Corrosion Barrier Development for LBE Corrosion Resistance

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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:

1. Fabrication/characterization of nanoporous alumina on steel

In the last quarter, a specialized sample holder was developed for the anodization of alumina on steel. In addition, it was determined that oxalic acid was the most appropriate acid for the anodization of these structures. The steel samples obtained from LANL were first cut into a number of pieces, each measuring 11mm x 8mm x 1.6mm, to allow multiple experiments. Special care was taken to ensure that the cutting process did not damage the samples. After investigation of several techniques, including laser cutting, the samples were cut using EDM wires. The cut steel pieces did not show any damage to the surface or the edges.

The steel samples were cleaned using acetone and methanol followed by DI water rinse, and then dried in nitrogen gas. A thin layer of titanium (10 nm thick) was deposited on the surface of the steel followed by deposition of a thick layer of 99.999% aluminum (1 um thick) using e-beam evaporation. The Ti layer was deposited for improved adhesion of aluminum on steel; direct deposition of aluminum on steel showed poor adhesion. The samples were then anodized in 0.3M Oxalic acid solution maintained at 15°C using a constant current density of 20mA/cm². The voltage time relationship, monitored during anodization, and shown in Fig.1, confirmed the formation of the porous layer of alumina. While the samples were anodized for 600 seconds, the data in Fig. 1 is shown only up to 80 seconds for clarity. Visual inspection of the samples (shown in Fig. 2) also confirmed the formation of the porous alumina layer. The samples were then coated with a thin layer of gold and characterized by SEM imaging. However, the surface of the sample was found to be very rough, which made high resolution imaging very difficult. We believe that it will be necessary to polish the surface of the steel samples before metallization in order to perform high resolution SEM imaging, which we are currently pursuing.

An important objective of this project is to develop a coating technology that will be able to provide corrosion resistance after thermal cycling. An important requirement for this is good adhesion of the coating film on the substrate under thermal cycling. Towards this goal, the samples were subjected to thermal cycling to 300°C and 400°C. A Lindberg/BlueM oven was used for this purpose and the samples were subjected to half hour cycles. Visual inspection of the samples showed the appearance of thermally cycled samples was same as the uncycled (room temperature) samples. Also, a preliminary scratch test using a pin showed the alumina coating to have good adhesion to steel after thermal cycling.

In order to accurately characterize the adhesion properties of the alumina coatings, scratch tests were performed on the thermally cycled and uncycled samples using a nano-scratch tester instrument at microphotronics corporation. The scratch test method is done by generation of scratches using a spherical stylus (Rockwell C Diamond, tip radius 2um). The stylus is drawn at a constant speed across the sample under either constant or progressive loading at a fixed rate. For progressive loading, the critical load is defined as the smallest load at which appreciable failure occurs on the sample; for the constant loading, the critical load corresponds to the load at which a regular occurrence of such failure is observed along the track. The scratch test is basically a
comparison test and the critical load depends on the mechanical strength (adhesion and cohesion) of a coating to the substrate. The critical load depends on parameters that might be directly related to the test itself like the loading rate, scratching speed and indenter tip radius and indenter material. It also depends on the coating substrate parameters that include the substrate hardness and roughness, coating thickness and roughness, friction coefficient between coating and indenter, internal stresses in the coating. A scanning force microscopy is used to obtain high resolution images in three dimensions and the quantitative lateral and depth measurements can be obtained in the scratched portions. The pre scan recording on the sample is done to include the effects of uniformity in the flatness of the sample, where the penetration depth is measured during the test. The post scan reveals the elastic recovery of the coating-substrate residue by providing the scratch path profile.

Fig. 1. A typical voltage-time relationship obtained during anodization of aluminum steel using 20mA/cm2 @ 15C.

Fig 2: Photograph of two anodized samples showing the porous alumina on steel
The results of the scratch tests for the thermally cycled samples (300°C and 400°C) and uncycled sample (room temperature) are summarized in Table 1. ‘Critical damage’ indicates the smallest force at which a recognisable failure occurs. ‘Delamination’ indicates the minimum force at which the film delaminates from the substrate. Fig. 3 shows a typical optical microscope image of the sample during scratch test. Fig. 4, 5 and 6 show the scratch test results for the samples 1, 2 and 3 respectively.

Table 1: Scratch test results obtained for alumina coating on steel under different thermal cycling conditions.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Temperature</th>
<th>Critical Damage</th>
<th>Delamination</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Room Temp</td>
<td>1.56mN</td>
<td>5.38mN</td>
</tr>
<tr>
<td>2</td>
<td>300°C</td>
<td>1.38mN</td>
<td>2.27mN</td>
</tr>
<tr>
<td>3</td>
<td>400°C</td>
<td>1.59mN</td>
<td>3.38mN</td>
</tr>
</tbody>
</table>

Fig 3: A typical optical microscopy image obtained during scratch test

The results obtained from the scratch tests are encouraging, demonstrating good adhesion of the nanoporous alumina coatings on steel for temperature cycling up to 400°C. Our next step is to subject the samples to further higher temperatures (up to 600°C) and evaluate the adhesion properties of the coatings.
Fig. 4 Scratch test results for thermally uncycled sample

Fig. 5. Scratch test results for sample thermally cycled to 300°C
2. Synthesis of Cr nanowires

The pores in the nanoporous alumina will be filled with Cr to provide increased corrosion resistance in LBE. These Cr nanowires will be synthesized electrochemically inside the pores. Towards this goal, a specialized sample holder was designed and fabricated that is similar to the one developed for anodization of steel. Following an extensive literature search, the following electrolytes were selected for the electrodeposition of Cr nanowires. Test runs are currently in progress to determine the suitable deposition parameters for Cr inside the pores.

\[ 100g/l \text{ CrO}_3 + 5g/l \text{ H}_2\text{SO}_4 @ 25^\circ C \]
\[ \text{current density} = 0.4mA/cm^2 \]

The next phase of the project will involve in accomplishing the following tasks:

- Create nanoporous alumina on polished steel surface for SEM imaging. Steel surface will be polished mechanically as well as electrochemically.
- Subject the samples to higher temperatures (500\(^\circ\)C and 600\(^\circ\)C) and obtain scratch test results to evaluate the adhesion of alumina layer to the substrate.
- Deposit chromium inside the alumina template.
- Formation of dense alumina on surface using sputtering or hydration.
- Characterization of nanoporous coatings.