Research Progress Report

Fabrication of Nanoporous Anodic Alumina Membrane for Nano-confining Solid Electrolyte Oxides

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Introduction

My research work involves fabrication of porous anodic alumina membrane mainly as a template for nanoconfinement of solid oxide electrolyte material for fuel cell application and synthesis of highly ordered one-dimensional nanostructure arrays. The overall objective of this project is to develop nanoporous anodic alumina template for nano-confining of solid electrolyte oxides. In this context, the specific objective is to develop thin and high quality ordered porous alumina membranes of controllable pore diameters and thickness by means of two-step anodization. The other objective is to minimize lengthy processing time with high mechanical strength and flexibility.

Methods

Anodization (anodic-oxidation) is a process similar to electrolysis in that it involves the use of two electrodes and an acid as an electrolyte, as shown in figure 1.1. The procedure to prepare the AAO membrane is well known. The anodizing process consists of immersing the aluminum to be anodized in a solution, referred to as an electrolyte, and passing a direct electric current through the electrolyte and into the aluminum, with the aluminum acting as the anode. Porous anodic alumina has self-organized hexagonal pore arrays obtained by the anodization in aqueous electrolyte of the most commonly used acids: sulphuric, oxalic and phosphoric. The properties of the electrolyte solution together with the value of an applied voltage have substantial effect on the final geometry of the porous structure.

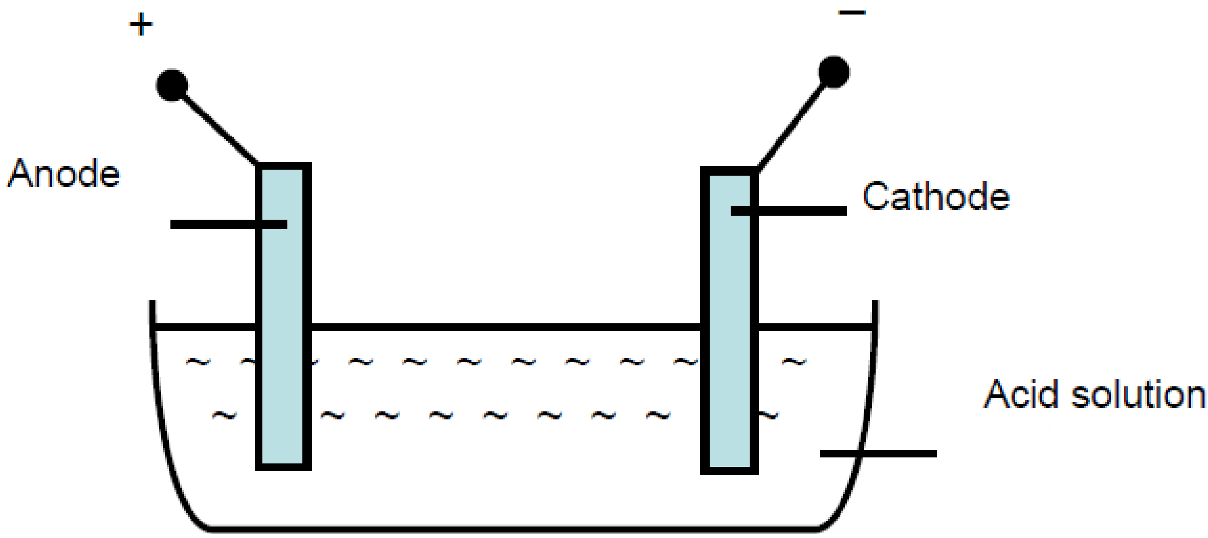

Figure 1. Schematic of electrochemical system for anodizing

Results

By systematically varying electrolyte type and concentration, anodization temperature, time, and voltage, we successfully synthesized thin and highly porous AAO with various pore sizes (60-100 nm), and wall thicknesses (10-50 µm).

As depicted in Figure 2, highly flexible with mechanical strength membranes were produced.

Figure 2. Images of highly transparent and intrinsically defect-free nanoporous morphology as prepared porous alumina membranes.

The structure of the bare alumina membranes was characterized with field emission scanning electron microscopy (ZEISS SEM), indicating that the pores are of cylindrical type but with different sizes at the front and back. Figure 3 shows the scanning electron microscope (SEM) micrographs of samples obtained by anodizing at voltage 40 V and 50 V in oxalic acid. A thin layer of carbon was sputtered onto the samples before the imaging was done since aluminum oxide is an insulator and would result in electron charging on the surface during SEM examination.

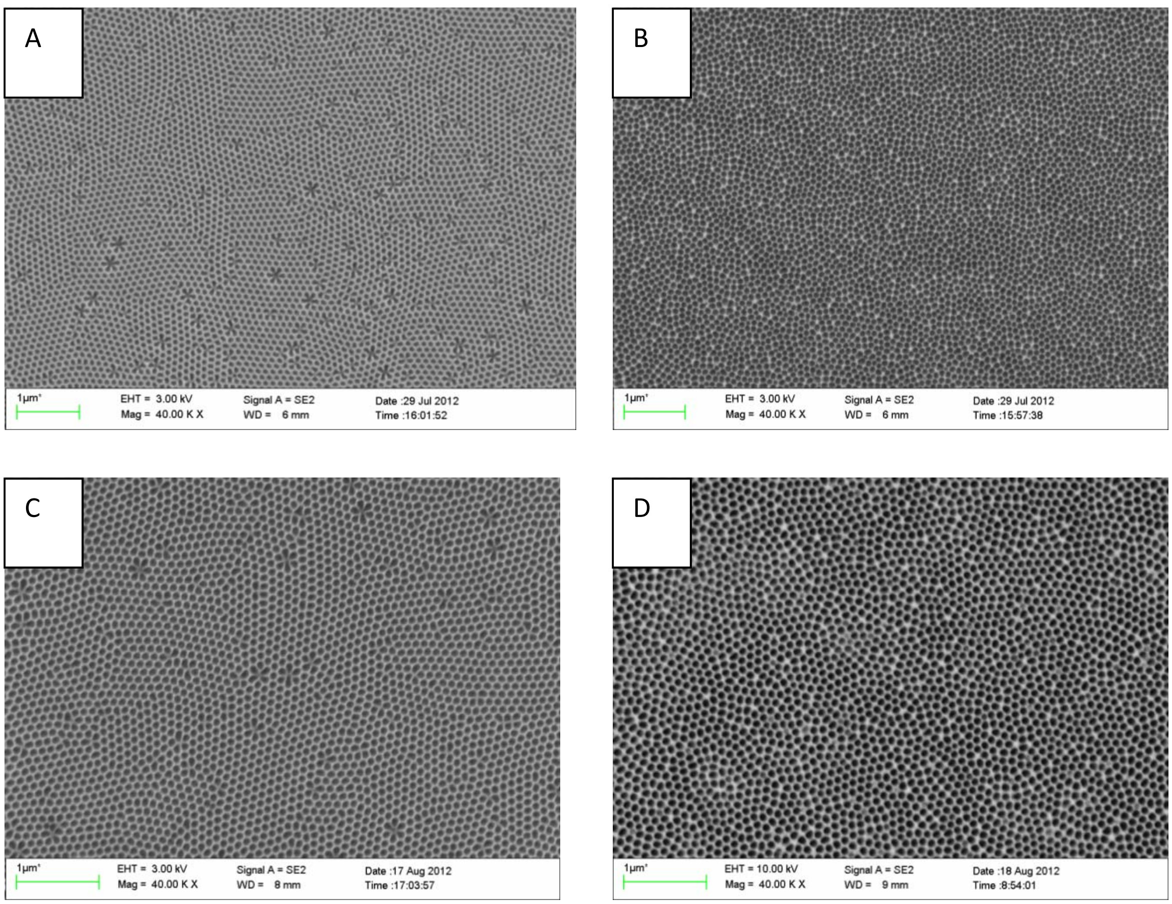

Figure 3. SEM images of AAO membranes grown in oxalic acid at 40 V ((a) back, b(front)) and 50 V ((c) back, d(front)).

The prepared porous membranes were further investigated with CDP infiltration by applying high vacuum deposition. The SEM images and EDS analysis were depicted in Figure 4. Cross sectional view on SEM image indicate that formation of CDP nanowires or nanorods. Further evidence of CDP deposition confirmed by EDS analysis as shown.

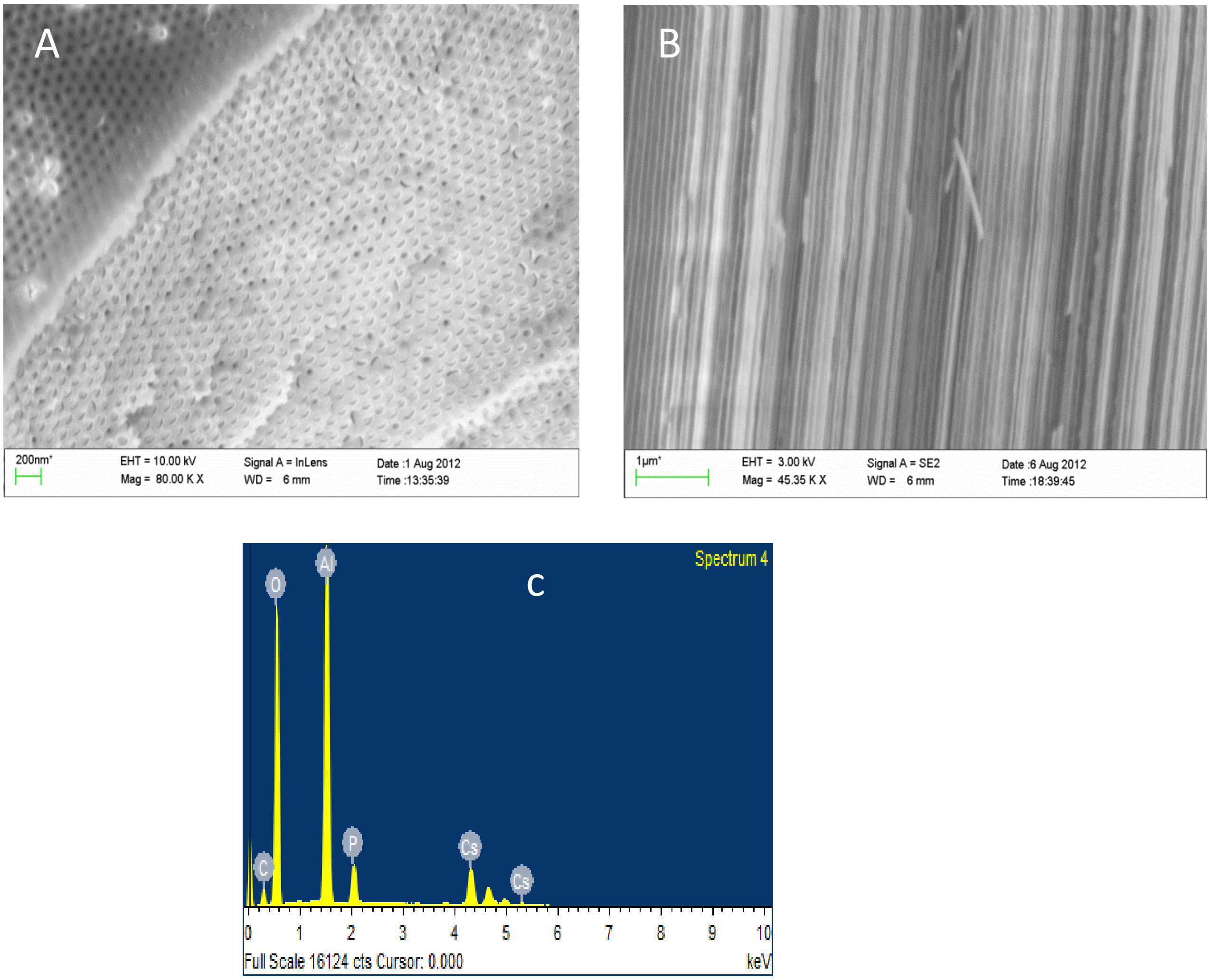

Figure 4. SEM images of porous AAO membranes infiltrated with CDP (a) top view, (b), cross sectional view and c) EDS results.

Conclusion

- Mechanically stable and reproducible membranes were fabricated
- A pore diameter of 60-80 nm with a thickness of 25-30µm was obtained at a voltage 50 V in the 2nd Anodization under 75 minutes
- Variation of time in the first anodization did not affect the pore diameter/membrane thickness
- A linear growth rate of the membrane thickness to the 2nd Anodization time was confirmed
- Able to fully infiltrate with cesium dihydrogen phosphate