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Corrosion Barrier Development for LBE Corrosion Resistance: Quarterly Report (April 2006)

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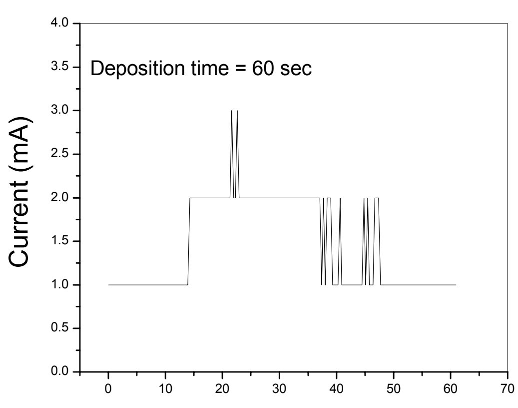
Quarterly Report (April 2006)
Task 23 (Das)

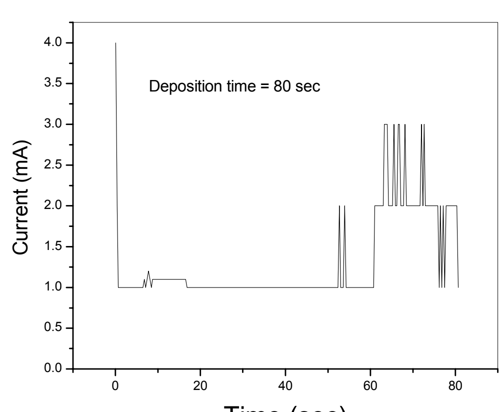
During the past quarter, one full time graduate student and one half-time undergraduate student researcher worked on this project. The major accomplishments during the past quarter are summarized below:

Deposition of Ni nanowire inside alumina template pores

As reported in the last quarterly report, synthesis of Cr nanowires was found to be problematic in terms of uniform coverage. Hence Ni was identified as the alternative metal to form the nanowires. The purpose of the metal nanowires is to provide structural integrity to the nanoporous alumina, as well as a second defense mechanism against corrosion by oxidizing in case the top alumina layer is compromised. Nickel was selected due to its established electrochemical synthesis procedure. While Ni can provide very good structural integrity to the porous alumina, one potential problem is its higher dissolution rate in LBE. However, since the Ni layer will be protected by a thick layer of *dense* alumina on top, this is not expected to pose a major problem. After a detailed consideration, it was decided that Ni filling of the porous alumina structure can provide very valuable information about performance of the coating layers under thermal cycling. In parallel, process development for uniform deposition of Cr nanowires will also continue, which if successful, will be included in the final coating structure.

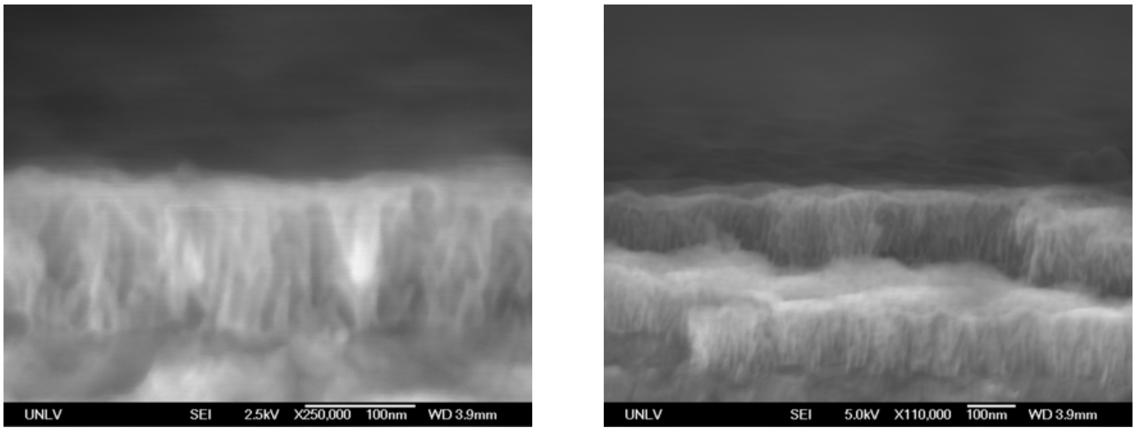
Deposition of Ni nanowires was achieved by using the following procedure. The electrochemical deposition of the Ni nanowire was carried out in a two-electrode arrangement with a Pt-mesh as anode, and a thin layer of Pt underneath the template acting as a cathode. A Watts solution was used (a mixture of $NiSO_4{\cdot}6H_2O$, $NiCl_2{\cdot}6H_2O$ and H_3BO_3) for the Ni source. The wires were grown at a potential difference of 1 V between the anode and the cathode. The deposition was done at ambient temperature. Two different deposition times (60 sec and 80 sec) were used for the wire growth. The deposition current as a function of time was used to monitor the growth rate of the wires. Time variations of deposition current are furnished below, which show uniform time dependence with occasional fluctuations common for electrochemical depositions.





Current-time characteristics during Ni nanowire deposition for different time lengths

The Ni nanowire samples were characterized by Field Emission SEM. The figures below show cross-sectional images of Ni nanowires deposited for different time lengths. The figures show excellent position uniformity and coverage. Thus the problem encountered previously with Cr nanowires is solved by the selction of Ni as the nanowire material. The next step will be to deposit a thick layer of dense alumina on top by sputter deposition, after which the samples will be ready for testing.



Cross-sectional FESEM images of Ni nanowires electrochemically deposited inside nanoporous alumina for different time lengths.

Investigation of Cr nanowire deposition uniformity

As mentioned previously, a significant problem was encountered with uniformity of Cr nanowires synthesized inside the alumina pores. This was somewhat puzzling since a well established recipe for the deposition of Cr was used. One explanation for the observed non-uniformity is the restricted space inside the nanopores that may not be conducive to the Cr synthesis chemistry. During the last quarter, further investigation of this phenomenon has been initiated in order to develop the process for more uniform deposition of Cr nanowires. Towards this objective, alumina structures with large pore diameters (100 nm) were synthesized by carrying out anodization in oxalic acid. Next, Cr will be deposited electrochemically inside the alumina pores and imaged to investigate the position uniformity.