Surface & Coatings Technology 304 (2016) 438–449

Contents lists available at ScienceDirect

Surface & Coatings Technology

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

Characterization of PEO nanocomposite coatings on titanium formed in electrolyte containing atenolol

Hossein Sharifi, M. Aliofkhazraei *, Ghasem Barati Darband, A. Sabour Rouhaghdam

Department of Materials Engineering, Faculty of Engineering, Tarbiat Modares University, P.O. Box: 14115-143, Tehran, Iran

ARTICLE INFO

Article history:
Received 6 April 2016
Revised 30 June 2016
Accepted in revised form 16 July 2016
Available online 18 July 2016

Keywords:
PEO nanocomposite
Atenolol
Corrosion
EIS
Wear
Nanoindentation

ABSTRACT

Nanocomposite coatings were formed on commercial pure titanium through plasma electrolytic oxidation (PEO) process in a phosphate-based electrolyte containing atenolol and alumina nanoparticles. The procedure was performed by direct current and current density was kept constant. The effects of atenolol concentration in the electrolyte on growth of coatings, absorption of alumina nanoparticles, morphology, chemical and physical structure and corrosion and mechanical behavior of coatings were studied by dynamic light scattering spectroscopy, scanning electron microscopy, energy dispersion X-ray spectroscopy, X-ray diffraction, potentiodynamic polarization test, electrochemical impedance spectroscopy, nanoindentation and pin-on-disk wear test. The existence of atenolol in the electrolyte was observed to increase the diffusion depth of alumina nanoparticles and decrease porosity. This behavior, in turn, led to an increase in charge transfer resistance by 211,420 Ω cm^2. Moreover, the addition of atenolol in alumina suspension decreased the wear rate by 130 times as it increased absorption of alumina nanoparticles and improved mechanical properties.

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1. Introduction

Titanium and titanium-based alloys are widely applied in aerospace, petrochemical, and biomedical industries because of their distinguished properties such as high strength to weight ratio, high melting temperature, appropriate mechanical properties, high resistance to corrosion, and biocompatibility [1]. To further improve titanium function in terms of mechanical properties, corrosion resistance, and biocompatibility, the rather new method of plasma electrolytic oxidation has recently been paid a great deal of attention [2]. The parameters of coating process which may be varied to achieve the best desired properties mainly consist of the electrolyte effect and electrical parameters of the PEO process. Among these parameters, the electrolyte and its additives are of great importance in the PEO process because of the role of their components in sparking process as well as the introduction of elements into the coating and affecting its properties. Recent studies regarding electrolyte and PEO process are mostly dedicated to the electrolyte additive effects [3,4]. It was reported that addition of cerium nitrate to the PEO electrolyte on titanium leads to the formation of a coating with proper biocompatibility properties [5]. The existence of sodium vanadate in the PEO electrolyte on titanium was seen to improve photocatalytic properties [6]. Moreover, the existence of iron sulfate in the PEO electrolyte on titanium causes a change in color to yellow and red [7]. Considering the application of corrosion inhibitors as electrolyte additives in other conversion coating processes such as anodizing [8,9], their use in PEO process has also recently attracted attentions. Oleinik et al. [10] investigated the effect of corrosion inhibitors in PEO process on aluminum-based alloys. They reported that existence of corrosion inhibitors raised hydrophobization. Another major advantage of PEO coating process in comparison with other conversion coatings such as anodizing is that the former is environmentally friendly [11,12]. Hence, it can attract more attention to employ non-toxic materials such as eco-friendly corrosion inhibitors as additives to the electrolyte to improve corrosion and mechanical behavior of PEO coatings on titanium. Atenolol is an antihypertensive with a chemical formula of $C_{14}H_{22}N_2O_3$ with a molecular weight of 266 g/mol [13]. The effect of atenolol as corrosion inhibitor was previously investigated. The results revealed that atenolol acts as an inhibitor against general corrosion and pitting in aluminum and Al—Si alloys in HCl media [14]. Accordingly, application of atenolol as a non-toxic additive to the electrolyte, which is readily accessible and inexpensive in PEO coating, on titanium surfaces may become a matter of considerable interest. Since atenolol is mainly used in medical and dental applications, it is necessary to use non-toxic materials in the coating process.

In addition, there are various methods to improve mechanical properties of PEO coatings on titanium including addition of hard ceramic nanoparticles suspended in an electrolyte [15], combination with other coating processes [16], application of new electrolyte compounds [17], performance of secondary heat treatments on the coatings [18], etc. The present work is conducted to investigate the effect of atenolol as an additive in the electrolyte (together with α-Al_2O_3 nanoparticles)

* Corresponding author.
E-mail addresses: maliofkh@gmail.com, khazraei@modares.ac.ir (M. Aliofkhazraei).

http://dx.doi.org/10.1016/j.surfcoat.2016.07.048
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on absorption of nanoparticles from the suspension, chemical structure, morphology and corrosion and mechanical behavior of properties of the oxide coatings obtained on commercially pure titanium (CP-Ti) surfaces through plasma electrolyte oxidation process.

2. Experimental procedure

2.1. PEO coating

CP-Ti sheets (ASTM-Grade 2) with dimensions of 50 mm × 50 mm × 1 mm were selected as the substrate material. Prior to the coating process, samples were polished until Ra < 1 μm, cleaned in acetone via ultrasonic, washed by deionized water and dried in warm air. The PEO process was carried out by means of a 20 kW DC power supply under the constant current density of 20 mA/cm^2 and for 12 min in an aqueous solution. In this process, the substrate (anode) is placed in a cell made of stainless steel (cathode) with a recirculating water cooling system. Since the effect of atenolol alone was to be considered in this project, the simplest electrolyte (single-component) was used. The electrolyte utilized in PEO process was composed of 8 g/L $Na_3PO_4 \cdot 12H_2O$ with/without 25 g/L α-Al_2O_3 nanoparticles and atenolol additive in deionized water. As shown in Fig. 1, the nanoparticles were analyzed by transmission electron microscopy (TEM; JEOL-2010). The average size of alumina nanoparticles was 22 nm. Several primary tests were carried out to acquire the concentration range of atenolol. It was observed that at concentrations higher than 4.5 g/L, sparks immediately reached a destructive state and the coating process would not be performed soundly. Thus, the maximum concentration of atenolol was set to be 4.5 g/L. To investigate the effect of atenolol additive, samples with atenolol concentrations of 0, 1.5, 3 and 4.5 g/L were provided. The denominated title for each sample is given in Table 1. The electrolyte temperature during the coating process was kept at 25 °C. All samples were washed with deionized water after the coating process and dried in warm air.

2.2. Characterization

Voltage variation during the sparking was automatically recorded by means of avomoter (digital multimeter; APPA 505). Samples were weighed before and after the coating (AND, GR-202, precision ± 50 μg balance). X-ray diffractometer (XRD, Philips; X'Pert MPD) working with Co Kα radiation (λ: 1.78897 Å) at a scanning rate of 0.02°/s was used for phase composition analysis. The elements distribution on surfaces and cross sections of the coatings were provided through energy dispersion X-ray spectroscopy (EDS; Oxford Instruments). The morphology of surfaces and cross sections was also studied by field emission scanning electron microscopy (SEM; Philips XL 30 and SIGMA/VP, Zeiss). The average size of alumina nanoparticles in the PEO suspensions with different concentration of atenolol after mechanical stirring for 24 h before use was determined by a dynamic light scattering spectroscopy (DLS; Zetasizer NanoZS, Malvern Instruments). Corrosion behavior of the coatings was evaluated by means of potentiodynamic polarization tests and electrochemical impedance spectrometry. Potentiodynamic polarization tests were carried out at a scan rate of 1 mV/s, from −300 to +3000 mV with respect to the corrosion potential (E_{corr}) using an EG&G Model 273A. Also, electrochemical impedance spectroscopy (EIS; Solarton 1260) in the frequency range between 65-kHz and 10 mHz with an amplitude of ± 10 mV, was employed. Both electrochemical tests were carried out in Ringer's solution (8.6 g/l NaCl, 0.3 g/l KCl and 0.33 g/l $CaCl_2 \cdot 2H_2O$), at 37 ± 0.5 °C. In these procedures, standard calomel electrode (SCE) and platinum were used as a reference and auxiliary electrodes, respectively. A nanoindentation testing system (Nanoindentation Tester CSM) with a Berkovich diamond indenter was employed to measure mechanical properties of PEO coating. For this purpose, the samples were cut, mounted, and mechanically polished down to the average roughness of 0.02 μm. The maximum load and its loading and unloading rates were adjusted to be 3 mN and 6 mN/min, respectively. Load–displacement curves were recorded and elastic recovery was calculated. Hardness and elastic modulus were determined using the Oliver and Pharr method [19]. In order to study the wear resistance of the coatings, the pin-on-disk test method was employed where the pin was 40 mm in length and 5 mm in diameter. The disk was a composite made of polymer in matrix and SiC as reinforcing particles. The normal load was 24 N, angular velocity was 50 rpm and wear distance for specimens with and without nanoparticles was 200 and 50 m, respectively.

Table 1
Samples code in different phosphate based electrolytes.

	Sample code							
Electrolyte components	A0	A1.5	A3	A4.5	AA0	AA1.5	AA3	AA4.5
Atenolol (g/L)	0	1.5	3	4.5	0	1.5	3	4.5
Alumina (g/L)	0	0	0	0	25	25	25	25

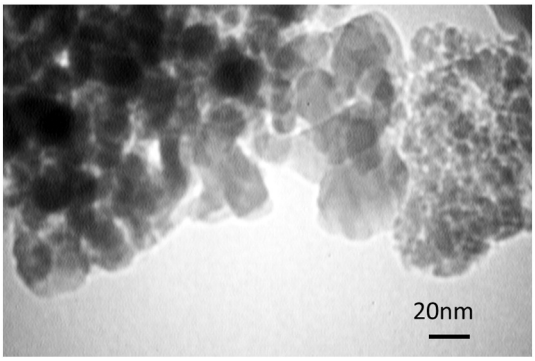

Fig. 1. TEM micrographs of alumina nanoparticles.

3. Results and discussion

3.1. Voltage-time curves

The voltage-time curves of the PEO coating process during sparking under constant current density for the coated samples at different electrolytes are plotted in Fig. 2. The voltage-time plot shows the effect of atenolol addition to the phosphate-based electrolyte and suspension. As shown in this figure, sparking during the PEO process occurred at lower potential ranges in the electrolyte compared with the suspension. The current density was kept constant; then, an increase in sparking voltage implies an increase in resistance of the system. Considering

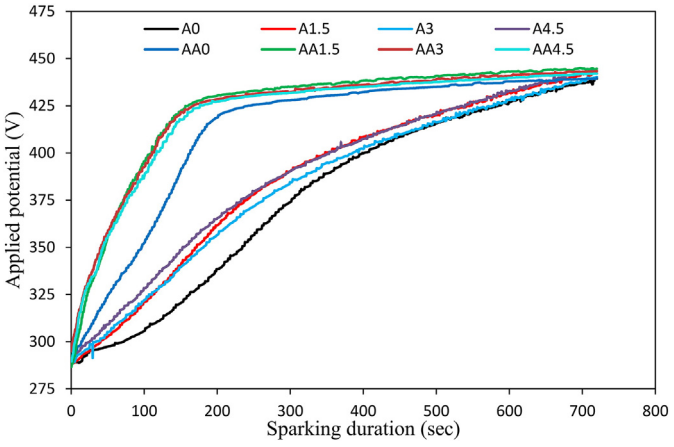

Fig. 2. The voltage-time curves of the PEO process during sparking under constant current density of 20 mA/cm^2 for 12 min on the coated samples at different electrolytes.

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