

Effect of Current Density on Anodised Titanium in Mixture of β -Glycerophosphate (β -GP) and Calcium Acetate (CA)

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Abstract

Anodic oxidation is an electrochemical method for the production of ceramic films on a metallic substrate. In this study, the surface morphology and crystallinity of titanium foil was modified by anodising in mixture of β -glycerophosphate disodium salt pentahydrate (β -GP) and calcium acetate monohydrate (CA). The experiments were carried out at high voltage (350 V), different anodising time (1, 3, 5 and 10 min) and current density (10 and 20 mA.cm⁻²) at room temperature. Anodised titanium was characterised by using glancing angle X-ray diffraction (GAXRD), field emission scanning electron microscope (FESEM) and focused ion beam (FIB) milling. The porosity and crystallinity of the oxide layer depended strongly on the current density.

Keywords: Titanium, Anodic Oxidation, Glancing Angle X-Ray Diffraction, Colour

Introduction

Anodic oxidation has been widely used to modify the surface characteristics and properties of titanium in order to improve the mechanical properties, corrosion resistance, biocompatibility, crystallinity and to eliminate the biotoxicity. Anodic oxidation of titanium allows for the controlled formation of a protective oxide surface layer which is much thicker than formed naturally [1]. The thickness of modified oxide layers can be 10 nm to 40 μ m [2]. These coatings may be dense or porous, and/or amorphous or crystalline,

depending on the parameters such as electrolyte type, solution concentration, applied potential, anodic time, current density and electrolyte temperature [1, 3]. By modifying the titanium surface in mixture of β -GP + CA, the thickness (> 1 μ m) and mineralogy of the oxide layer can be controlled. Moreover, the mixture of β -GP + CA electrolyte provides phosphorous and calcium ions which are embedded in the titanium surface during anodising [4]. This electrolyte mixture has been widely used as electrolyte for the anodising of titanium because of their role in modifying the surface of titanium to promote bone tissue growth and enhance anchorage of the implant to bone [5].

Experiment Method

High-purity titanium foils of dimension of 25 mm x 10mm x 0.5 mm were wet hand-polished using 1200 grit (~ 1 μ m) abrasive paper. Anodic oxidation was carried out in an electrochemical cell containing ~ 0.4 L of a mixture made from aqueous solution of β -GP and CA at ~ 25°C. These experimental parameters used are shown in Table 1.

Table 1: Parameters used for anodic oxidation.

Parameter	Values(s)
Electrolytes	0.02 M β -GP + 0.2 M CA
d.c voltage (V)	350
Current density (mA.cm ⁻²)	10, 20
Duration (min)	1,3, 5, 10

The anodising was done with a programmable power supply. The

mineralogical composition of the films was determined using GAXRD. The microstructure was examined using FESEM. The colour was recorded using digital camera. FIB milling was used for cross-sectional imaging.

Result & Discussion

Fig. 1 shows FESEM images of the anodised titanium. Fig 1 indicates that the higher current density facilitated the formation of both small and large pores at shorter times. The dielectric breakdown increases due to the increasing of the current density and leads to the faster polarization. Therefore, higher potential energy is required to breakdown the dielectric layer and it causes the pore size and pore number increase.

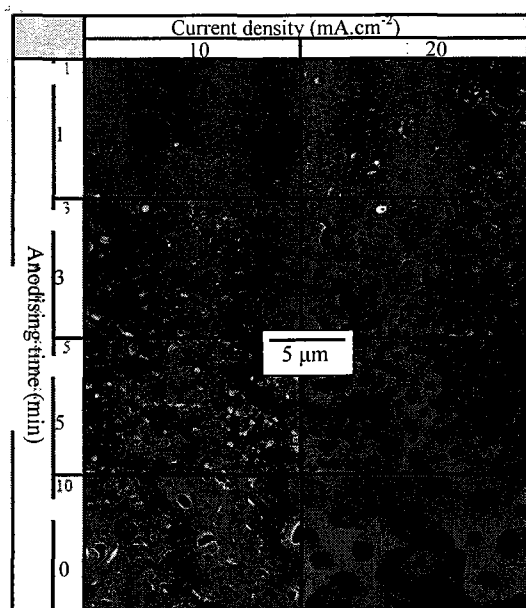


Fig. 1: FESEM micrograph of anodised surface in 0.02 M β -GP + 0.2 CA (350 V) at 10 and 20 mA.cm⁻² for 1, 3, 5 and 10 min.

The GAXRD pattern of anodised titanium at low current density (10 mA.cm⁻²) is shown in Figure 2. Amount of anatase and rutile increased as the increasing of anodising time. This study also revealed that the higher current density increased the amount of anatase and rutile. This is because the energy supplied to the system and temperature increases due to the increment

of current density. It is proved that anatase and rutile phases more stable than amorphous phase at higher temperature.

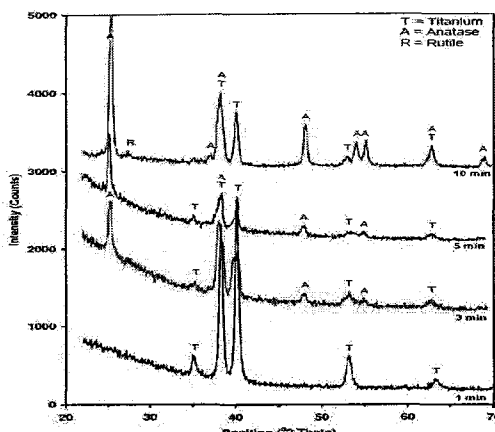


Fig. 2: GAXRD patterns of the samples anodised in 0.02 M β -GP + 0.2 M CA, current density 10 mA.cm⁻² at 350 V for 1, 3, 5, and 10 min.

Conclusion

As a conclusion, the porosity and crystallinity of titanium foil can be enhanced by increasing the current density because of the arching effect. Porous surface and anatase/rutile phases were formed at higher current density. Therefore, desired surface properties and crystallinity can be achieved easily by adjusting the current density of anodic oxidation to meet the clinical need.

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