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Comparison of the torsional fretting behavior of three porous titanium coatings for biomedical applications

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ABSTRACT

This research evaluated the torsional fretting wear proprieties of three porous bio-coatings modified with anodic oxidation, acid–base treatment, and alkali–heat treatment. Torsional fretting wear tests were carried out under a ball-on-flat contact configuration. Torsional fretting wear mechanisms were investigated by a combination of scanning electron microscope observations, energy dispersive X-ray analysis and X-ray diffraction analysis. Results showed that the torsional fretting wear resistance of anodic oxidation coating was better than that of acid–base coating and alkali–heat coating. Adhesive wear was the main damage mechanism of the three porous titanium coatings in partial slip regime. The combination of abrasive wear, delamination, and material transfer was the main wear mechanism of mixed and gross slip regimes.

1. Introduction

Titanium (Ti) and titanium alloys are frequently used as implant materials because of their excellent properties [1]. The morphology features of the implant play an important role in osseointegration rate [2]. Rough surface of dental implant provides larger contact area, good mechanical interlocking between implant surface and natural bone, further accelerates ossointegration, and is beneficial for stress distribution during mastication function [3,4]. In order to improve surface morphology of dental implants, researchers have explored many surface modification methods to form porous titanium, including sandblasting, acid etching, electrochemical treatment, and thermal spray coating, and so on [5–7]. The pores in titanium decrease the fracture strength and elasticity modulus of implant, which can effectively lightens or eliminates stress concentration and ultimately protects bone tissue around the implant when stress is imposed on an implant [8,9].

Dental implant repeatedly bears external forces in different directions during mastication functions; relative small displacement (less than 100 μm: fretting) occurs in all the interfaces of the implant system, including bone–implant and screw–implant interfaces [10–12]. Torsional fretting is inevitable at the osseointegration interface when dental implant suffers lateral occlusion force, which affects implant life [13]. This paper aimed to (i) investigate the torsional fretting behavior of the three kinds of porous titanium coatings in dental implant (ii) analyze the mechanism of torsional fretting damage (iii) provide scientific basis for choosing different surface treatment methods of porous materials to resist the torsional fretting damage.

2. Materials and methods

2.1. Preparation of porous titanium coating

Blocks of pure titanium ($1 \times 1 \times 1.25$ cm^3; ASTM standard) used in this experiment were purchased from the Northwest Nonferrous Metal Research Institute. The surface of each specimen was serially grinded in decreasing order of waterproof abrasive paper (400#, 800#, 1200#, and 2000#) and then polished to obtain flat, standardized surfaces. The specimens were ultrasonically cleaned three times each with petroleum ether, acetone, ethanol, and deionized water for 10 min. Finally, they were dried in room temperature.

2.1.1. Preparation of anodic oxidation (AO) coating

The sample was fixed on two pure titanium plates as anode and cathode. The titanium plates were then connected to the anode oxidation devices (Sichuan University Center for Biological Materials

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self-developed) separately. The poles and sample were immersed in 1 M H_2SO_4 solution for 5 min and underwent oxidation treatment under 40 V for 1 min. The voltage was gradually increased to 80 V and then another oxidation treatment was conducted for 1 min. Finally, the sample was washed three times with deionized water and allowed to dry naturally.

2.1.2. Preparation of alkali–heat treatment (AH) coating

2.1.2.1. Alkali treatment. The samples were immersed totally in 10 M NaOH solution and placed in a constant temperature oven (GHG-9246A; China) at 60 °C for 24 h. The samples were then cleaned three times with deionized water and dried in air.

2.1.2.2. Heat treatment. After drying in air, the sample was placed in a high-temperature sintering furnace (LHT-0817; Germany) at 600 °C for 1 h and then underwent furnace cooling.

2.1.3. Preparation of acid–base treatment (AB) coating

2.1.3.1. Acid treatment. The sample was immersed totally in acid solution, which was mixed with 37% HCl, 98% H_2SO_4, and deionized water (1:1:2). The mixture with the sample was heated in a constant temperature water bath (70 °C) for 30 min, cleaned three times with deionized water, and dried in air.

2.1.3.2. Alkali treatment. After drying in air, the sample was immersed totally in 6 M NaOH solution and heated in a constant temperature oven (70 °C) for 5 h. The sample was then cleaned three times with deionized water and dried in air.

2.2. Torsional fretting experiment

In this study, torsional fretting wear tests were carried out under a ball-on-flat contact configuration [13,14]. Three kinds of treated coatings and pure titanium (SU) were selected as flat specimens; ZrO_2 (Φ28.575 mm) was used as the counter-body during the fretting tests [15]. A constant rotary speed was set as 0.1°/s, and the torsional displacement amplitudes (θ) were 0.2°, 0.5°, 1°, 2°, and 5°. A constant normal load of 50 N was imposed during the tests, and the test cycles varied from 1 to 5000. The medium was deionized water with a temperature of 37 ± 0.5 °C. Friction torque was monitored by a six-axis force/torque sensor during the whole test.

2.3. Material characterization and analysis methods

2.3.1. Microhardness, surface roughness and wear depth

Microhardness of all samples was measured by a microhardness tester (MVK-H21; Akashi, Japan) with a Vicker diamond probe. Surface roughness and wear depth were measured by a step profilometer (AMBIOS XP-2; Ambios Tech, USA).

2.3.2. Morphological observation and energy dispersive X-ray (EDX) analysis

Material surface microstructure and torsional fretting damaged area were observed under an optical microscope (BX60M; JAPAN) and a scanning electron microscope (SEM; INSPECT F, FEI; The Netherlands). The chemical element contents of the damaged area and wear debris were measured using an EDX spectroscopy, which is an appendix of SEM.

2.3.3. X-ray diffraction (XRD)

Three kinds of coatings and substrate surface were analyzed by XRD (XPert PRO MPD, Philips).

3. Results

3.1. Surface features of porous coating

Fig. 1 shows the morphology characteristics of the 3 different coatings after surface treatment. AO coating formed relatively homogeneous honeycomb holes, which have a diameter of 50–300 nm. Some holes were connected to each other and fused into bigger holes. After AH treatment, the surfaces presented a structure of uniform needle plate, which was composed of nanoparticles. The pores between the plates mutually connected and formed the gaps with a width of about 300 nm. After the AB treatment, the surfaces showed a net-hole structure, which appeared like a coral reef. The inside meshes were connected to each other and constituted the 3D surface. The diameters of these meshes were not uniform; some meshes were as large as 1 μm.

The test result of the surface hardness of the three coatings was AO > AH > AB (Fig. 2). SEM analysis showed that the diameter of the holes was relatively small after AO treatment. The walls were relatively thick and exhibited strong resistance to the probe in the microhardness test. After the AB treatment, the surface showed porous mesh morphology. The anti-compressive ability of the surface was relatively weak; thus, the hardness was relatively low. Surface roughness was AO < AH < AB, which was similar to the results observed from SEM. As shown in Fig. 3, the maximum wear depth of the three coatings increased with the angular displacement. The torsional fretting wear depth of AO coating was less than the other two coatings under the same fretting test condition.

After AO treatment, the surface contained not only Ti, but also a relatively large amount of rutile and anatase titanium dioxide (Fig. 4). After AH treatment, the surface was mainly composed of

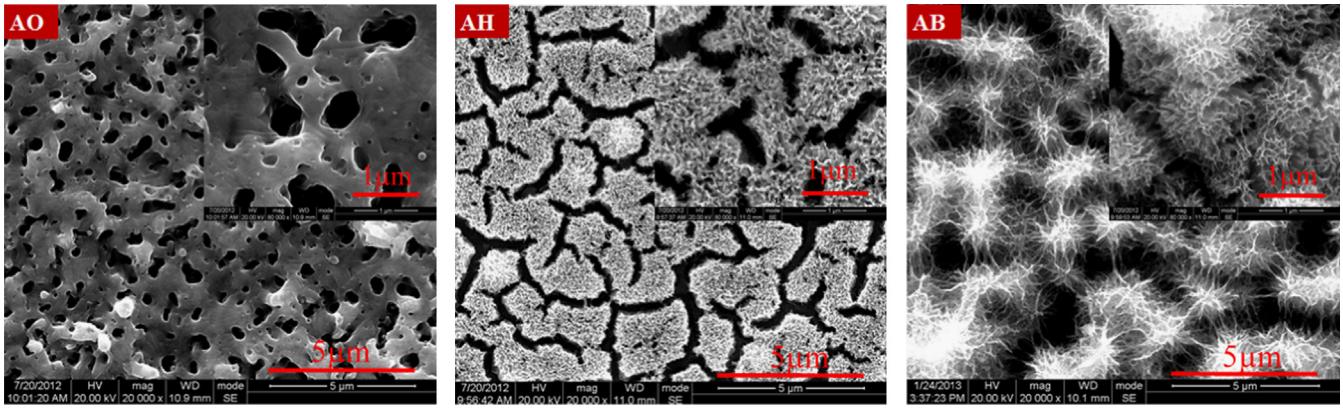

Fig. 1. SEM micrographs of the three coatings after surface treatments.