Influences of Laser Remelting on Microstructure and Thermal Shock Resistance of Al2O3-13wt.%TiO2 Coatings Deposited by Plasma Spraying

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Abstract. Al2O3-13wt.%TiO2 composite ceramic coatings were prepared by plasma spraying and laser remelting. Morphology, microstructure and phase composition were analyzed by scanning electron microscope (SEM) and X-ray diffractometer (XRD). Meanwhile, effects of laser remelting on thermal shock resistance of plasma-sprayed coatings were investigated and the thermal shock failure mechanism of coatings were discussed. Results demonstrate that after laser remelting, particles on the ceramic coating are refined and the lamellar structure disappears, thus increasing density and getting a remelting coating basically without defects like cracks. Laser remelting metastable phase γ-Al2O3 is transformed into a stable phase α-Al2O3. Due to low heat conductivity coefficient of ceramic materials, it is impossible to remelt the whole ceramic coating at laser remelting. An isometric crystal remelted zone with small grains, a sintered zone and a lamellar remained plasma-sprayed zone are formed on the remelting ceramic coating. Compared with plasma-sprayed coating, the laser-remelted coating has better thermal shock resistance. The thermal shock failure form of ceramic coating deposited by plasma spraying is basically corner peeling, while the thermal shock failure forms of laser-remelted ceramic coating includes both corner peeling and considerable local peelings in the center. Influences of laser remelting on thermal shock resistance of coating are mainly manifested as reduced initial failure resistance of coatings, decelerated crack propagation on coatings and changing failure mode of coatings.

1. Introduction
Thermal barrier coatings (TBCs), or known as thermal shielding coatings, are one of key technologies in modern aero-engine [1]. Based on high melting point, low vapour pressure, low thermal conductivity, low radiance and high reflectivity of ceramic materials, a layer of thermal insulation ceramic coating is prepared on high-temperature alloy surface at hot-end parts (e.g. flame tube, afterburner and turbine blade) of aero-engine. This thermal insulation ceramic coating can isolate high-temperature parts from high-temperature fuel gas to lower the operating temperature of high-temperature parts and protect high-temperature parts from high-temperature corrosion and ablation by fuel gas. Since TBCs serve in high-temperature environment or are influenced by violent changes of temperature, it is easy to cause early failure of TBCs due to thermal shock damages. Therefore, studying thermal shock failure process and mechanism of TBCs is of important significance to prolong the service life of coatings.

With advantages of fast deposition, high production efficiency, wide application range and low cost, plasma spraying solves spraying problems of refractory materials and ceramic materials [2]. It is the
mostly common preparation technology of TBCs in the world. However, inherent characteristics of plasma spraying determine the lamellar superposition structure of the coating. Moreover, the coating contains abundant pores and microcracks, thus restricting its application scope and service life. Wu et al. improved thermal shock resistance by preparing a nanostructured coating [3-5]. Lee et al. increased the thermal shock resistance by preparing a gradient thermal barrier coating [6]. Damani et al. discussed influences of thermal processing on thermal shock resistance of Al$_2$O$_3$ coating deposited by plasma spraying [7,8]. Laser remelting is a kind of laser surface strengthening technology that integrates laser technology and heat treatment. It can eliminate lamellar structure and most pores on the coating, form a uniform and dense ceramic coating, and protect performances of the coating, thus prolonging service life of workpieces [9-14].

In the present study, Al$_2$O$_3$-13 wt.% TiO$_2$ ceramic coatings were prepared on TiAl alloy surface by plasma spraying and plasma spraying-laser remelting combined technology. Morphology, microstructure and phase composition of coatings were investigated. A comparative study on thermal shock resistance between Al$_2$O$_3$-13 wt.% TiO$_2$ ceramic coatings prepared by plasma spraying and plasma spraying-laser remelting combined technology was carried out, which disclosed the thermal shock failure mechanism of coatings. This study is to explore superiority of laser remelting in preparation of TBCs.

2. Test Method
The $\gamma$-TiAl-based alloy (Φ25 mm×8 mm) melted by the High-temperature Material Research Center of China Iron and Steel Research Institute (CISRI) was used as the base material. The transition layer applied the NiCoCrAl superalloy powder with a granularity of -140~+325 meshes and dispersed by Y$_2$O$_3$ which was produced by Beijing General Research Institute of Mining and Metallurgy. Commercial Al$_2$O$_3$-13 wt.%TiO$_2$ ceramic powder with a granularity of 15~45 μm which was produced by Shenyang Ronghua and crushed irregularly by ordinary machine was applied as the ceramic material. The corresponding morphology of ceramic powders are shown in figure 1.

![Figure 1. SEM image of Al$_2$O$_3$-13wt.%TiO$_2$ ceramic powders](image)

Transition layer and ceramic layer were prefabricated by plasma spraying. The spraying equipment applied the 3710 mode plasma spraying system (Praxton, USA). Before spraying, samples were preprocessed by polishing, oil removing and abrasive blasting. Technological parameters of plasma spraying are listed in Table 1. Laser remelting applied SLCF-X12×25 type CO$_2$ laser machine and argon shield was applied at laser remelting. To reduce cracks on remelting coating like cracks, relevant low laser power and energy density were applied. Relatively optimized laser remelting technological parameters were gained from the test. Specifically, laser power, rectangular spot size, scanning speed and amount of overlap were 650 W, 5 mm×3 mm, 1 200 mm·min$^{-1}$ and 20%, respectively. The laser scanning was along 3 mm of the optical spots.

Morphologies of samples before and after thermal shock were observed by the JSM-7100F (JEOL) field emission scanning electron microscope. Thermal shock failure mechanism of ceramic coating
was discussed. D/max2500 X-ray diffractometer (XRD) (Rigaku, Japan) was applied for phase analysis.

Thermal shock resistance of ceramic coating was tested according to the aviation industrial standard: HB7269-96. In the thermal shock resistance test, samples were heated by SX2-4-9 chamber electric furnace under the temperature 850 °C. Samples were kept at 850 °C in the furnace for 15 min. Later, samples were collected from the furnace quickly and cooled in tap water under room temperature through water quenching. After dried in a drying oven, sample surfaces were observed and shot. This process was repeated until failure of samples. Coating surface with more than 30% area of peeling was evaluated as failure.

### Table 1. Plasma spraying parameters

| Process parameters     | Bond coating | Ceramic coating |
|------------------------|--------------|-----------------|
| Current (A)            | 710          | 850             |
| Voltage (V)            | 42           | 42              |
| Primary gas, Ar (PSI)  | 65           | 45              |
| Secondary gas, He (PSI)| 115          | 140             |
| Carrier gas, Ar (PSI)  | 45           | 45              |
| Powder feed rate (rpm) | 2            | 3               |
| Spray distance (mm)    | 110          | 110             |
| Traverse speed (mm·s⁻¹)| 100          | 100             |
| Coating thickness (μm) | ~100         | ~350            |

### 3. Test Results and Discussions

#### 3.1. Phase Component Analysis of Coating

Surface XRD spectra of Al₂O₃-13wt.%TiO₂ powder, plasma-sprayed coating and laser-remelted coating are shown in figure 2. It is observed that Al₂O₃-13wt.%TiO₂ powder mainly exists at stable phase α-Al₂O₃ and Al₃TiO₅ solid solution, accompanied with few metastable phase γ-Al₁₂O₃. The plasma-sprayed ceramic coating is mainly composed of γ-Al₁₂O₃, α-Al₂O₃ and Al₃TiO₅. In addition, few TiO₂ lose oxygen through redox in plasma jetting to produce TiO. γ-Al₂O₃ has a high content in the plasma-sprayed ceramic coating. Compared with Al₂O₃-13wt.%TiO₂ powder, plasma spraying transforms some α-Al₂O₃ into γ-Al₂O₃. This is mainly because the cooling rate of plasma spraying coating can reach as high as 10⁶~10⁸ °C/s and it is a typical process of fast solidification. Hence, it is easy to form a metastable phase on the coating. The formation of metastable phase is not only closely related with physical, chemical and thermal properties of base material and ceramics, but also is associated with melting degree, temperature, jetting velocity and particle distribution of ceramic particles as well as base temperature. In the process of fast solidification, particles in flux are overcooled. Under this circumstance, particle in flux meet homogeneous nucleation and nucleation ability of different phases in flux is determined by critical nucleation free energy of the solid phase rather than the free energy of different phases. Therefore, it is the phase with relatively low critical nucleation free energy rather than phase with low free energy that nucleate firstly. Since γ-Al₁₂O₃ has relatively low critical nucleation free energy in plasma spraying and it is easy to nucleate, while the nucleation rate of α-Al₂O₃ phase is small. Therefore, metastable phase γ-Al₁₂O₃ is the dominant component of the coating.

γ-Al₂O₃ disappears completely after laser remelting. Only α-Al₂O₃ and Al₃TiO₅ are retained in the coating. Under laser actions, the metastable phase γ-Al₁₂O₃ has been transformed to stable phase α-Al₁₂O₃ completely [14]. This can be explained as follows: in the laser remelting process, the instant high temperature when laser beam irradiates onto the ceramic coating surface may induce a high-temperature molten pool on the coating surface. When the temperature is higher than 1 200 °C, the metastable phase γ-Al₁₂O₃ might be transformed to α-Al₁₂O₃ irreversibly. The flux which is cooled more slowly than the plasma spraying technique will be solidified again as single α-Al₂O₃.
3.2. Microstructure of Plasma-sprayed Ceramic Coating

The cross-section morphology of plasma-sprayed coating is shown in figure 3. Figure 3(a) shows ceramic layer, bond layer and TiAl alloy base from the right to the left. The ceramic coating shows an evident lamellar superposition structure which is determined by characteristics of the plasma spraying. Plasma spraying forms a high-speed molten particle flow by heating materials to the molten or thermoplastic state based on plasma heat source. The particle flow impacts onto base or existing coating surface successively. It is flattened through transverse flowing of particles and solidified and cooled quickly, thus depositing into coatings [15]. When molten drops form a coating, behaviors of different molten drops are independent under ordinary spraying conditions due to the very high flattening speed and cooling and solidification speed. The spraying powder superposes onto the previous coating, thus forming the lamellar structure of the plasma-sprayed coating. Moreover, strong mechanical bonding interfaces were formed between the ceramic layer and transition layer as well as between the transition layer and base. The further enlarged view of ceramic tissues is shown in figure 3(b). The lamellar structure is clearer and the coating has an evident deep-shallow alternative lamellar structure.

Figure 2. XRD diffraction patterns: (a) original ceramic powder, (b) as-sprayed coating and (c) laser-remelted coating.

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According to the energy spectrum (figure 3(b)) analysis, the light tissue represents the TiO₂-rich zone and the deep tissue represents the Al₂O₃-rich zone, and there’s mutual dispersion of components between TiO₂-rich zone and Al₂O₃-rich zone. Since the melting point of TiO₂ is lower than that of Al₂O₃, TiO₂ powder is melted in the spraying process to bond abundant Al₂O₃. The melted TiO₂ and Al₂O₃ dissolve mutually to some extent [14]. Adding TiO₂ not only can decrease porosity and increase density of the coating, but also is conducive to increase bonding strength between the coating and transition layer or base material, and the bonding strength among Al₂O₃ particles in the coating. As a result, the mechanical properties of the coating are improved. Therefore, the Al₂O₃ coating containing TiO₂ has better tenacity and higher impact load resistance than pure Al₂O₃ coating.

3.3. Microstructure of Laser-remelted Ceramic Coating

Cross-section microstructure of the composite ceramic coating after laser remelting is shown in figure 4. Figure 4(a) shows ceramic layer, bond layer and TiAl alloy base from the right to the left. Due to collaborative effect of laser power, energy density, temperature field distribution in the zone of laser action, heat conductivity coefficient of ceramics and coating thickness, it is impossible to realize laser remelting of the whole ceramic layer and the remelted ceramic layer develops evident hierarchical structure features (figure 4(b)). According to different tissue morphologies, the coating can be generally divided into remelting zone (figure 4(c)), sintering zone (figure 4(d)) and remained plasma-sprayed zone [12].
Figure 4. Cross-sectional SEM morphologies of laser-remelted coating: (a) overview, (b) ceramic coating, (c) remelted zone and (d) sintered zone

Surface ceramic layer is melted by the instant high temperature produced during laser remelting and a high-temperature molten pool is formed. In the subsequent solidification process, the ceramic layer has a low heat conductivity coefficient and it is cooled slightly by the cold base. Therefore, heats in the molten pool are mainly dissipated through thermal radiation and convection of air. However, the molten pool is cooled relatively quickly, which is attributed to blowing protection during remelting. Because of the high degree of supercooling, isometric crystals with small grains are formed in the remelting zone. They are different from the columnar crystal tissues which are formed due to low cooling speed of molten pool and grow along the heat flow under general conditions [12-14]. Temperature in the layer below the remelting zone reaches the sintering temperature of \( \text{Al}_2\text{O}_3-13\text{wt.} \% \text{TiO}_2 \) ceramics, which is caused by heat conduction. In the sintering process, substances are driven by the reduction of surface energy and fill in neck space among particles and pores through different diffusion pathways, which further lead to gradual expansion of the neck space, linkage formation of fine particles, growth of crystal grains, reduction of pores and crystal boundaries gradually and increase of coating density. Finally, a sintering zone is formed. In the ceramic layer close to the transition layer, heats are difficult to be transmitted from the remelting zone to the ceramic layer due to the low heat conductivity coefficient of ceramic materials. Therefore, the ceramic layer has a low temperature and still maintains the typical lamellar structural characteristics of plasma spraying state [12].

3.4. Thermal Shock Test Results
The contrast test results of thermal shock resistances of the ceramic coatings prepared by plasma spraying and laser remelting under 850 °C water quenching are shown in figure 5. The number of thermal shock failures of plasma-sprayed ceramic coating and laser-remelted ceramic coating are 67 and 107 cycles, respectively. This confirms that laser remelting can improve thermal shock resistance of plasma-sprayed ceramic coating effectively.
Surface changes of plasma-sprayed ceramic coating in the process of thermal shock are shown in figure 6. Clearly, the plasma-sprayed ceramic coating presents strong thermal shock resistance in early period of thermal shock. The coating surface develops neither macrocracks nor peeling phenomenon after 43 heating cycles. It remains basically complete until the 44th thermal shock when the first macrocrack is developed at a corner of the coating. Once the crack is formed, the coating begins to peel off. With the continuous increase of thermal shocks, the macrocrack and peeling zone expand quickly on the coating surface. The peeling area on the coating surface reaches 30% after 67 heating cycles, indicating the coating failure.

Surface changes of laser-remelted ceramic coating in the process of thermal shock are shown in figure 7. Its failure mode differs significantly from that of plasma-sprayed ceramic coating. Failure of the plasma-sprayed ceramic coating is mainly manifested as corner peeling. However, the laser-remelted ceramic coating has both corner peeling and local peeling in the center. Moreover, the peeling zone on laser-remelted ceramic coating expands slowly with the increase of thermal shocks. Although both coatings begin to develop peeling phenomenon at similar number of thermal shocks, the total thermal shock life of laser-remelted ceramic coating is about 60% longer than that of the plasma-sprayed ceramic coating.
3.5. Thermal Shock Failure Mechanism Analysis of Coatings

Ceramic coatings finally may fail after repeated thermal shocks under high temperature. Although there are many causes of coating failure, internal stress produced in the heating cycles is the fundamental cause. Growth of thermally grown oxide (TGO) in the interface between transition layer and ceramic layer in the thermal shock process also can influence coating failure significantly. In addition, phase transformation stress can facilitate thermal shock failure of coatings to some extent.

Thermal stress in coatings is formed during heating cycles. It is the consequence of temperature difference between the coating and base materials as well as the difference of dilatation coefficient between the ceramic coating and metal transition layer and between the transition layer and base. In one heating cycle, volumes of ceramic layer and metal base change differently, which is related with their different dilatation coefficients. As a result, the coating surface develops different stress states when temperature increases and decreases. The surface ceramic layer is in the compressive stress state when temperature increases, but it is in the tensile stress state in the cooling process (figure 8(a)). In repeated thermal shocks, a residual tensile stress layer is formed on the coating surface, which may induce a bending effect of the coating (figure 8(b)). Influenced by this bending force, stress concentration occurs on the coating surface and the interface between the coating and transition layer. When tensile stress exceeds the breaking strength of coating, the coating surface is cracked. Cracks in longitudinal distribution are developed (figure 8(c)). When the interface stress exceeds bonding strength of the interface, cracks parallel to the coating are formed (figure 8(c)), finally causing peeling of the surface ceramic layer.

Influences of longitudinal cracks on thermal shock life of ceramic coating are different from those of horizontal cracks at the interface of bonding layer [16]. On the one hand, longitudinal cracks on coating surface make oxygen in air penetrate into coating and diffuse to the bonding layer, thus oxidizing the transition layer and forming a TGO layer. Such TGO layer further induces horizontal cracks at the bonding layer directly, and thereby influences thermal shock performance of the coating. On the other hand, production of longitudinal cracks on coating surface can relieve thermal stress which is produced in heating cycle, weaken extension force of horizontal cracks, and improve thermal shock resistance of the coating. However, horizontal cracks on bonding layer will propagate along the bonding layer in the thermal shock process once they are produced, causing peeling of the coating. Hence, the thermal shock life of the coating is shortened.

![Figure 7](image_url)
Figure 8. Schematic illustration of formation of thermal stress, surface and interface crack in coating subjected to a heating-cooling cycle during thermal shock cycling [16]: (a) temperature and stress history near the ceramic coating surface during a heating-cooling cycle, (b) stress-state in the coating after heating-cooling cycles and (c) resulting surface and interface crack due to heating-cooling cycles.

Microstructures of plasma-sprayed ceramic coating surface before and after the thermal shock under 850 °C are shown in figure 9. Compared with figure 9(a), figure 9(b) (unpeeling zone) shows evident networked cracks. Thermal shock resistance of coatings is determined by its tolerance to thermal stress under thermal shock conditions and its strength (including bonding strength between the coating and transition layer/base and the cohesion strength of the coating). Pores and weakly bonded laminated structure inside the plasma-sprayed ceramic coating are easy to form crack sources under the effect of cyclic thermal stresses. Since the coating lacks of an effective mechanism to relieve thermal stress, it is easy to develop cracks in the process of thermal shocks, especially horizontal cracks on the interface of bonding layer. Under cyclic thermal stresses, horizontal cracks propagate quickly and the coating peels off accordingly, manifested by poor thermal shock resistance [17,18]. Xu et al. pointed out that the plasma-sprayed coating has poorer resistance to high temperature oxidation than laser-remelted coating [19]. Moreover, TGO on the interface between ceramic layer and the transition layer of plasma-sprayed ceramic coating grows quickly during high temperature oxidation. Accordingly, TGO on plasma-sprayed coating surface grows quickly in the process of thermal shock. Although TGO membrane is important to hinder further oxidization of the transition layer and protect the base, excessive thick TGO member can weaken binding force of the transition layer and thereby accelerates peeling of the coating. The thermal shock test results also reveal that a large area of plasma-sprayed ceramic coating is peeled off and failed after relatively few thermal shocks (67 cycles).

Figure 9. SEM morphology of the coating: (a) as-sprayed coating and (b) plasma-sprayed coating after 67 cycles at 850 °C
Typical morphologies of laser-remelted ceramic coating before and after the thermal shock test are shown in figure 10. Microstructure of unpeeled surface after 107 thermal shocks is shown in figure 10(b). Laser-remelted ceramic coating develops similar networked cracks after thermal shocks with those on plasma-sprayed ceramic coating. However, distribution of networked cracks is denser and cracks are thinner, belonging to microcracks. Longitudinal cracks on surface are beneficial to relieve thermal stress in the process of thermal shocks. In particular, microcracks have been proved as stress releasing source and can improve tenacity of materials and relieve thermal stress in the process of thermal shock significantly, thus improving thermal shock resistance of materials. Moreover, laser remelting produces dense isometric crystal tissues, reduces pores and lamellar structures in coating, and changes direct diffusion of oxygen on lamellar interface or defects like pores. Although networked microcracks are produced in the process of thermal barrier, thermal expansion can close cracks effectively and retard direct diffusion pathways of oxygen during heating and heat retaining periods of thermal shock test. Accordingly, thermal shock resistance of the coating is improved [20]. However, ceramic materials which have poor shock tolerance and low breaking tenacity are easy to develop cracks, pores and even peeling phenomenon during rapid heating and cooling conditions in laser remelting. Additionally, the residual stress is large. Although relatively optimized technological parameters are applied during laser remelting of samples in the experiment, it is impossible to prevent defects like cracks and pores completely in laser remelting, especially in the overlap joint. Therefore, regions with more defects and large residual stresses are easy to have propagation of cracks in the process of thermal shock, which further causes local small-unit point peeling between the laser-remelted layer and remained plasma-sprayed layer. The peeling units increase gradually with the increase of thermal shocks and some peeling units connect into a large-scaled peeling. Nevertheless, most regions with few defects still keep good thermal shock resistance. In a word, although laser-remelted ceramic coating develops peeling at similar thermal shocks with the plasma spraying ceramic coating, its total thermal shock life is prolonged significantly.

![Figure 10: SEM morphology of the coating: (a) as-remelted and (b) laser-remelted after 107 cycles at 850°C](image)

**Figure 10.** SEM morphology of the coating: (a) as-remelted and (b) laser-remelted after 107 cycles at 850°C

Based on above analysis, laser-remelted ceramic coating is easier to develop local point peeling in the process of thermal shocks than plasma-sprayed ceramic coating, which is caused by some defects produced in the remelting process. This implies that laser remelting weakens initial thermal shock resistance of local regions of the ceramic coating, but the total thermal shock resistance is far superior to that of the original plasma-sprayed ceramic coating. If appropriate measures are used to further decrease defects (e.g. cracks and pores) in laser remelting, the thermal shock resistance of the coating can be improved significantly. Mixed spraying by adding rare earth elements into spraying powder and adding ceramic materials with low melting points into ceramic powder, changing output way of laser, controlling laser remelting parameters to form ordered phase in ceramic layer, controlling and improving coating quality and composition, and introducing in ultrasonic vibration into laser remelting all can control cracks and peeling problems during laser remelting process to some extent. Additionally, nanomaterials possess excellent performances beyond ordinary materials for its unique
structure and provide favorable conditions to improve coating performances. Laser remelting based on plasma-sprayed of nanostructured ceramic coating can form a nanoparticle coating. The remelting layer has strong tenacity by taking advantages of the dispersion strengthening mechanism of nanoparticles, which prevents cracking of laser-remelted ceramic coating [21,22].

4. Conclusions
In the present study, Al$_2$O$_3$-13wt.%TiO$_2$ composite ceramic coatings were prepared by plasma spraying and laser remelting. Effects of laser remelting on microstructure and thermal shock resistance of plasma-sprayed coating were investigated.

(1) The plasma-sprayed Al$_2$O$_3$-13 wt.% TiO$_2$ coating has a typical lamellar structure with low density. After laser remelting, particles on the surface ceramic layer are refined and lamellar structure disappears, while density is increased. There’s hardly defect (e.g. cracks) on the remelting layer. In the laser remelting process, metastable phase $\gamma$-Al$_2$O$_3$ is transformed to stable phase $\alpha$-Al$_2$O$_3$. After complete remelting of the ceramic coating, an isometric crystal remelting zone with small grains, sintering zone and lamellar remained plasma-sprayed zone are formed.

(2) Although laser remelting decreases the initial thermal shock resistance of local regions of the ceramic coating, it prolongs the total thermal shock life of the ceramic coating compared with plasma-sprayed ceramic coating. The plasma-sprayed ceramic coating basically fails by corner peeling, while the laser-remelted ceramic coating has considerable local peelings in the center in addition to corner peeling.

(3) Influences of laser remelting on thermal shock resistance of plasma-sprayed ceramic coating are mainly manifested by the reduced initial resistance to failure of coatings, relieved propagation rate of cracks, and different failure modes of coatings.

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6. References
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