

# Fabrication of Nanoporous Template of Aluminum Oxide with High Regularity using Hard Anodization Method

Hamed Rezazadeh, Majid Ebrahimzadeh, Mohammad Reza Zeidi Yam

**Abstract**—Anodizing is an electrochemical process that converts the metal surface into a decorative, durable, corrosion-resistant, anodic oxide finish. Aluminum is ideally suited to anodizing, although other nonferrous metals, such as magnesium and titanium, also can be anodized. The anodic oxide structure originates from the aluminum substrate and is composed entirely of aluminum oxide. This aluminum oxide is not applied to the surface like paint or plating, but is fully integrated with the underlying aluminum substrate, so cannot chip or peel. It has a highly ordered, porous structure that allows for secondary processes such as coloring and sealing. In this experimental paper, we focus on a reliable method for fabricating nanoporous alumina with high regularity. Starting from study of nanostructure materials synthesis methods. After that, porous alumina fabricate in the laboratory by anodization of aluminum oxide. Hard anodization processes are employed to fabricate the nanoporous alumina using 0.3M oxalic acid and 90, 120 and 140 anodized voltages. The nanoporous templates were characterized by SEM and FFT. The nanoporous templates using 140 voltages have high ordered. The pore formation, influence of the experimental conditions on the pore formation, the structural characteristics of the pore and the oxide chemical reactions involved in the pore growth are discuss.

**Keywords**—Alumina, Nanoporous Template, Anodization

## I. INTRODUCTION

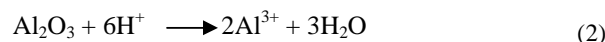
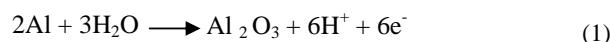
**A**NODIZING is accomplished by immersing the aluminum into an acid electrolyte bath and passing an electric current through the medium. A cathode is mounted to the inside of the anodizing tank; the aluminum acts as an anode, so that oxygen ions are released from the electrolyte to combine with the aluminum atoms at the surface of the part being anodized. Anodizing is, therefore, a matter of highly controlled oxidation the enhancement of a naturally occurring phenomenon. In 1995 Masuda *et al.* reported a two-step anodization which leads to a regular distribution of the pores due to a self-organization process [1].

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There is a great demand for the use of highly ordered nanohole arrays, which can be produced on a scale of several tens of nanometers through self-organization, in a diversity of applications, such as magnetic storage [2], solar cells [3], carbon nanotubes [4], catalysts[5] and metal nanowires[6]. Porous alumina films formed by anodic oxidation of aluminum have been intensively studied for use as mold to form nanostructured materials. Besides the growth of aluminum oxide in the anodization process, dissolution simultaneously happens at a much slower rate than oxide deposition. It strongly depends on the bath concentration and temperature during the process. The formation and dissolution of aluminum oxide during the electrochemical reaction can be expressed by Eq. (1) and Eq. (2), respectively. Reaction Eq. (1) shows the formation of aluminum oxide and reaction Eq. (2), the chemical dissolution of the oxide layer in oxalic acid.



According to Thompson *et al.*, this dissolution mechanism is due to a weakening of the Al-O bonds in the oxide lattice causing dissolution at the film-electrolyte interface [6]. Anderson has shown that, the electrical field is the main reason for the possibility of the ions to move through the barrier layer at all [7]. Under the influence of the high field, the hydroxide ions will move through the oxide to the interface metal-barrier layer. It will react with  $\text{Al}^{3+}$ , which is formed here and form aluminum oxide. If the oxide is formed by the hydroxide rather than the oxygen ions, positive hydrogen ions will move back through the film and into the electrolyte. This means that, the continuous formation of oxide, Eq. (1) is dependent on the ability of migration of aluminum and oxygen ions, through the barrier layer as shown in Fig. 1. In this experimental paper, we describe the set-up to grow nanoporous self-organized alumina templates using voltages [8-9]. We used two step hard anodization methods for fabrication of nanoporous anodic alumina template.

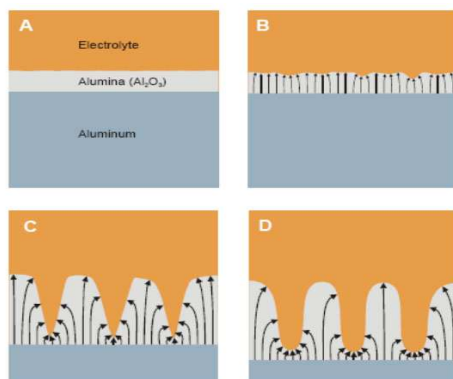


Fig. 1 Stages in the development of porous alumina during anodization [8]

## II. EXPERIMENTAL PROCEDURE

Fabrication of an ordered nanopore arrays is first introduced by Masuda and Fukuda [10]. Masuda and Fukuda used the two step anodization process. The steps of anodization process are applying electric current to a substrate in the presence of acid electrolyte to obtain an oxide layer with self-organized nanopores [11, 12]. Nanoporous alumina films are obtained by electrochemical oxidation of high-purity (>99.998%) aluminum foils. Prior to anodization, several cleaning treatments are employed. The substrate is first degreased in ethanol, followed by soft chemical polishing in acetone ( $\text{CH}_3\text{COCH}_3$ ) solution and inside the ultrasonic bath for 10 minute, which removes the native oxide layer, being then rinsed in deionized water. In the work here presented, in the first step, the anodization voltage is 40 V voltages during 30 min, and then a hard anodization procedure is used in order to achieve the desired organization of pore structure. The hard anodizations were performed at constant voltages of 90, 120 and 140 voltages during 1 hour. As electrolyte 0.3 M oxalic acid solution was used and the temperature kept constant within 0 °C.

## III. RESULTS

Fig. 2 shows the current - time, charge - time and voltage-time curves for aluminum anodization in 0.3 M oxalic acid and 140 V anodization voltage. In the beginning, the current is high due to the fact that the current only passes through the metallic aluminum. Then the current starts to decrease because of the formation of a thin non-porous oxide layer. This oxide layer has a higher resistance than the metallic aluminum. The fabrication of nanopores anodic alumina membrane from (99.998%) aluminum foil has been successfully demonstrated using two step hard anodization process. The structure and morphology of anodic films were greatly influenced by the voltage type and anodization duration [9]. In the modified two-step anodization process, the best result was obtained with using 40 V anodization voltages for the first anodization step, and then increase to 140 V for the second anodization step (Fig. 3).

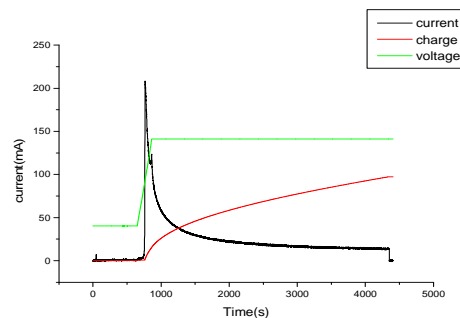


Fig. 2 Current-time curves for anodization process by 0.3 M oxalic acids and 140 V anodized voltage.

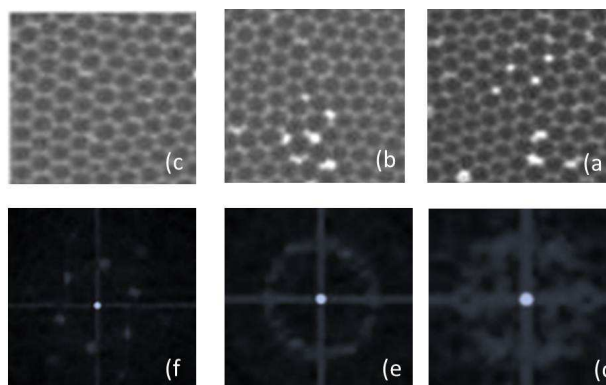


Fig. 3 SEM image and FFT image of nanoporous alumina formed in 0.3M oxalic acid ( $4 \times 4 \mu\text{m}^2$ ) a,d) 90V b,e)120V and c,f)140 V.

## IV. ANODIZING'S BENEFITS

### A. Durability

Most anodized products have an extremely long life span and offer significant economic advantages through maintenance and operating savings. Anodizing is a reacted finish that is integrated with the underlying aluminum for total bonding and unmatched adhesion.

### B. Color Stability

Exterior anodic coatings provide good stability to ultraviolet rays, do not chip or peel, and are easily repeatable.

### C. Ease of Maintenance

Scars and wear from fabrication, handling, installation, frequent surface dirt cleaning and usage are virtually non-existent. Rinsing or mild soap and water cleaning usually will restore an anodized surface to its original appearance. Mild abrasive cleaners can be used for more difficult deposits.

### D. Aesthetics

Anodizing offers a large increasing number of gloss and color alternatives and minimizes or eliminates color variations. Unlike other finishes, anodizing allows the aluminum to maintain its metallic appearance.

### E. Cost

A lower initial finishing cost combines with lower maintenance costs for greater long-term value.

### F. Health and Safety

Anodizing is a safe process that is not harmful to human health. An anodized finish is chemically stable, will not decompose; is non-toxic; and is heat-resistant to the melting point of aluminum (1,221 degrees F.). Fig. 4 shows the several product of Behin Sanaat Yam Company that for these products we use experiments results for anodization of alumina foil.

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Fig. 4 Thermal break windows of Behin Sanaat Yam Company with Aluyam Brand.

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