Control Porous Pattern of Anodic Aluminum Oxide by Foils Laminate Approach

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ABSTRACT

In this research, a novel, much simpler, and low cost method to fabricate porous pattern of the anodic aluminum oxide (AAO) based on the aluminum foils laminate approach were carried out. During our experiments, it was found that the pores of the AAO grew only on the upper foil, bi-directionally from both the top and the bottom surfaces. Experimental results further indicate that the upward porous pattern of the upper foil is determined by the surface structure of the bottom surface of the upper foil. The porous pattern of AAO can be controlled by a pre-made pattern on the bottom surface. Furthermore, no barrier removing process is required in this novel laminate method.

1. INTRODUCTION

Ordered porous membrane is a desirable material for wide applications such as sensors, optical devices, catalysts, and microfabricated fluidic devices. Anodic aluminum oxide (AAO) membrane [1-3] is one of the most attractive ordered nanopore materials and has been demonstrated to be a relatively inexpensive and high throughput template material in fabrication nanodevices such as patterned carbon nano tube (CNT) arrays, photonic crystals, quantum-dot arrays, and nanosensors [4-8].

Key technology that leads to the successful applications of AAO aforementioned is the capability of growing desired porous pattern on the AAO membranes. Li et al. [9] originally integrated the two-anodization process with the lithographic technique to fabricate hexagonally ordered two-dimensional nanopore arrays in anodic alumina. The pore distance can be arranged by changing the anodic electrolyte and the applied voltage. Yan et al. [10] used silica as the anodization barrier to grow highly ordered, patterned through-hole nanopore arrays. Aligned and patterned CNTs based on AAO nano-template with feasible applications in field emitters have recently been reported [7, 11-13]. Masuda et al. [14-15] fabricated ordered hole configuration in anodic porous alumina by pretexturing the aluminum. For these patterned nanopore arrays based on AAO template, additionally lithographic technique was always required. Although the incorporation of the lithographic technique into anodic porous alumina can be easily conducted, supplementary equipments and procedures are needed.

In this article, we propose a novel, much more convenient and cheaper method for the fabrication of porous pattern of the AAO by foils laminate approach. In this new foils laminate method, two tightly clamped aluminum foils, rather than the conventional single piece foil, are employed as the raw material for anodization. In general, an aluminum barrier layer exists at the bottom of the ordered AAO membrane after anodization. This barrier layer needs further removal to achieve a freestanding and through-pore AAO membrane. If an additionally aluminum sheet is attached to the bottom of the being anodized aluminum sheet and acts as a barrier layer during anodization, no additional barrier removing process is required. Furthermore, the porous pattern may be controlled by the structure of the two contacting surfaces.

2. CONTROL POROUS PATTERNS OF AN ANODIC ALUMINUM OXIDE

As shown in Figure 1, porous distribution of an anodic aluminum oxide depends on the surface roughness of the aluminum foil. An unpolished aluminum foil results in a relatively disordered porous pattern (Figure 1a). On the contrary, a polished aluminum foil can produce more orderly and complete porous pattern (Figure 1b). Form the atomic force microscopy (AFM) images of the aluminum foil surfaces as illustrated in Figure 2, it is found that the polished aluminum foil possesses flatter surface (Figure 2b), while the unpolished aluminum foil has parallel notches on its surface (Figure 2a). In contrast to Figure 1, it can be inferred that surface structure of an aluminum foil determines the porous pattern of the resulting anodic aluminum oxide. Based on this property, a novel and simple method to control porous patterns of AAO is presented.

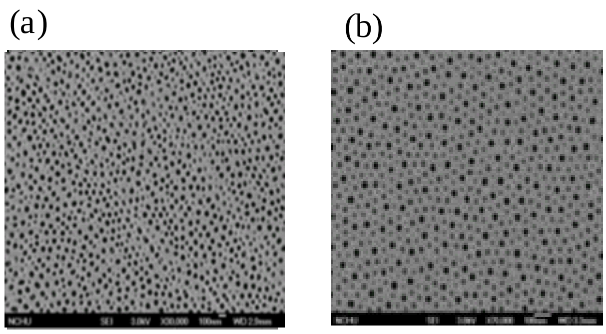


Figure 1. SEM image of the AAO made of (a) unpolished and (b) polished aluminum foils

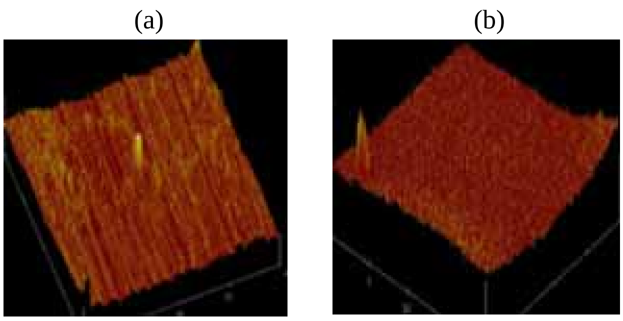


Figure 2. AFM image of the surface of (a) unpolished and (b) polished aluminum foils.

2.1 Experimental Procedures

The experimental procedures include aluminum foil preparation, electropolishing, aluminum foils clamping, anodization, and aluminum foils separation.

(1) Aluminum foils preparation

Aluminum foils (99.9995%, 175 μm thick) were used during the experiments. The aluminum was annealed at 400 °C for 3 h, vibrated by a super sonic vibrator for 1 m, then was cleansed with ethanol to degrease the surfaces.

(2) Electrolytic polishing

Electropolished aluminum will produce a bright finish as well as level surface. The aluminum foil is dipped into a bath solution in which the aluminum metal is electrically anodic. This bath consists of about 96% sulfuric acid, 85% phosphoric acid, and DI water with 1:1:1 ratio. The bath runs at 40° C for 14 min, being agitated by a magnet to ensure a high quality polishing.

(3) Aluminum foils clamping

The polished aluminum foil is vibrated with a super sonic vibrator for 1 m, and then is cleansed with ethanol to degrease the surfaces. Clamp two aluminum foils tightly together with a Teflon clamper as schematically illustrated in Figure 3.

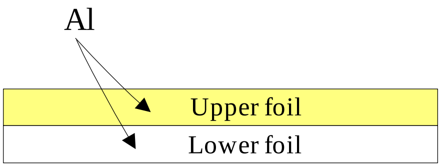


Figure 3. Schematic illustration of the aluminum foils clamping

(4) Anodization

Anodization is carried out under conditions of constant voltage 60 V in a 0.3 M oxalic acid solution at 0 °C for 7 h and being stirred by a magnet. After anodization (Figure 4), the sample is rinsed again with DI water, and then is dried with ethanol. A milky thin film can be found covering the top surface of the anodized sample and could be removed by DI water.

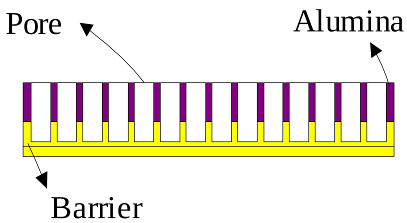


Figure 4. Anodized aluminum foils

(5) Aluminum foils separation

Take apart the barrier layer to obtain a patterned nanopore alumina.

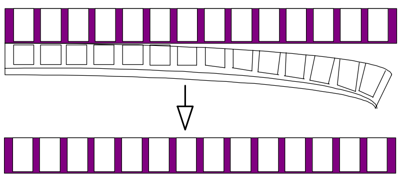


Figure 5. Aluminum foils separation

2.2 Experimental results and discussions

Five different experiments were conducted to investigate the dependency of the porous pattern on the surface structure of the laminate foils.

(1) Unpolished aluminum laminate

Tightly clamp two unpolished aluminum foils together as shown in Figure 6, followed by anodization. SEM images of the resulting AAO are shown in Figure 7. It can be found that the porous pattern on the top view of the upper aluminum oxide foil (Figure 7a) is similar to the pattern depicted in Figure 1(b), where the pattern resulted from an unpolished aluminum metal processed by conventional approach. The bottom view of the upper aluminum oxide foil is shown in Figure 7(b). The porous pattern spreads along a certain direction and is different from that of the unpolished aluminum sheet under conventional processing. It implies that the porous pattern on the bottom surface of the upper foil is determined by the structure of the bottom surface. The cross-sectional image of the aluminum oxide is shown in Figure 8. It can be seen that the pores grew from both the top and the bottom surfaces. Those pores grew from the top surface are much longer than those from the bottom surface. Ideally, the bottom surface was tightly clamped together with the top surface of the lower metal sheet; therefore, there should not be pore at the bottom surface of the upper metal sheet. We deduce that leakage between the foils may a feasible cause to have the upward pores grow in the notches of the unpolished surface.

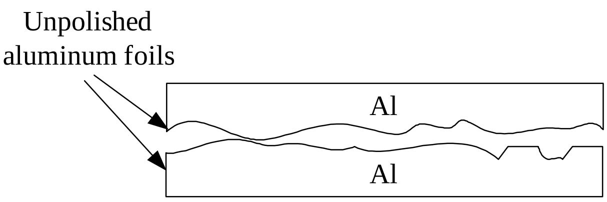
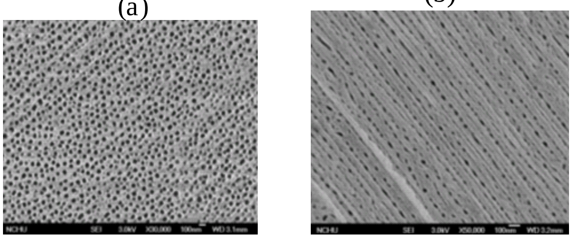


Figure 6. Schematic diagram of the unpolished aluminum laminate

(a) (b)



Top view of the upper foil Bottom view of the upper foil

Figure 7. SEM images of the porous pattern of the unpolished aluminum laminate

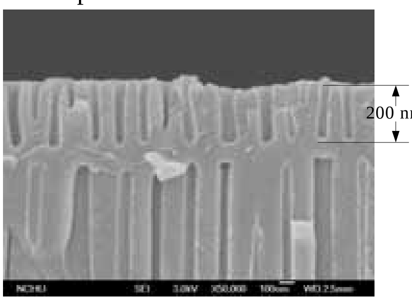


Figure 8. Cross-sectional image of the aluminum oxide of the unpolished laminate

(2) Lower aluminum foil polished laminate

Tightly clamp an unpolished (upper sheet) aluminum foil and a polished (lower sheet) aluminum foil together as shown in Figure 9 and then process anodization. Figure 10 is the bottom view of the upper aluminum oxide foil. The porous pattern is similar to that of the unpolished aluminum laminate shown in Figure 7. It infers that the upward porous pattern of the upper foil has not much strong relationship with the surface structure of the lower metal sheet.

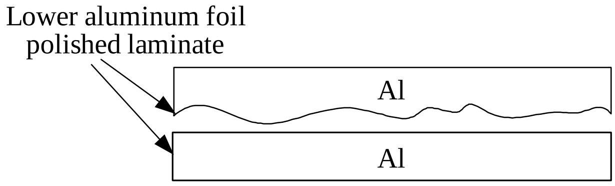


Figure 9. Schematic diagram of the lower aluminum foil polished laminate

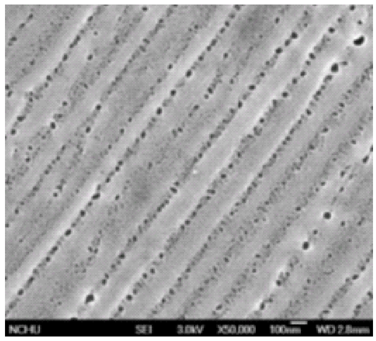


Figure 10. Bottom view of the upper foil for the lower aluminum foil polished laminate

(3) Upper aluminum foil polished laminate

Switched the unpolished and the polished foils of the second experiment as schematically illustrated in Figure 11 and then processed the anodization. The bottom view and cross-sectional profile of the upper aluminum oxide foil are illustrated in Figure 12 and 13, respectively. It can be observed that the porous pattern grew disorderly and is different from that of an unpolished aluminum metal under conventional anodization as shown in Figure 1(b). It implies that there is a strong dependency of the upward porous pattern of the upper foil on the surface structure of the bottom surface of the upper foil.

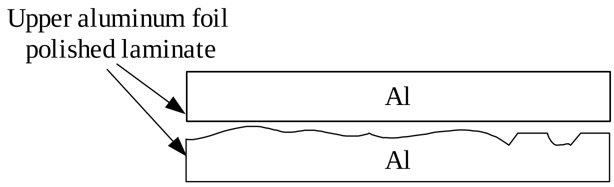


Figure 11. Schematic diagram of the upper aluminum foil polished laminate

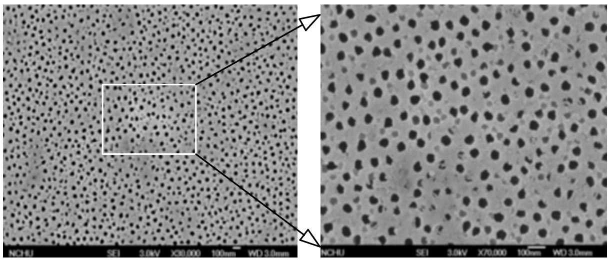


Figure 12. Bottom view of the upper foil for the upper aluminum foil polished laminate

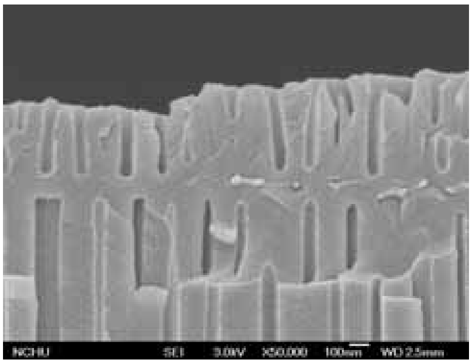


Figure 13. Cross-sectional image of the upper aluminum foil polished laminate

(4) Dual polished aluminum laminate

To verify the dependency of the upward porous pattern on the surface structure, two polished aluminum foils were clamped together (Figure 14) and then processed anodization. Similar to the upper aluminum foil polished laminate experiment, the porous pattern grew disorderly (Figure 15). The experimental results further indicate that the upward porous pattern of the upper foil is determined by the surface structure of the bottom surface of the upper foil.

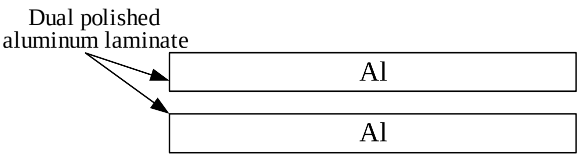


Figure 14. Schematic diagram of the dual polished laminate

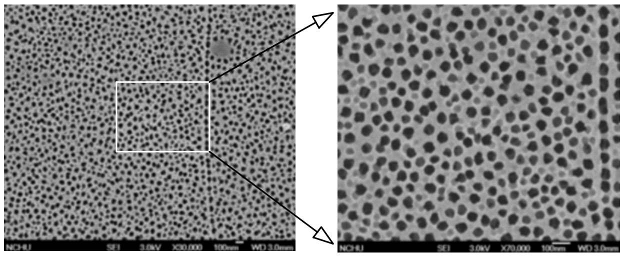


Figure 15. Bottom view of the upper foil for the dual polished laminate

(5) Upper aluminum foil pre-patterned laminate

Based on the above experimental results, additional experiment with the bottom surface of the upper foil being pre-patterned (Figure 16) were carried out. Figure 17 is the bottom view of the upper aluminum oxide foil. The porous pattern is found to be influenced by the shape of the pre-made pattern on the bottom surface.

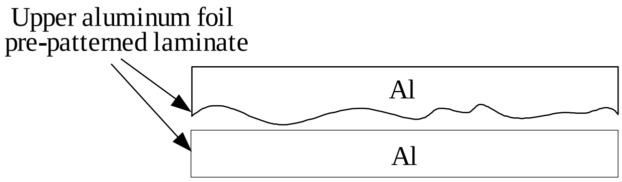


Figure 16. Schematic diagram of the Upper aluminum foil pre-patterned laminate

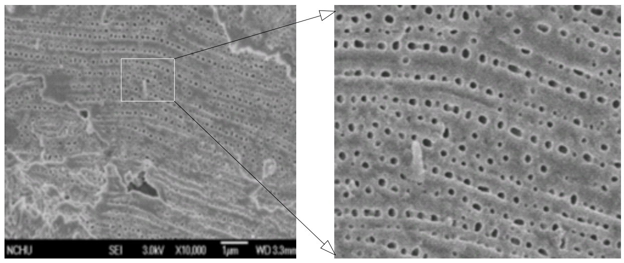


Figure 17. Bottom view of the upper foil for the upper aluminum foil pre-patterned laminate

3. CONCLUSIONS

In this research, a series of anodic aluminum oxide fabrication experiments based on the aluminum foils laminate approach were carried out. During the experiments, we found that the pores of the AAO grew only on the upper foil, bi-directionally from both the top and the bottom surfaces. Experimental results further indicate that the upward porous pattern of the upper foil is determined by the surface structure of the bottom surface of the upper foil. The porous pattern of AAO can be controlled by a pre-made pattern on the bottom surface. In addition, since the lower aluminum sheet attached to the bottom of the being anodized aluminum sheet acts as a barrier layer during anodization, no barrier removing process is required in this novel laminate approach. The developed work in this study can be further applied to the fabrication of nanofunction devices.

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