Task 18

Fundamental and Applied Experimental Investigations of Corrosion of Steel by LBE under Controlled Conditions: Kinetics, Chemistry Morphology, and Surface Preparation

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BACKGROUND

This effort is a continuation of the work of Task 3 (see page 10), and the same overview applies. Advanced nuclear processes such the transmutation of nuclear waste, fast reactors, and spallation neutron sources require advanced materials systems to contain them. In particular, a successful program in nuclear waste processing that includes transmutation in accelerator-driven systems and fast reactors requires structural materials that are stable in the presence of non-moderating coolants. A prime candidate for such a coolant is Lead Bismuth Eutectic (LBE).

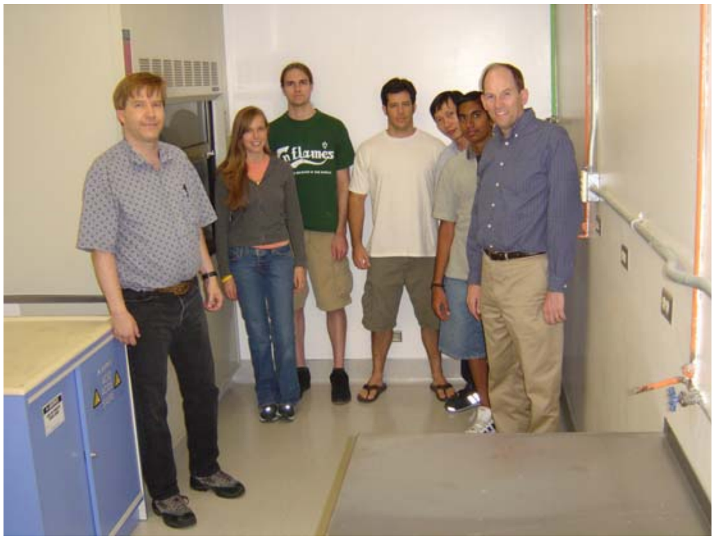
Materials in these systems must be able to tolerate high neutron fluxes, high temperatures, and chemical corrosion. For LBE systems, there is an additional challenge because the corrosive behaviors of materials in lead bismuth are not well understood. Most of the available information on LBE systems 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 in the LBE significantly reduced corrosion. However, a fundamental understanding and verification of oxygen's role in the corrosion of steels is incomplete.

RESEARCH OBJECTIVES AND METHODS

Previously, various steel samples that were exposed to lead bismuth eutectic were analyzed as part of the national program to develop LBE and allied technologies under TRP Task 3. In particular it was found that cold rolled 316L and a silicon modified HT9-like alloy EP823 had particularly low corrosion rates. The 316 systems and model iron/silicon alloys were compared using Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDAX) spectroscopy, and X-ray Photoelectron Spectrometry (XPS) sputter depth profiling. It was found that enhanced corrosion resistance was associated with underlying dramatic changes in corrosion layer composition and microstructure. From these observations, several possible mechanisms for the enhanced corrosion resistance were proposed.

To test these mechanisms:

1. It was suggested that some possible test samples be exposed in the DELTA loop (e.g. shot peened 316L) that are subsequently studied, along with other samples from the DELTA facility.
2. A high temperature lead-certified laboratory space (the High Temperature Materials Experiments Facility, HTMEF) was established where investigators are able to conduct molten LBE studies safely.
3. A Liquid Metal Corrosion Experiment (LMCE) is under construction, which will be a facility that allows the introduction, exposure, and extraction of materials test samples to hot (~1000°C) lead alloys (such as LBE) under careful atmospheric control. The facility will be made of refractory ceramics and metals, and thus will have better chemical control than steel based test systems. Lastly, it will be modular in design, allowing rapid changes to process parameters.
4. An existing mass selective ion beam apparatus is being upgraded to allow the implantation of isotopically labeled steel components into the samples, to allow monitoring of the diffusion of steel constituents in the corrosion layers and in the metal adjacent to the corrosion layers.
5. A set of gas phase experiments were started in sealed ampules and under controlled atmospheres to look at the formation of the corrosion layers and to track compositional changes in the corrosion layers and their substrates.


The High Temperature Materials Experimental Facility and Task 18 students and faculty.

RESEARCH ACCOMPLISHMENTS

Effort 1: Shot-peened (i.e., *in situ* cold worked) 316L steel samples were studied that were exposed at the DELTA loop at LANL. Shot-peening with smaller beads improved corrosion resistance: the smoother surface had fewer and smaller failures (see the top figure on the opposite page). This argues against the mechanical "keying" mechanism that was proposed earlier to explain the improvements in corrosion resistance observed with cold rolled 316L. On the other hand experiments at the Environmental Molecular Sciences Laboratory using a scanning Auger microprobe did not see evidence of improved chromium coverage in the cold worked material.

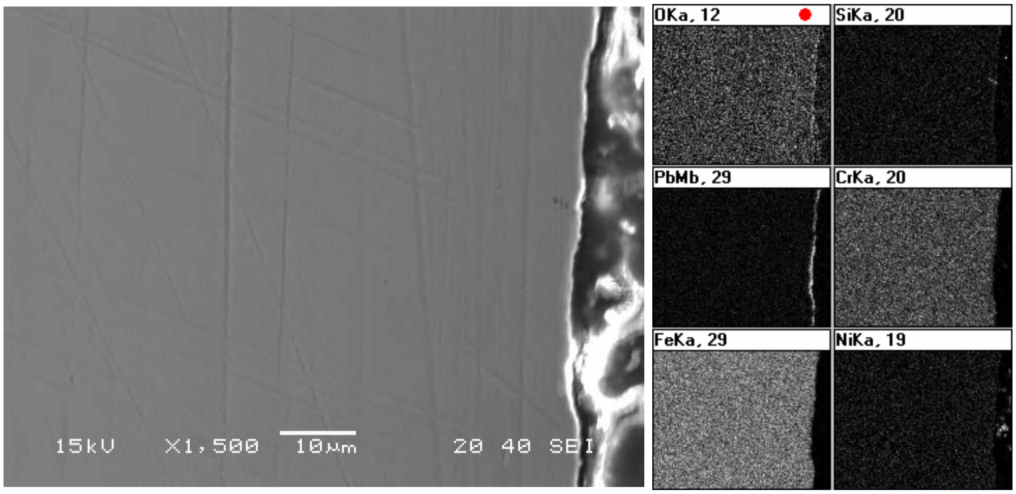
Effort 2: Plans were made to start experiments in the HTMEF (see figure on the previous page) in July 2005. The initial experiments will be gas phase experiments tracking the migration of alloy components during oxide growth at high temperature and low oxygen concentration.

Effort 3: The construction of the furnace section of the LMCE is essentially complete – while construction in the HTMEF continues.

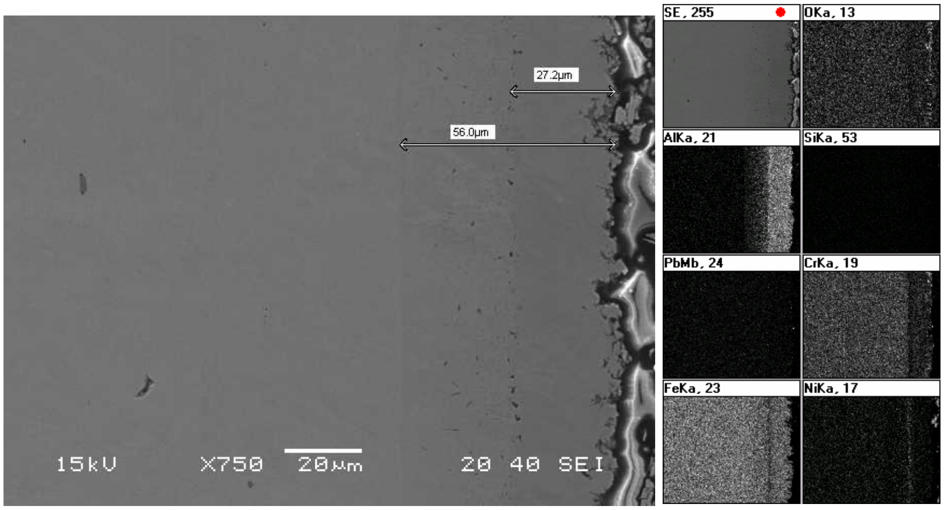
Effort 4: Aluminized 316L showed low corrosion but has a very complicated segregated surface/near surface region (see bottom figure). A paper is being prepared describing the effects of silicon in providing a protective layer. The aluminum results suggest that a significantly different protective layer may modify the underlying near-surface region in complicated ways. Ion beams have been obtained in the mass selective ion beam apparatus and the team is ready to start implanting stable isotopes to track diffusion in the metals and their oxides.

ACADEMIC YEAR HIGHLIGHTS

- "Application of X-ray photoelectron spectroscopy to the study of the lead-bismuth eutectic (LBE)-induced corrosion of stainless steel" by D.L. Perry, J.W. Farley, A.L. Johnson, D. Koury, B. Hosterman, U. Younas, and T. Ho was presented at a poster session at the ACS meeting in San Diego, March 13-17, 2005.



Shot-peened 316L exposed to LBE at the DELTA loop, LANL.



Aluminized 316L exposed to LBE at the DELTA loop, LANL.

Effort 5: The required instrumentation will be delivered to UNLV in July 2005 to start the gas phase experiments. Initial experiments will use a multiple zone tube furnace to heat an evacuated quartz ampoule to two different temperatures: one end will hold the sample under test and the second side will hold mixtures of copper and copper oxide at temperature to set the oxygen concentration. Later experiments will investigate the addition of LBE to the oxidation conditions.

FUTURE WORK

The certification of the HTMEF for lead work and the completion of the LMCE will continue. An investigation of the migration of isotopically labeled components in the oxidation and corrosion of the test alloys will be initiated. Investigations of samples exposed in the DELTA loop will be continued.

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