Applied Surface Science 258 (2012) 1929–1934

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Effects of TiN coating on the corrosion of nanostructured Ti–30Ta–*x*Zr alloys for dental implants

Won-Gi Kim[a], Han-Cheol Choe[b,c,*]

[a] Department of Dental Technology, Daegu Health College, Daegu, Republic of Korea
[b] Department of Dental Materials, Research Center of Nano-Interface Activation for Biomaterials, School of Dentistry, Chosun University, Gwangju, Republic of Korea
[c] Research Center for Oral Disease Regulation of the Aged, School of Dentistry, Chosun University, Gwangju, Republic of Korea

ARTICLE INFO

Article history:
Available online 27 July 2011

Keywords:
Nanotube
TiN coating
Corrosion behaviour
Biomaterials

ABSTRACT

Electrochemical characteristics of a titanium nitride (TiN)-coated/nanotube-formed Ti–Ta–Zr alloy for biomaterials have been researched by using the magnetic sputter and electrochemical methods. Ti–30Ta–*x*Zr (*x* = 3, 7 and 15 wt%) alloys were prepared by arc melting and heat treated for 24 h at 1000 °C in an argon atmosphere and then water quenching. The formation of oxide nanotubes was achieved by anodizing a Ti–30Ta–*x*Zr alloy in H_3PO_4 electrolytes containing small amounts of fluoride ions at room temperature. Anodization was carried out using a scanning potentiostat, and all experiments were conducted at room temperature. The microstructure and morphology of nanotube arrays were characterized by optical microscopy (OM), field emission scanning electron microscopy (FE-SEM) and X-ray diffraction (XRD). The TiN coatings were obtained by the radio-frequency (RF) magnetron sputtering technique. The depositions were performed from pure Ti targets on Ti–30Ta–*x*Zr alloys substrates. The corrosion properties of the specimens were examined using potentiodynamic test in a 0.9% NaCl solution by using potentiostat. The microstructures of Ti–30Ta–*x*Zr alloys were changed from an equiaxed to a needle-like structure with increasing Zr content. The interspace between the nanotubes was approximately 20, 80 and 200 nm for Zr contents of 3, 7 and 15 wt%, respectively. The corrosion resistance of the TiN-coated on the anodized Ti–30Ta–*x*Zr alloys was higher than that of the untreated Ti alloys, indicating a better protective effect.

© 2011 Elsevier B.V. All rights reserved.

1. Introduction

Titanium (Ti) and its alloys are widely used as a dental implant material in clinical dentistry and as an orthopedic implant material due to their good mechanical property, corrosion resistance, and biocompatibility. However, there are certain disadvantages such as poor osteointegration properties, high elastic modulus, and low corrosive-wear resistance [1–4]. In recent years, the formation of self-organized pores has been achieved on various valve metals such as Zr, Hf, Nb, and Ta including including Ti [5–10]. Self-organized porous TiO_2 has been produced in fluoride-containing electrolytes such as HF electrolytes, chromic acid–HF mixtures, $(NH4)_2SO_4$–NH_4F mixtures, H_2SO_4–HF mixtures and H_3PO_4–NaF mixtures [11–15]. Typically in these experiments, pores of a diameter of approx. 100 nm and a pore length of approx. 500 nm was reported. Nanotube titanium oxide formation on the Ti or Ti alloy surface is important to improve cell adhesion and proliferation in clinical use. It should be possible to control the nanotube size and morphology for biomedical implant use by controlling the applied voltage, alloying element, current density, anodization time, and electrolytes.

The titanium nitride (TiN) coatings are used widely in many dental and industry fields due to their high hardness, good wear resistance, good adhesion, excellent corrosion resistance, and low friction coefficient [16–18]. However, there still remains a need for improvement in the bone adhesion or in the mechanical properties of the Ti alloy to fabricate dental implants [19]. Many techniques such as physical vapor deposition (PVD), chemical vapor deposition (CVD), ion-beam deposition, and radio-frequency (RF) magnetron sputtering have been used to produce TiN films [20,21].

In this study, in order to improve the corrosion resistance of bio-implant for biocompatibility, we investigated the electrochemical behaviors of a TiN-coated Ti–30Ta–*x*Zr alloy before and after nanotube formation in 0.9% NaCl solution using electrochemical methods.

2. Experimental details

Ternary alloys were prepared from Ti (Grade 4, G&S Titanium, USA), Ta (99.95% purity, Kurt J. Lesker Company, USA),

* Corresponding author. Present address: Department of Dental Materials & Research Center of Nano-Interface Activation for Biomaterials, School of Dentistry, Chosun University, Gwangju, Korea. Tel.: +82 62 230 6896; fax: +82 62 230 6896.
E-mail address: hcchoe@chosun.ac.kr (H.-C. Choe).

0169-4332/$ – see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.apsusc.2011.06.151

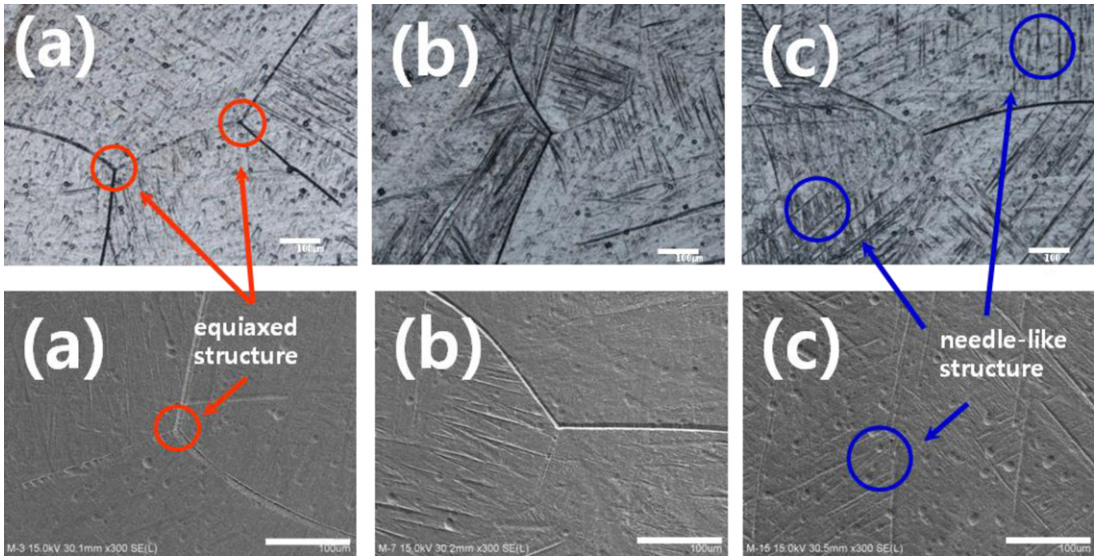

Fig. 1. OM and FE-SEM images of the Ti–30Ta–*x*Zr alloys (200×): (a) Ti–30Ta–3Zr; (b) Ti–30Ta–7Zr; (c) Ti–30Ta–15Zr.

and Zr (99.95% purity, Kurt J. Lesker Company, USA), having Zr contents 3, 7, and 15 wt%. Each alloy was melted six times to improve chemical homogeneity using a vacuum arc melting furnace. Heat treatment was subsequently performed at 1000 °C for 24 h in an argon atmosphere for homogenization of the microstructure, followed by water quenching. Specimens for electrochemical treatment were prepared by standard sequential metallographic polishing with emery papers and then given a polish with 0.3 μm Al_2O_3 slurry. All polished specimens were ultrasonically cleaned and degreased in acetone. Microstructures of the alloys were examined by optical microscopy (OM, Olympus BX60MF, Japan) and field emission scanning electron microscopy (FE-SEM, Hitachi 4800, Japan). The specimens for the OM and FE-SEM analysis were etched in Keller's solution consisting of 2 mL HF, 3 mL HCl, 5 mL HNO_3, and 190 mL H_2O. X-ray diffraction (XRD) was employed to identify the phases and crystallinity in the Ti–30Ta–*x*Zr alloys, using a diffractometer (X'pert Pro MPD, Philips, Netherlands) and Cu Kα radiation. The chemical composition of the nanotube were characterized by X-ray photoelectron spectroscopy (XPS, Kratos Axis Ultra DLD Multi-Technique).

Electrochemical experiments were carried out with a conventional three-electrode configuration having a platinum counter electrode, a saturated calomel reference electrode, and an anode working electrode. Before anodization, the samples were polished by standard ANSI silicon carbide papers of different grades ranging from 100 to 2000 polished, ultrasonically cleaned in deionized water and dried in flowing nitrogen. The sample was embedded with epoxy resin, leaving a square surface area of 10 mm^2 exposed to the anodizing electrolyte, which was 1.0 M H_3PO_4 containing NaF. Anodization treatments were carried out using a scanning potentiostat (Model 362, EG&G, USA).

TiN coating on the cleaned Ti–30Ta–*x*Zr alloys surface were performed by means of RF magnetron sputtering (CX-600S, COMDEL, Korea) system. Pure Ti (99.998%) was used for the coating. Before coating, the working chamber was evacuated to 1.0×10^{-6} Torr and kept at room temperature for 2 h. The RF power for Ti deposition was 100 W, and the working pressure was kept at 1.0×10^{-3} Torr. During sputtering of TiN, a reactive gas mixture of argon (Ar) and nitrogen (N_2) was used for deposition onto the metal alloy plate. The feed of Ar (99.9999% pure) and N_2 (99.9995% pure) gases was controlled by a mass flow controller. The mixture of gas was maintained by infusion of Ar and N_2 gas at a ratio of 1:7 (infusion volume; Ar: 5 sccm, N_2: 35 sccm) (sccm stands for standard cc/min) for 5 min. Pre-sputtering and deposition times were 20 and 60 min, respectively.

Electrochemical potentiodynamic polarization studies were carried out in 0.9% NaCl solution at 36.5 ± 1 °C using a potentiostat (EG&G Co., PARSTAT 2273, USA). A conventional three-electrode system with high-density graphite as counter electrode and saturated calomel electrode (SCE) as reference was used. The preparation of the sample was as described above. The sample edges were carefully covered with epoxy to avoid the possible crevice attack. The electrolyte was deaerated using high-purity Ar gas for 30 min before starting the experiment. Deaeration was continued at a uniform rate during the experiment. The potentiodynamic polarization test with a scan rate of 1.67 mV s^{-1} was carried out from −1500 mV to 2000 mV. The Tafel extrapolation was followed to determine the corrosion parameters; based on a software-based approximation. The all surface morphologies were observed by FE-SEM with high resolution.

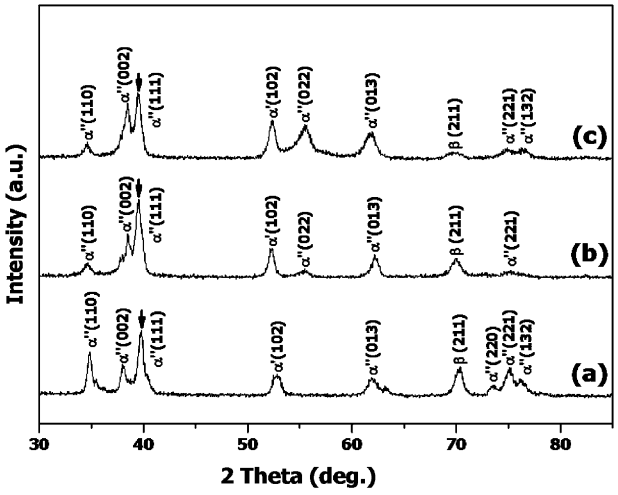

Fig. 2. XRD patterns of Ti–30Ta–*x*Zr alloys: (a) Ti–30Ta–3Zr; (b) Ti–30Ta–7Zr; (c) Ti–30Ta–15Zr.

Download English Version:

https://daneshyari.com/en/article/5362715

Download Persian Version:

https://daneshyari.com/article/5362715

Daneshyari.com