

Effect of povidone–iodine deposition on tribocorrosion and antibacterial properties of titanium alloy

Yu Yan^{a,*}, Yanbo Zhang^a, Qikui Wang^b, Hongwu Du^c, Lijie Qiao^a

^a Corrosion and Protection Center, Key Laboratory for Environmental Fracture (MOE), University of Science and Technology Beijing, Beijing 100083, China

^b Hebei General Hospital, Shijiazhuang, China

^c School of Chemistry and Biological Engineering, University of Science and Technology Beijing, Beijing, China

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ABSTRACT

Infection remains one of the most common causes for the early-stage failure of orthopaedic implants. Many methods have been developed to reduce the growth of bacteria. However, devices such as orthopaedic implants involve relative motion in several parts, and suffer wear from tribocorrosion processes. The surface pattern and texture can be damaged and the anti-bacterial efficiency reduced. In this paper, a two-stage method is reported. Povidone–iodine (PVP–I) was deposited on the titanium alloy surfaces and inside the pores to provide a longer release time. The results show that even under tribological tests, the anti-bacterial performance still remains satisfactory.

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1. Introduction

In surgery, disinfection, aseptic operation, and pre- and post-operative application of antibiotics are all used to reduce implant infection. However, infection remains common particularly among the elderly and diabetics, due to low post-operative resistance and the prevalence of drug-resistant bacteria [1]. After infection, the effectiveness of antibiotics is greatly reduced, making it necessary to irrigate the implant in the long term or to remove it [2]. It has been shown that infection is diagnosed in approximately 15% of revision total hip arthroplasty (THA) in the United States, corresponding to the third most frequently reported reason for revision. If a revision is needed, the cost is greater than the primary replacement. In addition, an extension of medical care is necessary for infected cases. Overall, the cost of infection can be increased dramatically and the patients need to undergo more operation and treatment [3].

Povidone–iodine (PVP–I) is recognized worldwide as the most secure and reliable antiseptic. It is biocompatible and a trace nutrient. The World Health Organization and various countries have targeted PVP–I research as a policy initiative [4]. PVP–I is a chemical complex with a carrier or enclosed by a ring structure to facilitate stability [5]. Clinical trials conducted on 222 patients with X-ray

fluorescence analysis revealed that six months after the external fixation pin was implanted, the PVP–I content was 50% of its initial value. After one year, this was 20% to 30% of the baseline. An additional series of trials showed that PVP–I-containing implants could effectively prevent and treat post-operative infection after orthopaedic surgery with no cytotoxicity or adverse reactions [6]. Other researchers studied the in-vitro antibacterial performances of titanium–I₂ and pure titanium, and reported that titanium–I₂ exhibited antibacterial activity and significantly reduced the incidence of pinhole infection. Therefore, titanium–I₂ shows good prospects as an antibacterial biomaterial [7].

Since titanium and titanium alloys were introduced to the medical field in the 1940s, they have been widely applied because of their good biocompatibility [8]. However, titanium alloys with no special treatments do not have particularly high wear resistance, and hence cannot serve as load-bearing interfaces. As a result, surface treatment of titanium alloys to improve their biocompatibility and mechanical properties is an active area of research [8–11]. The most commonly used surface modification method for titanium alloys is anodic oxidation. It has been shown that high potential favours the formation and growth of particulate anodic oxides [9]. This leads to the formation of a large number of coarse anodic titanium oxide particles, increasing the roughness of the sample surface and leading to the further formation of crystalline titanium oxide nanotubes. Indeed, the higher the potential, the higher the degree of crystallinity of the titanium oxide on the surface.

* Corresponding author. Tel.: +86 10 6233 4499; fax: +86 10 6233 2345.
E-mail address: yanyu@ustb.edu.cn (Y. Yan).

When surface anodic oxidation was applied to TC4 titanium alloy to prepare TiO_2 porous film, it was found that the formed TiO_2 oxide films were all rutile-type and anatase-type duplex oxide films [12]. As the voltage increased and the time progressed, the proportion of rutile-type TiO_2 increased and that of anatase-type TiO_2 decreased. Over time, the oxide film became predominantly composed of rutile-type TiO_2 . Komotori et al. [13] studied the effects of different friction velocities on the tribocorrosion performance of $\text{Ti}_6\text{Al}_4\text{V}$, and found that the higher the friction velocity, the lower the corrosion potential in the equilibrium process of the interaction between wear and corrosion. A high friction velocity was not conducive to the formation of a passive film. This reduced the fluctuations in corrosion potential. As a result, as the friction velocity became close to human walking speed, the repassivation ability of the titanium alloy surface declined.

Although titanium alloys normally are not used for bearing surfaces, they still involve relative motion [14]. Therefore, their durability, especially their antibacterial performance after the top surface is damaged by wear processes, is of great concern. Tribocorrosion is a material degradation process induced by the interaction of wear and corrosion [15–20]. For titanium alloys, their high corrosion resistance relies on the dense and protective oxide film. However, under tribological contact, the passive film can be locally or totally removed by the relative motion, resulting in an acceleration of the corrosion rate. In the present study, medical-grade $\text{Ti}_6\text{Al}_4\text{V}$ alloy was tested. Anodization was used to directly obtain a nanocrystalline TiO_2 porous film on the surface of a titanium alloy substrate. Then, electrochemical deposition was applied to deposit PVP-I in the pores. The tribocorrosion performance and antibacterial properties of the material were examined.

2. Materials and methods

The material used in the experiment was $\text{Ti}_6\text{Al}_4\text{V}$, corresponding to the United States ASTM F136 standard and of size $\varnothing 20 \times 2$ mm. Its chemical composition is shown in Table 1. The sample was polished sequentially with #280, #600, #1000, #1500 and #2000 sandpapers and then blow-dried. After ultrasonic cleaning in acetone for 15 min, the sample was subjected to chemical polishing for 60 s (hydrofluoric acid, HF mass fraction 1.5%) followed by ultrasonic cleaning in ionized water for 15 min, and then dried.

A steady-flow DC power supply was used to anodize the sample. The electrolyte was 0.5 mol/L H_2SO_4 . During the experiment, the $\text{Ti}_6\text{Al}_4\text{V}$ served as the anode, and the cathode was 316 L stainless steel. The DC voltage was held constant. The anode and the cathode were opposite each other and 5 cm apart throughout the experiment. A uniform solution concentration was maintained via magnetic stirring.

The morphology of the sample surface after anodization was observed with scanning electron microscopy (SEM). X-ray diffraction (XRD) analysis was applied to determine the phases of the sample surface. A wear tester (CETR UMT-2, US) and an electrochemical workstation (CHI 660E, Shanghai) were intergraded to examine the tribocorrosion properties of the material after different treatments. Electrochemical deposition was applied for PVP-I deposition.

The tribocorrosion tests were carried out using the apparatus shown in Fig. 1. The electrolyte used was a NaCl solution with a mass fraction of 0.9%. The reference electrode was an Ag/AgCl

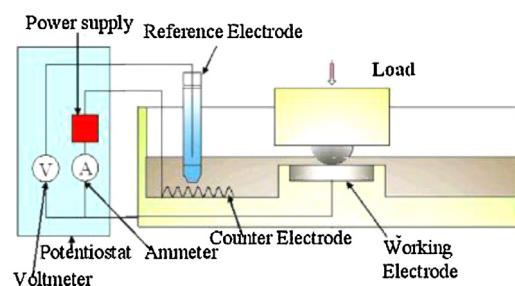


Fig. 1. Schematic diagram of the tribocorrosion tests apparatus.

electrode, and the counter electrode was a platinum wire. The tribological counterpart was made of Si_3N_4 , with a diameter of 5 mm. The frequency was 1 Hz, and the average velocity was 30 mm/s. The test temperature was maintained at 37 °C.

In the antibacterial experiment, the first colonies were transferred from solid lysogeny broth (LB) medium to liquid LB medium. They were then placed in a 37 °C THZ-C constant-temperature oscillator to culture the bacteria into an activated state. The incubator used was a Blue Pard water incubator set to 37 °C. All operations were performed on a sterile bench. Before the experiment, the sample was sterilized in a high-pressure steam sterilization pot at 121 °C for 20 min. The bacteria were *Staphylococcus aureus*. The LB medium was composed of 1 L deionized water, 10 g tryptone, 5 g yeast extract, and 10 g NaCl, and the pH was adjusted to 7.0 with 5 mol/L NaOH. For a solid medium, 15 g agar was added to the liquid LB. The steps of the experiment were as follows: (1) the sample was placed in a Petri dish, and after sterilization 100 μL bacterial suspension diluted 10-fold was added to the surface and covered; (2) the bacteria were cultured at 37 °C for 18 h; (3) the sample surface was washed with 5 mL PBS buffer; (4) after rinsing the sample surface, 20 μL of PBS buffer was added to the solid LB medium; (5) the bacteria were again cultured at 37 °C for 24 h.

3. Results and discussion

3.1. The impact of different treatments on the morphology and composition of $\text{Ti}_6\text{Al}_4\text{V}$

Constant-voltage anodic oxidation can be divided into three stages: (1) formation of an oxide-film barrier layer; (2) formation of nanopores in the porous layer; and (3) equilibrium growth of the nanopores [12]. In the first few seconds of the anodization, the current rapidly reached a peak value and then rapidly declined. A dense oxide film formed on the surface of the titanium substrate, which caused micro undulation. Consequently, the density distribution on the surface of the barrier layer became uneven, and the electric field was concentrated on the depressions of the oxide film surface. This increased the dissolution rate at the barrier layer in the depressions, resulting in a nanoscale porous structure on the oxide film.

Fig. 2(a) shows the sample after polishing. The $\text{Ti}_6\text{Al}_4\text{V}$ alloy is composed of two phases, α and β . The white projections are in the β phase, whereas the black parts are in the α phase. From Figs. 2(b) and (c), it is clear that under both 120 V and 130 V, the sample surfaces show a uniform porous morphology. The pore diameters are slightly larger under 130 V than 120 V. The former is in the range of 100 nm to 200 nm. Comparing (b) and (d), it is clear that after PVP-I deposition, the sample surface is still porous, but the pore sizes are slightly reduced. This is possibly due to the deposition of PVP-I inside the pores, which altered the diffusion and oxidation processes.

TiO_2 has three polymorphs—rutile, anatase and brookite. Of these, rutile and anatase TiO_2 are widespread. These two types of

Table 1
Chemical composition of $\text{Ti}_6\text{Al}_4\text{V}$.

Element	Al	V	Fe	C	N	H	O	Ti
Mass fraction (%)	6.1	4.05	0.2	0.03	0.02	0.008	0.12	Bal.

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