

Preparation of Controllable, Well-Ordered and Taper Porous Anodic Alumina Templates by Innovated Phosphoric Acid Hard Anodization

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In recent years, porous anodic alumina (PAA) has become one of the most commonly used templates for the fabrication of diverse nanomaterial such as nanorods, nanowires and nanotubes due to its high pore density, high aspect ratio and low cost.^[1,2,3] In general, the conventional fabrication of self-ordered Al₂O₃ pore arrays so-called mild-anodization (MA) require long processing time and the self-ordered phenomenon is restricted to a narrow operating window, limiting their actual application.^[4] Therefore, a new attempt for PAA fabrication, hard-anodization (HA), has successfully demonstrated.^[5] The HA process offers substantial advantages over MA in terms of processing time, faster oxide growth rate with improved long-range regular nanopores. However, researches about PAA membranes with H₃PO₄ HA have seldom been published; some cracks and defects accompanied by C₂H₂O₄ HA process resulted in poor mechanical properties either.

In our study, we report an innovation H₃PO₄ HA for long-range ordered and high growth-rate PAA templates. First, we discuss various anodization parameters including electrolyte, applied voltage and temperature which influence the morphology. The type and the concentration of the electrolyte for an applied voltage have to be selected suitably to obtain self-ordered aluminum pore growth. In our experiment, the applied voltage is as high as 160-210 V which exceeds previous studies. From Figure 1a, the concentration of the electrolyte in 2.5% display the best ordered. Then, we proved the relationship between anodization voltage and interpore distance (D_{int}) among our results and several researches.^[6] As shown in Figure 1b, D_{int} is linearly proportional to the applied potential but our results were found that a saturation phenomenon in HA process will occur during the higher anodization voltage is higher, the joule heat generated under PAA growth process can't distribute uniformly and rapidly. Therefore, the interpore distance reaches a saturation value which can't increase with larger applied voltage.

Subsequently, considering the ordering of PAA pores and their growth rate, the taper PAA templates are fabricated at 210 V in 2.5% H₃PO₄. According to our experiences, adjusting anodization time can be used to control the depth of alumina pore, while varying etching time can be used to control pore size and the aspect ratio. We demonstrated a representative fabrication process of taper PAA. Through the multi-step fabrications, the chemical etching treatment in 5% phosphoric acid after every anodization step makes the pore larger and the etching time (t) is the factor to control pore diameter. The highly-ordered taper PAA templates with pore diameter of 380 nm and pore depths of 750 nm, 1.5 μ m, 3 μ m were

successfully fabricated in Figure 2. The deeper PAA pores with a shape profile are tougher to fabricate because the procedures have to be divided into more steps in which the anodization and etching time are estimated hardly.

In summary, a new self-ordered regime of PAA was found during HA of aluminum substrates using 2.5% phosphoric acid and under potentials of 160-210 V at -10°C. We investigated the control factors of etching process and exhibited a taper template which could be used to fabricate a large area of nanotaper polymer structures for the use of an effective antireflective and self-cleaning surface for solar cells, cell phone monitor and others applications.

Reference

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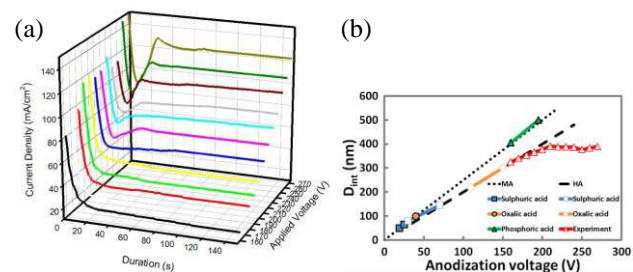


Figure 1. (a) Current-time transients under different anodization voltage in 2.5% H₃PO₄ (b) Summary of self-ordering voltages and corresponding interpore distance (D_{int}) in conventional MA and HA in different electrolyte.

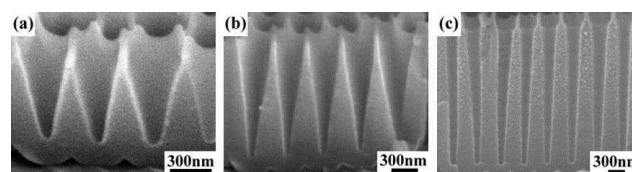


Figure 2. SEM images of our taper shape AAO with diameter 380 nm and various depth: (a) 750 nm; (b) 1.5 μ m; (c) 3 μ m.