Preparation and characterization of Si-containing titanium oxide film by micro-arc oxidation

Shih-Fu Ou[1,2,3],Fang-Yu Fan[2,4,5], Keng-Liang Ou [2,3,6*]

[1]Department of Mold and Die Engineering, National Kaohsiung University of Applied Science, Kaohsiung 807, Taiwan

[2]Research Center for Biomedical Devices and Prototyping Production, Taipei Medical University, Taipei 110, Taiwan

[3]Research Center for Biomedical Implants and Microsurgery Devices, Taipei Medical University, Taipei 110, Taiwan

[4]Department of Mechanical Engineering National Taiwan University

[5]Department of Dental Technology, Taipei Medical University, Taipei 110, Taiwan

[6]Graduate Institute of Biomedical Materials and Tissue Engineering, Taipei Medical University, Taipei 110, Taiwan

* corresponding author: Keng-Liang Ou

Micro-arc oxidation (MAO) has been widely used to surface modified titanium and its alloys for biomedical applications [1,2]. Also, Ca incorporating in the MAO film is beneficial to cell proliferation [3]. In addition, Si containing coating has been demonstrated to improve the cell adhesion [4]. Hence, this study prepared the Si, Ca, P containing electrolyte to fabricate titania films by MAO.

Commercial pure titanium (c. p. Ti) coupons (10×10×1 mm) were abraded with silicon carbide paper up to grade 800, cleansed with acetone for 5 min, followed by ethanol for 3 min in an ultrasonic bath, and finally dried at room temperature. The electrolyte contained 0.2M calcium acetate monohydrate ($Ca(CH_3COO)_2 \cdot H_2O$), 0.04M calcium glycerophosphate ($C_3H_7Na_2O_6P \cdot 5H_2O$), 0.04M Na_2(EDTA) and $Na_2SiO_3 9H_2O$ (0.04 M) as the source of Ca, P and Si for the MAO. Ti plates were micro-arc oxidized at a constant voltage of 300 V for 10 min using a direct current power supply, and a high density graphite plate was used as the cathode. The temperature during MAO was kept by a water cooled bath in a stainless steel tank. After micro-arc oxidized, the samples were rinsed with distilled water and dried in air. The surface morphologies of MAO films were observed using a scanning electron microscope (SEM; Model: PHILIPS XL30). The crystallinity and phase of the AOF were identified via glancing angle X-ray diffraction using a Kα radiation with an incidence angle of 1° and at a scanning speed of 2°/min (XRD; Model: PHILIPS X'Pert, X-ray tube PW3373/100 Cu LFF DK147515).

Fig. 1 shows the MAO film surface. The rough surface was consisted of large pores and small pores in size of nano-scale. The nano-pores were approximate 80-100 nm in diameter as shown in Fig. 2. The pores were formed by high energetic sparks during MAO. In addition, Ca, P and Si were detected from the MAO film except for Ti and O by EDX detection. The amounts of Ca in the MAO film greater than P were found. Previous studies indicated that P was easier to be incorporated into the MAO film as compared to Ca [] which was different in the results proposed in this study. Because phosphorus cations were forced under the applied electric field to migrate to anode but Ca anions moved toward the opposite direction. In this study, Ca^{2+} dissolved in the solution was gradually transformed into CaY^{2-} because Ca^{2+} was chelated by EDTA. The Violet parks also roughened the surface and the average surface roughness was measured as Ra 270nm. The XRD pattern shows the MAO film comprised anatase and rutile TiO_2.

References

1. H. Ishizawa, M. Ogino, J. Biomed. Mater. Res. 29 (1995) 65-69.
2. J. Y. Suh, B. C. Jang, X. Zhu, J. L. Ong, K. Kim, Biomaterials 24 (2003) 347-352.
3. J. Takebe, S. Itoh, J. Okada, K. Ishibashi, J. Biomed. Mater. Res. 51 (2000) 398.
4. Quanming W, Hongjie H, Yuqing Q, Zhengxiang Z and Junying S. J Mater Sci Technol, 2012, 28(2), 109-117.

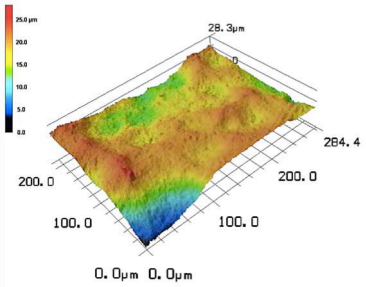

Fig. 1 Surface topography of the MAO film

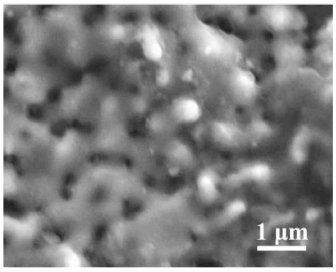

Fig. 2 Surface morphology of the MAO film