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Third Quarter Report, Covering January through March 2002

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Principal Investigators: Farley and Perry

Progress from June 2001-November 2001 was presented at the winter meeting of the American Nuclear Society in Reno (November 12-15, 2001), and incorporated into a refereed conference proceeding. A copy of the refereed conference proceeding has been provided to Tony Hechanova. In brief, the paper described the new program to examine the corrosive effects of lead-bismuth eutectic (LBE) on steels. We employed various types of surface studies (Scanning Electron Microscope [SEM], and X-ray Photoelectron Spectrometry [XPS]) to examine steel samples that had been exposed to LBE for various lengths of time at various temperatures. The goal is to understand the chemical reaction products and chemical reactions involved in the corrosion. More than 40 samples were examined. Preliminary results of our measurements were presented. We were able to find the atomic composition as a function of position on the sample. We examined both exposed and unexposed samples, and found significant differences.

On the unexposed steel, we found the expected surface dominance of Cr over Fe and Ni, as one expects for a passivated surface. No Pb or Bi is seen. However, we were surprised by the levels of Zn. On the exposed sample, we see the suppression of Cr with respect to Fe as seen in other corroding stainless steel systems. We also see some residual Pb and Bi.

Progress since November 2001

Sputter depth profiling was performed on samples of steel that were exposed to LBE by the IPPE laboratory in Moscow. In sputter depth profiling, the surface is sputtered away by ion milling. This allows analysis of the material farther down in the sample. This is the first time that sputter depth profiling has been carried out at UNLV.

After sputter depth profiling, survey XPS scans were taken. Preliminary analysis has been done of the survey scans. This yields a plot of the elemental abundance as function of depth, at one location on the steel-LBE sample.

In addition, XPS data has been taken at high resolution. This can reveal the oxidation state of the element. This data has not been analyzed yet.

We are currently engaged in a critical analysis of the literature: of studies of LBE from issues of JNM, and more generally in the literature of corrosion of steel, both by LBE and in conventional "rusting." The vast experience and expertise of Dale Perry has been of immense value in this regard. Perry and a student at Univ. California Berkeley have conducted a literature search, and Perry has the encyclopedia knowledge of the field to assess critically the various reports in the literature.

Our preliminary findings are as follows:

(1) In the corroded region, we find experimentally that the nickel is totally gone. This is consistent with the literature, which says that nickel dissolves first, then iron, and finally chromium.
(2) In our measurements, we find no chromium in the outer region of the oxidation layer. The literature says there should be some chromium deeper in, but we have not yet probed deeply enough into sample to find it. We will do that.

(3) We observe high abundances of iron at and near the surface of the oxidation layer. It increases once you get through the thin outer layer of surface contamination.

Dan Koury has started to write his Masters' thesis, and expected to receive his degree in August 2002.

Brian Hosterman joined the research group in fall 2001 as a new graduate student. Hosterman is participating in all of the activities of the group, including data-taking and data analysis with Dan Koury, and the writing of the manuscript for Journal of Nuclear Materials.

In addition to working with Koury, Hosterman is initiating another research thrust in the LBE program. He is planning to examine the LBE samples using the laser Raman system at UNLV. Laser Raman measures vibrational frequencies, and thus is very sensitive to chemical species, not just the element. Hosterman is working with UNLV postdoc Wayne Stanbery in becoming familiar with the laser Raman system. Hosterman made a first measurement of a glass cuvette to see if the cuvette had an appreciable Raman signal. Fortunately it did not, because this would have been a background signal.

Dale Perry has supplied some lead oxide standards, which will be examined by various techniques, including laser Raman. In recognition of the many contribution that Dale Perry has played in the LBE project, the Physics Department voted on April 2 to appoint him as an Adjunct Professor of Physics.