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Corrosion of Steel by Lead Bismuth Eutectic

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Task 3

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BACKGROUND

Materials for transmuter systems must be able to tolerate high neutron fluxes, great temperatures, and chemical corrosion. For lead bismuth eutectic (LBE) systems, there is an additional challenge in that the corrosive behaviors of materials in LBE are not well understood. Most of the available information on LBE systems has come from the Russians, who have over 80 reactor-years experience with LBE coolant in their Alpha-class submarine reactors. The Russians found that the presence of small amounts of oxygen (on the order of parts per million) in the LBE significantly reduced corrosion. However, a fundamental understanding and verification of its role in the corrosion of steels is incomplete.

RESEARCH OBJECTIVES AND METHODS

This research program will analyze various steel samples that have been exposed to LBE as part of the national program to develop LBE and transmutation technologies. The goal of this research is to understand the basic science of corrosion in the steel/LBE system. This information will be paramount in developing engineering efforts to control, avoid, and/or minimize the effect of corrosion of steels by LBE in transmuter and LBE systems. Additionally, this program provides UNLV researchers with hands-on experience that will be crucial in developing the UNLV LBE program, supporting the University's mission with the ISTC target complex, and the future development of additional facilities to examine LBE systems.

Investigators performed post-experiment testing and analysis on steel samples that have been in intimate contact with LBE. They employed surface analysis techniques that included Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDAX) spectroscopy, X-ray Photoelectron Spectrometry (XPS), and laser Raman spectrometry.

Allen Johnson and John Farley next to an X-ray Photoelectron Spectrometer (XPS).

These techniques, applied to the steel surface, have probed the surface morphology, elemental composition, and oxidation states as a function of position. Chemical alterations and resulting chemical species were studied at the steel surface. Additionally, the X-ray fluorescence microprobe at the Advanced Light Source at Lawrence Berkeley National Laboratory was used. This allowed spectroscopic characterization of the stainless steel before and after interaction with LBE to determine its composition.

RESEARCH ACCOMPLISHMENTS

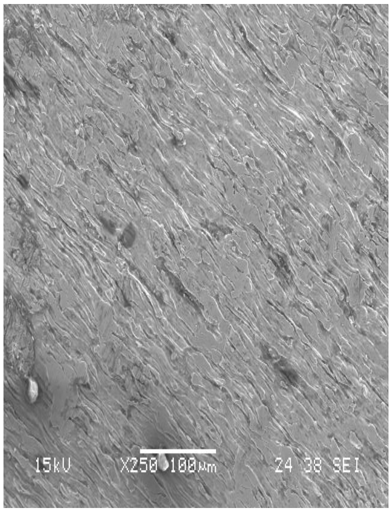
Using the technique of XPS/sputter depth profiling, researchers observed that the lead and bismuth were confined to the surface of the oxide – there was little or no penetration of the coolant into the protective oxide layer. Two forms of oxide layer were found on the 316 steel; on other alloys, a complicated structure composed primarily of iron oxide with an underlying oxide containing both chromium and iron was found. A chromium oxide layer on the metal substrate was also found on the 316L steel. This is similar to findings on the non-corroded starting materials. Ultimately, these analyses provide insights into the migration of materials, the composition of the protective oxide layer, and the basic science of the corrosion process. Overall, dramatic differences between the exposed and unexposed samples were found.

Images of the oxide layers of the 316 and 316L samples revealed a fairly uniform 10-micron thick oxide layer in the 316 samples and a 1-micron thick oxide in the 316L samples. The bottoms of the etch pit were optically examined and the 316L was seen to have a "patchy" bottom compared to the smooth bottom of the etch pits on 316 stainless. These results contrast findings from the material exposed at IPPE but are not unexpected from the literature.

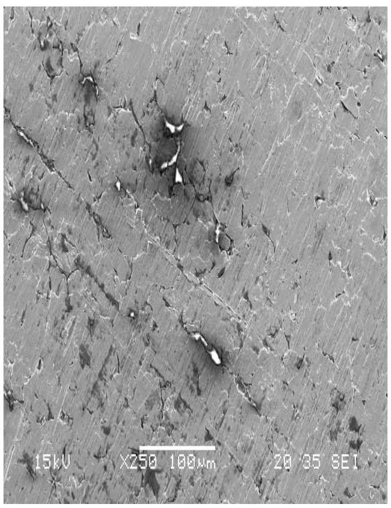
Further utilization of the XPS machine permitted experimentation on steel samples using Argon ions. The argon beam was used to mill the surface layers of the sample away, allowing researchers to examine the oxide and alteration layers as a function of depth.

Investigators took depth-profiling data on D9 steel that had been exposed to LBE. This is the first time that this type of steel was examined. This analysis enables researchers to separate out two effects and determine their effect on corrosion (composition of the steel vs. surface preparation).

Finally, an optical microscope was purchased and installed February 2003. This enhances researchers' abilities to make accurate distance measurements on steel samples.



Scanning Electron Microscope (SEM) image of the surface of 316 stainless steel before exposure to lead-bismuth eutectic (LBE).



SEM image of 316 stainless steel that is corroded from being exposed to LBE.

FUTURE WORK

Depth profiling of steel samples using the XPS technique will continue. Other steels such as D9, HT9, and T410 will be examined. Raman measurements will also be made; these are essential with regards to measuring chemical speciation.

HIGHLIGHTS

- "Surface Studies of Corrosion of Steel by Lead-Bismuth Eutectic" presented at the Federation of Analytical Chemistry and Spectroscopy Societies Annual Meeting , Oct 13-17, 2002, Providence, RI.
- "Surface Studies of Corrosion of Stainless Steel by LBE" presented at the meeting of the American Vacuum Society 49th international symposium, Denver, CO, Nov 3-8, 2002.
- Dan Koury completed a Physics Master of Science thesis "Investigation of the Corrosion of Steel by Lead-Bismuth Eutectic using Scanning Electron Microscopy and X-Ray photoelectron microscopy" December 2002.
- "Surface Studies of Corrosion of Stainless Steel by Lead-Bismuth Eutectic" presented at the ANS Student Conference, Berkeley, CA, April 2-5, 2003.
- Manuscript "Spectroscopic and Microscopic Investigation of the Interaction of Lead-Bismuth Eutectic (LBE) with 316 and 316L Stainless Steel at Elevated Temperatures" was submitted to refereed *Journal of Nuclear Materials*.

Samples will be characterized using a number of experimental techniques. Each technique has its own strength. Spectroscopic data can be combined with microscopic data, along with x-ray diffraction data, to fingerprint structural forms of the elemental species formed in the reactions. Plans to perform tests using synchrotron radiation (SR) based x-ray fluorescence (XRFF), a sensitive analytical technique capable of providing direct quantitative information on chemical compositions, are underway. X-ray fluorescence will provide a detailed map of the heavy metal ions being studied. Ultimately, data will then be analyzed and compared with the predictions from corrosion models.

Researchers also plan to expose samples to LBE in the new Molten Metal Small Experiments Facility (proposed 2004). By examining the samples before and after exposure to the LBE, researchers should be able to gain insights into the corrosion processes and kinetics. This should also allow the team to test the hypotheses postulated through the experiments with the already exposed materials.

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