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Mechanical properties of $Ti(C_{0.7}N_{0.3})$ film produced by plasma electrolytic carbonitriding of Ti6Al4V alloy

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ABSTRACT

Porous nanocrystalline $Ti(C_{0.7}N_{0.3})$ film on Ti6Al4V substrate was prepared by plasma electrolytic carbonitriding (PECN). The film was characterized and analyzed by using a variety of analytical techniques, such as XRD, SEM, EDX, TEM, FESEM, Rockwell C indenter, scratch tester, Vickers microhardness tester and ring-on-block tribometer. The results showed that the film was about 15 μm thick and its hardness was Hv 2369 at a load of 0.2 N. The adhesion of the film was characterized by Lc and Pc value, and was found to be about 42 N and more than 800 N, respectively. The friction coefficients and wear volume loss of the PECN-treated samples sliding against a steel counterpart were much less than those of the untreated Ti6Al4V. The film possessed a good wear-resistance and antifriction under oil-lubricated condition due to its high hardness, adhesion and fracture toughness. Also, the porous surface morphology of the $Ti(C_{0.7}N_{0.3})$ film contributed to the enhanced tribological resistance by promoting the formation of lubricant film and entrapping wear debris.

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1. Introduction

Titanium alloys are attractive materials due to its low density, very good resistance to corrosion and high relative strength. However, their resistance to frictional wear is poor. Surface engineering techniques are widely used to improve the surface hardness and wear resistance of titanium alloys, and they have been realized as promising methods to deposit TiC, TiN and Ti(C,N) hard films on titanium alloys. Up to now, various techniques have been developed to deposit or form titanium carbide/nitride films on titanium alloys, such as physical vapor deposition (PVD) [1,2], chemical vapor deposition (CVD) [3,4], ion beam assisted deposition (IBAD) [5–7], and plasma carbiding/nitriding [8,9]. However, the titanium carbide/nitride films produced by IBAD on Ti6Al4V alloy are too thin (generally less than 3 μm) and the load-bearing capacity is poor [7]. The adhesion of the titanium carbide/nitride films obtained by PVD and CVD are insufficient owing to the increased internal stresses in the films [10], and thereby delamination of the films has been observed under wear condition [11]. Although plasma carbiding/nitridng treatment of titanium at 700–900 °C leads to firmly adhered titanium carbide/nitride layer, the formation of the compound layer with the thickness more than 10 μm requires much higher temperature and longer treatment time [8,12], which results in worse fatigue behavior and earlier fracture due to the aging of the substrate material [7]. It has been proved that the improvement efficacy of the wear resistance and load-bearing capacity strongly depend on the thickness and adhesion of the hard films [13,14], thus alternative methods to produce thick and firmly adhered titanium carbide/nitride films on titanium alloy at low temperature are still worth exploring.

It is found that plasma electrolytic oxidation (PEO) can achieve a relatively fast conversion of titanium surface, at near-to-ambient bulk temperature, into a titanium oxide ceramic layer [15–17]. These titanium oxide layers amount to 10 μm in thickness and firmly bond to the titanium substrate. Based on the principle involved in PEO, a novel surface modification technology named as plasma electrolytic carbonitriding (PECN), has been developed for surface modification of pure titanium and thereby formation of thick $Ti(C_xN_{1-x})$ films in our previous work [18,19], however, still unclear how about the mechanical properties of the modified layer. In the present work, the mechanical properties of the PECN-formed $Ti(C_xN_{1-x})$ film on Ti6Al4V alloy are investigated, especially the adhesion, hardness, and friction and wear behavior.

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2. Experimental

Ti6Al4V plates with sizes of 30 mm × 10 mm × 1 mm were used as substrates. The plates were polished to a mirror finish with an aqueous silica suspension, and ultrasonically cleaned with acetone and distilled water. For PECN treatment, a pulse power supply was employed, and a Ti6Al4V plate was used as a cathode while a graphite plate was used as an anode in an electrolytic cell. A mixed organic solution of triethanolamine [$N(CH_2CH_2OH)_3$] and formamide [$HCONH_2$], and other proprietary ingredients (added primarily for adjustment of electrical conductivity) were chosen as an electrolyte. The applied voltage, pulse frequency and duty cycle were fixed at 600 V, 100 Hz and 40%, respectively, and the Ti6Al4V plates were discharge-treated for 2.5 h. The electrolyte bath was water-cooled and its temperature was maintained lower than 30 °C. After the PECN treatment, the obtained samples were washed with distilled water and dried at room temperature.

The phase components of the PECN-treated samples were analyzed with X-ray diffraction (XRD) using a Cu Kα radiation. Scanning electron microscopy (SEM) and field emission scanning electron microscopy (FESEM) were employed to observe the morphologies, thickness and grain sizes of the formed film.

Rockwell C indenter (conical diamond with 120° included angle and 0.2 mm tip radius) was used to evaluate the adhesion of the PECN-formed film. In the indentation method, the force which separates a film from a substrate, expressed by Pc value, can characterize the adhesion of a film [13]. A series of loads were applied to the surface of the PECN-treated sample, and the resulting damages of the film around the indentations were examined using SEM. As a comparison, the adhesion of the film was also investigated using a WS-92 automatic scratch tester. A diamond stylus of 200 μm radius with an apex angle of 120° was drawn over the film at a speed of 6 mm min^{-1} under a continuous progressive normal force. The smallest load at which the film is damaged is designated the critical load, Lc, which gives an indirect indication of the adhesion [20,21]. The scratch tester was equipped with acoustic emission monitoring equipment, which was used as an on-line failure monitor. Critical load thresholds and detailed morphology of the scratch pattern were correlated by SEM and backscattered electron microscopy (BSE) observations. Five scratch tests were performed to obtain mean and standard deviation values for each film.

Hardness measurements were performed on the surface of the PECN-treated sample using a Vickers microhardness tester at a load of 0.2 N. The hardness value is an average of 10 measurements. Fracture toughness of the film was roughly estimated from Vickers indentations at a load of 10 N. Friction and wear tests were carried out on a ring-on-block reciprocating sliding tribometer under lubricated condition as follows. The blocks (8 mm diameter) were made of the PECN-treated and untreated Ti6Al4V, respectively, and the rings (40 mm external diameter) were made of middle carbon steel with a hardness of HRC 48–52. The ring was rotated against the block at a speed of 0.42 m s^{-1}. Commercial 30# engine oil and 0.9% saline solution were used as lubricant, respectively, and drop rates of them were about 16–20 drops/min. The friction coefficients were calculated from the moment automatically recorded by the friction and wear tester according to the following relationship:

$$\mu = \frac{M}{R \times F} \quad (1)$$

where μ is the friction coefficient, M is the moment, R is the external diameter of the ring, and F is the applied load. The wear volume loss was determined by measuring the cross-sectional area of the wear scar with a profilometer. Worn surfaces were investigated using SEM coupled with energy-dispersive X-ray spectrometer (EDX). At least three repetitions of each test were carried out.

3. Results and discussion

The XRD pattern of the sample PECN discharge-treated for 2.5 h is shown in Fig. 1. The formed film is composed of $Ti(C_{0.7}N_{0.3})$. As shown in Fig. 2, the $Ti(C_{0.7}N_{0.3})$ film exhibits a porous surface morphology with a pore size of ~5 μm. These pores are well separated and homogeneously distributed over the surface of the film. When observed at high magnification (Fig. 3), the film matrix consists of much fine particles with the size of 40–60 nm, suggesting that the PECN-formed $Ti(C_{0.7}N_{0.3})$ film is nanocrystallized. From the cross-sectional view (Fig. 4), the thickness of the $Ti(C_{0.7}N_{0.3})$ film is ~15 μm. The surface and cross-sectional morphology and crystallization characteristics of the PECN-formed $Ti(C_{0.7}N_{0.3})$ film on Ti6Al4V are similar to those on commercial titanium [18,19].

The adhesion of the PECN-formed $Ti(C_{0.7}N_{0.3})$ film can be roughly evaluated using indentation test according to the damage type of the film [20]. Fig. 5 shows the indentation morphologies of the film subjected to different loads. No cracks and spallations can be observed around the indentation at a load of 800 N; however, the radial and annular cracks appear with increasing the applied

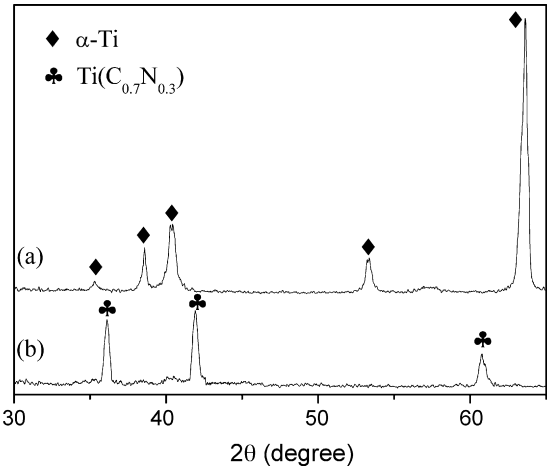

Fig. 1. XRD patterns of the PECN-treated samples for the discharge time of (a) 0 and (b) 2.5 h.

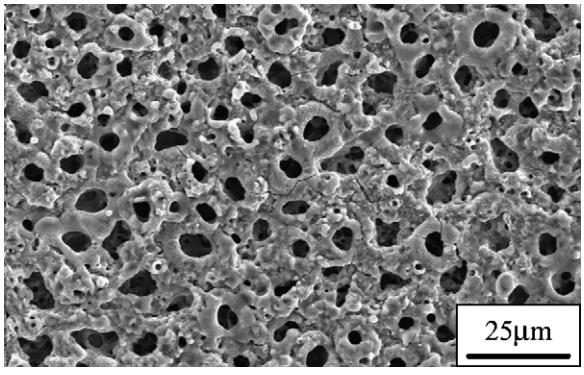

Fig. 2. SEM photograph of the sample PECN discharge-treated for 2.5 h.

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