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Corrosion Mechanisms and Kinetics of Steels in Lead-Bismuth Eutectic: Quaterly Report

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Summary of progress in first quarter of 2006
Task 18: Allen Johnson and John Farley

Personnel

Four graduate students in our group are pursuing degrees under this task. The students and the working titles of their thesis or dissertation are listed in the following table.

Student	Thesis/dissertation	Title
Brian Hosterman	masters thesis	Studies of corrosion of steel by lead-bismuth eutectic studied by micro Raman spectroscopy and x-ray diffraction
Dan Koury	doctoral dissertation	Study of pitting corrosion of steel by lead-bismuth eutectic
Thao Ho	doctoral dissertation	Study of corrosion of iron-silicon alloy steel by lead-bismuth eutectic
Umar Younas	masters thesis	Fundamental processes in lead-bismuth corrosion of steel studied by a small test facility

In addition, two undergraduate students, Jenny Welch and Karen Levy, are members of the research group.

Karen Levy, undergraduate biology major, joined the group during the quarter. Tehre is now a total of 6 students, including 4 graduate students and 2 undergraduate students.

Collaboration with LANL has been continued: task leader Ning Li, postdoc Jinsuo Zhang, graduate student Peter Hosemann, other people associated with DELTA loop at LANL.

Facilities

Room CHE 112C has been renovated into a High Temperature Materials Experimental Facility (HTMEF). The final renovation of the power and air conditioning was finished in March 2006.

Graduate student Brian Hosterman has assembled and debugged a laser Raman microscope for examination of corroded metal samples. The beam from an ion laser (Argon or Krypton) is focused on the sample, and the resulting fluorescence is analyzed by a spectrometer with an optical multichannel detector. Raman spectroscopy reveals the vibrational frequencies of samples, and has the advantages of smaller spot size (down to ~1 micron) and convenient access to the lower frequency vibrations expected in inorganic materials. The laser Raman microscope will be used for Brian Hosterman's master's thesis. Recent results obtained by Hosterman are discussed below.

Publication and presentations

Spectroscopic and microscopic investigation of the corrosion of 316/316L stainless steel by lead–bismuth eutectic (LBE) at elevated temperatures. II. Initiation of duplex oxide formation in D-9 alloy, Allen L. Johnson, Dan Koury, Jenny Welch, Thao Ho, Stacey Sidle, Chris Harland, Brian Hosterman, Umar Younas, Longzhou Ma and John W. Farley, presentation by Allen Johnson at the AFCI materials working group, March 2006, Santa Fe, NM.

Publication

Spectroscopic and microscopic study of the corrosion of iron-silicon steel by lead-bismuth eutectic (LBE) at elevated temperatures, Allen L. Johnson, Eric P. Loewen, Thao T. Ho, Dan Koury, Brian Hosterman, Umar Younas, Jenny Welch, and John W. Farley, accepted at Journal of Nuclear Materials (in press Dec 29, 2005). Page proofs were received during the first quarter of 2006.

Publications in preparation

Spectroscopic and microscopic investigation of the corrosion of 316/316L stainless steel by lead–bismuth eutectic (LBE) at elevated temperatures. II. Initiation of duplex oxide formation in D-9 alloy. Allen L. Johnsona, Dan Koury, Jenny Welch, Thao Ho, Stacey Sidle, Chris Harland, Brian Hosterman, Umar Younas, Longzhou Ma and John W. Farley (in preparation).

X-ray photoelectron studies of the lead-bismuth eutectic (LBE)-induced corrosion of stainless steels, D. L. Perry, D. Koury, B. Hosterman, J. W. Farley, A. L. Johnson, D. Parson, J. Manzerova, in preparation,to be submitted to Journal of Radioanalytical and Nuclear chemistry

Representative Experimental Results

A large number of studies of corroded samples were conducted using SEM, XPS, probe, and the TEM. Samples from the DELTA loop at LANL and from other sources were examined. Also, the investigation of 316 class stainless steel in LBE is continuing. Fig. 1 shows the Fe-Cr ratios of the protective thin oxide of a sample of cold-rolled 316 L stainless steel that has been exposed to LBE for 1000 hr at 550 C. The sample shows a Cr-rich oxide layer as revealed by XPS with sputter depth profiling. The top 100 nm shows an increasing Fe content, perhaps as a result of reaction of dissolved Fe with the oxide.

Corroded samples of D-9 steel have been in the process of being studies, which is a variant of 316 stainless steel that is optimized for resistance to swelling. The D-9 samples are notable for the process in which a localized failure of the protective oxide layer becomes widespread corrosion. The research group is examining the D-9 samples using a variety of surface microscopic techniques. A Wavelength Dispersion Spectroscopy

(WDS) map of an etched, corroded D-9 sample is shown in Fig. 2 below. It shows a rich morphology, with oxygen transport channels, anisotropic etching of the metal, and a strongly textured metal-oxide interface. A summary of the D-9 results was presented by Allen Johnson in March to the annual meeting of the AFCI research, and a detailed manuscript is in preparation for submission to the Journal of Nuclear Materials.

Raman spectroscopy reveals the vibrational frequencies of samples, and has the advantages of smaller spot size (down to ~1 micron) and convenient access to the lower frequency vibrations expected in inorganic materials. For example, wustite (FeO), hematite (Fe_2O_3) and magnetite (Fe_3O_4) are all iron oxides with Fe/O ratios near 1:1, but they have different vibrational frequencies. Typical data is shown in Figs. 3-5. The presence of the 670 cm^{-1} Raman peak in the corrosion layer of the 316 (annealed) sample (fig. 5) demonstrates that magnetite (Fe_3O_4, Fig. 4) and not hematite (Fe_2O_3, Fig. 3) was being formed on this sample, consistent with our previous results.

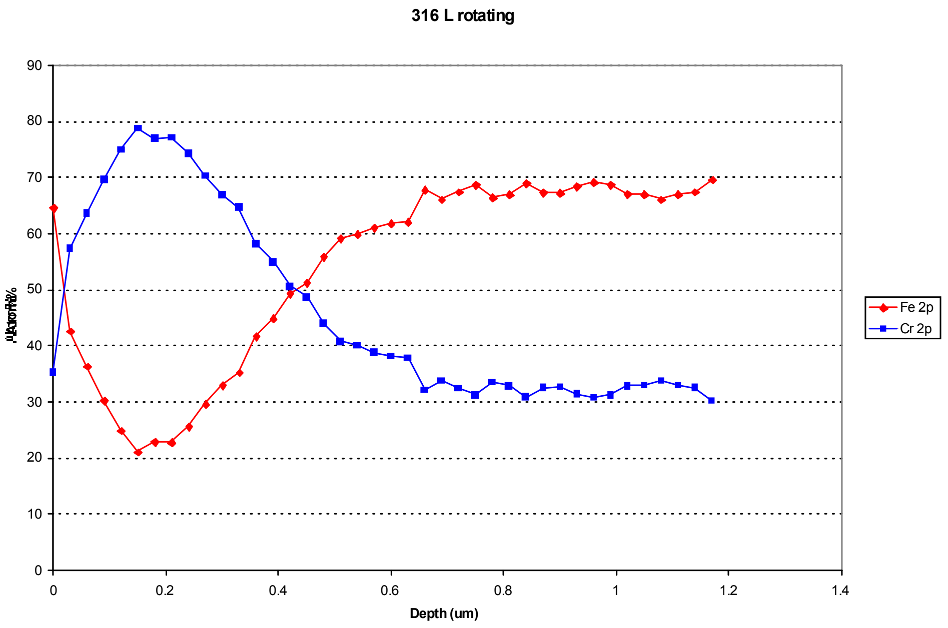

Figure 1. Iron/chromium ratio of the surface layer in cold rolled 316L stainless exposed to LBE for 1000 hrs at 550°C as determined by XPS sputter depth profiles. Note the increase in iron concentration in the top 100 nm of the oxide layer, perhaps due to reaction of dissolved iron with the surface oxide.

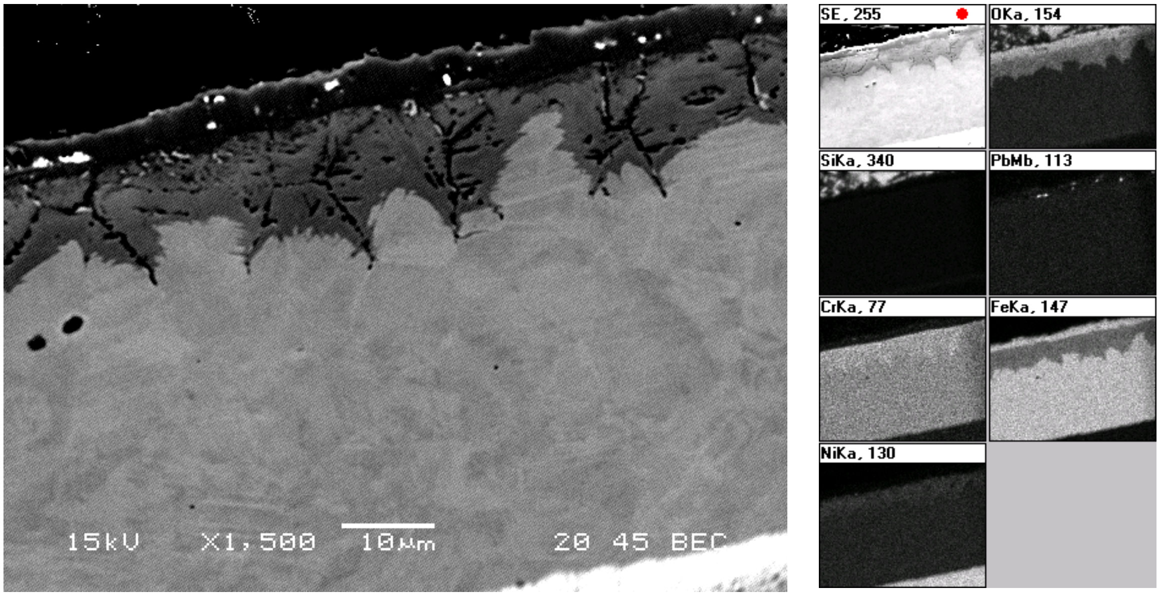

Figure 2. WDS image of D9 stainless as above. Note oxygen channels, etch ansisotropy, and highly irregular metal oxide interface.

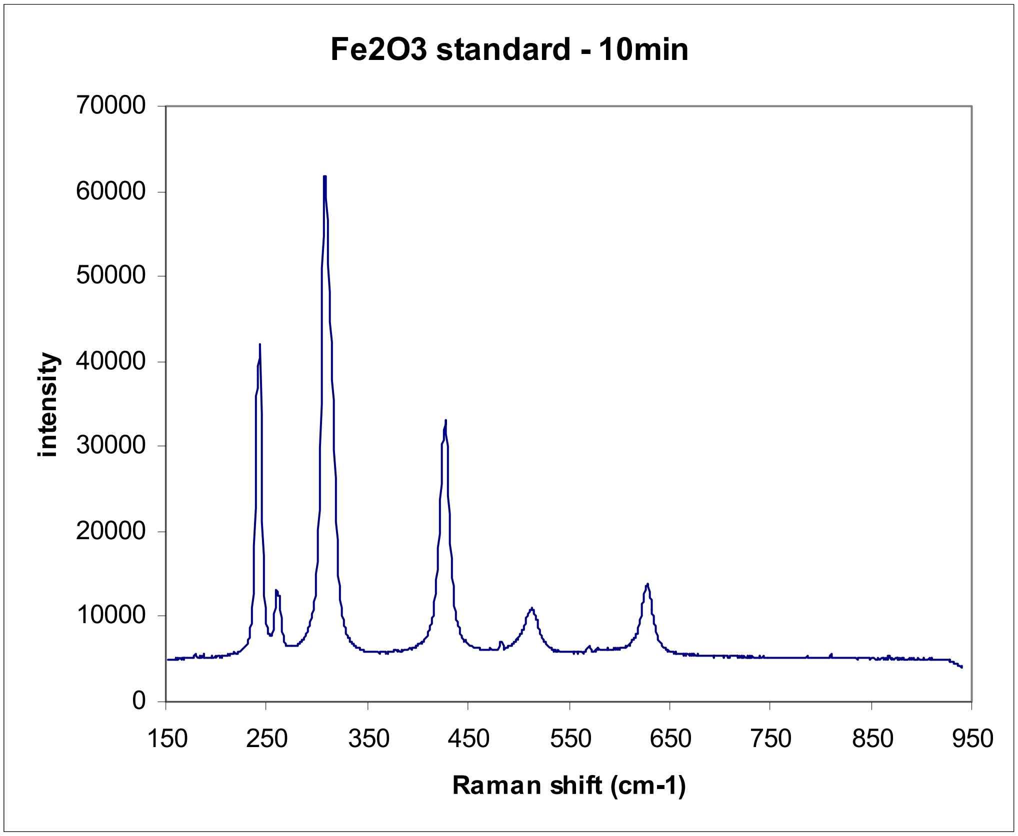

Figure 3. Raman spectrum of Fe_2O_3 standard.

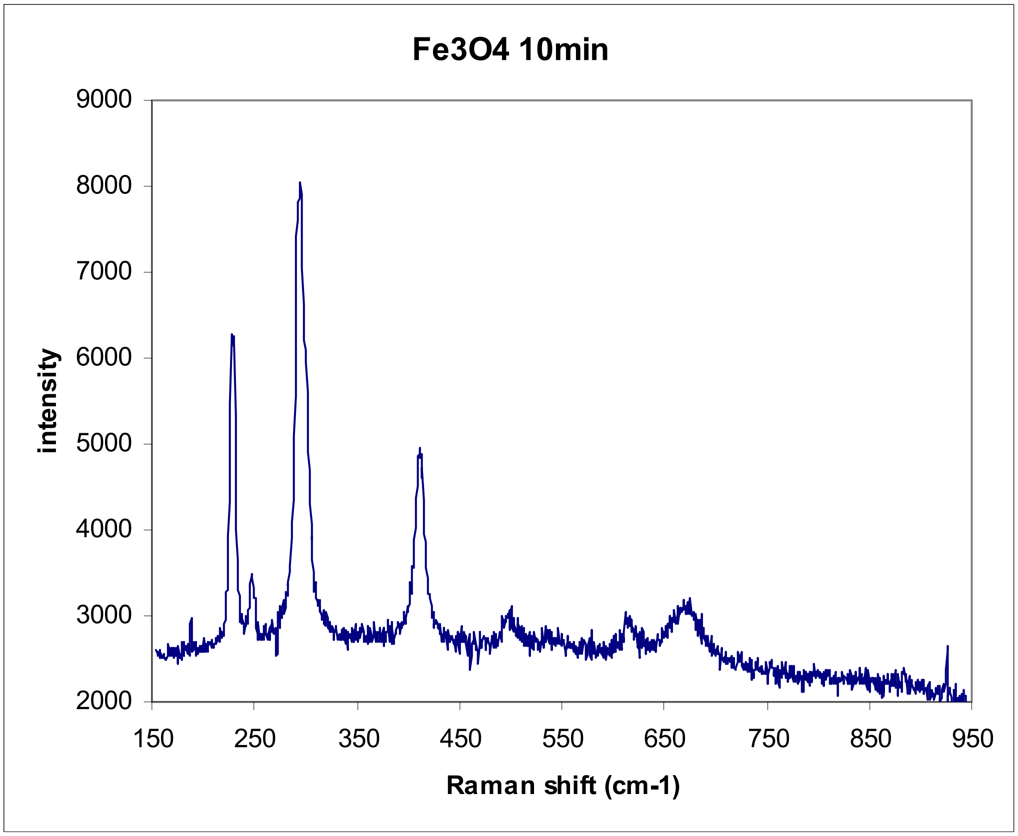

Figure 4. Note the new peak at 670 cm^{-1}. The other peaks are Fe_2O_3 peaks from impurities in the Fe_3O_4 standard.

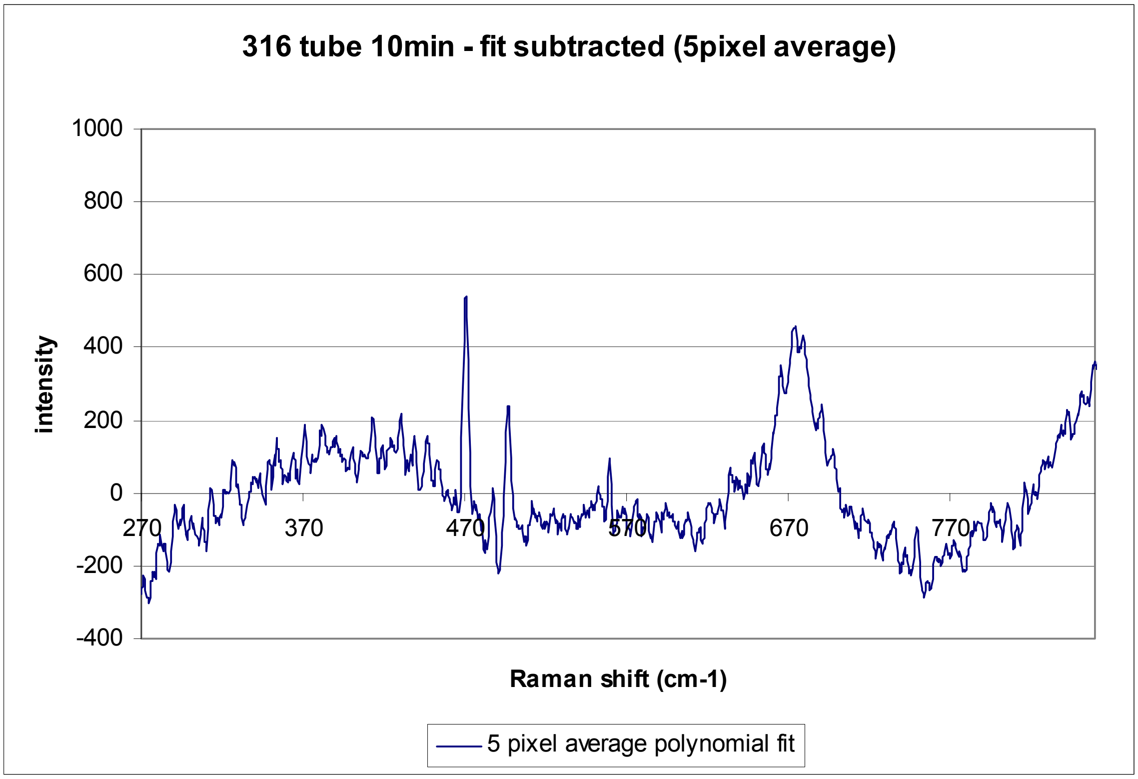

Figure 4. Annealed 316L exposed to LBE – 10 minute Raman spectra. Notice the peak at 670 cm^{-1}, characteristic of Fe_3O_4.