



Thick and macroporous anodic alumina membranes for self-lubricating surface composites

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Received 30 September 2004; received in revised form 8 November 2004; accepted 19 November 2004
Available online 7 January 2005

Abstract

Aluminum-base self-lubricating surface composites require thick and macroporous alumina membranes to add lubricants easily. The thick anodic alumina films obtained in sulfuric acid and oxalic acid are always microporous, while the macroporous films in general phosphoric acid are very thin. In this paper, thick and macroporous alumina layers were fabricated successfully with a phosphate-base solution. The anodizing process in this compound solution and the effects on the thickness of the oxide films were investigated in detail. The results indicate that the growth of porous layers in this solution undergo three stages during anodizing, same with in the general solution. This electrolyte is sensitive to anodizing temperature which affects current density in great degree.

The additive (including organic carboxylic acid and Ce salt) in the phosphate-base electrolyte plays an important role on the increase of film thickness. In this compound electrolyte, the pore diameter of the anodic alumina membranes is about 100 nm in average, pore density is 18 pores μm^{-2} and porosity 14%. The thickness can exceed 20 μm with appropriate anodizing time and temperature.

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Keywords: Anodization; Surface composite; Self-lubrication; Aluminum

1. Introduction

Anodic oxidation treatment, when applied to aluminum and its alloys [1,2], can increase the surface hardness and the anti-wear ability. But it also enhances the abrasion of the counterpart [3–5]. In order to

reduce the abrasion of the counterpart, a self-lubricating surface composite with low friction coefficient is desired. The self-lubricating surface composite is fabricated by depositing or synthesizing in situ some lubricants in the pores or on the surface of anodic porous film, which purpose is to improve the tribology properties of anodic oxide film when holding its high surface hardness and anti-wear ability. To get this type of composite layers, suitable porous alumina membranes must be fabricated firstly.

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The membranes for self-lubricating surface composite should be thick and hard to resist wear and macroporous to add lubricants easily. The anodic porous films are always fabricated in sulfuric acid, oxalic acid and phosphoric acid [6–11]. Whereas, the films obtained in sulfuric acid and oxalic acid are microporous which is uneasy to add solid lubricating materials; enlarging further films pores after anodization will decrease the film mechanical properties, furthermore, it is also confined by the width of wall of holes. The pore diameters of anodized aluminum films in phosphoric acid are larger than those of two solutions [12], and it will be easier to add the materials into the pores. However, the film thickness formed in phosphoric acid is only 1–3 μm with wear-resistance [13], which makes the films unsuited to be as the membranes for self-lubricating surface composites. Therefore, since it is difficult to obtain anodic macroporous membranes in sulfuric acid and oxalic acid, the only way is to thicken anodic films in phosphoric acid. Then, the key problem transforms to how to increase the thickness of the alumina films anodized in phosphoric acid.

In this paper, a compound phosphate-base electrolyte containing organic carboxylic acid and Ce salt for thick films with large pores was studied; the growth of anodic layers in mixed solution and the effects on film thickness were discussed.

2. Experimental

Annealed aluminum foils (99.99% purity, 3 mm \times 3 mm \times 0.5 mm) were used to grow anodic porous layers. High purity metal was preferred in order to minimize the influences of alloying agents. Prior to anodizing, the specimens were degreased and activated with sodium hydroxide at 60–70 $^{\circ}\text{C}$ for 3 min and then electropolished in a stirred alcohol and perchloric acid ($\text{C}_2\text{H}_5\text{OH}/\text{HClO}_4$) mixture for 3 min at 25 V, followed by ultrasonic rinsing in a cold distilled water for 5 min.

Anodization process was conducted in mixed electrolyte containing phosphoric acid (4 wt.%) and some additives (composed of 0.5 wt.% organic carboxylic acid and 0.05 wt.% Ce salt). After anodizing, each sample was washed thoroughly with distilled water and dried with an air jet.

An electrochemical reactor was designed and built to carry out anodizing experiments. Film thickness was measured with a scanning electron microscope (S-520). Film hardness was measured with HXD-1000 digital microhardness tester (load 50 g, loading time 15 s). Morphological parameters of membranes, such as pore diameter, and pore density were determined by the image analysis of the SEM (JSM-6700F) photos. Porosity was obtained from the ratio:

$$\text{Porosity } \% = \left(\frac{S_{\text{pore}}}{S_{\text{ox}}} \right) \times 100$$

where S_{pore} is the surface area of pores, and S_{ox} is the oxide geometric surface.

3. Results and discussion

3.1. Porous layer growth in phosphate-base mixed solution

The curve of voltage versus time during the growth of porous layers is shown in Fig. 1. After the initial increase in a short time, the curve shows a peak; then it drops and attains a constant value at length with the prolong of the anodizing time.

The above process is same as the general three stages of anodization. In the initial stage, the thin and dense aluminum oxide barrier film grows on the aluminum surface. Then the voltage increases rapidly. When reaches a value, the barrier film forms. However, the film thickness is not uniform, which

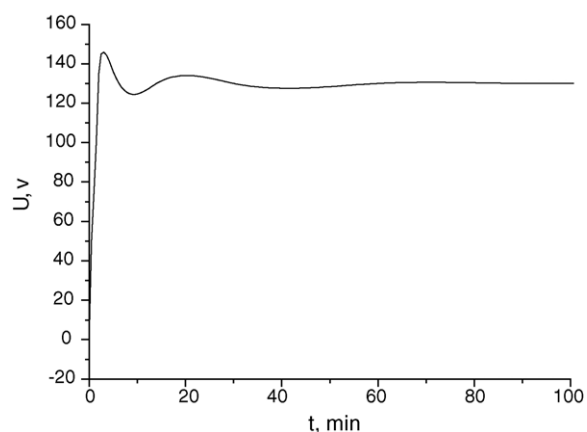


Fig. 1. Voltage against time curve at the constant current density.

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