Nanotube Morphology Change of Ti-Ta-Zr Alloy as Zr Content

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Abstract: Nanotube morphology change of Ti-Ta-Zr alloy as Zr content increase has been researched using various experimental methods. Ti-Ta-Zr(3, 7 and 15 wt%) alloys were prepared by arc melting and nano-structure controlled for 24 hr at 1000 °C in argon atmosphere. Formation of oxide nanotubes are conducted by anodizing a Ti-Ta-Zr alloy in H₃PO₄ electrolytes with small amounts of fluoride ions at room temperature. Electrochemical experiments were carried out with conventional three-electrode configuration with a platinum counter electrode and a saturated calomel reference electrode. The samples were embedded with epoxy resin, leaving a square surface area of 10mm² exposed to the anodizing electrolyte, 1.0M H₃PO₄ containing 0.8wt% NaF.

1.Introduction

Titanium dioxide (TiO₂) nanotubes have been formed by various methods including hydrothermal method [1], seeded growth [2], template-assistant deposition [3] and anodization [4]. Especially, anodization is a relatively simple method for nanotube formation and TiO₂ nanotube array control [5,6]. The electrochemical formation of novel highly ordered oxide nanotube layers has been reported for Ti anodization in fluoride-containing acid electrolytes at moderate voltage [7]. Nanotube formation on the TiO₂ is important to improve the cell adhesion and proliferation in clinical use. It is possible to control nanotube size and morphology for biomedical implant use. By using the various factor such as applied voltage, alloying element, current density, time and electrolytes.

In this work, nanotubes have been formed on a Ti-Ta-Zr alloy in a view to study the variation of tube demension with respect to Zr content.

2. Materials and methods

Ti (G&S TITANIUM, Grade. 4, USA) alloys containing Zr (Kurt J. Lesker Company, 99.95 % wt% in purity) up to 3, 7 and 15 wt% were melted six times to improve chemical homogeneity using the vacuum arc melting furnace. And heat treatment was carried out at 1000° C for 24h in order to homogenization in argon atmosphere. The specimens for electrochemical test were prepared by using various grit emery papers and then finally, polished with $0.3~\mu$ m Al_2O_3 powder. All of polished specimen was ultrasonically cleaned and degreased in acetone.

Microstructures of the alloys were examined by optical microscopy (OM, OLYMPUS BM60M, JAPAN) and field emission scanning electron microscopy (FE-SEM, HITACHI-3000, JAPAN). The specimens for the OM and FE-SEM analysis were etched in Keller's solution consisting of 2 m ℓ HF, 3 m ℓ HCl, 5 m ℓ HNO $_3$ and 190 m ℓ H $_2$ O. In order to identify the phase constitutions of the Ti-Ta-Zr alloys, X-ray diffracto-meter (XRD, Philips, X'pert Pro MPD) analysis with a Cu-K α radiation were performed.

Electrochemical experiments were carried out with conventional three-electrode configuration with a platinum counter electrode and a saturated calomel reference electrode. The sample was embedded with epoxy resin, leaving a square surface area of 10mm^2 exposed to the anodizing electrolyte, 1.0 M H₃PO₄ containing 0.8 wt% NaF. Anodization treatments were carried out using a scanning potentiostat (EG&G Co., Model 362, USA).

3. Conclusion

Microstructure properties observed by OM and FE-SEM were changed from lamellar structure to needle-like structure with increasing Zr content. Microstructures were changed from $\alpha+\beta$ phase to α phase through XRD and α phase increases according to the amount of Zr content. The composition of the alloys has a great influence on the two-size-scale structure, and two-size-scale structure appeared predominantly as Zr content increased, and small size nanotube was nucleated at pore spacing area. (*Corresponding author: hcchoe@chosun.ac.kr)

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