



Effect of TiO_2 content on properties of Al_2O_3 thermal barrier coatings by plasma spraying

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Abstract: Al_2O_3 thermal barrier coatings with different TiO_2 contents were deposited on 6061 aluminum alloy by plasma spraying. The corrosion resistance, thermal insulation property and phase composition of these coatings were investigated. The results indicate that all the feedstock powders exhibit phase transformation during the spray process. With the increase of the TiO_2 content in the powder, the corrosion resistance of the coating is enhanced but the thermal insulation property is decreased. This can be attributed to the higher thermal conductivity but lower melting point and brittleness of TiO_2 than those of Al_2O_3 , so it is easy for TiO_2 to disperse in the brittle Al_2O_3 substrate during spraying, in which these dispersively distributed TiO_2 play the role of hole sealing, releasing stress and reducing cracks.

Key words: plasma spray; corrosion resistance; thermal insulation property; phase transformation; TiO_2

1 Introduction

Atmospheric plasma spraying (APS) is widely used due to its high deposition rate as well as its production efficiency. The fabricated coating also exhibits high quality which enables this technology to play an important role in thermal spray technologies [1–3]. Al_2O_3 is widely used to fabricate thermal barrier coating due to its good chemical stability, corrosion resistance and thermal stability [4–6]. Thermally grown oxide (TGO) layer at the ceramic/bond coat interface was always formed because of the oxidation of the bond coat at elevated temperatures. The oxidation plays an important role in the failure of thermal barrier coatings (TBCs) [7,8]. One of the most effective measures to improve it is to fabricate an Al_2O_3 layer which is between the bond coat and top coat or as the top coat in the thermal barrier system [9–12]. Therefore, the research of Al_2O_3 coating is meaningful for improving the interface state.

Nowadays, the piston which is used in automotive industry is made of aluminum alloy which has a lower

melting point. If Al_2O_3 coating is fabricated on its surface, the lifetime of piston will be extended, and this method can also improve the thermal efficiency and save energy [13]. Therefore, it is important to research the thermal insulation of Al_2O_3 coating.

The Al_2O_3 thermal barrier coating whose temperature of working condition is very high will suffer hot-medium corrosion, so it is urgent to improve its corrosion resistance [14]. In 2005, SHEN et al [15] found that the TiO_2 coatings on steels exhibited excellent corrosion resistance. At the same year, CELIK et al [16] found that the corrosion resistances of Al_2O_3 and $\text{Al}_2\text{O}_3 + \text{TiO}_2$ were higher than those of $\text{Cr}_3\text{C}_2 + \text{NiCr}$, NiAl and NiCrAl coatings. In 2009, TIAN et al [17] concluded that nanostructured $\text{Al}_2\text{O}_3 - 13\% \text{TiO}_2$ coating had higher corrosion resistance than the conventional one. In 2010, TOMA et al [18] found that adding TiO_2 in Al_2O_3 increased its corrosion resistance. However, relatively little research has been done to study the mechanism for TiO_2 to improve the corrosion resistance of Al_2O_3 coating.

The influence of TiO_2 content on the corrosion resistance and thermal insulation of Al_2O_3 coatings, as

well as its mechanism was studied. In order to improve the thermal shock resistance and the bonding strength, a double layer structure whose bond coat is Ni(Al) and the top ceramic coat is Al_2O_3 or AT3 (Al_2O_3 –3% TiO_2) or AT13 (Al_2O_3 –13% TiO_2) was adopted.

2 Experimental

2.1 Materials and spraying

6061 aluminum alloy with the main elements of aluminum, magnesium and silicon was employed as the substrate. The powders used in this work were produced by Xiandao Plasma Powder Co., Ltd., China. And the particle size of Ni(Al), AT3 and AT13 powders was 38–58 μm ; the particle size of Al_2O_3 was 25–45 μm . The plasma spraying equipment used in this work was produced by PRAXAIR. After sand blasting and degreasing for substrate, a bond coat of Ni(Al) with the thickness of about 60 μm was fabricated on the substrate. And a top ceramic coat with the thickness of about 160 μm was fabricated on the bond coat. Their spraying parameters are given as Table 1.

2.2 Performance test methods

D8 X-ray diffractometer (Cu K_α radiation) was used to analyze the phase composition of the three kinds of ceramic layers and their powders. The continuous scanning speed was fixed at 10 ($^\circ$)/min. The ceramic layers and powders were characterized by JSM-6610LV scanning electron microscope.

Four materials (e.g., Al_2O_3 , AT3, AT13 coating and

aluminum alloy) were employed for electrochemical corrosion tests at 25 $^\circ\text{C}$ and 3.5% NaCl solution was chosen as the corrosive medium. This test was conducted in a three-electrode system and the type of the reference electrode was SCE ($\text{Hg}/\text{Hg}_2\text{Cl}_2$ -saturated KCl). The scanning rate was 5 mV/s, the polarization range was from –1 to 1 V (relative to the open circuit potential) and the frequency was 2.0 Hz, respectively.

Figure 1 shows the schematic diagram of thermal insulation performance test. When heating the samples with coating using a hot plate, the thermocouples were buried in their two sides. The temperature difference between the two sides was measured with the increased temperature of hot plate. After a period of time, the temperature field achieved a state of equilibrium and the temperature difference was the temperature insulation.

3 Results and discussion

3.1 Phase characterization

Figure 2 shows the comparison of phase composition between Al_2O_3 powder and the coating. The phase in Al_2O_3 powder is α - Al_2O_3 , while the phases in Al_2O_3 coating are α - Al_2O_3 and γ - Al_2O_3 . In other words, the phase transformation occurs obviously. In order to compare the relative content of different phases, the intensity of the highest diffraction peak is defined as 100, and the relative content is the ratio of the integrated intensity of the main peaks of various phases to that of the main phase. Table 2 gives the relative contents of different phases in Al_2O_3 powder and the coating, which

Table 1 Parameters of APS deposition

Powder	Primary gas (Ar) pressure/ $6.895 \times 10^3 \text{ Pa}$	Secondary gas (He) pressure/ $6.895 \times 10^3 \text{ Pa}$	Carrier gas (Ar) pressure/ $6.895 \times 10^3 \text{ Pa}$	Spray distance/ mm	Distance per step/ mm	Linear velocity/ ($\text{mm} \cdot \text{s}^{-1}$)	Powder feed rate/ ($\text{r} \cdot \text{min}^{-1}$)	Number of cycles
Ni(Al)	50	40	40	80	4	100	2.3	1
Al_2O_3	50	40	40	80	4	100	5.0	1
AT3	50	40	40	80	4	100	5.0	1
AT13	50	40	40	80	4	100	5.0	1

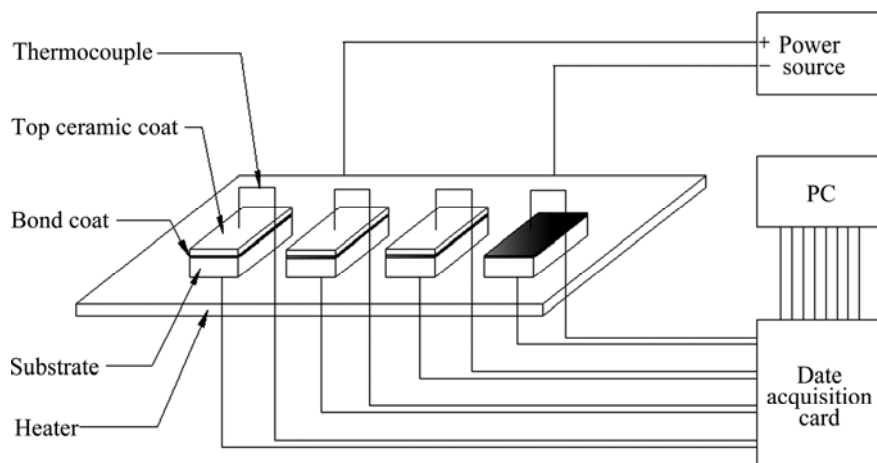


Fig. 1 Schematic diagram of thermal insulation performance test

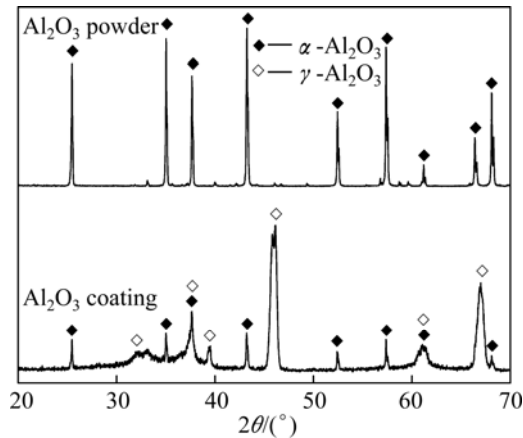


Fig. 2 Comparison of phase composition between Al_2O_3 powder and coating

Table 2 Relative contents of different phases in Al_2O_3 powder and coating

Sample	$w(\alpha\text{-Al}_2\text{O}_3)/\%$	$w(\gamma\text{-Al}_2\text{O}_3)/\%$
Al_2O_3 powder	100	0
Al_2O_3 coating	4.41	95.59

is measured using adiabatic method. The relative contents of $\gamma\text{-Al}_2\text{O}_3$ in Al_2O_3 powder and the coating are 0 and 95.59%, respectively. This can be attributed to the metastable phase in the coating which is formed after rapid heating and cooling [19–22].

Figure 3 shows the comparison of phase composition between AT3 powder and the coating. As can be seen in Fig. 2, the phases in AT3 powder are $\alpha\text{-Al}_2\text{O}_3$ and rutile TiO_2 while in AT3 coating they are $\alpha\text{-Al}_2\text{O}_3$, $\gamma\text{-Al}_2\text{O}_3$ and anatase TiO_2 . In other word, phase transformation also occurs obviously. Table 3 gives the relative contents of different phases in AT3 powder and coating. All the rutile TiO_2 transforms into anatase TiO_2 and the relative contents of $\gamma\text{-Al}_2\text{O}_3$ in AT3 powder and coating are 0 and 52.00%, respectively. Compared with Al_2O_3 coating, however, the relative content of $\gamma\text{-Al}_2\text{O}_3$ in AT3 coating decreases.

Figure 4 shows the comparison of phase composition between AT13 powder and coating. The phases in AT13 powder include $\alpha\text{-Al}_2\text{O}_3$, rutile TiO_2 and Al_2TiO_5 while in AT13 coating they are $\alpha\text{-Al}_2\text{O}_3$, $\gamma\text{-Al}_2\text{O}_3$, anatase TiO_2 and Al_2TiO_5 . Phase transformation occurs obviously. Table 4 gives the relative contents of different

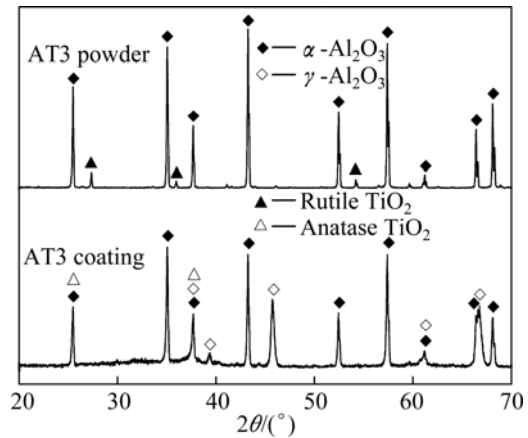


Fig. 3 Comparison of phase composition between AT3 powder and coating

Table 3 Relative contents of different phases in AT3 powder and coating

Sample	$w(\alpha\text{-Al}_2\text{O}_3)/\%$	$w(\gamma\text{-Al}_2\text{O}_3)/\%$	$w(\text{Rutile TiO}_2)/\%$	$w(\text{Anatase TiO}_2)/\%$
AT3 powder	97.09	0	2.91	0
AT3 coating	45.01	52.00	0	2.99

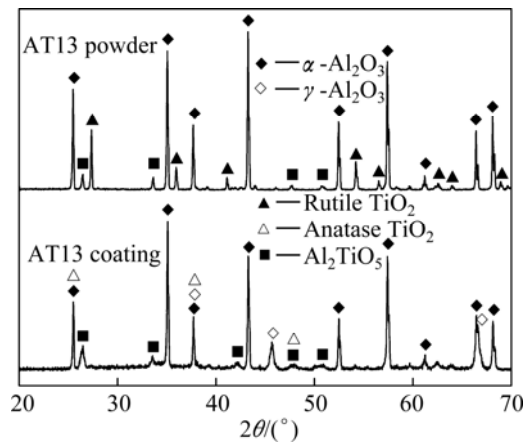


Fig. 4 Comparison of phase composition between AT13 powder and coating

phases in AT13 powder and coating. All the rutile TiO_2 phases transform into anatase TiO_2 . The relative content of $\gamma\text{-Al}_2\text{O}_3$ is 0 in AT13 powder while in coating it is 27.88%. The above results prove that the increase of the content of TiO_2 in the powder can inhibit the transformation from $\alpha\text{-Al}_2\text{O}_3$ to $\gamma\text{-Al}_2\text{O}_3$.

Table 4 Relative contents of different phases in AT13 powder and coating

Sample	$w(\alpha\text{-Al}_2\text{O}_3)/\%$	$w(\gamma\text{-Al}_2\text{O}_3)/\%$	$w(\text{Rutile TiO}_2)/\%$	$w(\text{Anatase TiO}_2)/\%$	$w(\text{Al}_2\text{TiO}_5)/\%$
AT13 powder	85.51	0	11.12	0	3.37
AT13 coating	57.30	27.88	0	11.32	3.50

Through the above analysis, it can be drawn that TiO_2 will inhibit the transformation from $\alpha\text{-Al}_2\text{O}_3$ to $\gamma\text{-Al}_2\text{O}_3$ during spraying process, and the degree of inhibition is enhanced with the increase of its content. And during spraying process, rutile TiO_2 will transform to anatase TiO_2 totally.

After 4 h of treatment in 400 °C, there is no phase transformation of the three kinds of coatings. The results are shown in Fig. 5. It can be concluded that Al_2O_3 , AT3 and AT13 coatings are very stable at 400 °C.

3.2 Surface and cross-sectional morphology analysis

Figure 6 shows the SEM images of all the powders. It can be seen that the shape of Al_2O_3 is blocky and others have the shape of cluster. Figures 7 and 8 show the SEM images of surface of three kinds of coating, with pores and cracks presented on them. The increase of pores and cracks enables corrosive medium easy to reach substrate, which can therefore reduce the corrosion resistance of the coating. Furthermore, cracks will reduce the thermal shock resistance which will make the coating fall off easily. As a consequence, avoiding pores and cracks in order to make coatings dense is one of the key concerns to fabricate coatings.

Figure 9 shows the cross-sectional SEM images of Al_2O_3 , AT3 and AT13 coatings. It can be seen that the interface is well combined and no obvious crack appears.

Pores exist in bond coat and top ceramic coat, and it is obvious that the bond coat is denser than the top ceramic coat. Ni(Al) , which is the material of bond coat, has low melting point. It can be fully melted in the process of spraying. And the molten state Ni(Al) has good liquidity, so it is dense after solidification via spraying onto the substrate. As the material of top ceramic coat, Al_2O_3 has high melting point, thus it is shown as fully-molten and semi-molten state in the process of spraying. The formation of pores can be possibly attributed to semi-molten state particles stacked overlapping each other and volume shrinkage during fully-molten material solidification.

Figure 10 shows the porosity and protective efficiency of the three kinds of coatings, and it is measured by Image-pro through cross-sectional SEM images of their top ceramic coat. It can be seen that with the increase of TiO_2 content in the Al_2O_3 powder, the porosity of coating decreases. The reason may be that low melting point TiO_2 disperses in the high melting point Al_2O_3 substrate during spraying, in which these dispersively distributed TiO_2 plays the role of hole sealing.

Figure 11 shows the EDX of cross-sectional images of the three kinds of coating. It scans from the top coat to substrate. It can be seen that O, Al and Ni exist in Al_2O_3 coating while O, Al, Ni and Ti exist in AT3 and AT13

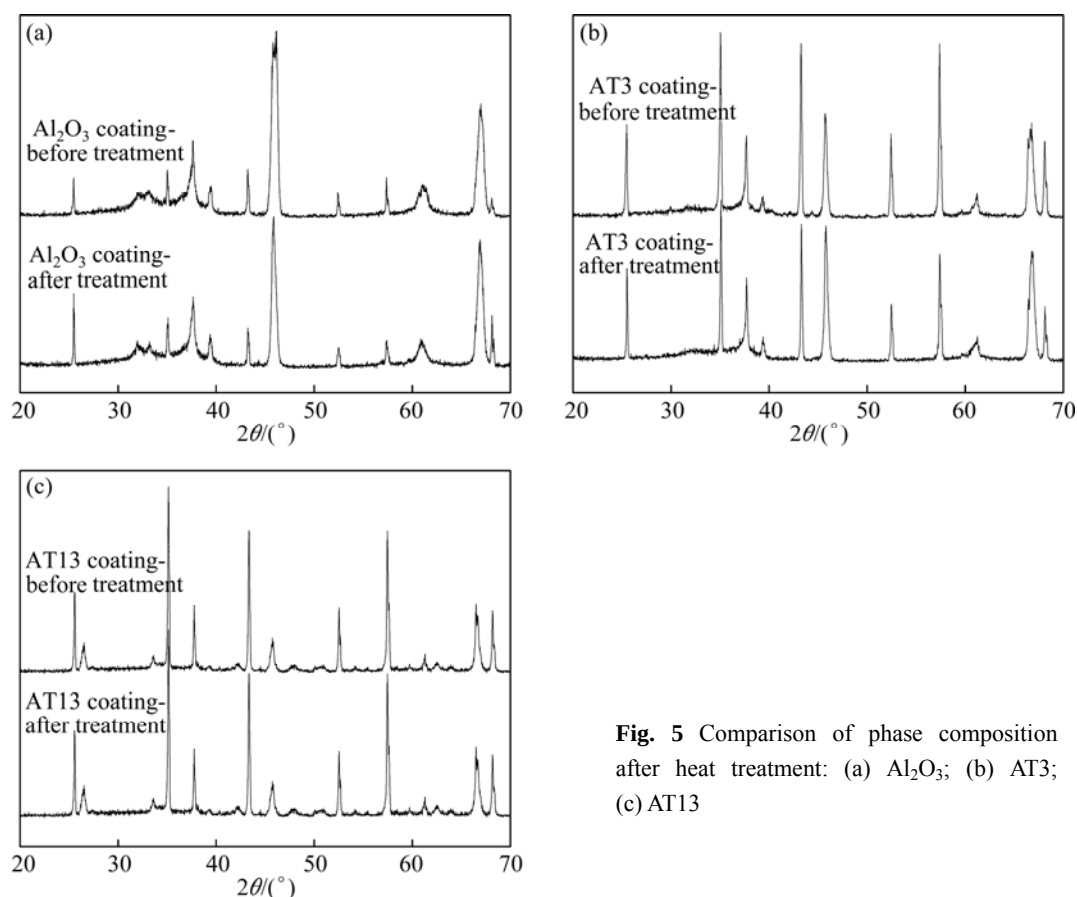


Fig. 5 Comparison of phase composition after heat treatment: (a) Al_2O_3 ; (b) AT3; (c) AT13

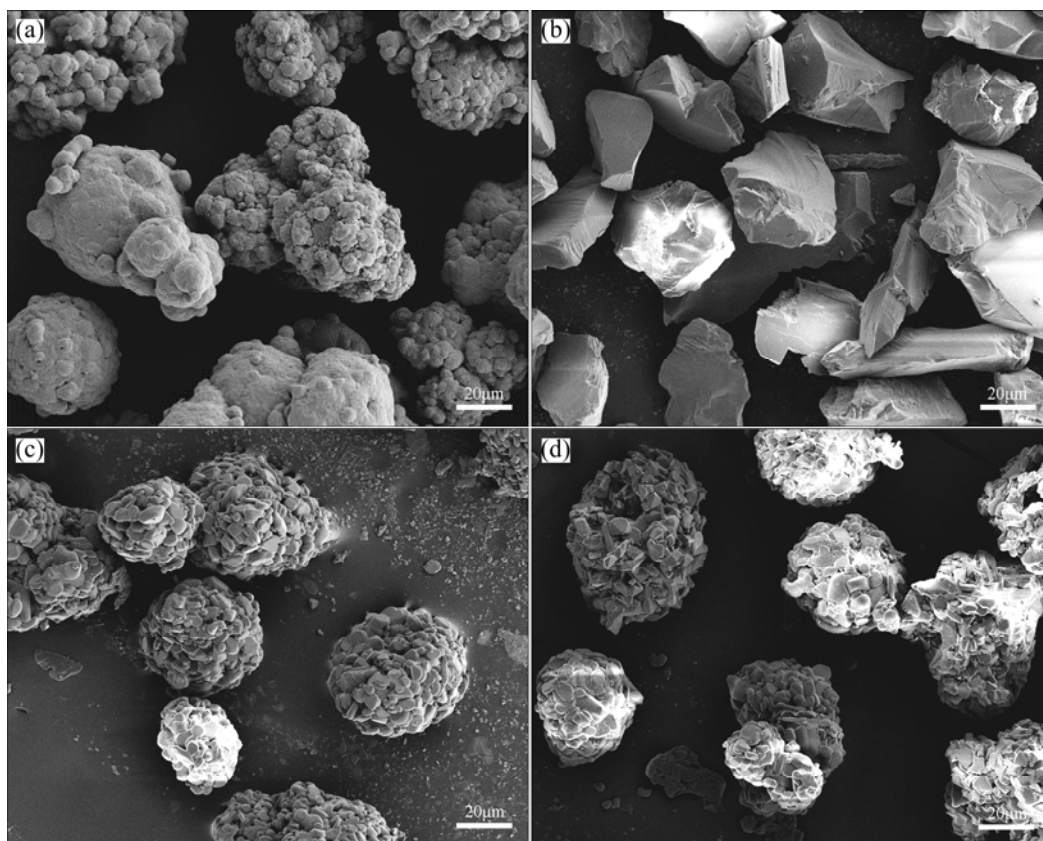


Fig. 6 SEM morphologies of powders: (a) Ni(Al); (b) Al₂O₃; (c) AT3; (d) AT13

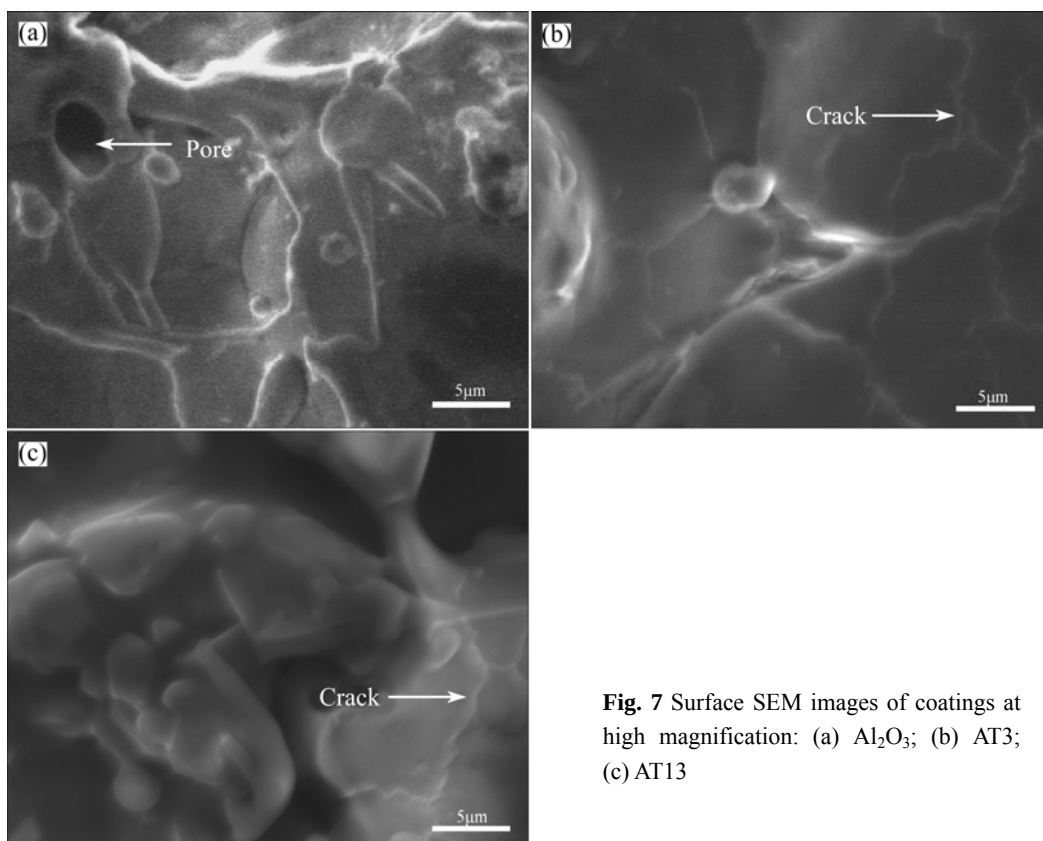


Fig. 7 Surface SEM images of coatings at high magnification: (a) Al₂O₃; (b) AT3; (c) AT13

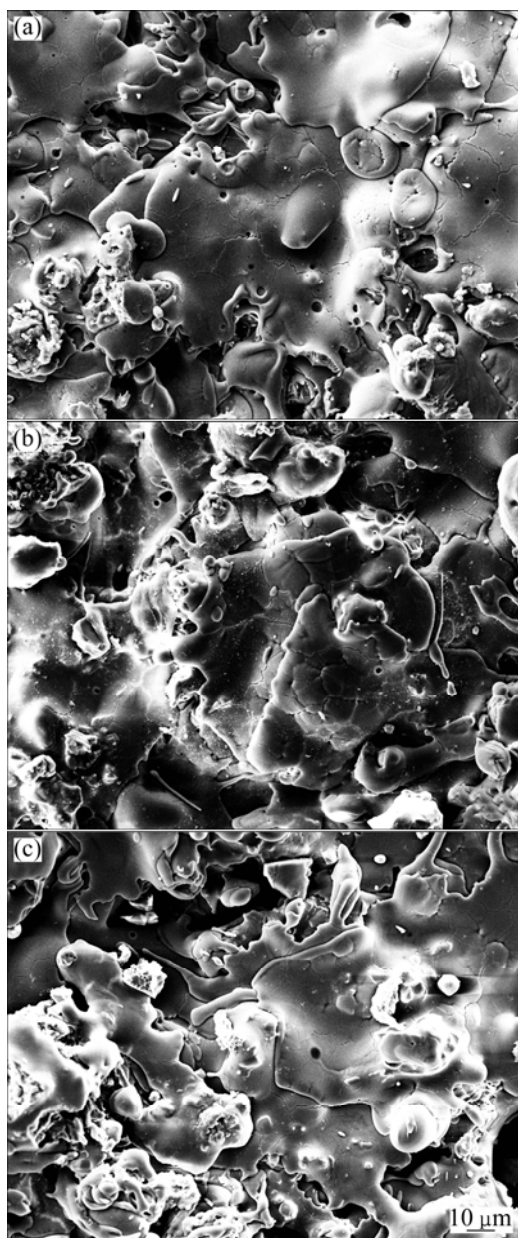


Fig. 8 Surface SEM images of coatings at low magnification: (a) Al_2O_3 ; (b) AT3; (c) AT13

coatings, and there is obvious difference of element content between substrate/bond coat or bond coat/top coat.

3.3 Corrosion resistance analysis

Figure 12 shows the potentiodynamic curves of the four samples (Al substrate, Al_2O_3 coating, AT3 coating and AT13 coating). The OCPs of substrate, Al_2O_3 coating, AT3 coating and AT13 coating are -0.78 , -0.80 , -0.88 and -0.87 V, respectively. With protection of coating for substrate, its φ_{corr} increases and corrosion current density decreases, indicating that its corrosion resistance is improved with protection of coating.

As can be seen in Fig. 12, the potentiodynamic

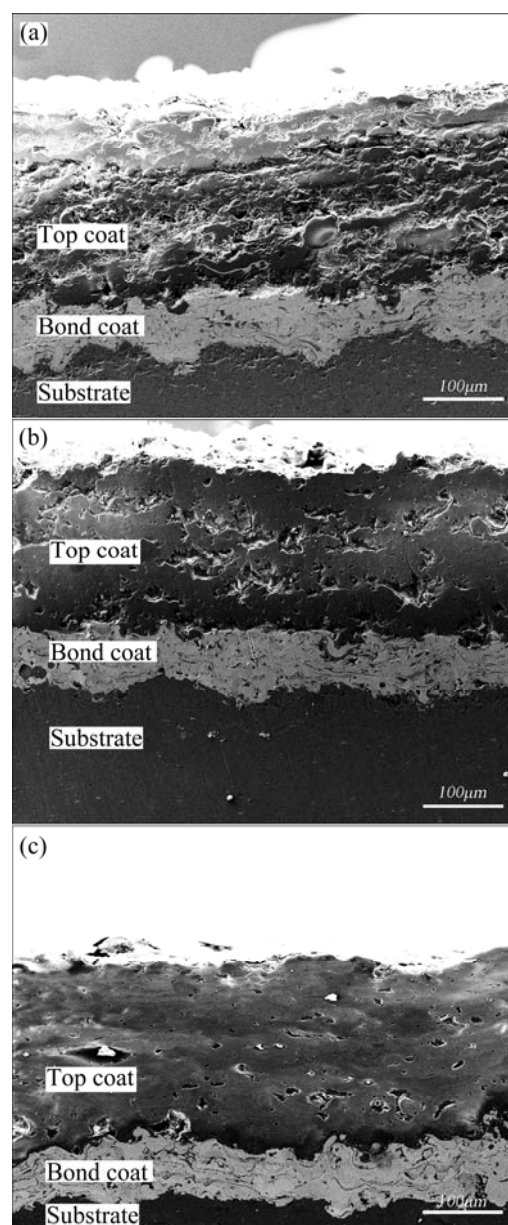


Fig. 9 Cross-sectional SEM images of coatings: (a) Al_2O_3 ; (b) AT3; (c) AT13

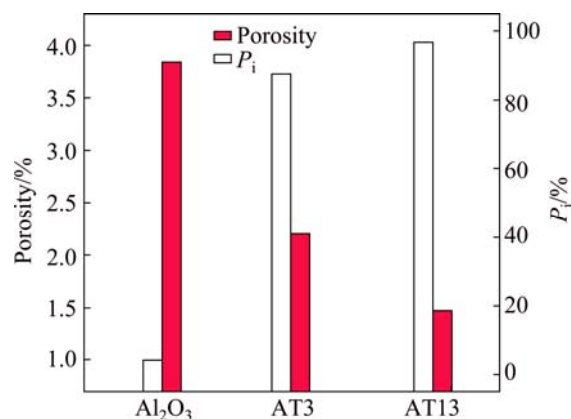


Fig. 10 Porosity and protective efficiency of three kinds of coatings

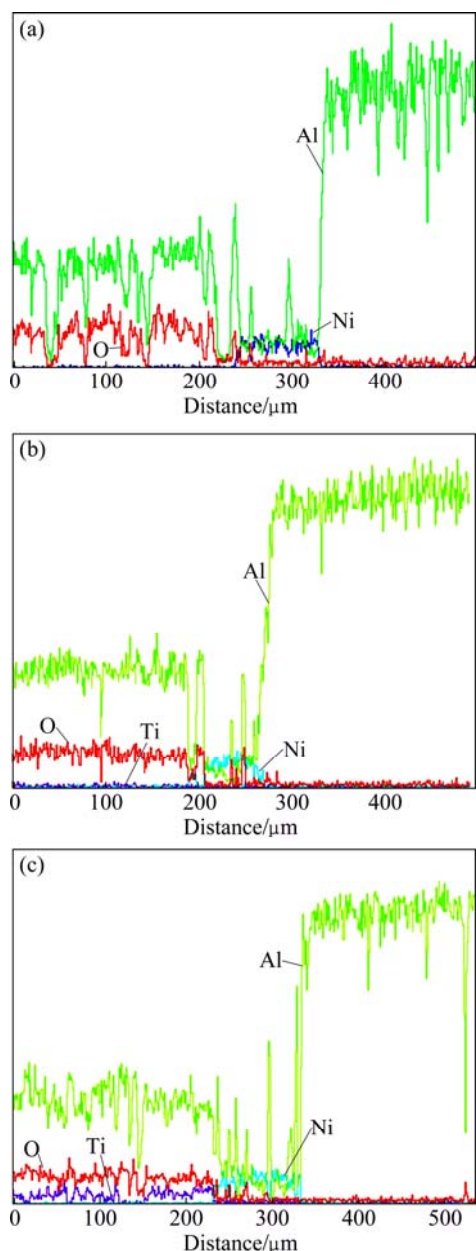


Fig. 11 EDX spectra of cross-sectional images of Al₂O₃ (a), AT3 (b) and AT13 (c)

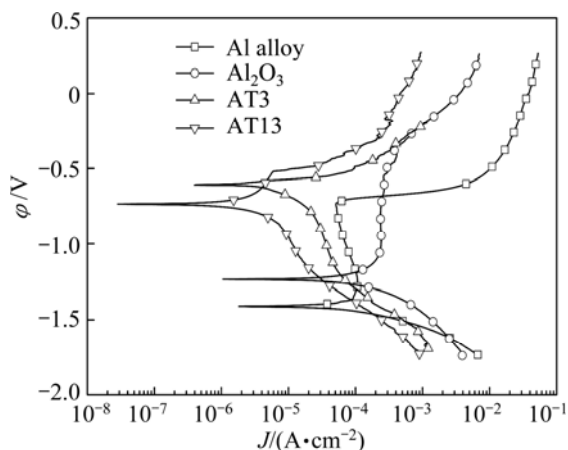


Fig. 12 Potentiodynamic curves of four samples

curves of substrate and Al₂O₃ have obvious passivation region, showing that they have undergone passivation phenomena. In generally, the Al substrate is easy to be passivated, and the dense Al₂O₃ should not have obvious passivation. However, the Al₂O₃ coating in this work also presents passivation. The possible reason is that Al₂O₃ coating has high porosity which leads to corrosive medium corroding the bond coat directly. From the potentiodynamic curves of AT3 and AT13, it can be seen that their passivations are not obvious. The main reason may be that TiO₂ with lower melting point plays a role of hole sealing. Meanwhile, the liquid TiO₂ disperses in the brittle solid Al₂O₃, leading to stress releasing and reducing the cracks. So it can be found that TiO₂ plays the role of hole sealing and reducing the cracks, making corrosive medium and substrate isolated, which prevents the passivation phenomenon.

After Tafel curve fitting, the corrosion resistance results of the four samples are given in Table 5. The results suggest that Al₂O₃ has the highest corrosion rate while AT13 has the lowest. Compared with uncoated substrate, the sample with coating has better corrosion resistance, and with the same coating parameters, the corrosion resistance of AT3 coating is higher than that of Al₂O₃ coating, but lower than that of AT13 coating.

Table 5 Corrosion resistance results of four samples

Sample	ϕ_{corr}/mV	$J_{\text{corr}}/(mA \cdot cm^{-2})$	$v_i/(mm \cdot a)$	$P_i/\%$
Al	-1.4099	0.0383	0.4492	—
Al ₂ O ₃	-1.2342	0.0369	0.4329	3.66
AT3	-0.6088	4.75×10^{-3}	5.57×10^{-2}	87.60
AT13	-0.7346	1.36×10^{-3}	1.59×10^{-2}	96.45

The protective efficiency of coatings, marked as P_i , can be calculated by

$$P_i = \left[1 - \left(\frac{J_{\text{corr}}}{J_{\text{corr}}^0} \right) \right] \times 100\% \quad (1)$$

where J_{corr} and J_{corr}^0 denote the corrosion current density of the coating and substrate [23], respectively. As shown in Fig. 10, with the decrease of porosity, the protective efficiency increases. The lower porosity will hinder corrosive medium corroding the bond coat, thus enhancing the protective efficiency.

3.4 Thermal insulation analysis

Figure 13 shows the dependence of temperature difference between the upper and lower surface of three kinds of coatings on time (after removal of the temperature of substrate and Ni(Al)). The temperature difference increases and reaches the steady state at 40 s. Al₂O₃ coating has the highest thermal insulation temperature of about 80 °C, and AT3 has better thermal

insulation temperature (approximately 70 °C) than AT13 (approximately 55 °C).

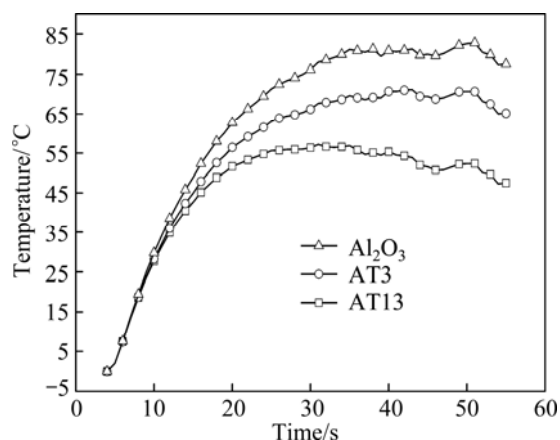


Fig. 13 Dependence of temperature difference between upper and lower surface of coatings on time

The thermal insulation property of the coating decreases with the increase of the content of TiO_2 in the powder. Thickness and thermal conductivity are two main factors to affect the thermal insulation property of coating. As the coatings have the same thickness in this work, the dominated reason for this phenomenon is the latter. One reason may be that TiO_2 has higher thermal conductivity than Al_2O_3 , meaning that increasing TiO_2 content will reduce the thermal insulation property of coating. Another one may be that the thermal conductivity of atmosphere is lower than Al_2O_3 , so the decrease of porosity because of hole sealing of TiO_2 will reduce the thermal conductivity of the coatings [24].

4 Conclusions

1) Phase transformation occurs during plasma spraying Al_2O_3 or Al_2O_3 and TiO_2 powders. The phase transformation in Al_2O_3 coating is $\alpha\text{-Al}_2\text{O}_3$ to $\gamma\text{-Al}_2\text{O}_3$. In AT3 coating, it is $\alpha\text{-Al}_2\text{O}_3$ and rutile TiO_2 to $\gamma\text{-Al}_2\text{O}_3$ and anatase TiO_2 . In AT13 coating, it is $\alpha\text{-Al}_2\text{O}_3$, rutile TiO_2 to $\gamma\text{-Al}_2\text{O}_3$ and anatase TiO_2 . TiO_2 will inhibit the transformation from $\alpha\text{-Al}_2\text{O}_3$ to $\gamma\text{-Al}_2\text{O}_3$, and with the increase of its content, the degree of inhibition is enhanced.

2) With the increase of TiO_2 content in the powder, the corrosion resistance of coating is enhanced. The possible reasons are that the melting point and brittleness of TiO_2 are lower than those of Al_2O_3 , so it is easy for TiO_2 to disperse in the brittle Al_2O_3 substrate during spraying, in which these dispersively distributed TiO_2 phases play a role of sealing hole, releasing stress and reducing cracks.

3) With the same thickness, the thermal insulation

property of coatings decreases with the increase of the content of TiO_2 in powder. The possible reasons are that TiO_2 has a higher thermal conductivity than Al_2O_3 , and these coatings have different porosities.

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TiO_2 含量对等离子喷涂 Al_2O_3 热障涂层性能的影响

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摘 要: 采用等离子喷涂技术在 6061 铝合金表面制备了不同含量 TiO_2 的氧化铝陶瓷涂层, 研究了涂层的相组成、隔热性能及耐腐蚀性。实验结果表明: 在喷涂过程中陶瓷层中均有物相的转变。随着粉末中 TiO_2 含量的增加, 涂层的耐腐蚀性增强但是隔热性下降。这可能是由于 TiO_2 的导热系数比 Al_2O_3 的导热系数高, 但其熔点比 Al_2O_3 的熔点低, 同时脆性较小, 在喷涂过程中, TiO_2 弥散分布在脆性的氧化铝基体中起到了封孔及释放应力、减少裂纹的作用。

关键词: 等离子喷涂; 耐腐蚀性; 隔热性能; 相变; TiO_2

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