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Corrosion of Steel by Lead Bismuth Eutectic: Year 1 Annual Report

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Task 3: Year 1 Annual Report

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Introduction

The goal is to investigate the corrosion of stainless steel by lead-bismuth eutectic (LBE), which has been proposed for use as a blanket coolant and a spallation target in the transmuter.

Personnel

Two graduate students, one undergraduate student, one national lab scientist, and two UNLV faculty members are working on the project.

Physics graduate student Dan Koury, at UNLV since fall 1999, has taken a great deal of data using the SEM and XPS techniques (explained briefly below), and is writing his Masters' thesis. Koury is expected to receive his degree in August 2002.

Brian Hosterman, joined the research group in fall 2001 as a new physics graduate student. Hosterman is participating in all of the activities of the group, and is initiating another research thrust in the LBE program. He is planning to examine the LBE samples using the laser Raman system at UNLV.

We are pleased to have Allen Johnson, a professor in the UNLV chemistry department, join our group. Before joining UNLV, he worked as a chemist for industry, and has a great deal of experience with the XPS technique. Johnson was the key faculty member at UNLV in purchasing and running the first XPS instrument in the state of Nevada. His expertise is very important to the task, and we are delighted to have him on the team.

Undergraduate student Denise Parsons has also joined the group. She has previously worked for the Nevada Test Site and the Yucca Mountain Project. She is a major help, assisting Johnson in data analysis of the XPS data.

Collaboration with DOE Laboratory facilities and personnel

Dale Perry, LBNL staff scientist, is a very important national lab collaborator. He has wide-ranging expertise in the analysis of nuclear materials, which has been of great use to our task. In addition, he is an expert in the chemistry of lead, and he gave a seminar at UNLV to the AAA community on that topic. He has supplied some lead oxide standards, which are very valuable in our analysis of the real-world samples. In recognition of the many contribution that Dale Perry has played in the LBE project, the Physics Department voted on April 2, 2002 to appoint him as an Adjunct Professor of Physics. This allows him to serve on the thesis committee of physics graduate students Dan Koury and Brian Hosterman.

Ning Li of LANL has been very helpful in providing interface with the national LBE program. In addition, he has supplied samples of steel, which were exposed to LBE for various times and various temperatures by Russian researchers under contract to LANL. We have benefited from reading his published and unpublished work on the corrosion of steel by LBE.

Stuart Maloy of LANL has been very helpful on an important technical issue: the delicate cutting of the samples without damage or the introduction of artifacts. Graduate students Dan Koury and Brian Hosterman visited the Maloy laboratory in spring 2002 to observe the technique. On the basis of that valuable visit, we are purchasing a cutoff saw suitable for metallographic use.

In addition, the facilities at the Advanced Light Source (ALS) at LBNL have been used by Allen Johnson and Denise Parsons. Their results are discussed below.

Analysis of samples

We have used a variety of techniques of surface analysis, including:

- Scanning electron microscopy (SEM), which provides a visual image of the surface morphology.
- Energy Dispersive X-ray Spectroscopy (EDAX) and
- X-Ray Photoelectron Spectroscopy (XPS), which provide elemental composition as a function of position. XPS can also provide information about the oxidation state of the element. Sputter depth profiling has been employed, in which 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.
- Laser Raman Spectroscopy, which measures vibrational frequencies, and thus is very sensitive to chemical species, not just the element. Brian Hosterman is starting to become familiar with this technique, working with UNLV postdoc Wayne Stanbery. Hosterman will start by seeking the Raman spectra of standards, before examining the LBE-exposed steel samples. The first data were taken after June 1, 2002.

Results

Our first results are a measurement of the thickness of the oxide layer. Our first experiments appeared to indicate that the oxide layer was about ten microns thick. We were cautious about these data because the data were taken using SEM on a cross-sectioned sample that was cut with a machine tool. We were afraid that this cutting technique might smear the oxide layer, and this later proved to be the case. Our later results were taken at the ALS, in which UNLV scientists sputtered through the oxide layer, showing the oxide layer to about one micron thick. This agreed with the results of an experiment conducted on a visit by Dan Koury and Brian Hosterman to the laboratory of LANL scientist Stuart Maloy, at which a metallographic cutoff saw was employed. The upshot is that we now find an oxide thickness of about one micron.

We have made two scientific visits to the ALS, and did preliminary studies on the lateral uniformity of the corrosion layer and depth profiling. We got one useful study out of the ALS work, which concluded (again) that the thickness of the oxide layer is roughly one micron.

At UNLV we performed two sets of studies using the XPS: (1) on an as-received surface of 316 and 316L steel, and (2) on surfaces that were cleaned by sputtering.

The first study was the unsputtered surface of 316 and 316L. We found mostly O and C, with the signal levels too low to say much about the metal components. This is not unusual for samples that have been exposed to environmental sources of oils, greases, vapors, etc.

A second study employed systematic cleaning of the surface using Argon ion sputtering. Just a minute of sputtering removed the outer layer of contamination. We found included lead and bismuth on the corroded samples, carbide on the uncorroded samples, oxidized carbon on the corroded samples, and varying amounts of chromium, iron, and other oxides, sometimes varying as a function of depth (i.e. iron). We found a lack of nickel and chromium in the corroded sample.

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. We have a great deal of data, which is being analyzed.

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 UC Berkeley have conducted a literature search, and Perry has the encyclopedia knowledge of the field to assess critically the various reports in the literature.

We will see if the metals were either dissolved into the LBE or left behind at the base of the corrosion layer as the corrosion layer grew. We have found that there are advantages and disadvantages of sputtering at grazing incidence and at normal incidence.

Dissemination of results: at scientific conferences

Dan Koury, Brian Hosterman, Denise Parsons and John Farley went to the ANS winter meeting in Reno in 2001, and presented a paper. Allen Johnson will give a talk at the AVS in the fall of 2002 in Denver. Dale Perry will make a presentation at the FACCS meeting in Boston this November.

Dissemination of results: publication

Our findings will be published in 2002 in the refereed conference proceeding in the Journal of Nuclear Materials. In addition, more will be incorporated in Dan Koury's master's thesis, expected August 2002. Finally, we have much more data (chiefly XPS data), currently being analyzed, which is enough for at least one or two more papers in the refereed scientific literature.

Travel

We have taken students to the national laboratories: Dan Koury and Brian Hosterman to LANL, and Denise Parsons to ALS/LBNL.

Importance of sample preparation

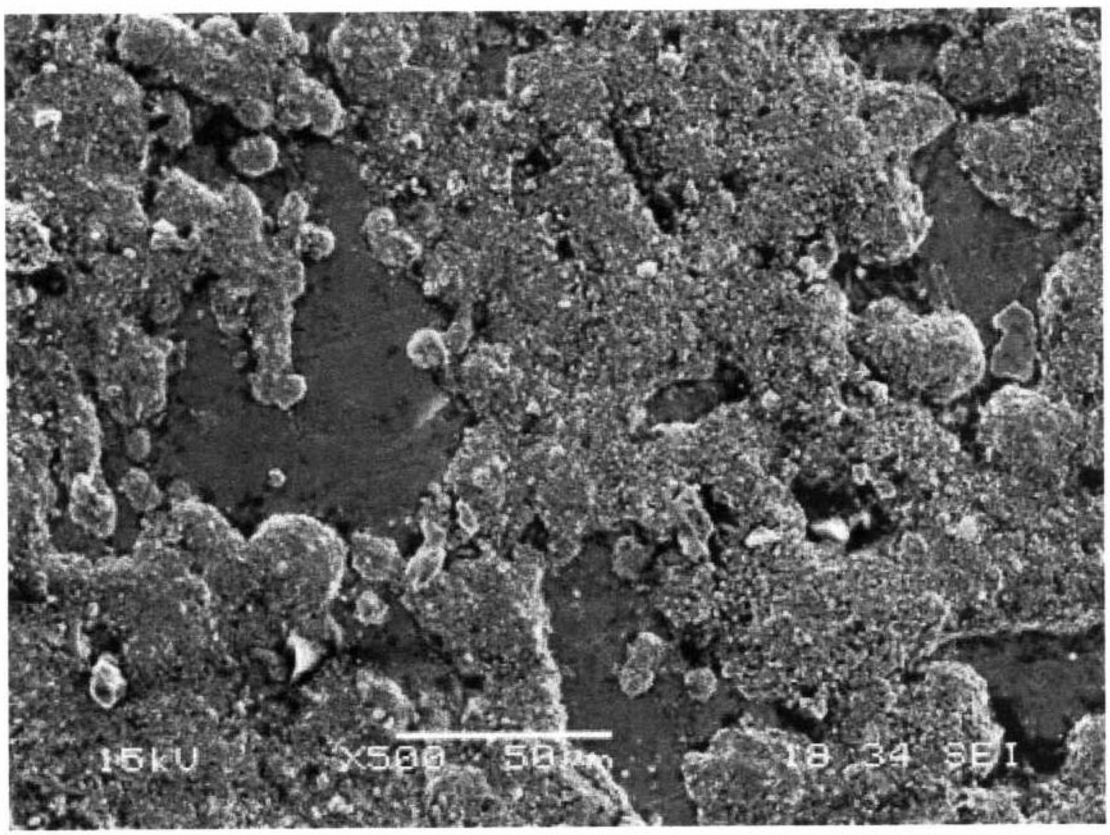
During the course of the first year, the importance of sample preparation became evident. Based in this important lesson learned, we purchased a metallographic saw, and an analytical balance.

This will benefit the UNLV project after the TEM facility is available, because proper sample preparation is essential there also.

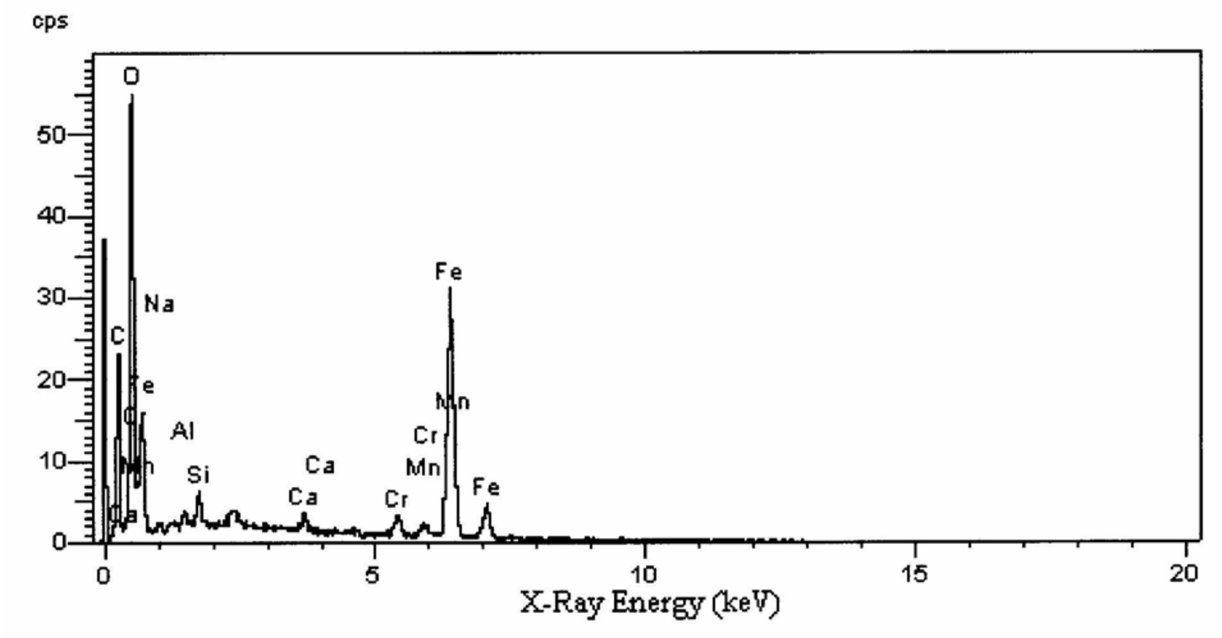
UNLV LBE loop

This research is highly relevant to the LBE loop that UNLV has recently obtained. We will work to relate our corrosion research to the experimental program at the experimental LBE loops at UNLV and around the world.

Representative Data



SEM of 316 steel sample exposed to LBE for 2000 hours at 450 C. EDAX analysis was performed on different highly localized areas. A quick glance shows that some areas are covered with corrosion.



EDAX spectrum of 316 stainless steel, exposed to LBE for 3000 hours at 550 C at IPPE Test Loop.