Chinese Journal of Aeronautics, (2016), 29(4): 1151–1158

Chinese Society of Aeronautics and Astronautics
& Beihang University

Chinese Journal of Aeronautics

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Effect of sodium tartrate concentrations on morphology and characteristics of anodic oxide film on titanium alloy Ti–10V–2Fe–3Al

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Received 6 July 2015; revised 25 July 2015; accepted 6 August 2015
Available online 2 November 2015

KEYWORDS

Anodic oxidation;
Coefficient of friction;
Concentrations;
Corrosion resistance;
Sodium tartrate

Abstract The effect of sodium tartrate concentrations on morphology and characteristics of anodic oxide film on titanium alloy was investigated. The alloy substrates were anodized in different concentration solutions of sodium tartrate with the addition of PTFE emulsion and their morphology and characteristics were analyzed. The anodic oxide film presented a uniform petaloid drums and micro-cracks morphology. Additionally, micro-cracks dramatically swelled with the increase of the tartrate concentrations. The thickness of the anodic oxide film increased with the concentrations until the concentration reached 15 g/L. The results of Raman analysis illustrate that all samples have similarity in the crystal structure, consisting of mainly amorphous TiO_2, some anatase TiO_2 and a small amount of rutile TiO_2. And the ratios of anatase TiO_2 and rutile TiO_2 increase with the concentrations until it reaches 15 g/L. Furthermore, the intensity of the peaks increases with enhanced concentrations until the concentration reaches 15 g/L. The corrosion resistance of the anodic oxide film is increased by the sodium tartrate with higher concentrations before 15 g/L. The coefficient of friction of the anodic oxide film reduces with the concentrations until the concentration reaches 15 g/L, then the coefficient of friction of the anodic oxide film increases with the concentrations.

1. Introduction

Thick, oxide-based film with potential for protection and functionalization of the surface can be obtained by anodization of titanium.[1–3] The anodic oxidation of the anodic oxide film reveals that their property largely depends on the concentrations, electrical source and temperature.[4–6] These factors have been widely researched in recent years.[7–9]

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Peer review under responsibility of Editorial Committee of CJA.

Production and hosting by Elsevier

http://dx.doi.org/10.1016/j.cja.2015.10.013

Due to the specific behavior of the incorporated anions, the morphology and crystallinity of the oxide layer are affected by concentrations in terms of the change of anodizing forming voltage.[10] Ohtsu et al. reported that high-concentration electrolyte crystallized the oxide layer as a result of the field crystallization effect.[11] In addition, electrolyte affected both the surface morphology and the crystallinity of the fabricated oxide layer.[12,13] Thus, the morphology, microstructure and corrosion resistance of the oxide layer are determined primarily by the electrolyte used. Sodium tartrate is the alkalescent electrolyte that is less destructive to the anodic oxide film. Therefore, sodium tartrate is the electrolyte widely used for anodic oxidation of Ti and alloys for the strong complexation to Ti. However, the effect of the sodium tartrate concentrations on the anodic oxide film is not clear. In this paper, anodic oxide film was fabricated on the Ti–10V–2Fe–3Al by using a pulse galvanostatic method.[14–17] As addition of the sodium tartrate, PTFE particles would obviously improve the corrosion resistance and wear resistance of anodic oxide film.

Thus, the purpose of this paper is to study the effect of sodium tartrate concentrations on morphology and characteristics of anodic oxide film on titanium alloy Ti–10V–2Fe–3Al by AC pulse power supply in the sodium tartrate with the addition of PTFE emulsion. And the mechanism of the enhancement of the properties of the anodic oxide film has been studied in detail. This paper can offer a theoretical of basis for the research in the future.

2. Experimental

2.1. Preparation of anodic oxide film

Titanium alloy Ti–10V–2Fe–3Al was cut into sheets with the dimension of 10 mm × 10 mm × 2 mm. Prior to anodization, samples were polished with silicon carbide paper which successively grades from 200 to 2000 grit followed by rinsing with acetone and deionized water successively and finally dried in air.

Anodic oxidation was carried out in a cell with a thermostat water bath and a magnetic stirring apparatus by using a pulse galvanostatic power source (WMY-IV). The output mode of the power source is pulsed power supply, shown in Fig. 1. In the figure, I is the current, t the time of anodization, T the time of pulse cycle, t^+ the time of pulse working period, and I^+ the pulse anodic current supplied. The Ti–10V–2Fe–3Al slice sample was used as anode, and a 1Cr18Ni9Ti stainless steel plate was used as cathode. The anode surface was less than 50% that of the cathode. The parameters of anodization process are given in Table 1. After the treatment, the coated samples were rinsed with water and then dried in the air.

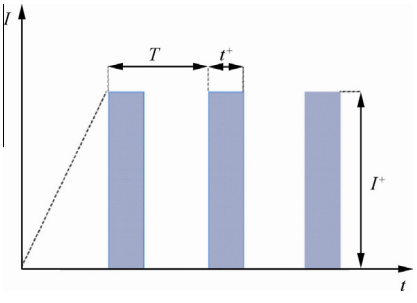

Fig. 1 Output mode of power source.

2.2. Morphology and microstructure of anodic oxide film

The surface morphology and thickness of the anodic oxide film were examined by using scanning electron microscopy (FE-SEM, XL30S, FEI, USA) and atomic force microscope (AFM, Dimension icon, Veeco, USA). The crystalline structure of anodic oxide film was determined by Raman spectroscopy (Raman, Horiba-HR800, Yvon Jobin, using a He-Ne laser without filter, 633 nm).

2.3. Corrosion resistance properties of anodic oxide film

Electrochemical tests were progressed in a traditional three-electrode system (an SCE as reference electrode, a platinum electrode as counter electrode and the oxide sample as working electrode) by using a potentiostat/galvanostat (AES, Parstat 2273, Princeton Applied Research, USA) in a 3.5% NaCl solution. The scanning rate was 0.5 mV · s^{-1} and the scanning range was from −0.5 V to +0.5 V versus the open circuit potential.

2.4. Wear resistance properties of anodic oxide film

Ball disc wear experiments were progressed by using a micro friction and wear machine (UMT-2, CTER, USA). All experiments were carried out by setting the force 3 N for 500 s and the rotation rate was 200 r/min. The diameter of Si_3N_4 grinding ball was 2 mm while diameter of friction being 8 mm. The results were characterized by coefficients of friction of the anodic oxide film.

3. Results and discussion

3.1. Effect of concentrations on thickness

The cross-section images of the anodic oxide film fabricated at different concentrations are shown in Fig. 2 (ρ is the mass of solute per unit volume).

The corresponding thicknesses of the anodic oxide film are 15.7, 16.6, 19.3, 19.4, 19.4 μm respectively, when the concentrations are 1, 5, 15, 30, 50 g/L (Table 2). It was obvious that the thickness of the anodic oxide film would increase with the concentrations until the concentration reached 15 g/L. Thickness of the anodic oxide film would be related to ultimate voltage[18,19] and the rate of the formation and corrosion of the anodic oxide film because of the weak alkaline of electrolyte.[20] At the beginning of the anodic oxidation, the rate of the

Table 1 Parameters of fabrication process.

Parameter	Value
Current density (A · dm^{-2})	5
Sodium tartrate solutions (g · L^{-1})	1, 5, 15, 30, 50
PTFE concentration(mL · L^{-1})	15
Anodization time (min)	40
Duty ratio (%)	20
Frequency (Hz)	1.3