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Corrosion Barrier Development for LBE Corrosion Resistance: Quaterly Report

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Quarterly Report

During the past quarter, one graduate student and two undergraduate student researchers worked on this project. The major accomplishments during the past quarter are summarized below:

Synthesis of Cr nanowires inside alumina template pores

With the demonstration of formation of nanoporous alumina on steel and its good adhesion to substrate under thermal cycling, the next project task was to synthesize Chromium nanowires inside the alumina pores. During the previous quarter, a specialized sample holder was developed towards this goal. Various techniques for the deposition of Chromium were investigated and electrodeposition was determined to be the most suitable approach due to the large aspect ratio of the pores. A challenge in using electrodeposition for porous alumina is the potential sealing of the pores in aqueous solutions at higher temperatures. To avoid this problem, a search was carried out for recipes for the deposition of Cr using non-aqueous or low temperature deposition schemes. Two such recipes were identified and experimentally tried out, the one selected is described below.

The process for the Cr wire nanodeposition was developed on silicon substrates using the following procedure. Silicon substrates were first degreased and cleaned using standard cleaning techniques. A back contact was next formed on the back of the silicon wafer by depositing 500 nm of aluminum followed by annealing at 250 degrees C for 30 minutes. Next, Pt/Al layers of thicknesses 10nm/900nm were formed on the top of the silicon wafers. The Pt layer was deposited to provide improved adhesion to the substrate as well as to act as a protective layer for silicon from the anodization process. The top Al layer was then anodized in 0.3M oxalic acid at 22 degrees C using a current density of 40mA/cm². The voltage time relationship monitored during anodization, shown in Fig 1, confirmed the formation of the porous alumina all the way down to the Pt layer. After this the samples were pore widened in 6% phosphoric acid solution for 7 minutes to clean up the residues from the anodization process. The samples were then thoroughly rinsed in DI water and dried in nitrogen gas. Following this, the samples were imaged in a Field Emission SEM; the top and cross-sectional views are shown in Figures 2 and 3 respectively. The irregularity of the pore structures in Fig. 2 is due to the single step anodization used for the test runs; the pore regularity can be significantly improved by using the two-step anodization process as demonstrated earlier.

Next the pores in the alumina layer were filled with Cr using the following procedure. A fresh chemical solution was prepared using 100 g/l Chromic acid solution, 5g/l of sulfuric acid solution and 1 liter of water. A specialized sample holder was designed and fabricated to carry out the electrodeposition of Cr. For electrodeposition, the back of the silicon substrate was used as the cathode and a platinum mesh was used as the anode (note that this configuration is opposite of that of anodization). A current density of 20 mA (60mA/cm²) was then applied and the process continued for 1 to 5 mins. Fig. 4

shows the voltage time characteristics monitored during the process for 5 minutes of electrodeposition. Next the samples were scribed and cross-sectional images were taken in a Field Emission SEM. Figure 5 shows the cross-sectional image of a sample showing complete filling of the pores demonstrating the feasibility of this technique. This process will be next transferred to steel substrates using a similar procedure.

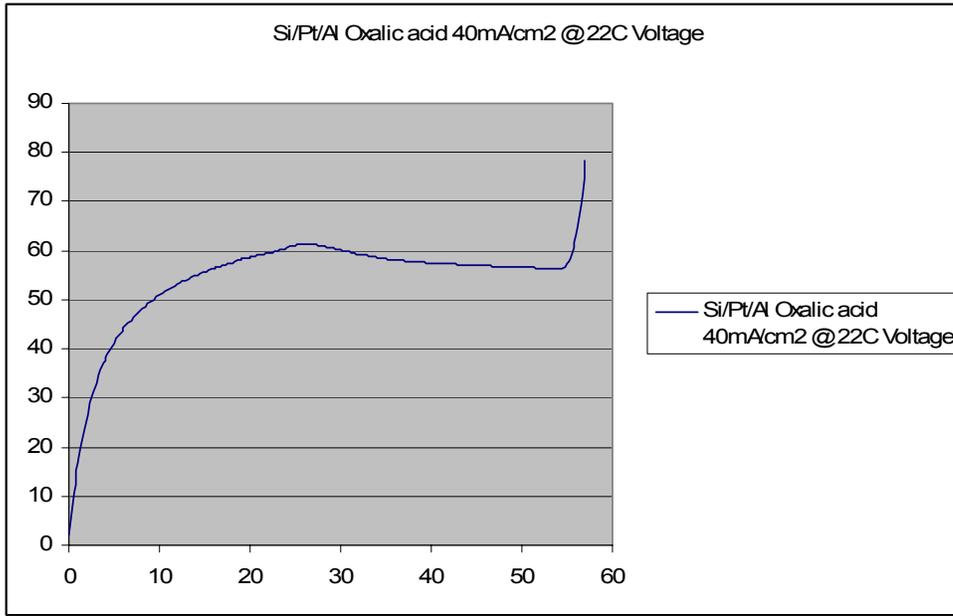


Fig 1. Voltage time relationship monitored during anodization

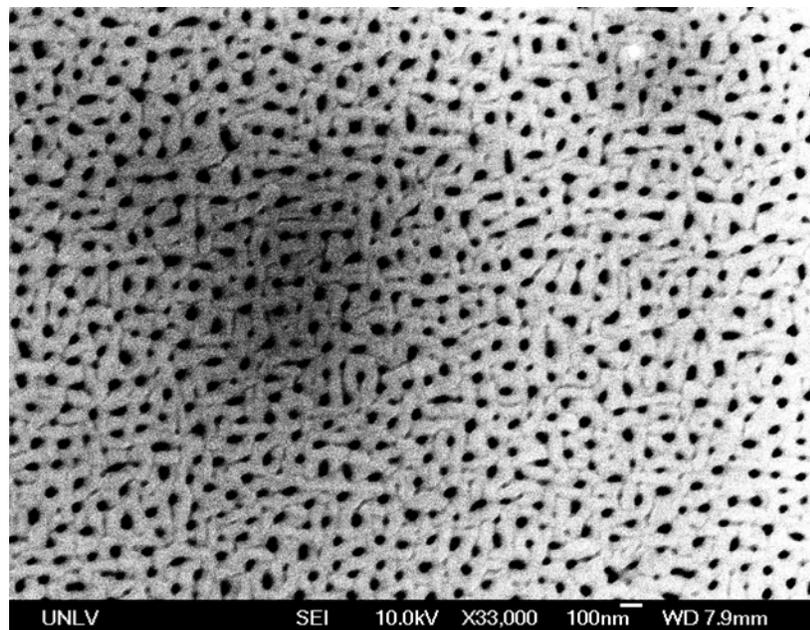


Fig 2. Top view of porous alumina sample.

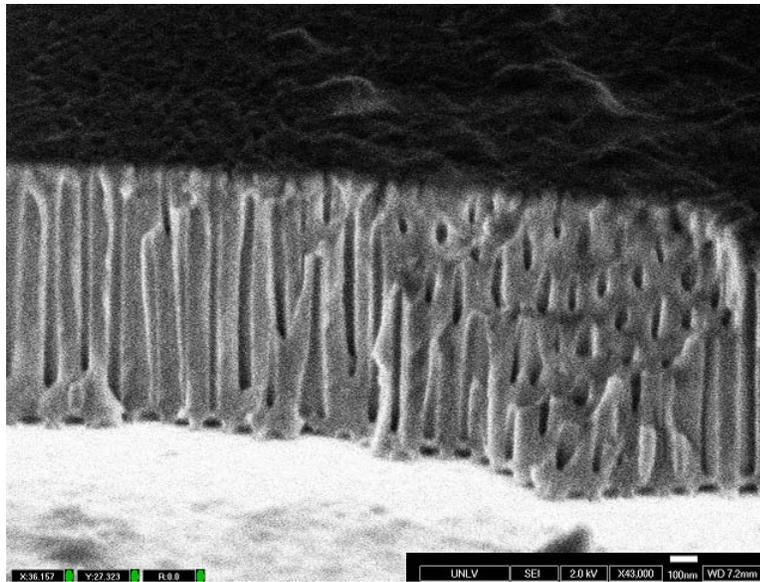


Fig 3. Cross sectional view of porous alumina sample

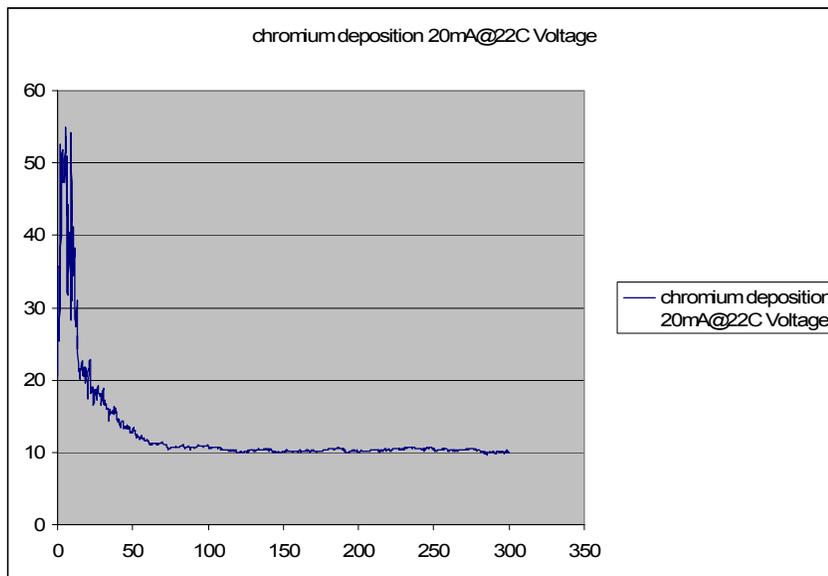


Fig 4. Voltage time characteristics monitored during chromium deposition.

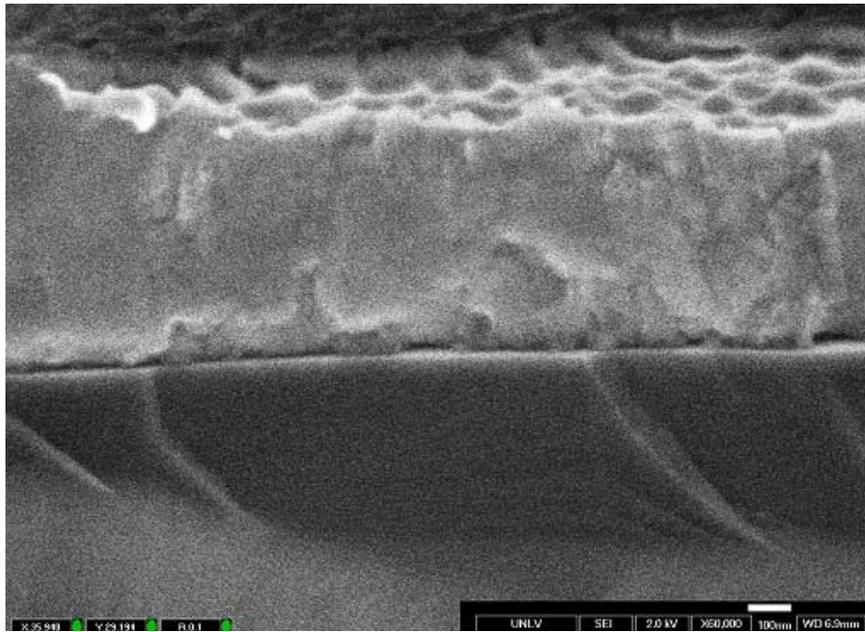


Fig 5. Cross-sectional image of Cr filled alumina pores