

Effects of Anodizing Parameters on the Formation of Titanium Dioxide Nanotubes

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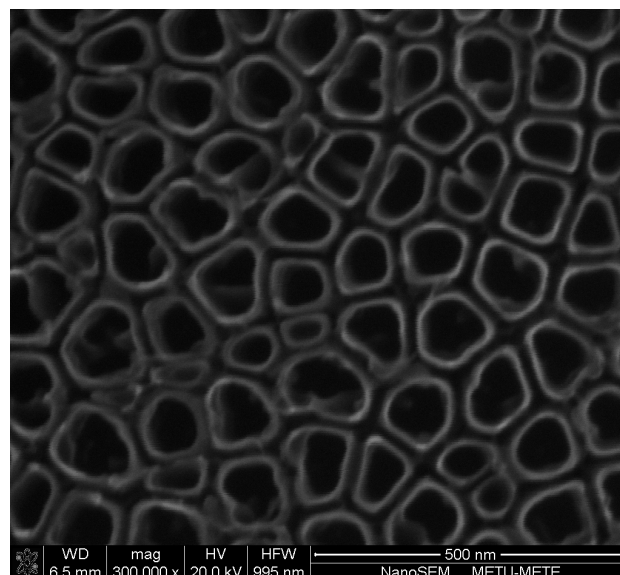
Titanium oxide has unique and excellent; optical, photochemical, biological and electronic properties [1]. These properties increase application areas of titanium oxide as; catalyst, gas sensing, biocompatible and corrosion resistant materials [2]. Titanium oxide thin films with nano-porous structures have gained great interest due to their large surface area and high reactivity properties in their applications [3]. Synthesis of titanium dioxide nanostructures may be achieved by various routes including sol-gel methods, template-assisted methods, hydro/solvothermal approaches, and by electro-chemical means.

The electrochemical method involves controlled anodic oxidation of titanium to form titanium oxide nanotube arrays on surfaces of titanium and its alloys. The TiO₂ nanotube arrays were grown in HF containing electrolytes or acidic HF mixtures [4]. They were also grown using buffered neutral electrolytes containing NaF or NH₄F instead of HF [5-6] and taking into account the importance of the pH gradient within the tube [6]. The lengths and diameters of titanium dioxide nanotubes can be affected by the applied voltage, current and duration of anodizing.

Titanium oxide nanotubes were fabricated by anodic oxidation of Ti6Al4V titanium alloy by applying 10 to 30 volts in this study. Aqueous solutions containing 1 molar H₂SO₄ and 0.1 to 0.5 wt. % hydrofluoric acid were used for 30 to 180 minutes anodizing time. The full factor analysis was used to determine experimental routes. The correlations of nanotube dimensions to experimental parameters were determined by Minitab program [7]. The nanotube diameters were compared by scanning electron microscopy (SEM) images and the average values were determined by image J program. The figure on the right shows the SEM images of Ti6Al4V anodized for 60 minutes under 20 V and 30 V potential differences in aqueous solutions containing 1 molar H₂SO₄ and 0.3 wt % HF. Nanotube diameters could be controlled between 10 to 150 nm by varying the hydrofluoric acid concentration. Anodizing time and voltage contributed to the reaction rate but did not have a significant influence on nanotube diameter.

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(a)



(b)

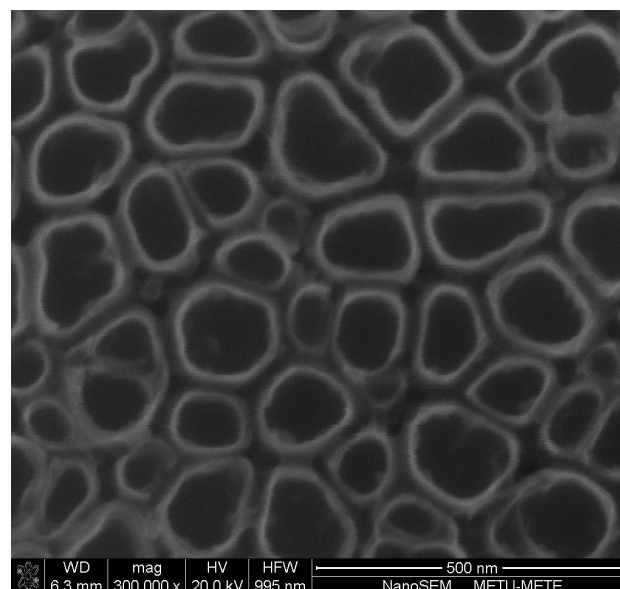


Figure: Scanning electron microscope images of Ti6Al4V anodized in aqueous solutions containing 1 molar H₂SO₄ and 0.3 wt % HF for 60 minutes by applying (a) 20 V, (b) 30 V potentials.

References

- [1] K. Satake *et al.*, Sens. Actuators B **20**, 111 (1994).
- [2] H. Gerischer, Electrochim. Acta **40**, 1277 (1995).
- [3] A. Ghicov, P. Schmuki, Chem. Commun. 2791 (2009).
- [4] Gong D, Grimes CA, Varghese OK, Chen Z, Dickey EC. J Mater Res 2001;16:3331.
- [5] Macak JM, Sirotna K, Schmuki P. Electrochim Acta 2005;50:3679.
- [6] Macak JM, Tsuchiya H, Schmuki P. Angew Chem 2005;44:2100.
- [7] Minitab15, "Statistical Software"