Restrained TGO Growth in YSZ/NiCrAlY Thermal Barrier Coatings by Modified Laser Remelting

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Abstract

Thermal barrier coatings (TBCs) are widely applied in high temperature gas turbines and jet engines to protect the superalloy components. Due to oxygen transfer through topcoat and the oxidation of bond-coat alloy, a thin layer of thermally grown oxide (TGO) will generate between the ceramic topcoat and the metallic bondcoat of TBCs after long-term operation. Cracking and spalling are often observed at the topcoat-TGO and TGO-bondcoat interfaces since the thickening of TGO layer may lead to strong tensile stress. Previously, laser remelting process has been developed as an effective way in restraining the TGO growth. In the present research, preheating laser remelting and sol-gel repairing laser remelting were respectively employed to further decelerate the TGO growth by reducing the number density and average size of vertical cracks in the topcoat. After 100h isothermal oxidation, the TGO growth rate of the modified laser remelted specimens are much lower than the conventional laser remelted specimens, which shows the potentiality of preheating and sol-gel repairing as effective promotions of laser remelting process in restraining the TGO growth.

Key word: Thermal barrier coatings; TGO; Laser remelting; Preheat; Sol-gel

1. Introduction

Thermal barrier coatings (TBCs) are widely applied in gas turbines and jet engines to protect the superalloy components, such as blades and vanes, from the high temperature combustion and harsh oxidizing atmosphere [1, 2]. Currently, TBC systems mainly consist of a ceramic topcoat for thermal insulation, and a metallic bondcoat providing the adhesion between the topcoat and the superalloy substrate [3, 4]. During the high temperature operation, a thin layer of thermally grown oxide (TGO) will generate between the topcoat and bondcoat, composed mainly of Al_2O_3 and some other oxides, such as chromia ($(\text{Cr}, \text{Al})_2\text{O}_3$), spinel ($\text{Ni}(\text{Cr}, \text{Al})_2\text{O}_4$) and nickel oxide (NiO), which are all formed due to the oxygen diffusion through the ceramic topcoat [5, 6].

Due to the mismatching of elastic modulus and thermal expansion coefficients, the growth of TGO will increase the tensile stress at the topcoat-TGO and TGO-bondcoat interfaces, leading to severe cracking and spalling of the ceramic topcoat [7, 8]. State-of-the-art TBC, yttria stabilized zirconia (YSZ), has a high oxygen permeability and has been identified as good oxygen ion conductor [3, 9]. Besides, the topcoat fabricated by air plasma spray (APS), contains various defects, such as the typical lamellar stacking microstructure, splat cracks and high volume porosity, which can also accelerate the oxygen diffusion through the topcoat [10-13]. In order to restrain the TGO growth, several kinds of surface-sealing treatments have been developed to optimize the surface condition of the YSZ topcoat, such as laser remelting, aluminum phosphate sealing, chemical densification and sol-gel process [14, 15]. Among them, laser remelting process has been proven to be effective, because the remelted dense layer at the topcoat surface can reduce the oxygen conduction and therefore slow down the TGO growth [16-19]. However, a segmented crack network

perpendicular to the topcoat surface will generate after remelting and solidification [20, 21]. Propagation of the cracks during the high temperature operation will result in a “shortcut” for oxygen transferring, and finally destroys the oxygen resistance of the remelted layer [22-24]. Increasing the temperature of the superalloy substrate can reduce the temperature gradient along the TBC system during the APS process and thereby minimize the size of cracks in the ceramic topcoat, which may also be effective during the laser remelting process [25-27]. Besides, due to the low oxygen conductivity and good high temperature stability, Al_2O_3 has been identified as a potential protecting layer on TBCs to modify the surface microstructure and provide the oxygen resistance [28-30]. Effect of Al_2O_3 on the microstructure and oxidization behavior of YSZ coatings has been studied [31-34]. However, researches are still lacking about minimizing the concentration and size of cracks in the laser remelted layer by Al_2O_3 modification.

In the present research, preheating of the substrate was employed to control the cracks in the laser remelting layer, and a sol-gel process was also conducted to generate a thin film of Al_2O_3 on the surface of the laser remelting layer to cover the cracks. Microstructure and oxidization behavior of these modified specimens were investigated. Effect of these two processes on the TGO growth was also discussed.

2. Experimental procedures

A nickel-based superalloy (Inconel, GH 4169, CISRI, Beijing, China) in the dimension of $10 \times 11 \times 5 \text{ mm}$ was selected as the substrate. Ni-25Cr-7Al-1Y powders with a particle size between $15 \mu\text{m}$ to $45 \mu\text{m}$ (BGRIMN, Beijing, China) and 7wt.% Y_2O_3 stabilized ZrO_2 powder with a particle size between $75 \mu\text{m}$ to $125 \mu\text{m}$ (BGRIMN, Beijing, China) were respectively

used as the raw materials for bondcoat and topcoat. The bondcoat was deposited by high velocity oxy-fuel spraying (HVOF, K2, GTV, Germany) and the topcoat was prepared by air plasma spraying (APS, F6, GTV, Germany). Specific parameters of the HVOF and APS processes are shown in table I. The TBC specimens were then laser remelted by a pulsed Nd: YAG laser (HYK-200, Hairun Creater, Beijing, China) with 1mm diameter standard circular pulses. The wavelength was 1064nm and the mean power was 80W. The scanning speed was 12mm/s and the overlap of the pulses was 40%.

Preheating laser remelting was applied to control the cracking of the topcoat after laser remelting. The specimens were preheated for 20min in an electric furnace (CMF 1100, MTI, Hefei, China) holding at 600°C. After dwelling, the specimens were immediately laser remelted with the same parameters mentioned above. On the other hand, Al₂O₃ coat was also adopted by sol-gel method on the surface of laser remelted specimens to repair the cracks. 20wt.% acidic Al₂O₃ sol (Sinopharm Chemical Reagent, Beijing, China) was coated on the surface of the specimens by a spin coater (pw-4a, Chemat Technology, Shanghai, China). Then the specimens were heat treated at 600°C for 5h with a heat rate of 5°C/min to remove the organics. Finally, the specimens were sintered at 1000°C for 8h to form the dense layer of Al₂O₃ [32, 33]. The laser remelting processes applied in the present research are summarized in table II.

In order to investigate the TGO growth in the specimens treated by different laser remelting processes, the specimens were furnace aged in air at 1000°C for 100h (CWF1200, Carbolite, Derbyshire, UK). Then the TGO layer was observed on the cross section of the specimens by SEM (Mira3 LHM, TESCAN, Brno, Czechia) and the thickness of them was also measured from the back scattered electron (BSE) images (JSM 7001F, JEOL, Tokyo,

Japan). Finally, the TGO growth rate was calculated by the TGO thickness and the oxidation time.

3. Results and discussions

Fig.1 shows the surface images of as-sprayed APS and laser remelted coating, respectively. The surface of laser remelted coating appeared to be smoother than that of APS coating. Meanwhile, some laser spots residual are remained on the surface after the laser remelting process. Furthermore, SEM images of the surface of the APS and the laser remelted coatings are shown in Fig.2. Specifically, as-sprayed coatings have a rough surface with melted zone, unmelted particles, cracks and pores. Although YSZ is a good solid electrolyte for oxygen ion conduction at high temperature, the defects will further enhance the oxygen conduction through the coating. Instead, the surface of the laser remelted coatings is much smoother and denser. Most of the pores are eliminated by the remelting. However, some vertical cracks generated during the cooling, which are mainly caused by the rapid solidification and large thermal gradient along the coating [19, 20]. Furthermore, Fig.3 shows the cross section microstructure of the laser remelted coatings. A dense remelted layer formed at the surface of the coating in contrast to the defective and porous unmelted part beneath.

After 100h oxidation at 1000°C, TGO of different thickness have formed at the topcoat and bondcoat interface for both coatings with and without laser remelting. Fig.4 shows the BSE images of their cross sections. Clearly, TGO in the non-remelted specimens is thicker than that of laser remelted ones, confirming that laser remelting can restrain the TGO growth rate. It should be noticed that the TGO thickness is not uniform. This is mainly affected by two factors; the one is the lamellar structure of the APS coating and the random cracks, voids

and pores and the other is the roughness of the bond coat surface. These random distributed defects will lead to the variation of oxygen diffusion and non-uniform TGO growth. The rough bond coat surface with convex and concave also induces a non-uniform TGO thickness layer [35, 36].

In order to accurately evaluate the TGO growth, we defined a specific TGO thickness (δ) as the ratio of the cross section area and the length of the TGO layer, as shown in Fig.5. The measurement of the area of TGO layer was conducted using the Photoshop software while the length of TGO was obtained by AutoCAD at the same place in the BSE image. For each specimen, the area and length of the TGO were measured at 10 different places. Then the average value of the TGO thickness was calculated and is shown in table III.

Specifically, the TGO thickness of non-remelted and laser remelted specimens are $5.03 \pm 1.17\mu\text{m}$ and $4.34 \pm 0.73\mu\text{m}$. The TGO growth rate calculated by the TGO thickness and time are respectively $0.0503 \pm 0.0117\mu\text{m/h}$ and $0.0434 \pm 0.0073\mu\text{m/h}$ for non-remelted and laser remelted specimens, which are shown in Fig.6. A 14% reduction of the TGO growth rate is achieved by the laser remelting process, indicating that the laser remelted dense layer is beneficial to the oxygen resistance of the YSZ topcoat. However, as mentioned above, the vertical cracks in the dense remelted layer, which are generated by the rapid solidification and large thermal gradient, may limit the oxygen resistance or even cause the damage of the layer [20-22].

To control the cracking of the dense remelted layer, preheating was applied. As shown in Fig.1, the surface of preheating laser remelted specimen seems to be even smoother than that of specimen without preheating. SEM images of the surface of laser-remelted specimens with

and without preheating are shown in Fig.7. It can be observed that the cracks on the surface of preheated specimens are clearly less than that of specimens without preheating. Furthermore, the crack proportion of the surface can be quantitatively evaluated using the surface image analysis from SEM images. As shown in Fig.8, the area of all the cracks on the surface can be selected by Photoshop software and the crack proportion can be calculated according to the pixels. The evaluation and calculation were applied at five different section for each specimen which is shown in Fig.9. As summarized in table IV, crack proportions on the surface of laser remelted specimens with and without preheating are $4.34 \pm 0.43\%$ and $6.46 \pm 0.39\%$, respectively. This indicates that preheating is an effective way to reduce the crack proportion on the remelted layer, which may lead to better oxygen resistance.

After 100h oxidization at 1000°C , the thickness and the growth rate of the TGO could be calculated from the SEM images of the cross section of the preheating laser remelted specimen (Fig.4c). As shown in Fig.6 and listed in table III, the TGO thickness in preheating laser remelted specimen is $3.7 \pm 0.72\mu\text{m}$ and the TGO growth rate is $0.0370 \pm 0.0072\mu\text{m/h}$, respectively, which are about 15% lower than the laser remelted specimen without preheating, and about 26% lower than the as-sprayed coating. Clearly, preheating can improve the effect of laser remelting process in restraining the TGO growth.

In present study, effect of Al_2O_3 sol-gel repairing laser remelting is also investigated. In the surface image of sol-gel repairing laser remelted specimen (Fig1), the laser spots disappeared from due to a cover of new Al_2O_3 coating. As shown in Fig.10a the SEM image of the surface of the Al_2O_3 sol-gel repairing laser remelted specimen reveals that the cracks on the dense remelted layer are mainly sealed by a thin film, although there are still some small cracks remained due to the shrinkage of the gel during densification. EDX measurement

results (Fig.10b, Fig.10d) indicate the composition of film is alumina and the Al_2O_3 infiltrates into the cracks. However, XRD test could not identify the $\alpha\text{-Al}_2\text{O}_3$ phase (the thermodynamically stable phase above 1000°C) on the surface since the amount of Al_2O_3 is too small. Since Al_2O_3 is an oxygen barrier material restraining the oxygen diffusion across the coating, low TGO growth rate can be expected in Al_2O_3 sol-gel repairing laser remelted specimens [37-40].

After high temperature oxidation test, the TGO layer in the Al_2O_3 sol-gel repairing laser remelted specimen is even thinner (shown in Fig.4d), for which the TGO thickness is $3.8 \pm 1.00\mu\text{m}$ and the TGO growth rate is $0.0380 \pm 0.0100 \mu\text{m/h}$, respectively. Comparing with as-sprayed coating, the reduction of TGO growth rate is about 24% lower, which is similar with that of preheating laser remelting process.

In summary, TGO growth rate of laser remelted, preheating laser remelted and sol-gel repairing laser remelted specimens have been obviously reduced in comparison to as-sprayed APS coatings. It is well known that the oxygen transportation from the atmosphere to the topcoat-bondcoat interface through the YSZ coating has two major mechanisms. One is the oxygen diffusion in the gas phase via the interconnected cracks, pores and voids; the other one is the oxygen ion migration and conduction by means of a discrete hopping process through the YSZ lattice [21]. In the present research, the biggest TGO growth rate for as-sprayed coating is owing to both of the two mechanisms. For laser remelted specimen, the pores are eliminated and the crack concentration and size are also controlled. Thus, the gas phase diffusion is retarded and the TGO growth rate is smaller than that of as-sprayed coating. For preheating laser remelted specimen, the crack concentration in the dense remelted layer is reduced by about 30%, therefore the gas phase diffusion is further retarded and the TGO

growth rate is even smaller. For sol-gel repairing laser remelted coating system, the YSZ grains are covered by the oxygen barrier Al_2O_3 film and the cracks are also partly sealed. Therefore, both oxygen transportation mechanisms are retarded. The TGO growth rate is smaller than that of the laser remelted specimen.

Comparing with the as-sprayed coating, the TGO thickness (after 100h at 1000°C) reduction of laser remelted, preheating laser remelted and sol-gel repairing laser remelted specimens are 13%, 26% and 24% respectively. These results are compatible with some previous researches, such as 13% (100h at 1000°C) after conventional Al_2O_3 covering [41], 28% (100h at 1100°C) after nanostructured Al_2O_3 /YSZ compositing [37] and 26% (240h at 1200°C) after laser remelting [21]. It should be noticed that the TGO thickness reduction (26%) of laser remelted coating after 240h oxidation at 1200°C is twice more than that only laser remelted (13%) in the present research. The difference may be caused by the oxidation temperature and time.

4. Conclusions

In order to restrain the TGO growth in TBCs, three different methods based on laser remelting process were employed to YSZ/NiCrAlY TBCs. The TGO thickness and growth rate in the specimens were evaluated discussed. Laser remelting can reduce the TGO growth rate by about 13%. Preheating laser remelting can reduce the surface crack proportion generated during the laser remelting, thereby further decreases the TGO growth rate decreased by about 26%. Moreover, Al_2O_3 film can cover the surface of YSZ coating and seal the cracks. Therefore, Al_2O_3 sol-gel repairing laser remelting can decrease the TGO growth rate by 24%.

These results provide new perspectives to restrain the TGO growth in YSZ/NiCrAlY thermal barrier coatings.

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Table I

Parameters of the HVOF and APS processes

Parameters	HVOF	APS
Current (A)	/	625
Power (KW)	/	47
Primary gas flow (L/min)	(O ₂) 900	(Ar) 38
Secondary gas flow (L/min)	(kerosene) 0.43	(H ₂) 13
Carrier gas (L/min)	(Ar) 7	(Ar) 4
Spray distance (mm)	380	110
Powder feed rate (g/min)	80	35

Table II

Laser remelting processes for the TBC samples.

No.	Process	Laser remelting	Preheat at 600°C	Sol-gel Repairing
1	APS			
2	Laser remelting	√		
3	Preheating laser remelting	√	√	
4	Sol-gel repairing laser remelting	√		√

Table III

TGO thickness of APS, laser-remelted, preheating laser-remelted and sol-gel repairing laser-remelted specimens after 100h oxidation at 1000°C

Process	APS	Laser remelting	Preheat laser remelting	Sol-gel Repairing
TGO Thickness (μm)	5.03 $\pm$ 1.07	4.34 $\pm$ 0.73	3.70 $\pm$ 0.72	3.80 $\pm$ 1.00

Table IV

Crack area ratio to laser-remelted and preheating laser-remelted coating surface

Laser remelting			Preheating laser remelting		
section	Crack ratio (%)	Average (%)	section	Crack ratio (%)	Average (%)
1	6.27	6.46±0.39	1	3.73	4.34±0.44
2	6.52		2	4.25	
3	5.72		3	4.97	
4	6.59		4	4.32	
5	6.71		5	4.45	

Figure captions

Fig. 1. Surface images of APS, laser-remelted, preheating laser-remelted and sol-gel repairing laser-remelted coating

Fig. 2. SEM images of the surface of (a) APS coating and (b) laser-remelted coating

Fig. 3. Microstructure of the cross section of laser-remelted coating

Fig. 4. TGO morphology of the specimens after 100h oxidation at 1000°C (a) APS; (b) laser-remelted; (c) preheating laser-remelted and (d) sol-gel repairing laser-remelted

Fig. 5. A scheme drawing of the calculation method of TGO thickness by Photoshop-AutoCAD

Fig. 6. TGO growth rate of APS, laser-remelted, preheating laser-remelted and sol-gel repairing laser-remelted specimens at 1000°C

Fig. 7. SEM images of the surface of (a) preheating laser-remelted coating and (b) laser-remelted coating

Fig. 8. A scheme drawing of the calculation method of crack area proportion by Photoshop

Fig. 9. SEM images of sections for crack area proportion calculation on the surface of (a) preheating laser-remelted and (b) laser-remelted coating

Fig. 10. (a) SEM image; (b) EDX spectra; (c) SEM cross section and (d) EDX Al map of sol-gel repairing laser remelted coating

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