4-2005

Fundamental and applied experimental investigations of corrosion of steel by LBE under controlled conditions: kinetics, chemistry, morphology, and surface preparation: quarterly report (January 2005-April 2005)

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Quarterly Report: January 2005-April 2005

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Summary of accomplishments

- Started occupation of the High Temperature Materials Experimental Facility.
- Presented a paper at the meeting of the American Chemical Society in San Diego in March 13-17.
- Currently reviving the ion beam for implantation experiments.
- Currently analyzing 38 steel samples from the DELTA loop from LANL.

Introduction

This project has four components: (1) the fabrication of a materials test apparatus with unique capabilities, (2) comparative studies of steel corrosion under gas phase conditions comparable to the Lead Bismuth Eutectic (LBE) oxygen control conditions, (3) isotope labeling studies, and (4) collaborative efforts with other workers in the field.

(1) Materials test apparatus

We have started to build the High Temperature Materials Experimental Facility in room CHE 112C. We expect to locate 2-3 experiments there in the initial phase, and up to 6 at a later stage of the facility. The facility features a stainless steel hood for high temperature apparatus, a filtered laminar flow hood for sample manipulations and preparations, an acid work/storage area, steel bench space, gas storage, safety equipment, and space for equipment racks and computer workstation(s). This equipment is partially in place, and will continue to be delivered over the next month.

The LBE materials exposure facility is being constructed. We are currently sending parts out for bid.

(2) Comparative gas phase studies

We have submitted a PO for an appropriate tube furnace for the gas phase experiments.

They will use both thermal decomposition of unstable oxides (lead oxide, copper oxide) and the Oxygen Control System (built at KALLA) for atmospheric controls. We have arranged for access to the quartz tube capsule fabrication station in the physics glass shop.

(3) Isotope labeling studies

The isotope labeling experiments are progressing. The ion implantation facility experienced a number of control system malfunctions which have been repaired, and test experiments are planned for April.

(4) Collaborative work

In collaboration with LANL, we have been characterizing samples exposed to LBE in the DELTA loop at a temperature of 520C and for exposure times of 267 or 400 hours. We recently received 38 samples. Mounting and SEM/EDX investigations are ongoing, and will be reported as they become available.

We had particular interest in the shot-peened 316L samples, which should be roughly comparable to the cold rolled samples exposed at IPPE and examined by us. We found in the 267 hour DELTA loop exposed samples a very thin oxide layer, on the order of 1 micron thick (figure 1). However, the corrosion was not uniform and in some regions we found pitting, sometimes shallow pits with oxide at the bottom and sometimes deep pits, also oxidized. In regions where the LBE was not flowing (void regions near the hold down fixtures) more significant corrosion features were found: pits, detached oxide, evidence of significant removal of metal and lead infiltration (figure 2).

The 400 hour shot-peened specimen was similar (figure 3). In some cases we see simultaneous formation of lead, iron, and chromium oxides of porous morphology. In stagnant regions we see lead infiltration, breakdown of the crystal matrix of the alloy, and mixed (primarily chromium) oxides (figure 4).

Shot-peening with smaller beads improved corrosion resistance: the smoother surface had fewer and smaller failures (figure 5). This argues against the mechanical “keying” mechanism we proposed earlier to explain the improvements in corrosion resistance observed with cold rolled 316L.

The salient feature of these specimens is the absence of any obvious healing of oxide failures in the stagnant regions during LBE exposure. This problem is less important in the flowing (non-stagnant) regions: although oxide failure do occur, evidence of healing is observed in some cases.

Aluminized 316L shows low corrosion, but a very complicated segregated surface/near surface region (figure 6). We have a paper in preparation on 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.

As we have seen before with samples from another exposure facility (INEEL), there is evident contamination of the LBE by particulate matter, at least at cold temperatures. These particulates are not associated with observable oxygen by EDX, which argues that they were dissolved in the LBE in metallic form (i.e., not oxide particles) at the operational temperature. More detailed studies will be necessary to resolve this issue.

Figure 1.

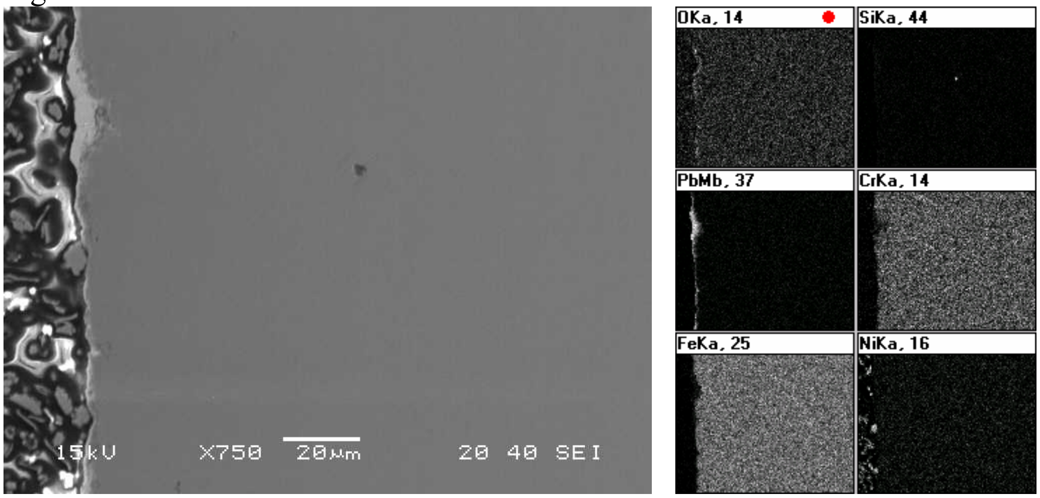

Figure 2.

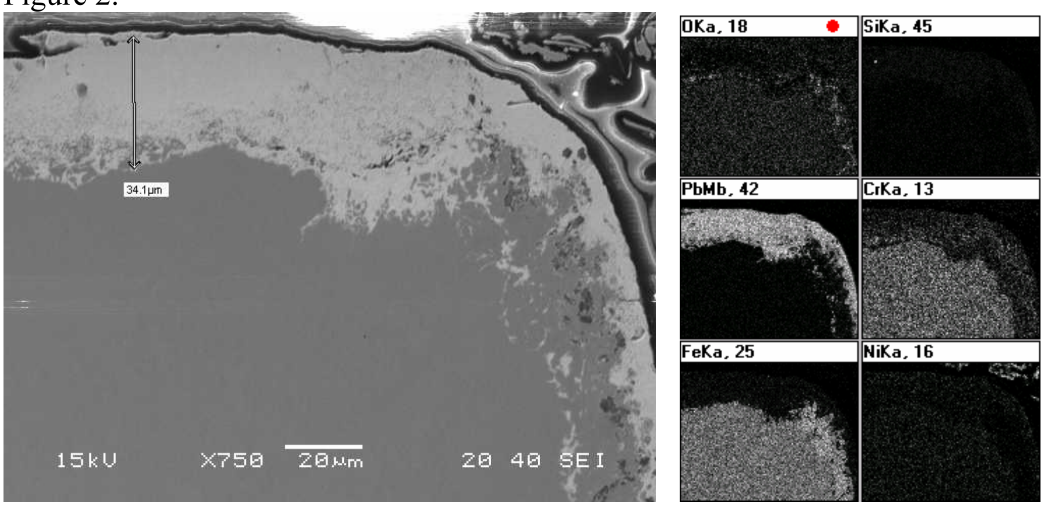

Figure 3.

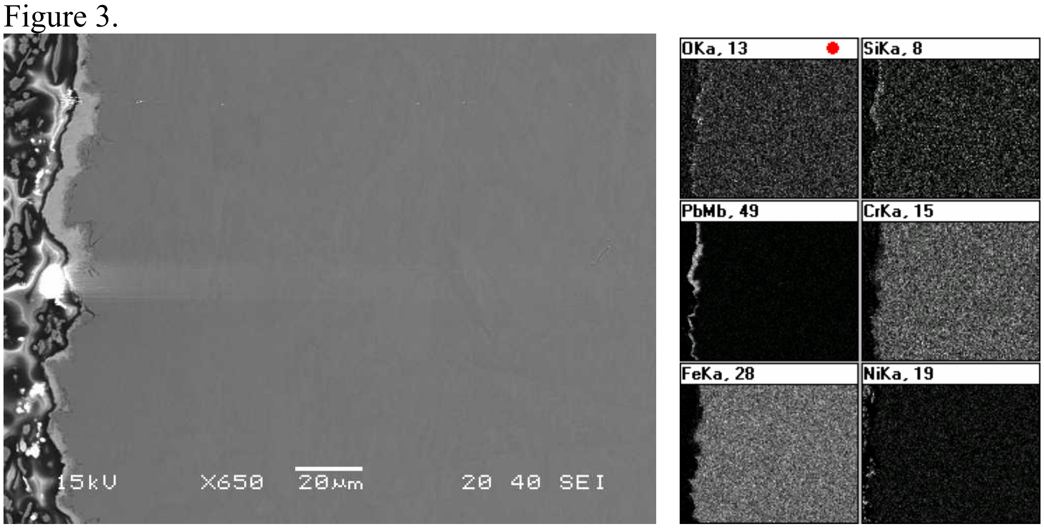

Figure 4.

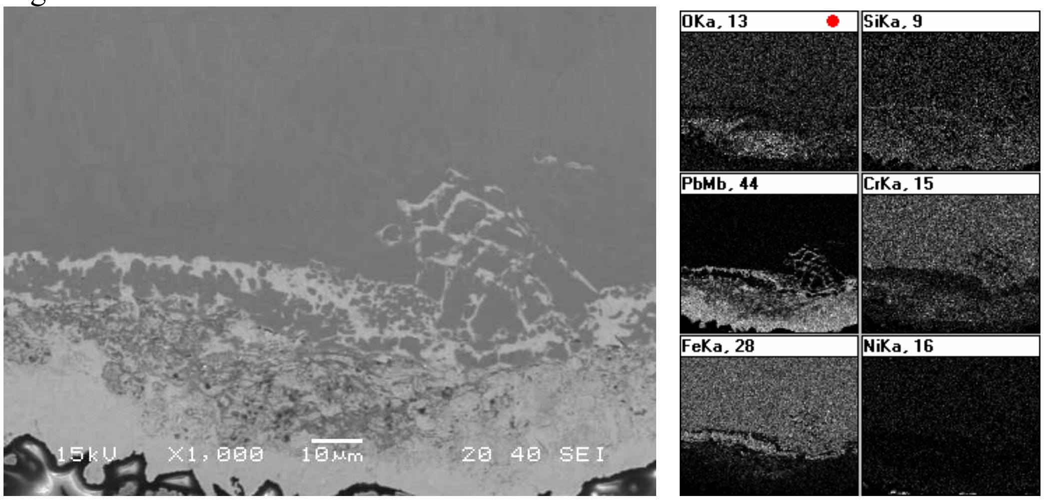

Figure 5.

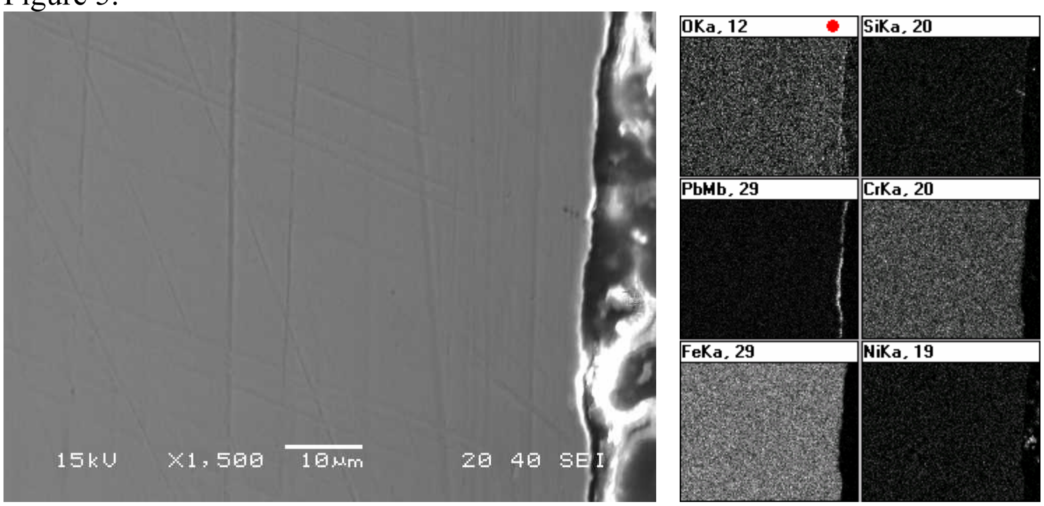

Figure 6.