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Fundamental and applied experimental investigations of corrosion of steel by LBE under controlled conditions: kinetics, chemistry, morphology, and surface preparation: quarterly report

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Fundamental and applied experimental investigations of corrosion of steel by LBE under controlled conditions: kinetics, chemistry, morphology, and surface preparation.

Quarterly Report: Project start - October 2004

Principal Investigator (PI):

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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 made progress in the preparation of a laboratory at UNLV for the collocation of multiple experiments utilizing molten lead alloys. We have pending a review of the final plan for the space renovation, and have started baseline environmental studies for lead activities at UNLV.

We are midway in the process of generating the engineering drawing for the test facility, and have started purchasing the standard parts for the device.

(2) Comparative gas phase studies

We accepted delivery of an Oxygen Control System from KALLA, Germany; a nuclear laboratory in Germany with a large program in lead and LBE technology. Training of students occurred during the delivery and installation, and useful discussions were started with Drs Muller and Weisenburger of KALLA.

We have set up a tube furnace for gas phase studies, and are doing initial characterizations.

(3) Isotope labeling studies

Isotope labeling can be used to measure diffusion of materials under realistic conditions. We plan two methods for the introduction of isotope labels into the steels systems under study.

For oxygen, chemical methods using O¹⁸ enriched reactants are possible. We have purchased O¹⁸ enriched water and plan to electropolish our steel samples to introduce a labeled oxide layer than then can be tracked using Secondary Ion Mass Spectroscopy (SIMS).

For iron and chromium we intend to use the ion beam/mass separator located in Prof. Farleys' laboratory. The apparatus has been brought back on line, and we expect test experiments (probably using the O¹⁸ water) soon.

Contact has been initiated with EMSL laboratory at DOE's Pacific Northwest Laboratory, where we expect to do the SIMS studies.

(4) Collaborative work

A major effort has been underway to aid efforts at LANL and INEL.

We have examined over 30 samples by SEM for LANL on a professional courtesy basis, and have another 19 samples which we have mounted for examination here at UNLV. Those samples include some motivated by our previous studies under the TRP program.

We are collaborating with Dr. Loewen of INEL in the investigation of the effects of silicon on the corrosion of steel by LBE. We have 4 samples of iron with varying amounts of silicon up to about 4% which have been exposed to oxygen controlled LBE in an isothermal test system at INEL.

We have found significant new results – the silicon is found in three different forms in this system. These results are clearest in our Xray Photoelectron Spectrometry (XPS) Sputter Depth Profiles (SDPs) (Appendix).

In the oxide layer at the surface of the iron the silicon is in the form of a silicate $-\operatorname{SiO_4-2}$ (binding energy 102eV in XPS studies). At the bottom of the oxide layer/start of the metal layer the silicon is in the form of silica $-\operatorname{SiO_2}$ (binding energy 103.5eV in XPS). In the metallic iron the silicon is in metallic form (binding energy of 99eV in XPS).

At the bottom of the oxide layer we found both silica and carbide inclusions in the iron grain boundaries. Iron and silica in the grain boundaries indicate significant mobility of silicon and oxygen under the test conditions. Further, we found both lead and lead oxide droplets at the bottom of the oxide layer, indicating failure of the oxide to protect the iron in localized areas. A paper covering this work is expected to be submitted by the end of the year.

Other program activities

Attendance at the AFCI semiannual meeting, September 04.

Publication of a manuscript in Journal of Nuclear Materials July 04

Meetings with LANL staff in September, August, May – sample studies, the formation of a theory/experiment collaboration, and issues with the planned test facilities discussed.

Appendix: Studies of Iron/Silicon alloys in LBE

Starting composition of samples

Metal	Concentration (wt%)												
	Fe	Si	P	S	Mo	Cu	Cr	Al	Ti	C	Mn	Ni	Fe
Fe	Bal	0.05	0.007	0.016	0.017	0.03	0.04	0.031	0.002	0.02	0.31	0.03	99.447
Fe-1.24% Si	Bal	1.24	0.006	0.001	0.01	0.03	0.09	0.005	0.003	0.01	0.04	0.08	
													98.485
Fe-2.55% Si	Bal	2.55	0.003	0.001	0.1	0.03	0.08	0.003	0.006	0.017	0.12	0.15	
													96.940
Fe-3.82% Si	Bal	3.82	0.022	0.025	-	-	-	-	-	0.011	0.24	-	
													95.882

Changes of Si conc. at the intersection of bulk and oxide layer

Sample	Si in bulk	Si at the surface of sample (after SDP)	Differences of Si in bulk of samples in sequence		
	(1)	(2)	(3)	(1)-(2)	(2)/(1)
Fe 44	0.05	0		0.05	0
Fe 1 44	1.24	0.03	1.19	1.214	0.021
Fe 2 44	2.55	0.09	1.31	2.457	0.0365
Fe 3 44/3 43	3.82	2.43	1.27	1.391	0.6359

XPS/SDP studies

Sample	%Fe	%Si		Binding Energy (eV)		
Fe 44	100	0	Cycle	Fe	Si	O
	tering: 5,664 s		2	710		530
~ thickness 3	.3984µm		13	710 , 707		530
70K 65K 60K	~~~~	Fe O	15	710, 707		530
50X 50X 40X		Si Pb	17	707		530
15 40K		TO TO				
20X 15X 10X						
58 0 1416 2022 4240	564 700 846 942 1320	12744 14100				
*Ovida lavar	thistraga (by	WDC): hard to				
determine	unckness (by	WDS): hard to				
determine						

Sample	%Fe	%Si		Binding Energy (eV)				
Fe 1 44	99.974	0.026	Cycle	Fe	Si	O		
	uttering: 14,16	0 s	2	710	102	530		
~ thickness 8	<mark>.496µm</mark>		15	710, 707	102	530		
90K 80K 80K 70K		Fe	27	707	102	530		
70K 60K 60K 50K		O Si	45	707	103, 99	530		
E 400 8 400 100		Pb						
20K 20K 20K								
10K 5K 0 -0,000 1416 2032 4248	5564 7000 8468 8612 11236	12344 19400						
	TME- SECONDS	WDS): around						
1.2µm	unckness (by	w DS). around						

Sample	%Fe	%Si		Binding En	ergy (eV)	
Fe 2 44	99.420	0.093	Cycle	Fe	Si	O
	uttering: 11,53	2 s	2	710	102	530
~ thickness 6	<mark>5.9192μm</mark>		5	710	102	531
			8	710, 707	103	531
1004 10		Fe O	12	710, 707	103	532, 531
75K 70K 65K		Si	16	707	103	532, 531
556 508 456 456		Pb	20	707	103	533,531
208 208 208 208			21	707	103	533,531
10W 50 0 -5,000			26	707	103	533 , 531
1416 2822 4248	5664 7060 8466 9912 11321 TBME - SECONDS	12744 14160	30	707	103.5 , 99	533 , 531
	thickness (by	WDS): around	40	707	103, 99	533
1.2μm						

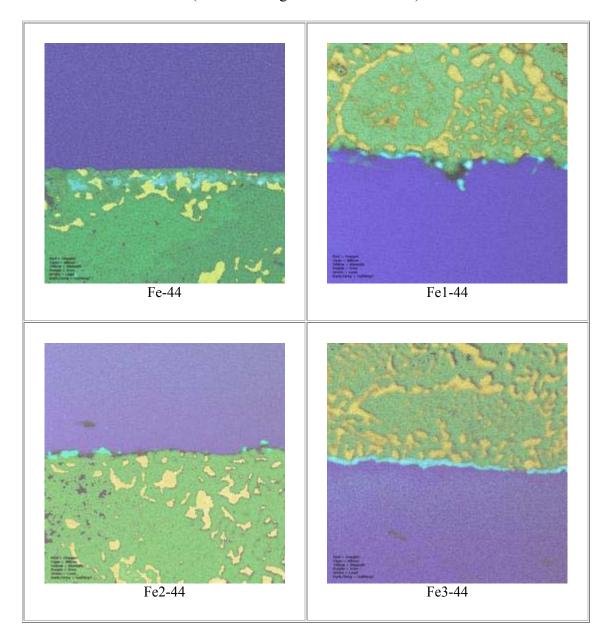
Sample	%Fe	%Si		Binding Energy (eV)			
Fe 3 44	97.571	2.429	Cycle	Fe	Si	O	
	ttering : 14,16	0 s	2	710	102	531	
~ thickness 8	<mark>3.496µm</mark>		8	710	103	531	
766 706 656		Fe	11	710, 707	103	531 , 533	
50K	O Si		16	710,707	103	533 , 531	
45K 40K 10K 10K 10K		Pb	19	707	103	533 , 531	
200 200 100			26	707	103 , 99	533 , 531	
10k 5k			45	707	99	533 , 531	
-5,000 416 2822 4268	5664 7080 8466 9612 11226 TMS-SECONDS	12744 14100					
*Oxide layer	thickness (by	WDS): 1.5μm					

Sample	%Fe %Si			Binding Energy (eV)			
Fe 3 43	97.298	2.702	Cycle	Fe	Si	O	
*Time of sput	tering: 14,160	S	2	710	102	530.5	
~ thickness	<mark>8.496µm</mark>		8	710,707	103 , 99	531	
70K- 65K-		Fe	13	710, 707	103 , 99	531 , 533	
55K 55K		O Si	18	710, 707	103 , 99	531 , 533	
E 328		Pb	24	707	103 , 99	531,533	
2004 2004 1006			33	707	103, 99	531,533	
SK-			52	707	103, 99	531, 533	
9416 2832 4348	5664 7080 8496 9912 11228 TBSC-55CCMDS	12744 14160					
*Oxide layer	thickness (by	WDS): <mark>1.5μm</mark>					

WDS Maps of Fe-Si samples

Legend: Oxygen Silicon Bismuth Iron Lead Dark/Grey = (no signal)

All maps are 68 μm per side. (Click on images for full-size view)



Notes:

- -Fe 44: Oxide layer has been dissolved into LBE. (???)
- -Fe 144 and Fe 244: The oxide layer is at the surface of the bulk as a barrier to stop LBE intrusion into the bulk. However, we can see that the Si is not enough to make a full barrier. Hence, in this case LBE still can attack the bulk to some extent.
- -Fe 344: Si now is concentrated enough to make a nearly full barrier to protect the bulk from corrosion by LBE.
- There are other interesting features of Fe244 and Fe344 WDS spectrum: Underneath of oxide layer we can clearly see the Si-depleted zone. Especially, with Fe 344, underneath of Si-depleted zone we have Si-enriched zone and Si-depleted zone consequently.