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Torsional fretting wear of a biomedical Ti6Al7Nb alloy for nitrogen ion implantation in bovine serum

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A B S T R A C T

Ti6Al7Nb is a high-strength titanium alloy used in replacement hip joints that possesses the excellent biocompatibility necessary for surgical implants. Ti6Al7Nb treated with nitrogen gas (N_2) plasma immersion ion implantation–deposition (PIII–D) was investigated. Torsional fretting wear tests of untreated and nitrogen-ion-implanted Ti6Al7Nb alloys against a Zr_2O ball (diameter 25.2 mm) were carried out under simulated physiological conditions (serum solution) in a torsional fretting wear test rig. Based on the analyses of the frictional kinetics behavior, the observation of 3D profiles, SEM morphologies and surface composition analyses, the damage characteristics of the surface modification layer and its substrate are discussed in detail. The influence of nitrogen ion density on the implantation and torsional angular displacement amplitudes were investigated. The results indicated that ion implantation layering can improve resistance to torsional fretting wear and thus has wide potential application for the prevention of torsional fretting damage in artificial implants. The damage mechanism prevented by the ion implantation layer on the Ti6Al7Nb alloy is a combination of oxidative wear, delamination and abrasive wear. An increase in ion implantation concentration inhibited detachment by delamination.

1. Introduction

Many clinical conditions, including but not limited to arthritis, trauma and congenital or acquired joint diseases, lead to a loss of cartilage tissue in human joints. The intrinsic avascular and aneural nature of cartilage provides little scope for natural self-repair. As a result, millions of people require treatment to repair damaged cartilage, usually at the major load-bearing articular joints such as the hips and knees [1].

Early clinical results provide evidence that loosening of one or both components of the Total Joint Replacement Surgery (TJR) occurs in 10% to 70% of patients 10 years after surgery [2]. Recent large-scale studies report that 10–20% of new joints must be replaced within 15–20 years, with aseptic loosening accounting for approximately 80% of these revisions [3]. Joint contacts experience very complex kinematics during walking, which is a combination of rolling and sliding. Three different sliding modes (transversal, radial, and circumferential) have been defined in the literature [4,5]. Torsional wear is one of the main motion modes of joints, and torsional fretting can be defined as the relative motion induced by reciprocating torsion under an oscillatory vibratory environment. Several solutions have been proposed to understand torsional contact and the mechanics of its behavior [6–8].

Titanium alloys are attractive materials for the manufacture of implants for medical and dental applications due to their superior biocompatibility and outstanding corrosion resistance compared with other conventional metallic materials [9,10]. Commercially pure titanium and Ti6Al4V alloy are the most commonly used materials in the manufacture of implants. Due to controversy around the potential toxic effects of vanadium compounds, V-free alloys such as Ti6Al7Nb have been recently developed for biomedical applications [11,12]. Ti6Al7Nb alloy is widely used because of its advantages in biocompatibility, mechanical properties and corrosion resistance [13,14]. Plasma immersion ion implantation–deposition (PIIID) is a rapidly developing surface modification technique that has shown effectiveness in modifying the physicochemical characteristics of thin films and mixing layers. In PIII–D, the target is typically enshrouded in self-excited plasma generated by applying a large negative voltage to the target [15,16].

However, the torsional wear behavior of Ti6Al7Nb and its implantation-treated nitrogen ion layer have not been clearly discussed. For a given torsional contact configuration, the resulting contact zone kinematics were found to profoundly influence accumulation, compaction and displacement of the debris particles generated during contact.

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When the torsional angular displacement amplitude is very small, accommodation by elastic deformation and a small plastic deformation are observed, and torsional fretting occurs in the partial-slip regime (PSR). When the torsional angular displacement amplitude is very large, which means that gross slip occurs throughout the process, torsional fretting occurs in the gross slip regime (GSR). The intermediate state is a mixed fretting regime (MFR). One dramatic phenomenon that occurs in MFR is a change in wear morphology from annular to an entire worn zone, accompanied by the disappearance of the sticking zone as a function of the number of cycles [7,8,20].

In this research, a new testing method was applied to reveal and compare the torsional fretting behavior of untreated and nitrogen-ion-implantation-treated biomedical Ti6Al7Nb alloy.

2. Experimental procedures

2.1. Materials

Biomedical Ti6Al7Nb alloy ((wt %): 5.88Al, 6.65Nb, 0.03Fe, 0.10C, 0.20O, 0.07N, 0.02H and 87.05Ti) was used in the present investigation as a rod supplied by the Northwest Institute for Nonferrous Metal Research, Xi'an, China. The rod was machined flat to dimensions of $10\times10\times25$ mm^3. One side (10 mm $\times$ 25 mm) of each sample was ground with 1500-grit diamond paper and then polished to a roughness of about R_a=0.5 μm before plasma immersion ion implantation [11].

2.2. PIII–D treatment

Ti6Al7Nb alloy was laid on a stainless steel substrate attached to an insulated stainless steel electrode in the center of the vacuum chamber, and a negative voltage was applied to the electrode.

The facility was equipped with an RF plasma source, hot filament glow discharge source, vacuum arc source, etc. The chamber was 1200 mm in height and 1000 mm in diameter. Before PIII, the samples were sputter-cleaned with argon plasma ion bombardment. The pre-treatment instrumental parameters were: RF forward energy=1000 W with reflected power of approximately 20 W, bias voltage=2.5 k V, gas flow=10 sccm, and clean time=40 min. Nitrogen (N_2) was bled into the vacuum chamber and nitrogen plasma was sustained using an RF power supply with a power of 1000 W, a work pressure of 5.5×10^{-4} Torr, and gas flow of 20 sccm. The implantation time was 40 min and the nitrogen-ion densities of implantation were 3×10^{17},5×10^{17}, 7×10^{17} and 9×10^{17}. After the immersion tests, optical microscopy was used to observe the surface morphology.

2.3. Fretting wear tests and analysis method

Torsional fretting wear tests of untreated and plasma implantation nitride Ti6Al7Nb alloy against a Zr_2O ball (with diameter of 25.2 mm and a roughness of Ra=0.2 μm) were performed under simulated physiological conditions in a torsional fretting wear test rig [17]. The test medium was a 20% bovine serum solution (Shanghai Bao Man Biological Technology Co., Ltd.). All tests were performed at 25 °C in naturally aerated solution. Following immersion tests, an optical microscope was used to observe the surface morphology. Angular displacement of the contact pair was measured and controlled by a sensor in the motor system and then acted on as the control signal fed back to the control unit of the tester. In this study, torsional fretting tests were performed under a normal load of 100 N at a constant rotary speed of 0.2°/s. The torsional angular displacement amplitudes were set at 0.5°, 5°, 15°, and 45°, and the number of cycles varied from 1 to 1000. After the torsional fretting wear tests, the 3D profiles were observed using a profilometer (Nano Map-Dual Mode), and surface morphologies and chemical analyses were investigated using a scanning electron microscope (SEM, KYKY2800) with an energy-dispersive X-ray spectrum (EDX).

3. Results and discussion

3.1. Surface analysis

After PIII treatment without auxiliary heating, all surface samples were gold in color, typical of titanium nitride, although the formation of this phase was not detected by XRD. Fig. 1 includes SEM images of the original polished and nitrogen-ion PIIID-processed surfaces.

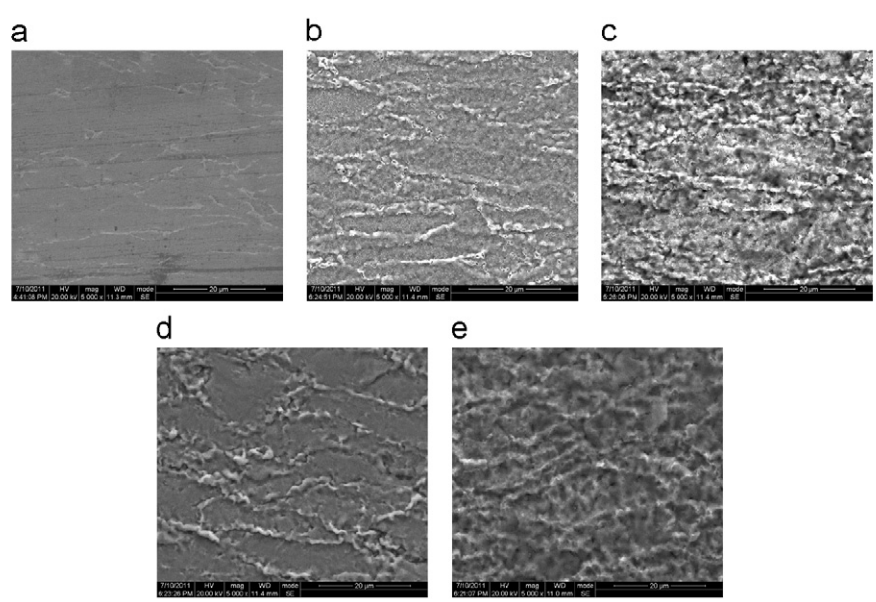

Fig. 1. Surface SEM morphologies of pre-test specimens. (a) substrate, (b) $3\times10^{17}N^+$, (c) $5\times10^{17}N^+$, (d) $7\times10^{17}N^+$ and (e) $9\times10^{17}N^+$.