



Effect of N-ion implantation and diamond-like carbon coating on fretting wear behaviors of Ti6Al7Nb in artificial saliva



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Received 15 December 2015; accepted 28 March 2017

Abstract: The tribology behaviors of Ti6Al7Nb, its alloy with N-ion implantation, and its alloy with diamond-like carbon (DLC) coating were investigated in artificial saliva. Fretting wear tests of untreated, N-ion implanted and DLC coated Ti6Al7Nb alloys plate against a Si₃N₄ ball were carried out on a reciprocating sliding fretting wear test rig. Based on the analysis of X-ray diffraction, Raman spectroscopy, 3-D profiler, SEM morphologies and frictional kinetics behavior analysis, the damage behavior of surface modification layer was discussed in detail. The results indicated that the fretting wear behavior of Ti6Al7Nb alloy with N-ion implantation was increased with the dose increase of the implanted nitrogen ions. Moreover, the DLC-coated Ti6Al7Nb alloy with low ion implantation could improve the fretting wear behavior greatly. In addition, the Ti6Al7Nb with DLC coating had better corrosion resistance due to the special compact structure. All results suggested that the Ti6Al7Nb with DLC coating had better wear resistance than that with N-ion implantation in artificial saliva.

Key words: Ti6Al7Nb alloy; ion implantation; diamond-like carbon coating; fretting wear behavior

1 Introduction

Titanium and its alloys are known as the attractive materials for the manufacture of implants for medical and dental applications due to low density, high specific strength, excellent corrosion resistance and good biocompatibility compared with other conventional metallic materials [1–4]. Thus, titanium alloys become the first choice used for artificial bones, dental and surgical implants artificial dentures [5,6]. However, some factors (low hardness) limit the applications of titanium and its alloys. During the long-term clinical applications, it is found that the surface of the oxide film was easy to be peeled off in the contact fretting due to the poor wear resistance, resulting in the abrasive wear acceleration of the implants [7,8]. As a result, metal ions could be released from the implant alloy and were detected in tissues close to titanium implants. Even though the number of released metal ions is small, it can cause local inflammation of the tissues surrounding the implant [8,9]. The crack initiation, wear and adhesion of these implants could happen in the presence of small amplitude oscillatory sliding motion, which are the main reason of

the failure of titanium alloy implants [10].

Commercially pure titanium and Ti6Al4V alloy are the most widely used materials for the manufacture of implants. Due to the potential toxic effects of vanadium compounds, many researchers have studied the medical titanium alloy with good overall properties in recent years. V-free alloys like Ti6Al7Nb alloy have been recently developed for biomedical applications. Ti6Al7Nb alloy is widely used because of its advantages in biocompatibility, mechanical properties and corrosion resistance [11–14]. Meanwhile, some surface technologies are used to modify the surface of titanium alloy to obtain more suitable properties [15–18]. Currently, there are many methods (ion implantation, DLC coating and so on) to improve surface behaviors of titanium alloys for the application of hard tissue repair alternative materials. The common method for the improvement of the wear resistance is to form a hardness coating at the surface of titanium alloy. Plasma immersion ion implantation-deposition (PIIID) is a rapid and effective surface modification technique to modify the physicochemical characteristics of the surface [19,20]. Some surface coatings, especially diamond-like carbon (DLC) coatings, are well known as low-friction coatings.

During the past decade, the fretting wear of DLC coating has been widely investigated [21–24]. The thermal degradation of DLC coatings can cause a transformation from sp^3 to sp^2 bonds, resulting in low wear resistance. In addition, the tribological behavior of DLC coatings at lower frequencies is better than that of TiN, but it still could not provide very high levels of protection [25–27]. Thus, the further understanding on the tribological behavior of DLC coatings is expected.

In this work, the fretting wear behaviors of Ti6Al7Nb alloy and its alloy with N-ion implantation, DLC coating in artificial saliva were investigated. Ti6Al7Nb alloys were implanted with different nitrogen doses ($1 \times 10^{17} \text{ N}^+/\text{cm}^2$, $13 \times 10^{17} \text{ N}^+/\text{cm}^2$) and diamond-like carbon (DLC) thin film. The fretting wear behaviors of titanium alloy in artificial saliva at 37 °C (body temperature) on a fretting test system had been carried out, the fretting motion was induced by the action of an oscillating normal force in a sphere-on-flat contact. The X-ray diffraction, Raman spectroscopy, 3-D profiler, SEM morphologies, and the fretting running were analyzed in detail. This study focused on the fretting running behaviors and damage mechanisms.

2 Experimental

2.1 Materials

The material used for this study was the biomedical Ti6Al7Nb alloy, with a chemical composition (mass fraction, %): 5.88 Al, 6.65 Nb, 0.03 Fe, 0.10 C, 0.20 O, 0.07 N, 0.02 H and 87.05 Ti. The samples were cut into 10 mm × 10 mm × 25 mm for tests. All specimens were ground and polished with 0.05 μm alumina powder to obtain the roughness of $R_a=0.5 \mu\text{m}$. The Ti6Al7Nb alloy was implanted with N-ion with the concentrations of 1×10^{17} and $13 \times 10^{17} \text{ N}^+/\text{cm}^2$. Then, the specimen surface implanted with N-ion with the concentration of 1×10^{17} and $13 \times 10^{17} \text{ N}^+/\text{cm}^2$ was plating DLC films. The DLC films plating process like this: at first, plant Cr in the sample surface, then plant CrN and CrSiN in the Cr surface, which was used to form the gradient layer of hardness and reduce the film crack under load due to the hardness gap of the substrate and DLC film, at last plant DLC films in the CrN surface. The silicon nitride (Si_3N_4) counter-body (the sphere specimen) was a bearing sphere (AISI 52100), 10 mm in diameter. The hardness of the Si_3N_4 specimen was about HV 740, and its average

roughness R_a reached approximately 0.1 μm. Before the tests, the Si_3N_4 sphere was carefully cleaned by acetone.

The artificial saliva (0.4 g NaCl, 0.4 g KCl, 0.795 g $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 0.78 g $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 0.005 g $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$, 1 g urea, 100 g distilled water) was used to simulate the oral environment.

2.2 Surface treatments (nitrogen ion implantation and DLC coating)

The samples were sputter-cleaned with argon and deionized water, and then laid on stainless-steel substrates attached to an insulated stainless-steel electrode in the center of the vacuum chamber. Next, a negative voltage was applied to the electrode. Two doses of nitrogen ion (1×10^{17} and $13 \times 10^{17} \text{ N}^+/\text{cm}^2$) were implanted into Ti6Al7Nb alloy, which was carried out at the high energy ion implantation and enhanced deposition system of the Lanzhou Institute of Chemical Physics, China. The specific ion implantation parameters were shown in Table 1.

Prior to deposition tests, the specimens were polished mechanically and ultrasonically cleaned in alcohol for about 15 min. Afterwards, Cr/CrN/CrSiN/DLC multi-layered coatings were deposited on Ti6Al7Nb alloys with nitrogen ion implantation using a combined arc ion plating and magnetron sputtering process in the same chamber. For the deposition tests, the bottom vacuum of chamber was kept to $4 \times 10^{-3} \text{ Pa}$, access to Ar gas to 1 Pa, a bias voltage of −700 V, and use the producing glow clean substrate for 10 min. The targets of Cr and Si were of high purity (99.99%). For Cr film, the target was deposited with argon (99.999%) inlet with a flow of 40 mL/min. For CrN/CrSiN film, argon and nitrogen (99.999%) were introduced into the chamber with a flow of 10 and 40 mL/min, respectively. For DLC film, argon and CH_4 (99.999%) was introduced into the chamber with a flow of 20 and 40 mL/min, respectively. The deposited time of Cr, CrN, CrSiN and DLC film was 30 min for each layer. During the deposition process, the pressure was kept at 0.5–06 Pa. During the whole deposition process, the substrate must not be heated and the sputtering power was set to 1.1 kW.

2.3 Electrochemical corrosion tests

The electrochemical tests were performed on a PARSTAT 2263 electrochemical workstation. The test

Table 1 Parameters of N-ion implantation technology

Source	Filament current/A	Arc		Suppression		Accelerate	
		Voltage/V	Current/A	Voltage/V	Current/A	Voltage/kV	Current/mA
Cleaning source	12.5	42.5	1.0	200	6.5		
Gas source	11.2	66.5	0.2	1000	0.6	40	8.0

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