

Formation of an antibacterial oxide film on Ti-Nb alloy by anodizing oxidation

Shih-Fu Ou^{1,2,3}, Fang-Yu Fan^{2,4,5}, Keng-Liang Ou^{2,3,6*}

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

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

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

⁴Department of Mechanical Engineering National Taiwan University

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

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

Titanium and its alloys are widely used as dental and orthopaedic implant materials.¹ Nevertheless, for long-term implantation loosening of the prosthetic devices caused by “stress shielding effect” of the inserted implant on adjacent bone can occur.² To alleviate this effect, the present authors have developed the Ti-30Nb-1Fe-1Hf alloy with high strength and low elastic modulus (UTS: 914MPa, 0.2% Proof stress: 862MPa and E: 62GPa)³. The anodized Ti-30Nb-1Fe-1Hf alloy with an oxide film containing Ca and P was demonstrated to have good biocompatibility in our previous study⁴. However, anodic oxide film does not possess the properties to protect against bacterial adhesion or infection. Bacterial adhesion to implant surfaces is a major source of inflammation and infection. When bacterial adhesion occurs on implant surfaces, it is very difficult to be clinically treated because of the formation of bacterial biofilms, which can obstruct the immune response, systemic antibiotic therapy, and the integration of the indwelling implants with the surrounding tissue. In the present study, the Ti-30Nb-1Fe-1Hf alloy was treated with an improved anodic oxidation process by adjusting the electrolyte composition to incorporate Ca, P and Ag into the anodic oxide film. The relationship between microstructure and anti-bacterial ability against *S. aureus* of the oxide film was evaluated.

The material used in this study is Ti-30Nb-1Fe-1Hf plate. 10×10×1 mm coupons were abraded with silicon carbon papers up to grade 800, and were then cleansed with acetone for 5 min followed by ethanol for 3 min in an ultrasonic cleaner, and finally dried at room temperature. The electrolyte for anodizing treatment was composed Ca, P and Ag containing solution. The pH of the solution was around 7.8. The Ti-30Nb-1Fe-1Hf plate was galvanostatically anodized at a constant current density of 50 mA/cm² up to 300 V using a direct current power supply, and a high density graphite plate was used as the cathode.

After anodizing, an oxide film with craters formed on Ti-30Nb-1Fe-1Hf alloy, as shown in Fig. 1. The average surface roughness of the anodic oxide film was approximately 440 nm. The pore size, composition, and adhesion strength of the anodic oxide film were strongly affected by the voltage imposed during galvanostatic anodization. A thin oxide film formed and intimately contacted the substrate prior to sparking. When the anodizing voltage was higher than 200 V, the thickness of anodic oxide film increased rapidly and numerous craters formed as a result of sparking. The Ca/P ratio and the contents of Ca and P in the oxide film increased with increasing anodic potential. A great amount of Ca and P contained in the anodic oxide film anodized to 300V and the ratio of Ca to P was about 1.98. However, only a trace of Ag was detected.

With regard to in vitro test, the anodic oxide film exhibited no cytotoxicity. No significant antibacterial activity was achieved for the anodic oxide film anodized to a relatively low voltage. However, the anodic oxide film with antibacterial was achieved when the anodizing potential was up to 250 V.

References

1. Van NR. Titanium: the implant material of today. *J Mater Res* 1987;22:3801-3811.
2. Rietbergen BV, Huiskes R, Weinans H. *Clin Orthop* 1992;274:124-134.
3. Hon YH, Wang JY, Pan YN. *Mater Trans* 2004;45:2449-2455.
4. Shih-Fu Ou, Hsin-Hua Chou, Chao-Sung Lin, Ching-Jui Shih, Keng-Liang Ou, Yung-Ning Pan. *Appl Sur Sci* 2012; 258: 6190-6198.

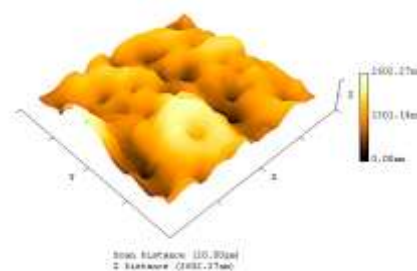


Fig. 1 Surface topography of the AOF anodized to 300 V.