

# A novel $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite coating on $\gamma$ -TiAl alloy and evaluating the oxidation performance



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## ABSTRACT

A novel  $\text{TiAl}_3/\text{Al}_2\text{O}_3$  composite coating was prepared on  $\gamma$ -TiAl alloy. The process included two steps: (1)  $\text{TiAl}_3/\text{Al}_2\text{O}_3$  composite powders were prepared by high energy ball milling of pure Al and nano- $\text{TiO}_2$  powders, followed by a heat-treatment; (2) the as-prepared composite powders were deposited on  $\gamma$ -TiAl substrate by cold spray. The cyclic oxidation was conducted at 900 °C to test the performance of the composite coating. The results showed that the composite coating had good crack resistance and effectively decreased the oxidation rate of the substrate.

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## 1. Introduction

$\gamma$ -TiAl based alloys possess a favorable combination of low density, high stiffness, high yield strength and good creep resistance. Therefore, they are promising candidates for use in advanced structural applications, such as parts in automotive engines and turbine blades in aircraft engines [1,2]. However, the poor oxidation resistance at temperature above 750 °C has hindered their practical engineering applications [3,4].

$\text{TiAl}_3$  coating which can form a stable and continuous  $\text{Al}_2\text{O}_3$  scale during oxidation has attracted great concerns because of its good compatibility with  $\gamma$ -TiAl substrate. Aluminizing such as pack cementation and hot dip process is one of preparation methods of  $\text{TiAl}_3$  coating [5,6]. The other one is deposition of an aluminum layer followed by a heat-treatment, such as sputtering aluminum [7], electroplating aluminum [8], thermal spray aluminum [9], and cold spray aluminum [10,11]. The formation of  $\text{TiAl}_3$  phase of these two methods is attributed to the reaction between the inward diffusion of Al and  $\gamma$ -TiAl substrate. Therefore, the  $\text{TiAl}_3$  coating prepared by these two methods is a kind of diffusion coating.

This conventional  $\text{TiAl}_3$  diffusion coating is apt to produce cracks because of its low ductility and high coefficient of thermal expansion (CTE) comparing to that of  $\gamma$ -TiAl alloy [12,13]. Therefore, how to enhance the toughness of  $\text{TiAl}_3$  is a main matter of concern.

In the study of  $\text{NiAl}/\text{Al}_2\text{O}_3$  composite coating, the toughness and strength of  $\text{NiAl}$  can be improved by adding  $\text{Al}_2\text{O}_3$  particles [14–16]. However, there are no reports on the study of the performance of  $\text{TiAl}_3/\text{Al}_2\text{O}_3$  composite coating because it is impossible to prepare  $\text{TiAl}_3/\text{Al}_2\text{O}_3$  composite coating by conventional diffusion method. In this paper, a novel overlay  $\text{TiAl}_3/\text{Al}_2\text{O}_3$  composite coating was prepared on  $\gamma$ -TiAl alloy by high energy ball milling and cold spray. The performance of the composite coating was tested by a cyclic oxidation at 900 °C.

## 2. Experimental

The nominal composition of the  $\gamma$ -TiAl substrate was Ti-47Al-2Cr-2Nb-0.15B (at.%), which was provided by Titanium Alloys Division, Institute of Metal Research, CAS. The raw materials used for high energy ball milling was pure Al and  $\text{TiO}_2$  powders. Al powders (Changsha Tianjiu metallic materials Co., Ltd, China) were sieved to –400 + 500 mesh.  $\text{TiO}_2$  powders were P25 without any further treatment.

Al/ $\text{TiO}_2$  composite powders (40 wt.%  $\text{TiO}_2$ ) were fabricated by high energy ball milling using of a shaker mill (QM-3A, Nanjing NanDa Instrument Plant, China). The high energy ball milling was done with 5 mm agate balls, ball-to-powder mass ratio of 5:1, milling speed of 1200 rpm, and total milling time of 2 h. After high energy ball milling, the as-prepared composite powders were subjected to a heat treatment at 750 °C for 8 h in an annealing furnace under argon gas flow of 40 ml/min. After the heat treatment, the composite powders turned to be agglomerate. Therefore, the

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agglomerates were smashed using the high energy ball milling for 15 min as aforementioned conditions.

The heat-treated powders were deposited on  $\gamma$ -TiAl substrate by cold spray. The cold spray device was manufactured by Institute of Metal Research, CAS. The De Laval nozzle was a rectangle exit equipped with a cross section of 2 mm  $\times$  10 mm and a throat of 2 mm  $\times$  2 mm. The parameters for the cold spray process were 550 °C, 2.0 MPa, and 10 mm for gas temperature, gas pressure and standoff distance, respectively.

The cyclic oxidation tests were conducted in a muffle furnace at 900 °C for 100 cycles. The samples were kept at 900 °C for 1 h and cooled down to room temperature for 15 min as a cycle. The mass changes of all samples were measured at regular intervals with a balance. The sensitivity of the balance is  $10^{-5}$  g.

X-ray diffraction (XRD) analysis was conducted on D/max-2500pc (RIGAKU, Japan). Scanning electron microscopy (SEM-EDS) imaging was obtained using JSM-6301F (SHIMADZU, Japan) and Inspect F50 (FEI, USA).

### 3. Results and discussion

#### 3.1. Morphology and microstructure of the composite powders

Fig. 1 shows the SEM images of ball-milled Al/TiO<sub>2</sub> composite powders (a)(b) before and (c)(d) after the heat-treatment. Fig. 2 shows the XRD pattern of ball-milled Al/TiO<sub>2</sub> composite powders (a) before and (b) after the heat-treatment. From Fig. 1(a), it could be seen that the as ball-milled particles exhibited irregular shapes with an average size of 60  $\mu$ m. From the cross-section of the particle in Fig. 1(b), the microstructure of the particle was homogeneous without observable second phase. It indicated that the chemical composition of the particle was uniform. During the high energy ball milling, ductile aluminum particles were successively flattened, cold welded, fractured and re-welded while the brittle nano-TiO<sub>2</sub> particles were difficult to deform. So they were trapped and embedded in the aluminum particles. With continued milling, the nano-TiO<sub>2</sub> particles got uniformly dispersed in aluminum particles [17,18]. From the XRD pattern as shown in Fig. 2(a), it could be

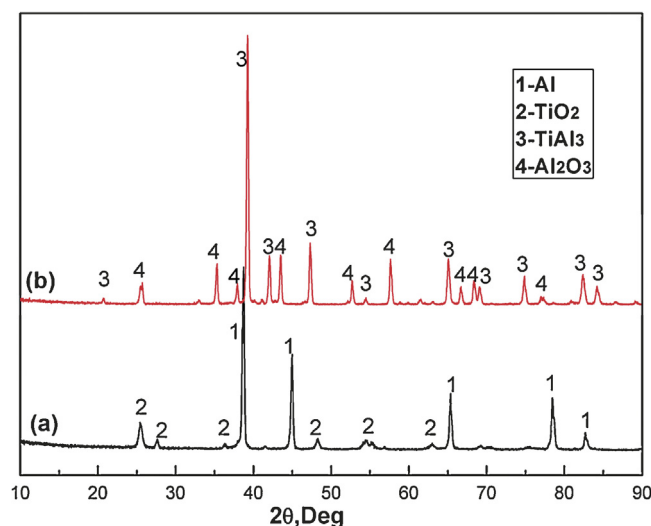
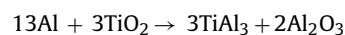


Fig. 2. XRD pattern of ball-milled Al/TiO<sub>2</sub> composite powders (a) before and (b) after the heat-treatment.

seen that the diffraction peaks of Al and TiO<sub>2</sub> phases were observed and no other diffraction peaks were found, which indicated that no reaction occurred during the high energy ball milling.

From Fig. 1(c), the shapes and size of the particles after the heat-treatment were similar to that of the as ball-milled particles. The particle was composed of two phases as shown in Fig. 1(d). The EDS analysis indicated that the gray parts (Ti: 23.0 at.%, Al: 64.3 at.%, O: 12.7 at.%) were TiAl<sub>3</sub>-rich and the dark parts (Ti: 6.1 at.%, Al: 48.3 at.%, O: 45.6 at.%) were Al<sub>2</sub>O<sub>3</sub>-rich. The XRD pattern in Fig. 2(b) further demonstrated that the particle was composed of TiAl<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> phases. The formation of TiAl<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> phases was attributed to the following reaction:



It was reported that the formation of Al<sub>2</sub>O<sub>3</sub> and the titanium-rich phases required the composite powders to be heated to a

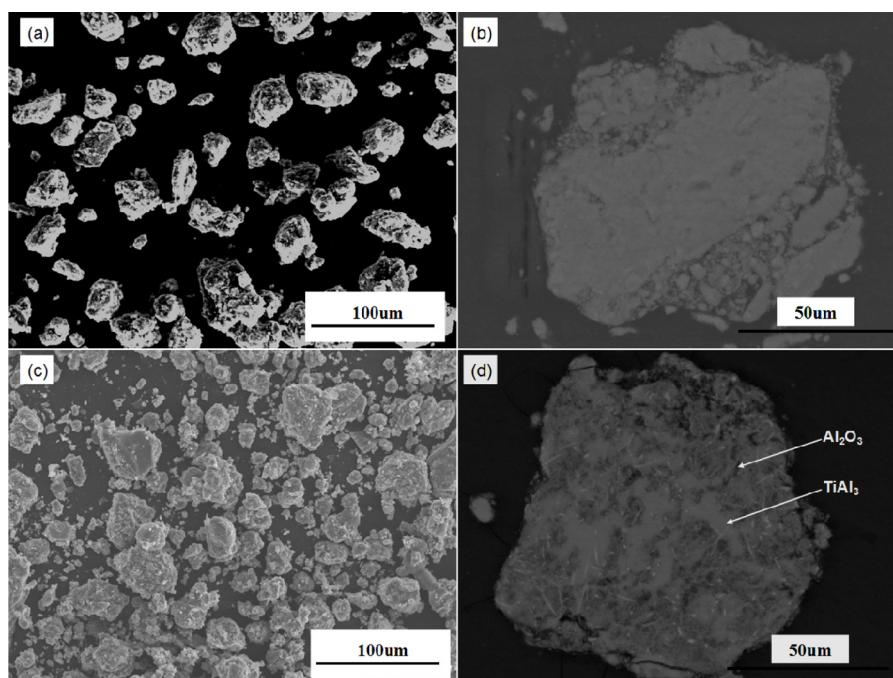


Fig. 1. SEM images of ball-milled Al/TiO<sub>2</sub> composite powders (a)(b) before and (c)(d) after the heat-treatment.

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