is too high, the phase transformation will occur repeatedly during the use of the coating, accompanied by 3–5% volume expansion, which will lead to the failure of the coating [32–34]. Phase stability of pyramid columnar coating is better than the dense layered coating.



Figure 8. EBSD images of TBC YSZ-2 after heat treatment at 1550 °C/100 h: (**a**) quality maps; (**b**) orientation maps; (**c**) phase maps; The orientations are defined by tetragonal lattice symmetry.

3.3. Texture

EBSD technology was used to analyse the local crystal texture of PS-PVD coatings. Figure 5a–c shows that YSZ-1 coating exhibited mainly a dense layered structure, in which the coating as a whole had no preferred orientation. The monolayer structure had a clear crystal orientation. The powders were melted completely and changed to a molten state. The resulting droplets were confined and accelerated to impact the substrate, where they deformed and flattened into splats. This process is similar to the traditional APS. There were many particles with no preferred orientation in the layered structure, and the particle composition was complex. Some powder materials in the plasma stream were evaporated and nucleated homogeneously in supersaturated state to form nanoclusters. The particles entering the plasma jet were melted and evaporated, but some large particles cannot be completely transformed into steam during the heating process. Some droplets solidified before colliding with the substrate or the growing coating surface [35,36].

As shown in Figure 6a–c, in the area of the pyramidal columnar coating closest to the bottom of the column, about 10 micron, the preferred orientation of the grains was not obvious, which was consistent with the dense structure layer near the bottom, and its basement layer consisted of small and randomly grown grains with a diameter of about 600 nm. In the EB-PVD YSZ coatings, this layer is called equiaxed crystalline grains, which has the characteristics of small grain size and thin thickness [37]. The grains in the quasi-equiaxed grains were randomly oriented. The growth direction of columnar structure was obvious above the quasi-equiaxed crystalline grains region. With the increase of coating thickness, columns were formed. Single column showed a clear crystal orientation, but different columns had different orientations, which made the growth orientation of the whole coating grain present random distribution.

The continuous growth and coalescence of the coating are closely related to the surface free energy, surface diffusion and volume diffusion, which depend on the substrate deposition temperature and

volume diffusion [38–40].

the total energy of the system. The growth process of PS-PVD coatings is similar to that of EB-PVD, which is also affected by atomic adsorption effect and surface diffusion, and substrate temperature has a great influence on the deposition. During the EB-PVD deposition process, when the substrate temperature was low, due to the self-shadow effect and the insufficient diffusion of deposition atoms on the substrate surface, the deposited coating was dome columnar structure with more pores at the grain boundary; In the appropriate temperature region, the coating structure was affected by the deposition growth controlled by surface diffusion, which can be diffused for a considerable distance and reduce the shadow effect. Each grain was epitaxial to form columnar structure with specific orientation. The defect density in the grain was low and the crystal structure was compact. When the temperature is too high, the recrystallization structure will be formed, which is mainly controlled by

The deposition process of the pyramid PS-PVD coating was mainly vapor deposition. The gaseous atoms adsorbed on the substrate diffused and migrated to nucleate, and the crystal-line nucleus adsorbed atoms and grew up to form island structure. With the continuous growth of the "island"-like structure on the substrate surface, it gradually covered the whole substrate surface, and then the adjacent island structures would affect each other [8,25,31]. In the process of further deposition, there was growth competition among different orientation crystals. The crystals with low surface energy were dominant to choose specific orientation and continue to grow, which will develop into columnar structure. The direction of crystal growth depended on many factors, including substrate temperature, energy and condensation rate. The slow-growing columns stopped growing near the adjacent columns, and the gap between them still existed [40]. The high column preferentially adsorbed the vapor atoms, and the shadow effect increased with the growth of the coating due to the insufficient surface diffusion caused by the high deposition rate. The result is that the microstructure of pyramid was separated by gaps. Because the columnar structure of pyramid coating was formed by the growth and competition of grains with different orientations, different columns had different orientations, and the coating as a whole did not exhibit preferred orientation.

The properties of the crystallized materials depend not only on the microstructure of the material, but also on the characteristic distribution of grain boundaries, such as intergranular fracture, creep, intergranular corrosion and impurity segregation. After high temperature thermal aging treatment, the microstructure (grain size, pores and cracks) and phase composition of the YSZ coatings changed significantly, and the texture of the YSZ coatings also changed accordingly. The preferred orientation has an important effect on the life of YSZ coating under the long-term service at high temperature. As shown in Figures 7a–c and 8a–c, after 1550 °C/100 h, the grains of the dense coating grew obviously, and a clear grain boundary was formed between the grains. The as-sprayed coating YSZ-1 had no preferred orientation, while the single column structure of the as-sprayed TBC YSZ-2 had a preferred orientation. During the high temperature aging treatment process, each grain appeared a slip phase transition, and the degree of interaction between dislocations was also different. This interaction would inevitably cause grain deformation and the preferred orientation of the coating. During the sintering process of coatings, due to high temperature and volume change caused by phase transformation, the growth of grains loss directionality, and the overall coating had no preferred orientation.

4. Conclusions

The effects of PS-PVD experimental processing parameters on the microstructure, phase and texture of the coatings with typical dense layered and columnar structures were investigated systematically.

(a) In the process of pyramid columnar coating deposition, the quasi equiaxed grains with random orientation were deposited firstly. With the increase of coating thickness, columnar structure were formed. Single column exhibited a specific crystal orientation, but different columns had different orientations, and the coating as a whole did not show a preferred orientation. The dense layered structure coating with no preferred orientation was mainly composed of melted droplets and unmelted/semi-melted particles. In addition to droplet deposition, condensation of vapor materials was also found in the coating.

(b) Compared with the dense layered coating, columnar coatings had a higher content of tetragonal phases. After heat treatment at 1550 °C for 100 h, the tetragonal phase content of the coatings decreased, the columns became smooth, the feather like structure of columnar coating disappeared, and some micro-nano grains of feather like structure also disappeared due to high temperature sintering and diffusion. The grain size of the as-sprayed dense coating was about 600 nm, and the grain size grew obviously with the extension of heat treatment time. After high temperature aging treatment, the preferred orientation of the two kinds of coatings disappeared.

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