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Fretting Wear Behavior of Double-glow Plasma Cr-Nb Coating on γ-TiAl alloy

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Abstract: Double-glow plasma (DGP) coatings are recommended for metallic components to mitigate the damage induced by complex loading conditions. In this paper, Cr-Nb alloyed layer was formed onto the TiAl substrate via a double-glow plasma process to enhance its anti-fretting wear performance. Nano-indentation and scratch tests were used to evaluate the mechanical properties of the coating. The fretting wear behavior of the coating was investigated using a pin-on-plate fretting rig by rubbing against the Si_3N_4 ball. The 7μm thick Cr-Nb coating was well bonded to the substrate with the 2μm thick diffusion layer. The hardness of the coating was 9.5 GPa, which was 1.6 times greater than that of the uncoated TiAl substrate. Scratch tests showed that the critical load of Cr-Nb coating was 17.6 N. The fretting wear mechanism of the coatings was discussed in detail in this paper.

Keywords: TiAl; Double-glow plasma; Nano-indentation; Fretting wear; Chromium-Niobium.

1 Introduction

Intermetallic alloys are an emerging class of materials which may serve in a variety of structural material applications[1]. Three classes of intermetallics have actually matured sufficiently to offer a competitive balance of properties in the structural materials area: the $L1_2$ structured nickel aluminides[2], the B2 structured iron aluminides[3], and the $L1_0$ structured gamma titanium aluminides (γ-TiAl)[4]. Among these intermetallic compounds, γ-TiAl alloys are especially attractive, due to their low density, high specific yield strength, high specific stiffness, good oxidation resistance, flame retardancy and good creep properties. They also represent an example of how fundamental and applied research can go along with industrial development, leading to development of a new class of advanced engineering materials[4, 5].

γ-TiAl alloys show good potential for low-pressure turbine blades applications[6]. General Electric company for example, equipped the last stages of their low pressure turbine GEnX jet engine with cast TiAl blades [4, 7, 8]. However, γ-TiAl blades might be subjected to fretting damage at the dovetail joint, resulting from the natural high frequency blade vibration and alternating centrifugal force. The most damaging effect of fretting is significant reduction in fatigue capability of the fretted components. It was reported that the reduction in fatigue strength of Ti-47Al-2Nb-2Mn with 0.8 vol.% TiB_2 by fretting can be as much as 20%[6]. Careful blade design can reduce fretting damage in most cases, but not completely eliminate it, due to the angled blade–disk dovetail attachment geometry promoting micro-slip motion. Hence, surface engineering solutions like ion implantation[9] and physical vapor deposition[10] are applied as an efficient and economically viable solution. Among them, the double-glow plasma (DGP) surface alloying technique, known as Xu-Tec process[11], is an effective method of improving the micro-hardness, oxidation resistance and wear resistance of metals and alloys. In recent years, Cr and Nb coatings have been used to improve mechanical and tribological properties of TiAl alloys. The mechanism by which TiAl alloy is being strengthen is by replacement of Ti and Al atoms by Cr atoms in the Ti-Al-Cr crystallographic compound, resulting in increase of hardness[12, 13]. Moreover, Nb has a high alloying capacity; it improves the microstructure stability and provides high temperature strength[14].

In order to allow a full exploitation of TiAl alloys in turbine blade applications, further research is needed to understand their fretting damage mechanism, including study of candidate coatings and surface engineering solutions.

In this study, Cr-Nb coating was deposited on TiAl substrate by DGP surface alloying technique. The microstructural morphology, phase composition and elements distribution of the modified layer were measured. Fretting wear performance and wear regime under room temperature were analyzed and elaborated.

2 Experimental Procedures

2.1 Specimen Preparation

Casting TiAl alloy used in this study was produced by induction shell melting, and the chemical composition of the alloy is presented in Table 1. The ingot was cut into 14mm × 14mm × 3mm specimens, which were used as the substrate material acting as a cathode during coating deposition process. The specimens were mechanically polished, cleaned with ultrasonic cleaner and then dried using compressed air before the deposition.

Table 1 The chemical composition of the γ-TiAl alloy (wt %).

Ti	Al	V	Cr	Nb	O	C	N
Base	46.5	≤1.5	≤1	≤0.20	≤0.015	≤0.1	≤0.05

The operating procedure of the DGP surface alloying technique was described elsewhere[11], hence only the most important stages are outlined here. Prior to the alloying treatment, the TiAl specimens were subjected to plasma surface cleaning and activation in argon at 20 Pa pressure for 15 min, and then heated for 3 h at 35 Pa at the temperature of 900-950°C. A Cr-Nb target (Cr: Nb = 2: 1, wt %) prepared via the powder metallurgy method was used as the source electrode for supplying the alloying elements during deposition. The specimens were placed on a platform inside a double walled, water-cooled vacuum chamber, and the anode and cathode were each connected to the DC power supplies. The potential difference between the cathode and the source electrode resulted in an unequal potential, leading to the hollow cathode effect. Once the given voltage was applied, both the cathode and the source electrodes were surrounded by the glow discharge. As a result, Cr and Nb ions were sputtered from the source electrode by argon ions bombardment and deposited onto the specimens due to the bias gradient. The alloying elements were sputtered from the source electrode, accelerated towards the sample and forced to diffuse into the sample's surface. The parameters of DGP surface alloying technique are summarised in Table 2.

Table 2 DGP process parameters.

Item	Parameter
Processing temperature (°C)	900-950
Processing time (h)	3
Work-piece pressure (Pa)	30-40
Distance between source and cathode (mm)	18-20
Source voltage (V)	800-900
Cathode (substrate) voltage (V)	500-600

2.2 Microstructure analysis

Surface and cross-sectional microstructure morphologies of Cr-Nb coated TiAl samples were investigated using JSM-6360LV scanning electron microscope (SEM). Surface chemical composition and the alloying elements' distribution were measured using an energy dispersive spectrometer (EDS). The morphology and the depth of the fretting wear scars were investigated using Carl Zeiss EVO MA15 SEM and Bruker NPFLEX white light interferometer (WLI) respectively.

2.3 Mechanical properties measurements

2.3.1 Nano-indentation test

Nano-indentation tests were performed using MicroMaterials Nano-Test Platform® equipped with a Berkovich indenter, in order to measure the coating hardness and elastic modulus.

The experiments were conducted by driving the indenter at a constant loading rate of 0.5mN/s into the sample's surface with the maximum applied load of 100 mN, and the holing time of 10 s at a peak load. Each indentation experiment was repeated at least five times.

2.3.2 Scratch test

The scratch tests were performed using TriboTechnic scratch tester. A 0.2mm radius Rockwell C diamond indenter was drawn across the coating surface under a normal load ramped linearly from 0 to 50 N, enabling the measurement of the adhesion strength and intrinsic cohesion of Cr-Nb coated TiAl samples. The schematic diagram of the scratch tester is shown in Fig. 1.

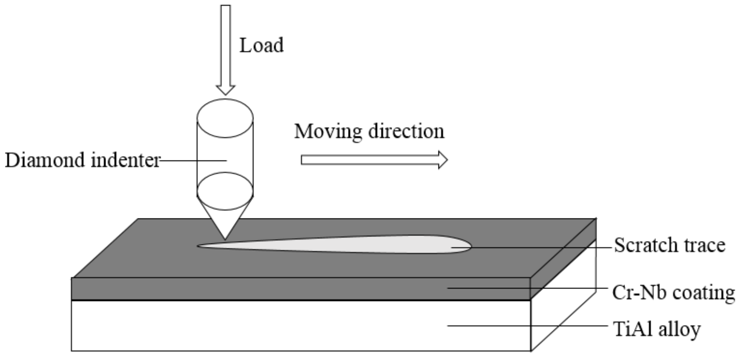


Fig. 1 The schematic diagram of the scratch tester.

2.4 Fretting wear experiments

Fretting wear tests were conducted on uncoated and Cr-Nb coated TiAl samples using a pin-on-plate fretting rig in air at room temperature (22 ± 2°C), and room relative humidity (58 ± 4 %). Si_3N_4 balls with the average hardness of HRC 75 and a diameter of 5 mm were used as the counterface material. The schematic diagram of the fretting rig is shown in Fig. 2.

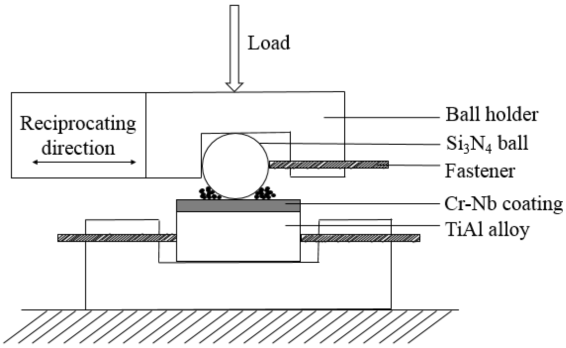


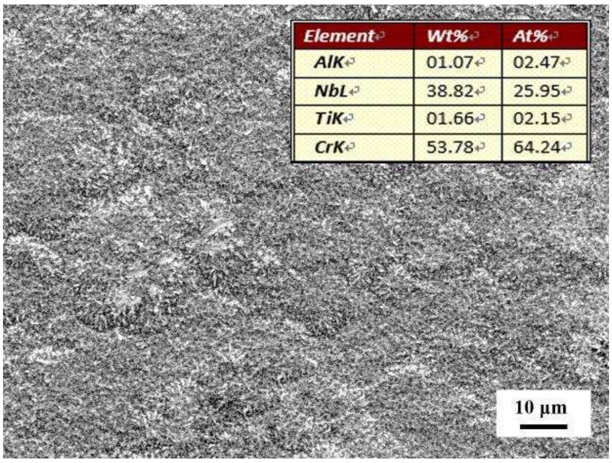
Fig. 2 The schematic diagram of the fretting rig.

Fretting tests were conducted at a load of 30 N, frequency of 5Hz, and slip amplitudes of 50, 100 and 150μm for 2500-10000 cycles. Both, the ball and the flat surfaces were rinsed with acetone before testing in the fretting apparatus. After completion of the experiment, the specimens were ultrasonically cleaned in acetone. and their surface characteristics were investigated. Each fretting experiment was repeated at least five times.

3 Results

3.1 Microstructure and phase composition

Fig. 3 shows the morphology and chemical composition of Cr-Nb coated TiAl sample. It was observed that the coating surface appears as a dense, non-porous and homogeneous structure without defects or cracks. The content of Cr and Nb, recorded by EDS was 52.78 wt % and 38.82 wt % respectively.



Element	Wt%	At%
AlK	01.07	02.47
NbL	38.82	25.95
TiK	01.66	02.15
CrK	53.78	64.24

Fig. 3 Morphology and EDS chemical composition of Cr-Nb coated TiAl sample.

Fig. 4 shows the cross-section SEM image of Cr-Nb coated TiAl sample. The coating exhibited a continuous and compact structure and was well bonded to the substrate. As shown in the EDS line scans (Fig. 5), the coating was divided into two discrete zones, i.e., a deposited layer and an inter-diffusion layer. The total thickness of the Cr-Nb coating was about 7μm, consisting of 5μm deposited layer and 2μm inter-diffusion layer.

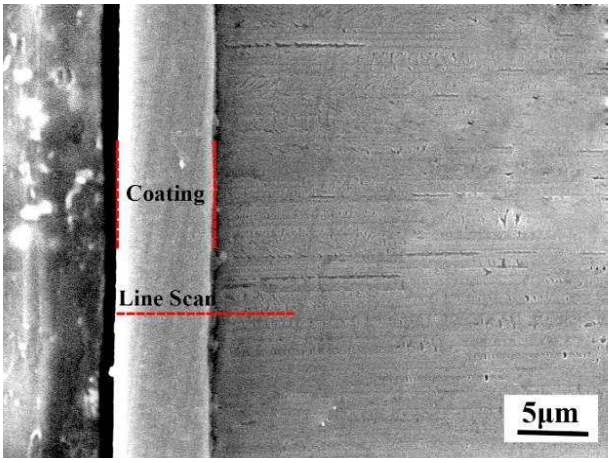


Fig. 4 Cross-sectional SEM morphology of Cr-Nb coated TiAl sample.

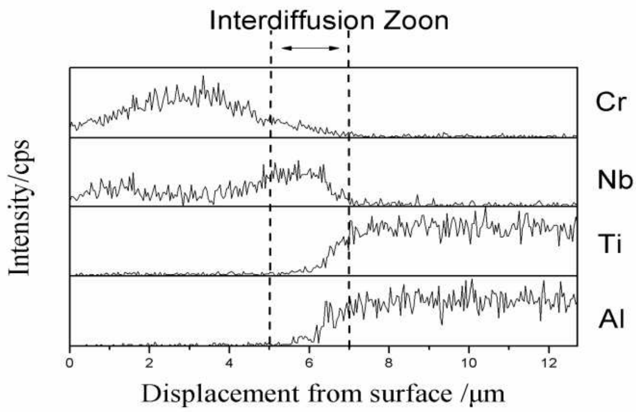


Fig. 5 EDS line scans of Cr-Nb coated TiAl sample.

3.2 Nano-indentation

Fig. 6 illustrates a typical load-displacement curves for uncoated and Cr-Nb coated TiAl samples. It is noted that the maximum indentation depth was no more than 10 % of the coating thickness, to avoid the influence of a ductile substrate on the measurements[15, 16]. Fig. 7 shows the hardness and the elastic modulus calculated using Oliver-Pharr method based on the force-depth data obtained from nano-indentation experiments[17].

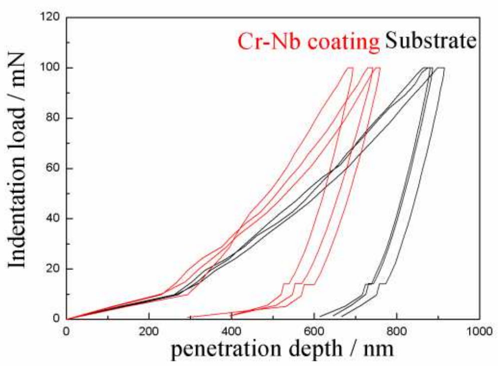


Fig. 6 Load-displacement curves of uncoated and Cr-Nb coated TiAl samples.

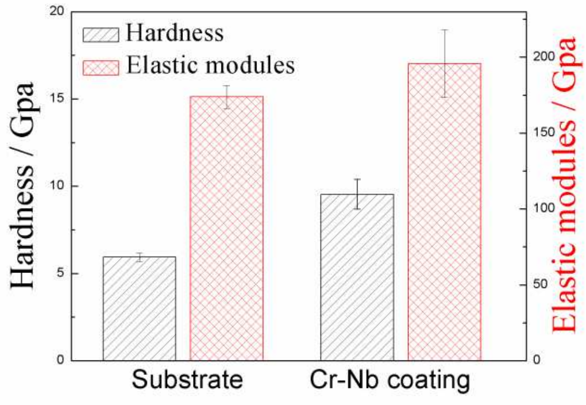


Fig. 7 Hardness and elastic modus of uncoated and Cr-Nb coated TiAl samples.

Compared with uncoated TiAl substrate, the hardness of Cr-Nb coating was improved from 5.9 GPa to 9.5 GPa. Same as the hardness, the average elastic modulus of the coated sample was improved from 174GPa to 196GPa.

It has been proven in the literature, that the elastic strain to failure ratio (H/E) and plastic deformation resistance factor (H^3/E^2) can be more suitable than the hardness alone for predicting the wear resistance of materials[15, 17]. As shown in Fig. 8, the H/E and H^3/E^2 ratios improved from 34.1×10^{-3} and 6.9×10^{-3} respectively for the substrate, to 48.8×10^{-3} and 22.8×10^{-3} for the coated samples, suggesting improved tribological performance of Cr-Nb coated TiAl substrate.

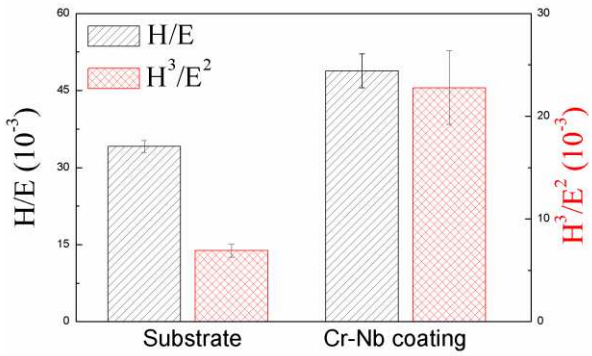


Fig. 8 The H/E and H^3/E^2 ratio of uncoated and Cr-Nb coated TiAl samples.

2.4 Scratch test

Fig. 9 shows a typical result of the scratch test experiment. Cr-Nb coating was subjected to progressively applied load from 0 to 50 N. At a certain critical load, the coating would start to fail, which was identified as a coating critical failure load. The critical load for Cr-Nb coating was observed at 17.6 N.

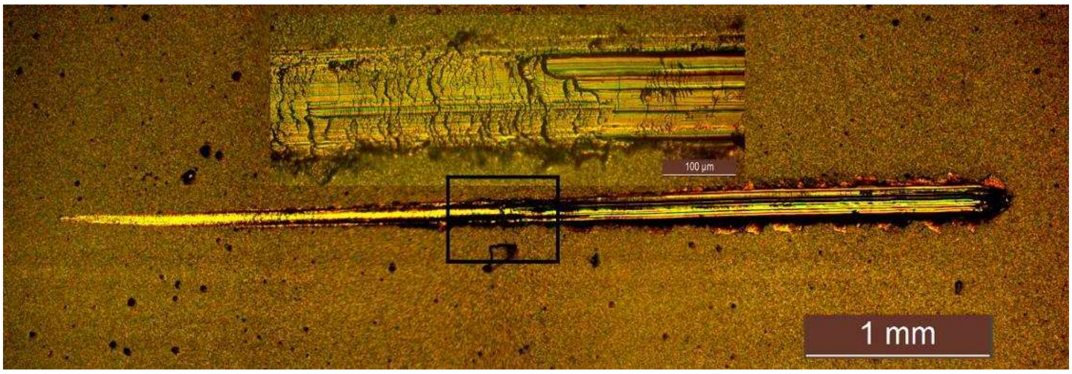


Fig. 9 Optical Microscopy image of scratch-test track.

2.5 Fretting wear tests

The evolution of the fretting loops shape made up by the tangential force and the displacement amplitude recorded for each fretting cycle, is one of the most important and basic information that can be recorder during fretting wear experiment. Fig. 10 shows the fretting loops of uncoated and Cr-Nb coated TiAl samples, obtained during 10000 cycles tests at load of 30 N, a constant frequency of 5Hz, under three different displacement amplitudes (50, 100 and 150 μm). According to the RCFM (Running Condition Fretting Map)[18] and MRFM (Material Response Fretting Map)[19] concepts, there are three fretting regimes which can be identified from the evolution of the fretting loops: (1) Stick regime (closed, narrow loop); (2) Mixed stick-slip regime or partial-slip regime (elliptical loop); (3) Gross-slip regime (quasi-rectangular loop)[10].

As shown in figures 10a and 10d, the fretting regime of the uncoated TiAl sample in the low amplitude range was partial regime, while that of the Cr-Nb coating was stick regime. The fretting regime of uncoated and Cr-Nb coated TiAl samples were all in gross-slip regime in the intermediate and high displacement ranges.

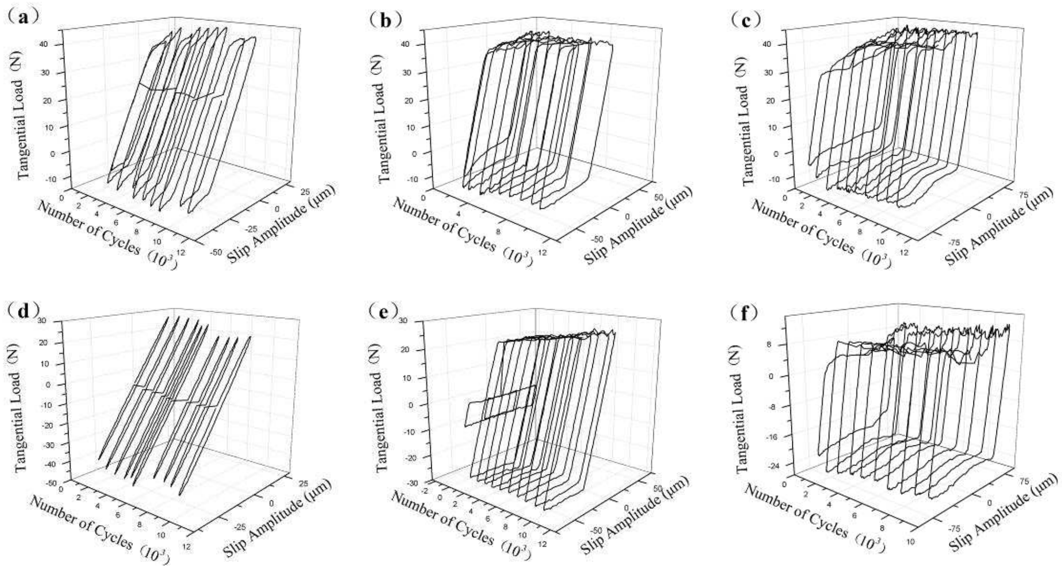


Fig. 10 Fretting loops of uncoated and Cr-Nb coated TiAl samples recorder under three fretting displacement amplitudes. Substrate: (a) 50μm, (b) 100μm, (c) 150μm; Cr-Nb coating: (d) 50μm, (e) 100μm, (f) 150μm.

Fig. 11 shows the friction coefficient evolution of uncoated and Cr-Nb coated TiAl samples recorded during 10000 cycles tests at load of 30 N, a constant frequency of 5Hz, under three different displacement amplitudes (50, 100 and 150 μm). Under low displacement amplitude values, the average friction coefficient of the Cr-Nb coating was 0.9, which was the same as the uncoated TiAl. For the intermediate and high amplitude values, the average friction coefficient of the Cr-Nb coating was 0.85, which was slightly lower than that of the uncoated TiAl substrate ~ 0.9.

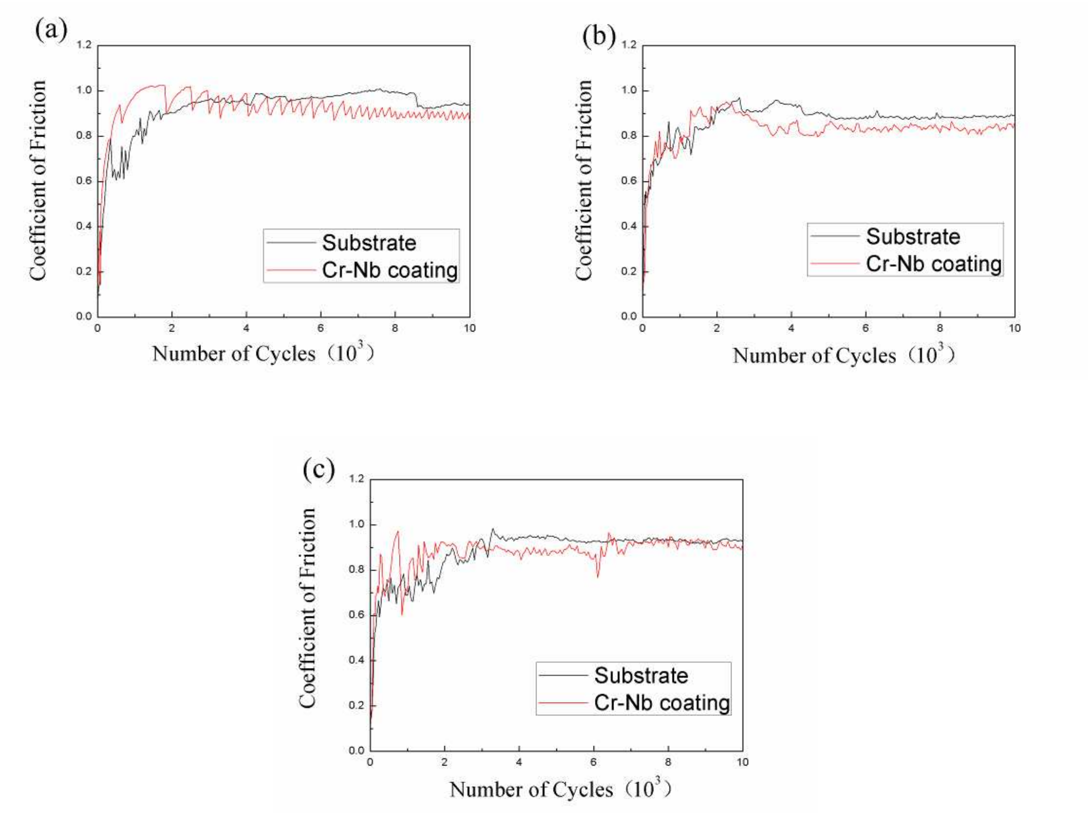


Fig. 11 Friction coefficient of uncoated and Cr-Nb coated TiAl samples under different fretting amplitudes (a) 50μm, (b) 100μm, (c) 150μm.

Fig. 12 shows the wear depth of uncoated and Cr-Nb coated TiAl samples measured after 10000 cycles tests at load of 30 N, a constant frequency of 5Hz, under three different displacement amplitudes (50, 100 and 150μm). The fretting wear depth of uncoated and Cr-Nb coated TiAl alloy was generally increasing with increasing number of cycles and displacement amplitudes. The observed wear was lower for the coated specimens.

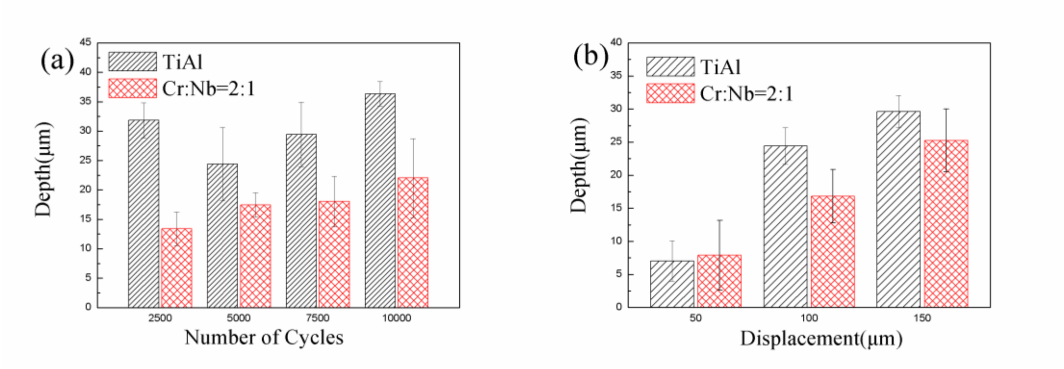
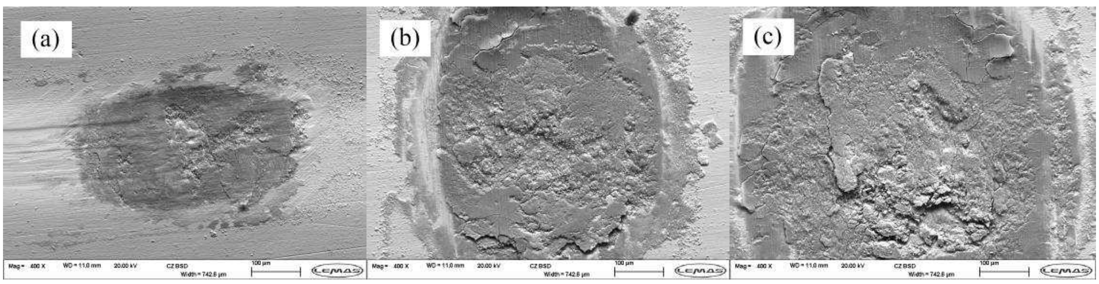


Fig. 12 Fretting wear depth of uncoated and Cr-Nb coated TiAl samples as a function of (a) fretting cycles and (b) displacement amplitudes.

Fig. 13 shows the surface morphologies of wear scars of uncoated and Cr-Nb coated TiAl samples. For the low amplitude range, the wear scar of uncoated TiAl was a shallow pit with adhesive wear features appearing in the central region of the scar, with some fine particles ejected outside the contact region. In contrast, only minimal damage was observed on Cr-Nb coated sample under the same test conditions. Under intermediate and high amplitude values, wear scars were morphologically similar, characterized by wear debris, scratches, plastically deformed asperities, and cracks, as the depth and volume of wear scars were gradually increasing with higher displacement amplitudes. However, the observed extent of wear was larger for uncoated TiAl samples.



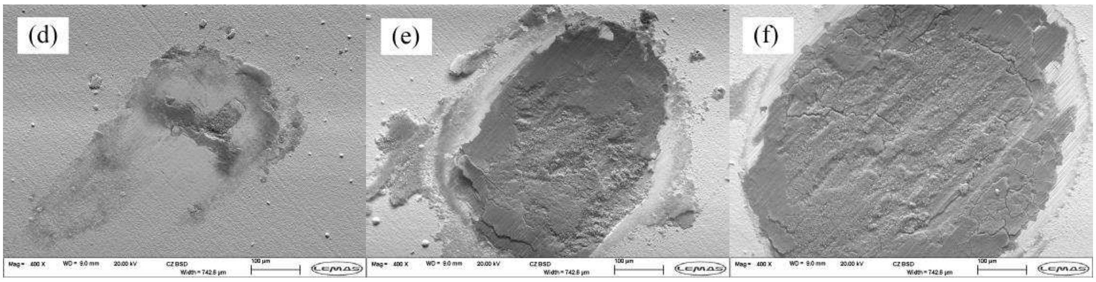


Fig. 13 Surface morphologies of wear scars. Substrate: (a) 50μm, (b) 100μm, (c) 150μm; Cr-Nb coating: (d) 50μm, (e) 100μm, (f) 150μm.

4 Discussion

The results showed that Cr-Nb coating was deposited successfully on the TiAl alloy via a double-glow plasma process. The morphology of the Cr-Nb coated Ti-Al sample demonstrated uniform and compact layer with a large number of globular and acicular particles on the surface. The surface morphology was caused by the nature of growth of the sputtered atoms clusters during DGP process. It was also found that the chemical composition of the Cr-Nb coating surface was different from that of the sputtering target, which was due to the differences in sputtering rates of elements[17]. Additionally, the final composition of the coating was affected by the diffusion of alloying elements from the substrate to the coating[15]. Moreover, Nb is known to be soluble in both, the γ and α_2 phases, and has been found to be particularly advantageous for strengthening of γ-TiAl engineering alloys[7].

The cross-sectional SEM morphology and EDS results showed that Cr and Nb were distributed gradually in the transition layer due to the high temperature induced diffusion. Furthermore, Ti and Al were both detected in the diffusion layer as well, confirming the outward diffusion process of elements into the coating zone. This was beneficial to the interfacial bonding strength of the coating[20, 21], as confirmed by the scratch-test results. The nano-indentation results showed that the surface hardness of Cr-Nb coating was improved from 5.9GPa to 9.5GPa compared with the TiAl substrate.

It has been shown that fretting wear and fretting fatigue are two main mechanisms of damage of coatings in turbine blade applications, that can lead to the catastrophic failure of components[22]. Moreover, from a phenomenological analysis, the gross-slip and partial-slip regimes have been associated with the wear phenomena, while the stick-slip regime with cracking[10]. The contribution of the Cr-Nb coating to reducing the partial-slip regime in favor of the stick-slip regime was proven in this study (Fig. 10 and Fig. 13). This in return, decreased the amount of energy dissipated in the contact region and lead to lower wear rates. As a result, the wear depths of Cr-Nb coating were all lower than that of the uncoated TiAl under the same test conditions, which can be also attributed to the enhanced mechanical properties and good binding force of the coating.

In summary, it has been shown that Cr-Nb coating can reduce not only the friction loss and fretting wear of the TiAl alloy, but also limit its fretting fatigue damage and prevent cracking. Hence, double-glow plasma Cr-Nb coatings are potential candidates for performance improvement of TiAl alloy in low-pressure turbine blades applications.

5 Conclusions

A uniform, non-porous and compact Cr-Nb coating without defects or cracks was successfully formed onto the TiAl substrate via a double-glow plasma process. The following conclusions are drawn from the results of this study:

(1) The total thickness of the Cr-Nb coating was about 7μm including 2μm inter-diffusion layer, consisting of Cr, Nb, Ti and Al elements.

(2) The hardness of the Cr-Nb coating was 9.5 GPa, which was 1.6 times greater than that of the uncoated TiAl alloy.

(3) The fretting loops recorded for uncoated and Cr-Nb coated TiAl alloy showed that Cr-Nb coating reduces the region of gross-slip in favor of the partial-slip regime.

(4) The wear of Cr-Nb coating was increased with increasing displacement amplitudes, however it was lower than that of the uncoated TiAl under all loading conditions.

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