

## Oxidation Behavior of Al-Y Coating on $\gamma$ -TiAl at 900 °C

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### ABSTRACT

In this study, an Al-Y coating was applied to enhance the property of oxidation resistance on the surface of  $\gamma$ -TiAl alloys by rf magnetron sputtering. The isothermal oxidation tests were investigated at 900 °C for 100 h. The coating characteristics and microstructure of coating after isothermal oxidation were inspected by XRD and SEM methods respectively, and coating bonding strength was investigated by scratch tester. Results show that  $\text{Al}_2\text{O}_3$  and  $\text{Y}_2\text{O}_3$  were produced on the surface of  $\gamma$ -TiAl alloys to enhance oxidation resistance during the isothermal oxidation test. Diffusion layer with rich element of Al provides abundant Al to surface for formation of a continuous  $\text{Al}_2\text{O}_3$  layer which increases oxidation resistance effectively. Oxides of  $\text{YAlO}_3$  and YAG were formed during the oxidation tests, which were beneficial for the strength, hardness and fracture toughness of crystal grain of  $\text{Al}_2\text{O}_3$ . Furthermore, Element of Ti diffused into surface from substrate was restrained by Al because of the formation of  $\text{TiAl}_3$  and TiAl, which avoid the layer-broken by titanium oxides with porous structure

**Keywords:** Oxidation behavior; Magnetron sputtering; High temperature; Al-Y coating;  $\gamma$ -TiAl alloy; Diffusion

### 1. INTRODUCTION

$\gamma$ -TiAl alloys are widely used in aero engines and automotive components due to the outstanding creep properties (more than 900 °C), high melting point which is more than 1450 °C, and the preminent specific modulus that is around 160 GPa to 180 GPa [1]. However,  $\gamma$ -TiAl alloys are limited to apply in industry because of inadequate oxidation resistance [2-6]. Therefore, a lot of researches were carried out to enhance the property of oxidation resistance. Additional elements such as Si, Cr and Nb can measurably increase the oxidation resistance, but mechanical properties of these materials will be deteriorated in the process of further alloying. Thus, surface modification technique can be an excellent method which contributes to form steady oxide scales to increase oxidation resistance of  $\gamma$ -TiAl alloys, and make sure it can be used steadily in high temperature environment.

Various attempts on surface treatment of TiAl alloy have been applied to improve the property of oxidation resistance [7-10]. M Taheri et al. [11] have been succeeded in forming NiCrAlY or CoNiCrAlYSi coating on the surface of TiAl alloy to improve the oxidation resistance. However, the formation of  $\text{TiO}_2$  on the surface of TiAl alloy is not good enough to protect the substrate. Mutual infiltration of the coatings and TiAl alloy was rigorous. The interdiffusion of elements can induce to form brittle interlayer, and the new formation debases the adhesive force between coatings and substrate at high temperature [12-15].

Furthermore, ceramic coatings were used in TiAl alloy to improve the property of oxidation resistance. Such as  $\text{Cr}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ , which have been used to enhance the long time steady application in high temperature environment [16-22]. Nevertheless, smash and fragmentation of ceramic coatings cannot be averted because of the diffusion and lose of salutary elements in high temperature environment [23-25].

About the methods, there are many techniques to prepare excellent coatings, such as magnetron sputtering, electron deposition and CVD [26-29]. The magnetron sputtering is one of the easiest ways for forming film and coating among these methods. Based on high ionisation of argon gas, the ions strike target as well as elements of the target were sputtered toward workpiece. So, beneficial layer was formed on the surface of workpiece [30,32].

In this paper,  $\gamma$ -TiAl alloy were subjected to coating via rf magnetron sputtering. A preminent coating was applied on the surface of TiAl alloy to improve the property of oxidation resistance. The aim of the study is to reveal Al-Y coating and demonstrate the effect of Al-Y layer on surface of  $\gamma$ -TiAl in oxidation resistance.

## 2. MATERIALS AND METHODS

The base material was  $\gamma$ -TiAl alloy with the chemical composition contained 46.5% Al, 2.5% V, 1% Cr and balance Ti, which was purchased from Baoji Hong Ding metal materials Co., Ltd. The  $\gamma$ -TiAl alloy was cut in cuboid (10mm  $\times$  10mm  $\times$  8 mm) by wire cutting. Samples used in research were polished by waterproof abrasive paper of 1200 meshes, and then cleaned in acetone by ultrasound equipment to dislodge any surface contamination.

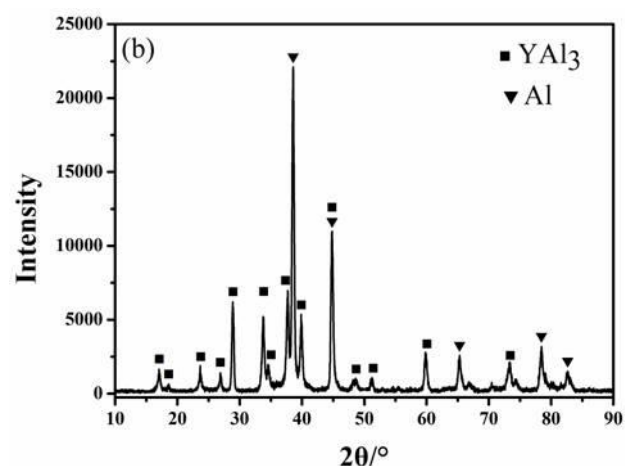
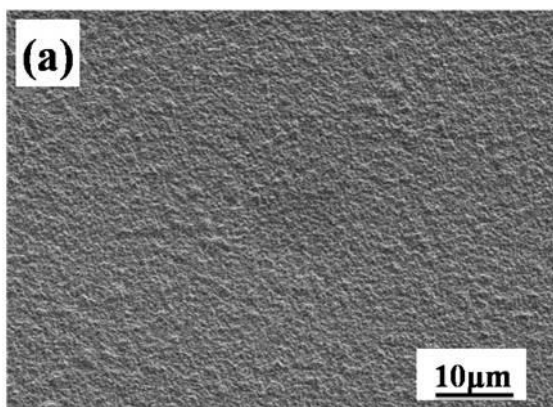
The coating of Al-Y was fabricated via rf magnetron sputtering. Argon was provided as the background gas during the process. The deposition power and duration of the Al-Y layer was 200 W and 4 h, respectively. Pressure was in the range of 4 -6 Pa. The distance between target and sample was 25 mm. Al-Y alloy with the chemical composition of 70% Al and 30% Y was used as the target with the diameter of 90 mm. Finally, vacuum annealing was applied on the specimen at 600 °C for 10 h.

After vacuum annealing process, three pieces of Al-Y coated samples were detected by isothermal oxidation test during 100 h at the temperature of 900 °C. The microstructure of Al-Y coating was examined by a machine named Scan electron microscopy (FEI, Quanta450, USA). The X-ray diffraction (Rigaku, DMAX-RB12KW, Japan) with copper K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) was applied to determine the main crystal phase compositions of Al-Y coating. Scratch tester (BangYi, WS-2005, China) was used to measure the adhesion strength between Al-Y layer and  $\gamma$ -TiAl alloy.

## 3. RESULTS AND DISCUSSION

### 3.1 Coating characterization

Fig. 1 is the surface morphology of the Al-Y coating. The SEM pictures show that Al-Y coating is dense and uniform (Fig. 1a), and there are no drawbacks like holes and cracks on the surface or interface. Fig. 1b shows the XRD pattern of the specimens, and angle of X-ray diffraction is 10° to 90°. The XRD results manifested that the existing phases of Al-Y layer were Al and YAl<sub>3</sub>. The cross-section morphology of Al-Y coating shows that the thickness of Al-Y coating is 19.5  $\mu\text{m}$ , and content of Al and Y remained unchanged at the range of 0 -16.2 $\mu\text{m}$ . At the range of 16.2 -19.5 $\mu\text{m}$ , elements of Al and Y diffused into substrate, which contribute to enhance the bonding strength between coating and substrate. During magnetron sputtering treatment, ion bombardments aggrandize vacant sites to enhance the coating bonding strength. And the diffusion element distribute raked in the Al-Y coating and interfaces [33].



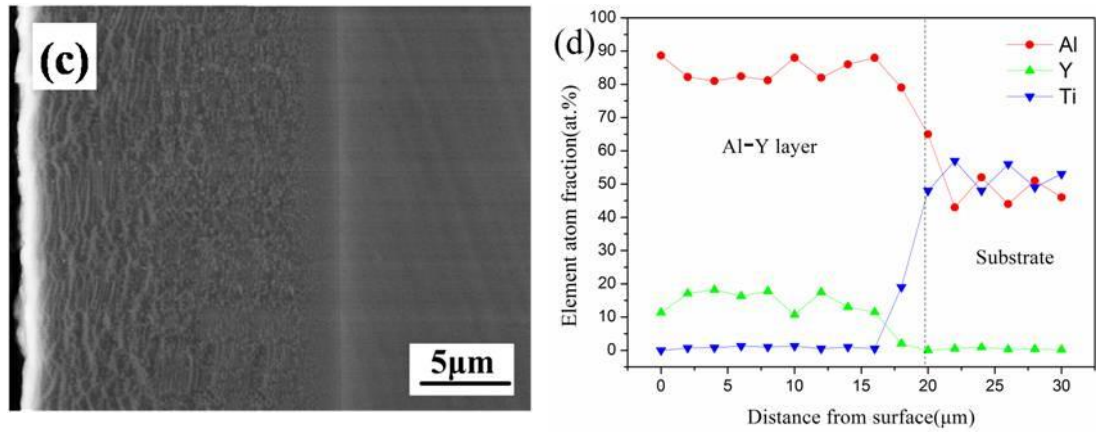


Figure 1: (a) surface sem picture, (b) xrd picture, (c) cross-section picture and (d) line-scanning picture of al-y coating.

### 3.2 Micro-Structure analysis of Al-Y coating after oxidation test

Surface morphology of  $\gamma$ -TiAl alloy without Al-Y coating are exhibited in Fig. 2(a) after isothermal oxidation test at 900 °C for 100 h. Substrate without Al-Y coating was destroyed seriously by Oxygen and loose oxide layer appear on the surface of it. Fig. 2(c) shows the surface morphology of the Al-Y layer undergoes isothermal oxidation test at 900 °C for 100 h. No holes or cracks can be found on sample with Al-Y coating. Mountain-like hillocks and pits on the surface of the film were discovered. The reason is that phase transition of  $\text{Al}_2\text{O}_3$  crystal phase occurred ( $\theta$ - $\text{Al}_2\text{O}_3$  to  $\alpha$ - $\text{Al}_2\text{O}_3$ ), and shrinkage stress from volume change during phase transition process led to wrinkling of the film. The results analyzed with XRD (Fig. 4) manifest that substrate without Al-Y coating oxidized seriously because of oxide of  $\text{TiO}_2$  produced with porous structure, and the considerable solubility of oxygen in Ti and weak bonding between many Ti-O oxides. However, Al-Y coating reacts with oxygen, and dense layer consisted of  $\text{Al}_2\text{O}_3$  and  $\text{Y}_2\text{O}_3$  was produced to control the diffusion of oxygen during the oxidation test. Thus, oxidation resistance of  $\gamma$ -TiAl alloy is increased ideally.

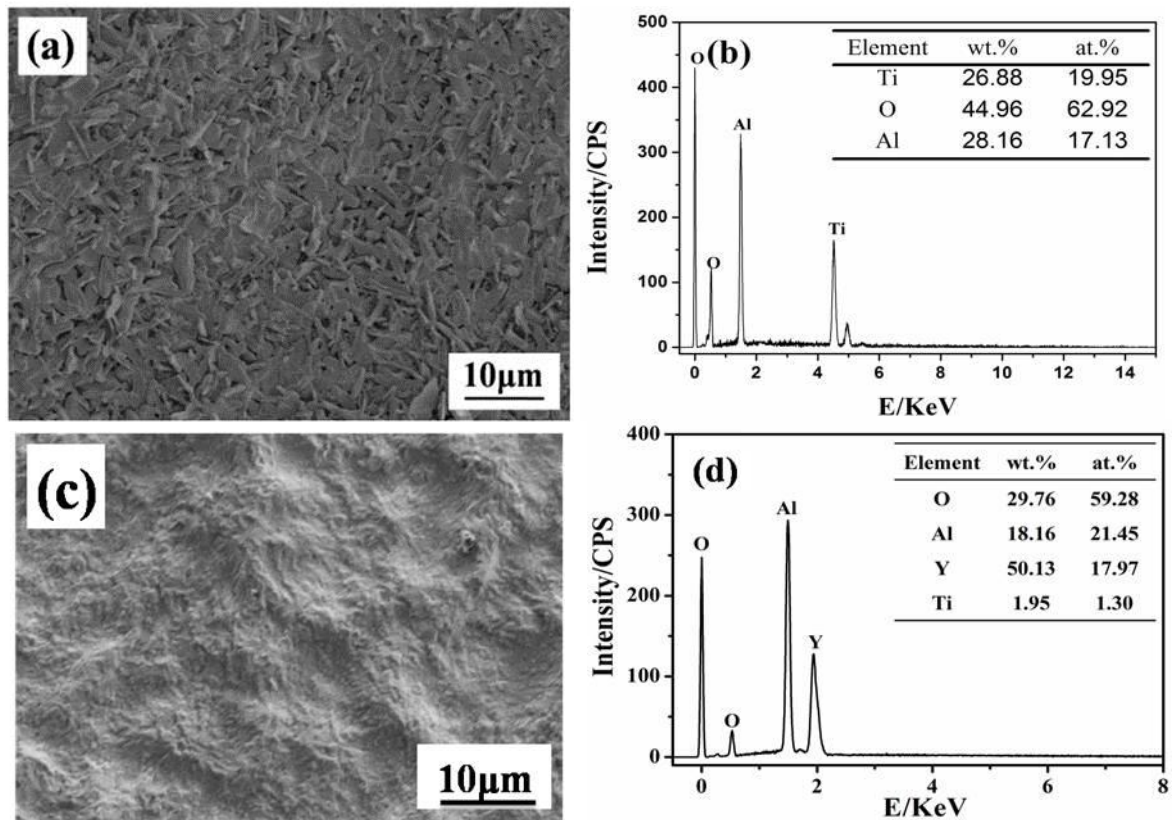
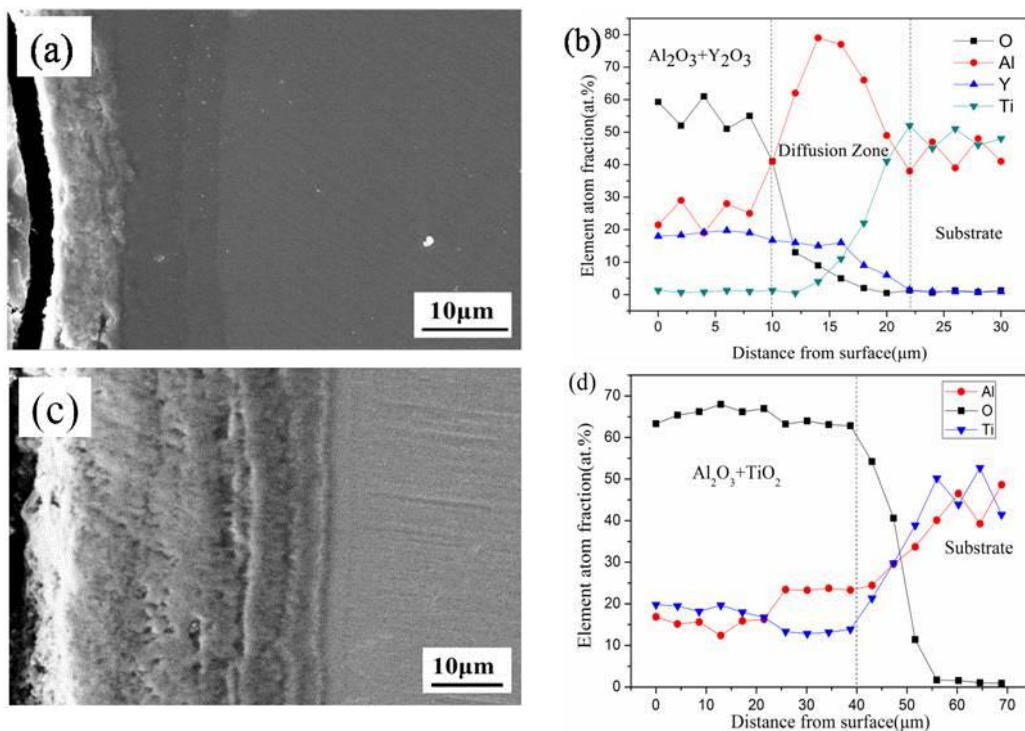


Figure 2: Surface pictures and EDS pictures after oxidation test(a), (b) substrate and (c), (d) Al-Y coating.

The cross-section morphology and EDS results of  $\gamma$ -TiAl alloy with and without Al-Y coating after isothermal oxidation test at 900 °C for 100 h are shown in Fig. 3. The oxide layer of substrate with holes and cracks was discovered, and substrate sample was destroyed seriously by Oxygen and loose oxide layer appear on the surface of it. The EDS results indicated that thickness of oxide layer was 40 $\mu$ m, and at the range of 40 -56 $\mu$ m, the content of oxygen reduced sharply. However, the oxide layer of  $\gamma$ -TiAl alloy with Al-Y coating was existent without holes and cracks, and gradient layer with Al was shown under the oxide layer. The EDS result (Fig.3 b) compared with Fig.1d indicated that oxygen diffused into Al-Y layer and reacted with Al and Y to form oxide in the range of 0 -10 $\mu$ m. The content of oxygen reduced sharply in the layer with rich Al at the range of 10 -21 $\mu$ m. The diffusion distance of Y was 22.3 $\mu$ m, and this relatively further distance compared with the coating before oxidation test (about 19.5 $\mu$ m in Fig.1d). Y and Al diffused into substrate furtherly because of the high temperature treatment during oxidation test. The outward diffusion of Ti was suppressive due to the integration of Al-Ti intermetallics such as TiAl and TiAl<sub>3</sub>, which was verified by the XRD result.



**Figure 3:** Cross-section pictures and line-scanning pictures after oxidation test (a), (b) Al-Y coating and (c), (d) substrate.

Fig. 4 shows the XRD patterns on the surface of specimens with and without Al-Y coating after isothermal oxidation at 900 °C for 100 h. The result (Fig. 4b) analyzed with Fig. 2(c) shows that that a dense film of Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> were formed to protect substrate. Furthermore, crystal phases contained YAlO<sub>3</sub> and YAG (Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>), and YAlO<sub>3</sub> is metastable phase which reacts with Al<sub>2</sub>O<sub>3</sub> to YAG at high temperature. Melting point of YAG is 1940 °C with higher strength and creep resistance at high temperature. The strength, hardness and fracture toughness of crystal grain of Al<sub>2</sub>O<sub>3</sub> were improved by YAG.



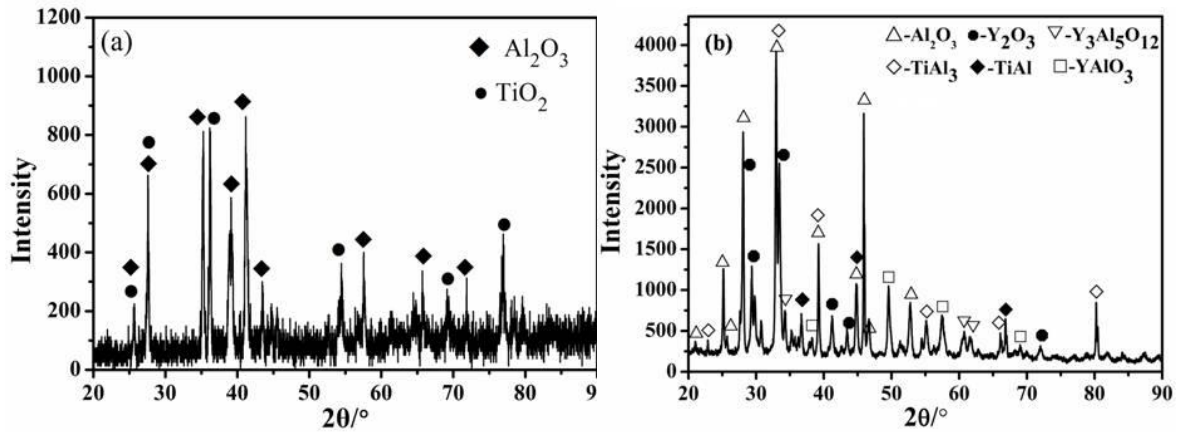


Figure 4: XRD patterns after isothermal oxidation test (a) substrate, (b) Al-Y coating.

### 3.3 Oxidation mechanism

Fig. 5 represents mass change curves of  $\gamma$ -TiAl alloy with and without Al-Y coating after isothermal oxidation test at 900 °C. The samples with and without Al-Y coating were tested under same conditions and were charted in same image for comparison. Results indicate that the mass gain of  $\gamma$ -TiAl alloy without Al-Y coating is more than 17.724 mg cm<sup>-2</sup> after 100 h of oxidation test, which indicates that oxidation resistance of substrate is poor. The main reason of mass gain of the sample without Al-Y coating is surface oxidation. Nevertheless, mass gain of  $\gamma$ -TiAl alloy with Al-Y coating is 3.725mg cm<sup>-2</sup> after 100 h, which is less than the sample without Al-Y coating. The result of mass change curve indicates that Al-Y coating exhibits wonderful oxidation resistance at 900 °C.

Fig. 6 illustrates the changes of coating according to results of XRD and SEM during oxidation test. The outward diffusion of Ti element and inward diffusion of Al and Y elements lead to a gradient zone. Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> were formed to protect titanium alloy from oxygen on the surface, diffusion layer with rich element of Al provided abundant Al to surface for formation of a continuous Al<sub>2</sub>O<sub>3</sub> layer. Y and Al diffused into substrate to enhanced bonding strength between coating and substrate. Element of Ti diffused to surface and was restrained by Al because of the formation of TiAl<sub>3</sub> and TiAl. In gradient zone, oxides of Al-Y are beneficial for Al<sub>2</sub>O<sub>3</sub>.

In a word, this specific structure contributes to spallation resistance, and enhances the property of oxidation resistance of  $\gamma$ -TiAl.

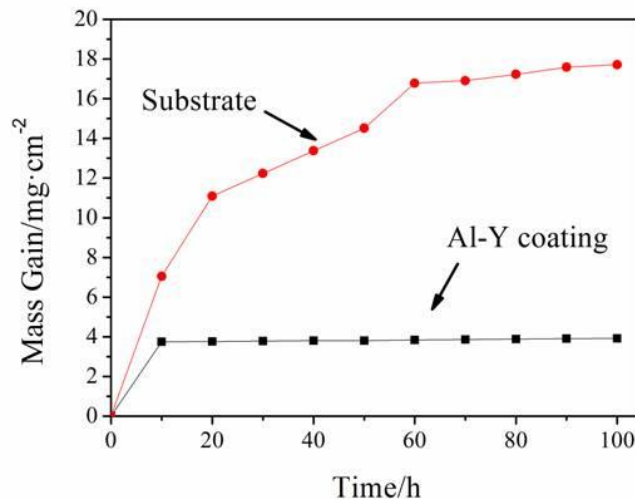


Figure 5: Mass change curves under isothermal oxidation test.

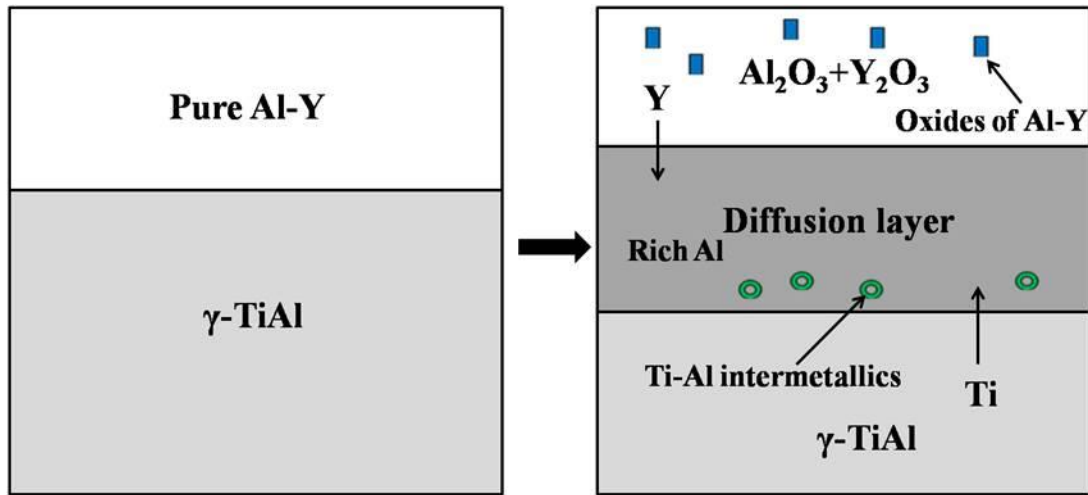


Figure 6: Schematic illustration for element evolution mechanism of Al-Y coating during oxidation test.

### 3.4 Scratch tester

Availability of Al-Y protective coating was provided by the bonding strength between coating and substrate during the process of service. Thus, scratch test was performed via WS-2005 scratch tester. After the breaking off coating, acoustical signal was produced by acoustic emission sensor, and translated into curve after amplification treatment. Acoustical signal began to wave when cracks appeared on the surface of material. Cracks were enlarged from crystal boundary and vacancy in Al-Y coating without desquamation. Fig. 7a is the picture of acoustic emission curve which indicates that bonding strength between Al-Y coating and substrate is 51.8 N, and the bonding strength between coating and substrate is universally adequate for application [24, 25]. Fig. 7b shows the morphology of Al-Y coating after scratch test. Scratch was uniformly distributed without disbanding. When the elements of Al and Y were sputtered on the substrate and the temperature of isothermal oxidation test was increased, atomic migration occurred by kinetic energy and heat energy, and Al and Y diffused into substrate to improve bonding strength between coating and substrate. The proof of deformation from Al-Y coating scratch test reveals that bonding strength between Al-Y coating and substrate is great, and Al-Y coating with perfect quality was produced.

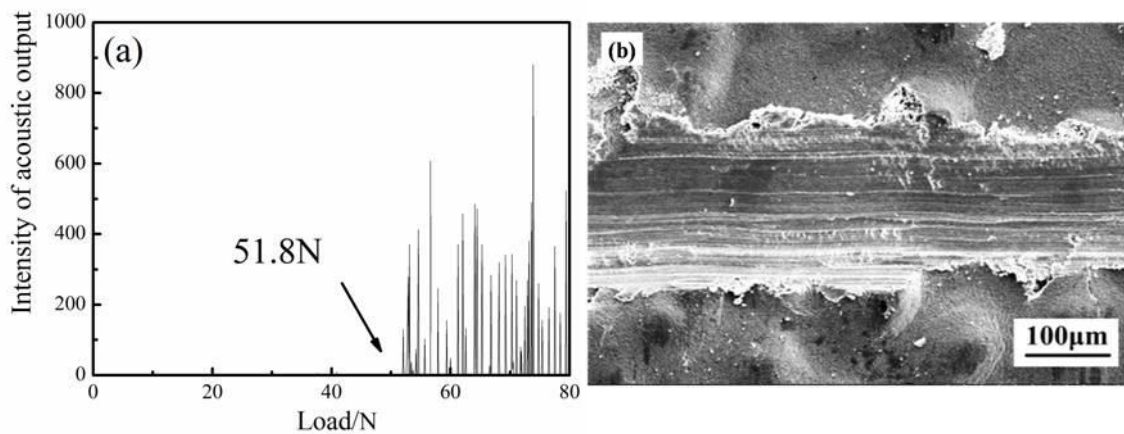


Figure 7: Acoustic emission curve and scratch morphology of Al-Y coating.

### 4. CONCLUSIONS

In this work, the Al-Y coating was fabricated on the surface of  $\gamma$ -TiAl to enhance the property of oxidation resistance, and isothermal oxidation tests were experimented at 900 °C for 100 h. Results show that  $\text{Al}_2\text{O}_3$  and  $\text{Y}_2\text{O}_3$  were formed to protect titanium alloy from oxygen. Diffusion layer with rich element of Al provides abundant Al to surface for forming a continuous  $\text{Al}_2\text{O}_3$  layer. Element of Ti diffusion to surface from substrate was restrained by Al because of the formation  $\text{TiAl}_3$  and  $\text{TiAl}$ . In diffusion zone,  $\text{YAlO}_3$  and  $\text{YAG}$

were beneficial for the strength, hardness and fracture toughness of crystal grain of Al<sub>2</sub>O<sub>3</sub>. Mountain-like hillocks and pits on the surface of the film were discovered because of the phase transition of Al<sub>2</sub>O<sub>3</sub> crystal phase.

## 5. ACKNOWLEDGMENTS

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