



## In vitro studying corrosion behavior of porous titanium coating in dynamic electrolyte



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### ABSTRACT

Porous titanium (PT) is considered as a promising biomaterials for orthopedic implants. Besides biocompatibility and mechanical properties, corrosion resistance in physiological environment is the other important factor affecting the long stability of an implant. In order to investigate the corrosion behavior of porous titanium implants in a dynamic physiological environment, a dynamic circle system was designed in this study. Then a titanium-based implant with PT coating was fabricated by plasma spraying. The corrosion resistance of PT samples in flowing 0.9% NaCl solution was evaluated by electrochemical measurements. Commercial pure solid titanium (ST) disc was used as a control. The studies of potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) show that the pores in the PT play a negative part in corrosion resistance and the flowing electrolyte can increase the corrosive rate of all titanium samples. The results suggest that pore design of titanium implants should pay attention to the effect of dynamic process of a physiological environment on the corrosion behavior of implants.

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### 1. Introduction

Porous titanium (PT) and its alloys have been proved to be a promising biomaterials for orthopedic implants and hard tissue replacements. Many advantages of PT implants, such as good biocompatibility, low density, adjustable mechanical performance, bone tissues in-growth ability, have been reported by many different groups [1–8]. Meanwhile, as an implanted materials, it should possess high corrosion resistance in highly corrosive body environment. Because the failure of an implant due to corrosion has remained as one of the challenging clinical problems [9–14].

With respect to PT and its alloys, several researches have reported their corrosion behaviors in simulated body fluids. Seah et al. [15] has showed that the corrosion rate of porous titanium is significantly higher than that of solid titanium in 0.9% aqueous NaCl solution. Then seah [16] studied the influence of pore morphology of porous titanium on corrosion and indicated that small, isolated pores trap ionic species and exhaust the supply of oxygen; on the contrary, an open, interconnected pore allows the free flow of species resulted in higher resistance to pitting. Xu et al. [17] fabricated porous NiTi alloys by microwave sintering and evaluated its corrosion resistance in Hank's solution, and found that the corrosion current density of the porous NiTi alloy is

about three times higher than that of the dense NiTi. Sun et al. [18] carried out a comparative study on corrosion behaviors of porous and dense NiTi alloys in a 0.9% aqueous NaCl solution and suggested that the porous NiTi alloys are more susceptible to localized corrosion than dense NiTi. The other studies also indicate that pores have strong effects on corrosion characteristics of porous Ti alloys which are less corrosion resistant than solid ones [19].

However, all the studies were carried out in an immobile electrolyte, which ignored the flowing of body fluid encountered by implants in hosts. We believe that it is not accurate to employ a static model to simulate the dynamic process. Meanwhile, corrosion of a material would behave differently in flowing or static electrolyte. Abd Rabbo· et al. [20] studied the corrosion behaviors of Ti in KBr electrolyte at different flow rates and found that the breakdown potential ( $E_b$ ) measured in static solution decreases as the flow rate increases. The pit growth at all values of flow is noticeably fast compared with the rate of growth in static solution. Nescic et al. [21] pointed out that surface inhibitor films forming on a metal surface can reduce corrosion rate. However, the films can be removed locally or globally by chemical dissolution or mechanical force. In dynamic electrolytes, shear stress from flowing of electrolyte is connected with the onset of the film removal.

To the best of our knowledge, there is no study on the corrosion of PT in flowing electrolyte for simulating the circulating of body solution. In this study, we designed a circle system for realizing the flow of electrolyte at a certain rate. In this circle system, we employed electrochemical

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measurements to study the corrosion of PT coating fabricated by plasma spraying in our previous work [22] in a flowing 0.9% aqueous NaCl solution at 37 °C. Solid titanium (ST) was used as a control for the tests. The results of this study would guide the pore design for PT implants.

## 2. Materials and methods

### 2.1. Preparation of PT coating

In this study, we used plasma spraying to fabricate PT coatings on titanium discs ( $\Phi 14 \times 1$  mm). The detail process has been described in Ref. [22]. Simply, the main process includes the following three steps. First, a special three-layer sphere (HA core,  $\text{CaCO}_3$  middle layer and Ti outside layer) was prepared by layer-to-layer process; second, the three-layer spheres were sprayed on titanium discs; finally, the as-sprayed samples were soaked in the 1 M HCl solution for formation of porous structure.

### 2.2. Preparation of electrode

For electrochemical test, the PT samples were encapsulated in epoxy resin. Following the fixation of copper conducting wires on one side (without porous coating) of the titanium discs, the samples were embedded in epoxy resin only with the porous coatings exposing in the air. Meanwhile, ST discs used as control were embedded in epoxy resin as well. For all samples, after the epoxy resin solidified, the non-embedded sides were polished with 1500 grit water-proof emery papers under running water, cleaned by sonication in acetone, washed in ethanol and distilled water, and then dried in an oven at 60 °C. The area of electrode is about 12 mm<sup>2</sup>.

### 2.3. Design of dynamic circle system

For simulating the flow of body fluid in human bodies, we designed a dynamic circle system. The schematic of the system was shown in Fig. 1. A peristaltic pump supplied the power for sustaining flow of electrolyte. A storage tank was used for storing 0.9% aqueous NaCl solution. A glass

electrolytic cell with sealing cover was connected to the storage tank by silicone tubes. Some holes were reserved in the sealing cover for fixing the work electrode, reference electrode, counter electrode, thermometer and glass tubes for entrance and exit of  $\text{N}_2$ . The thermometer, glass tubes and all electrodes were firmly placed in the holes by rubber plugs. During the electrochemical tests, the electrolytic cell always was immersed in a 37 °C water bath.

### 2.4. Electrochemical tests

The electrochemical measurements were performed using an electrochemical workstation (Solartron1287 + 1260), according to the ASTM G5. The process underwent in flowing or static electrolyte. For in flowing electrolyte, the electrochemical tests were performed in the dynamic circle system keeping the flowing of electrolyte at 2 mL/100 mL min (means 2 mL NaCl solution pouring per minute from 100 mL NaCl solution stored in the storage tank) according to normal physiological flow rate of human skeletal muscle tissue. In static electrolyte, the measurements were carried out in a baker placed in a constant temperature water bath. We defined the samples PT and ST in static electrolyte as PT-s and ST-s, respectively, and the samples in dynamic electrolyte as PT-d and ST-d. The electrolyte was 0.9% NaCl solution of which the temperature was kept at 37 °C. A saturated calomel electrode and a platinum wire with a surface area of  $1 \times 1.5$  cm<sup>2</sup> were used as the reference electrode and counter electrode, respectively. Potentiodynamic curves were measured by scanning the potential from  $-0.5$  V below the open circuit potential (OCP) to  $+1.2$  V at a scan rate 1.67 mV/s. Tafel approximation was employed to determine the corrosion potential and corrosion current density. The electrochemical impedance spectroscopy (EIS) measurements were operated at OCP, hold potentiostatically stable, in the frequency range of 100 kHz to 1 mHz using a 10 mV peak-to-peak voltage excitation. EIS spectra were interpreted by the software ZSimpWin 3.10. Before measurements of polarization curves and EIS, the working electrode was immersed in the test solution at OCP for 30 min to attain a stable state. Each electrochemical test was repeated three times with the fresh sample and electrolyte. For all assessments, prior to beginning to test,  $\text{N}_2$  was aerated into the electrolyte for 30 min for removal of oxygen.

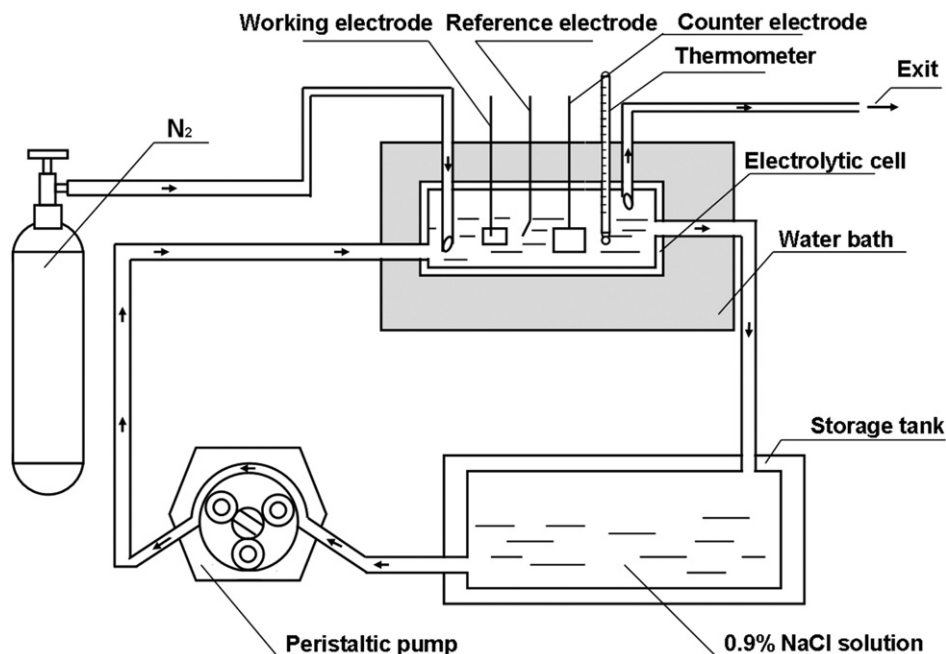


Fig. 1. Schematic of the dynamic circle system.

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