



Effect of nitrogen ion implantation dose on torsional fretting wear behavior of titanium and its alloy



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Abstract: Various doses of nitrogen ions were implanted into the surface of pure titanium, Ti6Al7Nb and Ti6Al4V, by plasma immersion ion implantation. Torsional fretting wear tests involving flat specimens of no-treated and treated titanium, as well as its alloys, against a ZrO₂ ball contact were performed on a torsional fretting wear test rig using a simulated physiological medium of serum solution. The treated surfaces were characterized, and the effect of implantation dose on torsional fretting behavior was discussed in detail. The results showed that the torsional fretting running and damage behavior of titanium and its alloys were strongly dependent on the dose of the implanted nitrogen ions and the angular displacement amplitude. The torsional fretting running boundary moved to smaller angular displacement amplitude, and the central light damage zone decreased, as the ion dose increased. The wear mechanisms of titanium and its alloys were oxidative wear, abrasive wear and delamination, with abrasive wear as the most common mechanism of the ion implantation layers.

Key words: titanium alloy; ion implantation; fretting; wear mechanism

1 Introduction

Titanium and its alloys, e.g., Ti6Al4V, are widely used as implant materials in clinical applications such as components for artificial joints and dental implants, because these materials are known to be favorably biocompatible and can provide the required mechanical properties. However, titanium and its alloys are soft, with low surface shear resistance, because of adhesion concerns, stress concentration, and dimension effect problems. These characteristics limit the application of titanium and its alloys to several specific areas, so hardening of titanium surface is performed [1,2]. To improve wear resistance or modify the surface properties, titanium surfaces are subjected to various surface treatments, such as thermal spraying, plasma vapor deposition, anodic oxidation, ion implantation, glow discharge nitriding, and laser treatment [3,4]. CRESSMAN et al [5] and MUTYALA et al [6] pointed out that Ti6Al4V surface coated with MnPO₄ or NiPO₄ is insufficient to prevent high friction and wear in gross or

mixed fretting situation. Plasma immersion ion implantation (PIII) is a promising alternative for improving and functionalizing the surfaces of various types of titanium and its alloys through ion implantation. Nitrogen is a kind of good curing agent. After nitrogen ion implantation, titanium, and its alloys surface will form a nitriding layer, its main composition of titanium nitride is a kind of high melting point, high hardness clearance compounds [7]. Nitrogen ion implantation is cost effective and enhances the wear resistance and anti-corrosion properties of titanium [8]. In the past three decades, nitrogen implantation has been extensively investigated. Given the potential toxic effects of vanadium compounds, vanadium-free alloys such as Ti6Al7Nb have recently been developed for the application of artificial joints instead of Ti6Al4V [9,10].

This paper focused on one type of rotary motion, namely, the torsion, which commonly occurs at human joints and other rotating parts [11]. Most tribological studies have focused on linear sliding wear behavior. However, torsional wear with small angular displacement has been ignored because small frictional

displacement is difficult to control, measure, and characterize, particularly in cases involving non-linear motion and small contact area.

BRISCOE et al [12,13] found that torsional contact is more detrimental to the wear resistance of polymethyl methacrylate (PMMA). WANG et al [14,15] investigated the torsional wear behavior of various composite materials containing MC nylon, polytetrafluoroethylene composites filled with poly (phenyl-hydroxybenzoate), and hexagonal boron nitride. In our previous study, we showed the damage and running behavior of various materials (carbon steel, aluminum alloys, UHMWPE, and natural cartilage) [16–18]. All related previous studies were based on a single material, and did not investigate how modified layers or coatings affect torsional wear damage.

In the present study, pure titanium and its alloys were implanted with PIII at various doses. The mechanical properties of the modified layers were measured, and the effects of the implantation dose on torsional fretting wear behavior were discussed.

2 Experimental

2.1 Materials and plasma immersion ion preparation

Pure titanium (TA2), Ti6Al7Nb, and Ti6Al4V (Table 1) were selected as the test substrate materials (rods provided by the Northwest Institute of Nonferrous Metal Research, Xi'an, China). The rods were cut to specimens with dimensions of 10 mm × 10 mm × 25 mm. One side of each plate (10 mm × 25 mm) was polished continuously using 60, 200, 600, 1000, and 1500 grit diamond paper and flannel to a roughness of $R_a=0.5 \mu\text{m}$ before being placed in a plasma immersion ion implanter.

Table 1 Chemical compositions of TA2 and two titanium alloys (mass fraction, %)

Material	Al	Nb	Ta	Fe	V	C	N	O	Ti
TA2	–	–	–	0.30	–	0.10	0.05	0.25	Bal.
Ti6Al7Nb	6.0	6.97	0.36	0.22	–	0.10	0.07	0.20	Bal.
Ti6Al4V	6.2	–	–	0.16	4.10	0.041	0.08	0.16	Bal.

2.2 Nitrogen PIII

PIII treatment was performed using a new plasma immersion ion implanter (Tongchuang, Chengdu, China). The chamber of the implanter was 1.2 m in height and 1.0 m in diameter, and a negative voltage was applied to the electrode. The facility was equipped with a classical radio frequency (RF) plasma source, hot filament glow discharge source, and vacuum arc source, etc. The substrates were laid on stainless steel substrates attached to an insulated stainless steel electrode at the center of

the vacuum chamber. Prior to implantation, the plates were sputter-cleaned via argon plasma ion bombardment. The pretreatment instrument parameters were as follows: RF in forward energy, 1000 W with a reflected power of approximately 20 W; bias voltage, 2.5 kV; gas flow, 10 mL/min; and cleaning time, 40 min. Then, nitrogen was passed into the vacuum chamber, and nitrogen plasma was sustained by RF power supply at a power of 1000 W, work pressure of $7.33 \times 10^{-2} \text{ Pa}$ and gas flow of 20 mL/min (main arc voltage of 72 V with a current of 0.2 A, suppress voltage of 1 kV with current of 0.5 mA, extraction voltage of 1 kV with a current of 7.5 mA, and accelerating voltage of 50 kV with a current of 8 mA). After immersion, the morphology of the surfaces was observed using an optical microscope (OM). Nitrogen ion (N^+) implantation doses of 1×10^{17} , 3×10^{17} , 5×10^{17} , 7×10^{17} , and $9 \times 10^{17} \text{ cm}^{-2}$ were selected.

2.3 Torsional fretting wear test and analysis

The wear tests of the no-treated and implanted plates against a Zr_2O ceramic ball used for artificial joints and teeth (diameter of 25.2 mm, $R_a=0.05 \mu\text{m}$, and hardness of HV 1100) were performed under a simulated physiological condition, on a torsional fretting wear test rig [16]. The test medium was a 20% bovine serum solution (Shanghai Bao Man Biological Technology Co., Ltd.), similar to human blood condition [19,20]. All the wear tests were performed at room temperature. The rotation speed was controlled to be 0.001 r/min by a lower motor. Torsional speed was kept constant at 0.01 rad/s. Each test was run for 1000 cycles. Various degrees (0.5° , 5° , and 15°) were set as the angular amplitudes, and a constant load of 100 N was selected as the normal load.

Based on the analyses of frictional kinetics behavior under fretting wear, the profiler observation of 3D profiles (Nano Map-Dual Mode 3D), and SEM (SEM-Quanta 200) morphology and surface chemical analyses (EDX, EDAX-7760/68ME), the wear damage characteristics of the surface modification layer and its substrate materials were discussed in detail. The influence of the dose of nitrogen ion implantation on torsional fretting behavior was also examined.

3 Results

3.1 Surface characterization

The XRD patterns of titanium and its two kinds of alloy samples after nitrogen ion implantation are shown in Fig. 1. The XRD peak intensity, peak shape, and chemical composition varied with the dose of nitrogen ion implantation. Compared with bare titanium and the alloy samples, the peak intensities of samples shown in Fig. 1(b) gradually weakened, and a new XRD pattern

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