

# Surface Roughness and Hydrophilicity of Titanium after Anodic Oxidation



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**Abstract:** Anodic oxidation was applied to prepare the nanostructured titanium surface with different roughnesses and hydrophilicities. The morphology was characterized by scanning electron microscopy (SEM). The surface roughness was tested by atomic force microscopy (AFM), and the hydrophilicity was assessed from the contact angle between the deionized water and sample surface at room temperature. The results show that surface morphologies change remarkably with applied voltage and oxidation time during anodic oxidation. Under optimized oxidation conditions, well-ordered nanotube arrays were fabricated on the Ti surface. Roughness values increase with increase of the oxidation time, ranging from several dozen to several hundred nanometers, while the influence of voltage on surface roughness is not obvious. The hydrophilicity increases initially with the increase of oxidation time, but then decrease. The variations of surface morphology, roughness and hydrophilicity are correlated to the reactions occurring during the anodic oxidation.

**Key words:** titanium; anodic oxidation; roughness; hydrophilicity

Titanium and its alloys are widely applied as implant materials in many medical fields including dentistry and orthopedics due to their excellent mechanical properties, high corrosion resistance and good biocompatibility<sup>[1,2]</sup>. It was reported that these favorable properties resulted from the chemical stability and the structure of the titania (TiO<sub>2</sub>) film which grew spontaneously on the surfaces of Ti and its alloys upon exposure to air. However the TiO<sub>2</sub> film is bioinert, which usually leads to the insufficient osseointegration<sup>[3,4]</sup>. Therefore, the surface modification of Ti and its alloys is required to enhance their bioactivity.

A number of methods (e.g., sand blasting, acid etching, hydroxyapatite coating) have been employed to modify the Ti implant surfaces. However, a major disadvantage of these approaches is that they are either incapable of

producing highly controllable surface structure, or prone to impose toxicity on the cells/tissues due to the harmful chemical residuals on the implant surface. Anodic oxidation is a low-cost, efficient, versatile technique to produce oxide coatings with highly controllable nanostructures. The anodic oxidized coatings usually have strong interfacial adhesion due to their direct in-situ growth on metal substrates, and the discharging-associated thermal effect also makes the coating rigid. During recent years, TiO<sub>2</sub> coatings with controllable nanostructures have been produced on Ti surface by anodic oxidation and proved to have good bioactivity<sup>[5]</sup>. Therefore, the anodized TiO<sub>2</sub> coatings have great potential as bioimplant materials in clinic.

The nanostructure of the oxidized TiO<sub>2</sub> coatings has been

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proved to play a critical role in improving the bioactivity. However, till now, the mechanism of the beneficial effect of nanostructures on bioactivity is not well understood, and there is still no consensus on the optimal nanostructures for the biomedical application [6]. The bioactivity of biomaterials are known to strongly depend on their surface properties, especially surface roughness and hydrophilicity, which are two important factors regulating a number of biological events including protein adsorption, cell attachment and other aspects of cell behaviors including proliferation and differentiation [7]. Therefore, the investigation of the influence of surface nanostructures on the surface roughness and hydrophilicity will definitely help understanding how the surface nanostructures affect the bioactivity, thus, providing more insight into the design of the optimal surface on the implant surface.

In the present paper, to conduct the systematic research on the relationship between roughness, hydrophilicity and the anode oxidation induced nanostructures, TiO<sub>2</sub> coatings with different types of nanostructures were prepared by adjusting anode oxidation conditions (e.g. voltage and oxidation time), and the influence of nanostructures on surface roughness and hydrophilicity was also investigated.

## 1 Experiment

Commercially pure Ti plates with dimension of 10 mm×10 mm×1 mm were mechanically polished by 1000# abrasive paper, and then ultrasonically cleaned successively in acetone, ethanol and deionized water. Anodic oxidation was carried out in potentiostatic mode at room temperature using a direct current (DC) voltage source (WYK-150, Yangzhou, China) in 1.0 mol/L NaF electrolyte. The Ti plate was used as anodic electrode, and rectangular graphite was used as cathode electrode. The distance between anodic and cathodic electrodes was about 40 mm. After anodic oxidation, all samples were ultrasonically cleaned with deionized water, and then air-dried. Samples without anodic oxidation were used as the control group.

The surface morphologies of samples were observed using scanning electron microscope (SEM, HITACHI S-4800). The surface roughness was characterized by atomic force microscope (AFM, Agilent 5500). The hydrophilicity was assessed from the measurements of the contact angle between the deionized water and sample surface at room

temperature. Drop volumes of 2  $\mu$ L were chosen to avoid gravitation-induced shape alteration and to diminish the evaporation effects. The drop image was acquired using a digital camera (1280×960 pixels) attached to a microscope and processed by an image analysis software.

Samples were prepared in quintuplicate for each group. Standard deviations were plotted as error bars for the data points on all figures. Statistical difference was determined by Students t-test. Difference with  $p < 0.05$  was considered to be significant.

## 2 Results

### 2.1 Surface morphologies of anodized Ti

Fig.1 shows surface morphologies of the Ti samples treated by anodic oxidation at 10 V for 10 min, 30 min, 1 h and 4 h. From these surface images, it can be seen, when Ti samples are anodic oxidized at 10 V for 10 min, nanopores and nanotube arrays with diameters of 20~30 nm are obtained on the surface (Fig.1a). The diameters of nanopores and the nanotube arrays increase with the increasing of oxidation time (Fig.1b). When the oxidation time is 1 h, only nanotubes with inner diameters of 40~50 nm are formed, and uniformly distributed over the entire surface (Fig.1c). However, as the oxidation time is further prolonged to 4 h, the surface structure and the diameters of nanotubes remain unchanged (Fig.1d).

Upon increasing the voltage from 10 V to 20 V, the surface morphologies of the Ti samples treated for different time are shown in Fig.2. It can be seen that after anodic oxidation for 10 and 30 min, only nanopores are obtained and uniformly distributed over the entire surface on Ti substrate (Fig.2a), which is different from the case at a voltage of 10 V where both nanopores and nanotubes are formed simultaneously (Fig.1a, Fig.1b). The diameters of nanopores also increase with the increase of oxidation time (Fig.2b), which is in good agreement with the results shown in Fig.1. When the Ti samples are treated for 1 h, some nanotubes and collapsed structure are observed on the Ti surfaces (Fig.2c). As the oxidation time is further prolonged to 4 h, mainly collapsed structure is distributed over the entire surface. In addition, the diameter of nanostructures of samples treated at 20 V is much larger than that treated at 10 V (Fig.2d).

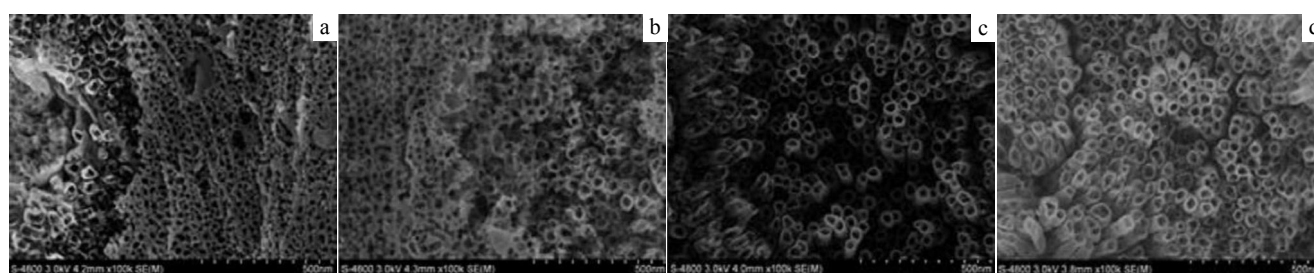


Fig.1 Surface morphologies of the Ti samples treated by anodic oxidation at 10 V for 10 min (a), 30 min (b), 1 h (c), and 4 h (d)

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