Laterally Controlled Metal Electrodeposition in Nanoporous Alumina Membranes

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Nanomaterials have attracted considerable interest in recent years. One of the methods for producing nanostructures is ‘template synthesis’, enabling the preparation of a variety of micro- and nano-materials of desired morphologies.[1] When mesoporous templates are used, the growth of the new phase proceeds in the empty space of the solid matrix. Templates with pores of uniform diameter can be used for preparing nanocylindrical materials (nanorods, nanotubes) comprising polymers, metals, semiconductors, carbon, etc..

Most of the cylindrical templates studied to date consist of two types of materials: ‘track-etch’ polymeric membranes and nanoporous alumina membranes. The latter, usually produced by anodic oxidation of planar Al sheets under special conditions, have a typical naturally occurring self-ordered structure comprising a parallel nanocylindrical pattern. We have recently prepared nanoporous alumina membrane on Al substrates with planar or curved geometries.

Planar Al_2O_3 membranes were treated with 3-mercaptopropyl trimethoxysilane, which serves as an adhesive layer, and coated with evaporated Au on one side. The membranes were then used as templates for Cu electrodeposition in the pores, with the Au serving as the cathode (Figure 1). Control of the Cu electrodeposition in the pores was demonstrated in both the vertical and the lateral directions. Lateral control of the Cu growth was achieved by a novel scheme involving application of a lateral potential gradient[2] on a thin Au film evaporated on the membrane, used as the cathode (Figure 2). This process provides a model system for controlled electrodeposition and gradient formation in nanoporous templates.

Formation of sizeable metal gradients in nanoporous insulating membranes is shown to occur under conditions where essentially no gradient is formed on similar bare electrodes. The latter is attributed to the permanent resistivity of the thin Au between the pores, which does not disappear upon Cu deposition, as it does when thin bare electrodes are used.

References

1. C. R. Martin and D. T. Mitchell, in *Electroanalytical Chemistry* Vol. 21, A. J. Bard and I. Rubinstein (Eds.), Marcel Dekker: NY, 1999.
2. K. M. Balss, B. D. Coleman, C. H. Lansford, R. T. Haasch, P. W. Bohn, *J. Phys. Chem. B* **2001**, *105*, 8970.

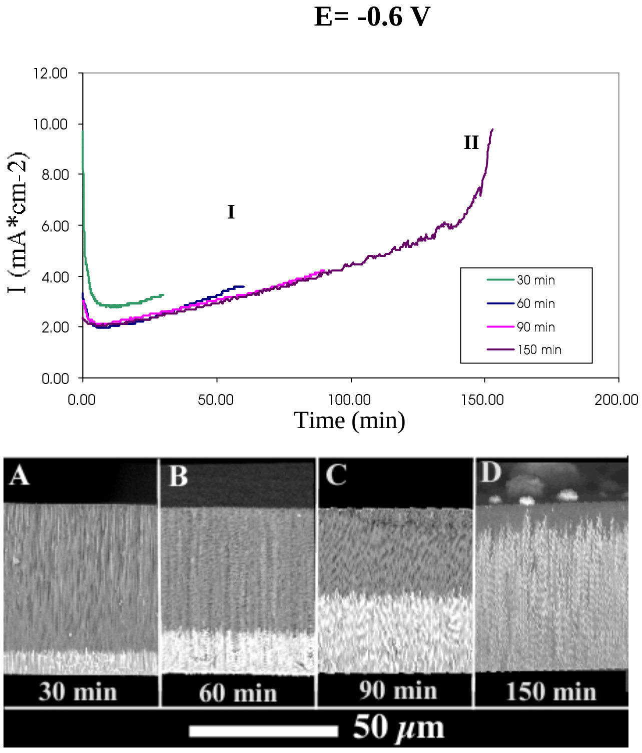

Figure 1. Bottom: SEM images (back-scattered electrons) showing cross-section of Cu-filled membranes following regular Cu electrodeposition for several time periods (indicated). Top: Reduction current density as a function of time of deposition. Electrodeposition was carried out at –0.6 V vs. Hg/Hg_2SO_4.

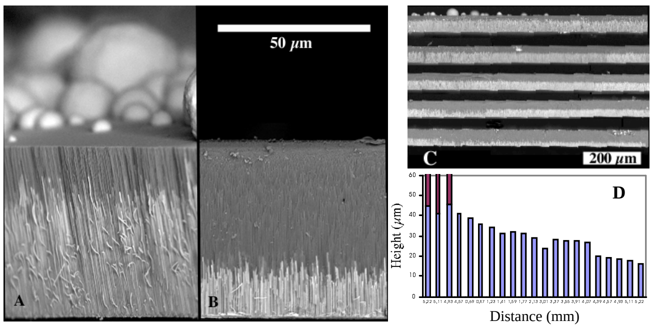

Figure 2. (A), (B): SEM images (back-scattered electrons) showing the edge points of a Cu filled membrane, deposited using a lateral gradient of –0.8 V to –0.6 V, respectively. (C), top to bottom: SEM image showing the entire cross-section of the membrane filled with electrodeposited Cu gradient. (D) A graph showing average measured heights of the deposited Cu (the region where Cu is deposited outside the membrane as ‘mushrooms’ is marked).