Appendix C1

SEM/EDS Data for Unused and Test #2, Day-30 Aluminum Coupons

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This appendix shows SEM/EDS results for the metal aluminum coupons in three different exposure catagories: (1) unused, (2) suspended or unsubmerged, and (3) submerged. Unused refers to the coupon condition that existed before being subjected to ICET test conditions. Suspended or unsubmerged refers to coupons located above the water level of the solution during ICET tests. Suspended coupons contacted the solution only during the 4-hour spray period at the start of the test. In addition, the surface of the suspended coupons may also be affected by moisture in the test chamber gas space. Submerged refers to the coupons that were immersed in the test solution for the entire test.

The coupon samples were collected on March 7, 2005 (the date Test #2 was shut down), and were examined by SEM/EDS on March 21, 2005. The aluminum coupon samples were dried in air before being coating with carbon for SEM examination. SEM results illustrate the surface condition of the aluminum coupons. In addition, EDS results provide spectra and semiquantitative elemental analyses of the coupon surface and the corrosion and deposition products that were found to be present.

Laboratory session from March 21, 2005

Aluminum Sample



Conditions: 15-kV, 1-nA beam current, Aperture=2

Metal	Sample	Test
Aluminum:	Al-095	Unused Al-095
	AI-098	Submerged Al-098
	Al-121	Unsubmerged Al-121

Note: All samples were carbon coated

Sample Al-095 (Unused)

Image:	A1095001	$40 \times$	Overview SEI	Figure C1-1
	A1095002	150 ×	SEI surface	Figure C1-2
	A1095003	150 ×	BEI surface	Figure C1-3
	A1095004	$1000 \times$	SEI on contam. "flowers" in image 003	Figure C1-4
EDS:	AIEDSI		Contaminant "flower" on Al	Figure C1-5
Image:	A1095005	$1000 \times$	SEI metal surface	Figure C1-6
	A1095006	1000 ×	BEI metal surface w/ numerous inclusions	Figure C1-7
EDS:	AIEDS2		Bright inclusion in Al	Figure C1-8
	AIEDS3		Average composition at 1000 ×	Figure C1-9

Sample Al-098 Test #2 (Submerged)

Image:	A1098007	40 ×	Overview on corroded surface	Figure C1-10
	A1098008	150 ×	On corrosion product (SEI)	Figure C1-11
	A1098009	150 ×	BEI on same area	Figure C1-12
· EDS:	AlEDS4		Smooth corrosion product surface	Figure C1-13
Image:	Al098010	$1000 \times$	SEI on surface & crystals	Figure C1-14
	Al098011	$1000 \times$	BEI same area	Figure C1-15
EDS:	AIEDS5		Crystals on Al corrosion product	Figure C1-16
Image:	A1098012	150 ×	SEI corroded Al surface (hole in corrosion layer)	Figure C1-17
	A1098013	1000 ×	SEI close-up of corroded surface in hole	Figure C1-18
EDS:	AIEDS6		Wormlike feature on corroded Al in image 013	Figure C1-19

Sample Al-121 Test #2 (Unsubmerged)

Image:	Al121014	$40 \times$	Overview of surface SEI	Figure C1-20
	Al121015	40 ×	BSE same area	Figure C1-21
	Al121016	150 ×	SEI on circle in image 015	Figure C1-22
	All21016annotated		Annotated Photoshop picture of EDS sample locations.	Figure C1-23
	Al121017	150 ×	BEI same area	Figure C1-24
EDS:	AIEDS7		Dark splotch (in image 017)	Figure C1-25
	AIEDS8		Wormlike crystals (see All21016annotated below)	Figure C1-26
	AIEDS9		Surface away from crystals (see Al121016annotated below)	Figure C1-27
Image:	A1121018	1000 ×	SEI wormlike crystals (see All21016annotated below)	Figure C1-28

C1-4



Figure C1-1. SEM image at 40× magnification for an unused aluminum coupon. (Al095001)



Figure C1-2. SEM image at 150× magnification for an unused aluminum coupon. (Al095002)



Figure C1-3. Backscattered SEM image at 150× magnification for an unused aluminum coupon. (Al095003)



Figure C1-4. SEM image close-up (1000×) of contaminant "flower" on an unused aluminum coupon, as shown in Figure C1-3. (Al095004)



Figure C1-5. EDS counting spectrum for the contaminant "flower" shown in Figure C1-4. (AIEDS1)



Figure C1-6. SEM image at 1000× magnification for an unused aluminum coupon. (Al095005)



Figure C1-7. Backscattered SEM image at 1000× magnification for an unused aluminum coupon. (Al095006)



Figure C1-8. EDS counting spectrum for the white spots shown in Figure C1-7. (AlEDS2)



Figure C1-9. EDS counting spectrum for the average composition of an aluminum coupon shown in Figure C1-7. (AIEDS3)



Figure C1-10. SEM image at 40× magnification for a Test #2, Day-30 submerged aluminum coupon. (Al098007)



Figure C1-11. SEM image at 150× magnification for a Test #2, Day-30 submerged aluminum coupon. (Al098008)



Figure C1-12. Backscattered SEM image at 150× magnification for a Test #2, Day-30 submerged aluminum coupon. (Al098009)



Figure C1-13. EDS counting spectrum for the smooth corrosion product shown on the surface of an aluminum coupon in Figure C1-11. (AIEDS4)



Figure C1-14. SEM image at higher magnification (1000×) for a Test #2, Day-30 submerged aluminum coupon. (Al098010)



Figure C1-15. Backscattered SEM image of at higher magnification (1000×) for the same area of a submerged aluminum coupon shown in Figure C1-14. (Al098011)



Figure C1-16. EDS counting spectrum for the crystal corrosion product, as shown as white granules in Figure C1-15. (AIEDS5)



Figure C1-17. SEM image at 150× magnification for a Test #2, Day-30 submerged aluminum coupon showing a hole or defect in the corrosion layer. (Al098012)



Figure C1-18. SEM image close-up (1000×) of the surface within the corrosion gap shown in Figure C1-17 on a submerged aluminum coupon. (Al098013)



Figure C1-19. EDS counting spectrum for the wormlike corrosion product shown in Figure C1-18. (AIEDS6)



Figure C1-20. SEM image overview at 40× magnification for a Test #2, Day-30 unsubmerged aluminum coupon. (Al121014)



Figure C1-21. Backscattered SEM image overview at 40× magnification for a Test #2, Day-30 unsubmerged aluminum coupon. (Al121015)



Figure C1-22. SEM image at 150× magnification for the circular corrosion patches shown in Figure C1-21 for a Test #2, Day-30 unsubmerged aluminum coupon. (Al121016)



Figure C1-23. SEM image at 150× magnification for a Test #2, Day-30 unsubmerged aluminum coupon illustrating EDS sampling locations. (Al121016annotated)



Figure C1-24. Backscattered SEM image at 150× magnification for the circular corrosion patches shown in Figure C1-21 for a Test #2, Day-30 unsubmerged aluminum coupon. (Al121017)



Figure C1-25. EDS counting spectrum for the dark patch shown in Figure C1-24. (AIEWDS7)



Figure C1-26. EDS counting spectrum for the wormlike crystals shown in Figure C1-22. (AIEDS8)



Figure C1-27. EDS counting spectrum for the coupon surface away from wormlike crystals shown in Figure C1-22. (AIEDS9)



Figure C1-28. SEM image close-up (1000×) of wormlike crystals shown in Figure C1-22 for a Test #2, Day-30 unsubmerged aluminum coupon. (Al121018)

Appendix C2

SEM/EDS Data for Unused and Test #2, Day-30 Copper Coupons

Figures

Figure C2-1.	SEM image for an unused copper coupon. (C411052) C2-5
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Figure C2-3.	SEM image for an unused copper coupon (C411054) C2-6
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Figure C2-14.	EDS counting spectrum for the light patches shown in Figure C2-11.
	(CEDS32)
Figure C2-15.	EDS counting spectrum for the lathlike crystals shown in Figure C2-12.
	(CEDS33)

Tables

Table C2-1.	The Chemical Composition for CEDS31 (Figure C2-13)	C2-12
Table C2-2.	The Chemical Composition for CEDS32 (Figure C2-14)	C2-14

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This appendix shows SEM/EDS results for the metal copper coupons in three different categories: (1) unused, (2) suspended or unsubmerged, and (3) submerged. *Unused* refers to the coupon condition before being subjected to ICET test conditions. *Suspended* or *unsubmerged* refers to coupons located above the water level of the solution during ICET tests. Suspended coupons contacted the solution only during the 4-hour spray period at the start of the test. In addition, the surface of the suspended coupons may also be affected by moisture in the test chamber gas space. *Submerged* refers to the coupons that were immersed in the test solution for the entire test.

The coupon samples were collected on March 7, 2005 (the date Test #2 was shut down), and examined by SEM/EDS on April 4, 2005. The copper coupon samples were dried in air before being coated with carbon for SEM examination. SEM results present the surface condition of the copper coupon. In addition, EDS results provide a semiquantitative elemental analysis of the coupon surface and the corrosion products that are present.

C2-1

Laboratory session from April 4, 2005

Copper Samples



Conditions: 15-kV, 1-nA beam current, Aperture = 2

Metal	Sample	Test
Copper:	C411	Unused C411
	C152	Test #2 Unsubmerged C152
	C127	Test #2 Submerged C127
NT - A 11	1 <u>(</u> 1	

Note: All samples were carbon coated.

Sample C411 Copper Blank (Unused)

Image:	C411052	40 ×	SEI	Figure C2-1
	C411053	150 ×	SEI	Figure C2-2
	C411054	$1000 \times$	SEI	Figure C2-3

Sample C152 Test #2 Unsubmerged Copper

Image:	C152055	$40 \times$	SEI	Figure C2-4
	C152056	150 ×	SEI	Figure C2-5
	C152057	1000 ×	SEI	Figure C2-6
	C152058	1000 ×	BEI	Figure C2-7
EDS:	CEDS30		Dark spot on T2D30 unsubmerged Cu sample	Figure C2-8

Sample C127 Test #2 Submerged Copper

Image:	C127059	40 ×	SEI	Figure C2-9
	C127060	150 ×	SEI	Figure C2-10
	C127061	150 ×	BEI	Figure C2-11
	C127062	1000 ×	SEI	Figure C2-12
EDS:	CEDS31		Dark spot on T2D30 submerged Cu	Figure C2-13
	CEDS32		Light spot on T2D30 submerged Cu	Figure C2-14
	CEDS33		Lathlike crystals on T2D30 submerged Cu	Figure C2-15



Figure C2-1. SEM image for an unused copper coupon. (C411052)



Figure C2-2. SEM image for an unused copper coupon. (C411053)



Figure C2-3. SEM image for an unused copper coupon (C411054).



Figure C2-4. SEM image for a Test #2, Day-30 unsubmerged copper coupon. (C152055)



Figure C2-5. SEM image for a Test #2, Day-30 unsubmerged copper coupon. (C152056)



Figure C2-6. SEM image for a Test #2, Day-30 unsubmerged copper coupon. (C152057)





Figure C2-8. EDS counting spectrum for one of the dark spots shown in Figure C2-7. (CEDS30)



Figure C2-9. SEM image for a Test #2, Day-30 submerged copper coupon. (C127059)



Figure C2-10. SEM image for a Test #2, Day-30 submerged copper coupon. (C127060)



Figure C2-11. Backscattered SEM image for a Test #2, Day-30 submerged copper coupon. (C127061)



Figure C2-12. SEM image for a Test #2, Day-30 submerged copper coupon. (C127062)



Figure C2-13. EDS counting spectrum for the dark patches shown in Figure C2-11. (CEDS31)

The results from the chemical composition analysis for CEDS31 are given in Table C2-1.

Table C2-1. The Chemical Composition for CEDS31 (Figure C2-13)

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Group Sample Comment Condition	: NRC : Metals : Dark sp : Full Sc Live Ti Acc. Vc Stage P Acq. Da	ID# : 31 pot on T2D30 cale : 20KeV ime : 60.0 colt : 15.0 Point : X=42. ate : Mon P	submged Cu V(10eV/ch,2F 000 sec / KV / 970 Y=70.69 Apr 4 13:48	Kch) Aperture Probe Cur: 96 Z=11.0 3:30 2005	# : 1 rent : 5.5461 00	E-10 A
Element O K Na K Mg K Al K Si K P K Ca K	Mode Normal Normal Normal Normal Normal Normal	ROI (KeV) 0.25- 0.77 0.81- 1.27 0.97- 1.57 1.19- 1.83 1.50- 2.05 1.75- 2.38 3.39- 4.30	K-ratio(%) 38.8311 1.2974 4.9621 0.7808 0.9519 11.7319 3.3210 	+/- 0.0031 0.0009 0.0004 0.0003 0.0003 0.0033 0.0012	Net/Backgrou 5041 / 484 / 2689 / 421 / 478 / 3637 / 811 /	111d 52 136 34 79 116 37 5
Element Ma O 5 Na Mg 1 Al Si P 1 Ca Total 10	ASS% At 59.978 72 2.826 2 11.431 9 1.789 1 1.887 1 1.887 1 6.716 10 5.372 2	comic% ZAF 2.8049 0.9785 2.3873 1.3800 9.1315 1.4595 .2876 1.4515 .3046 1.2557 0.4809 0.9027 2.6032 1.0249	Z 5 0.9908 0.9 0 1.0457 1.3 5 0.9845 1.4 5 1.0179 1.4 7 0.9952 1.2 7 1.1946 0.7 9 1.0161 1.0	A 1 9877 1.000 9215 0.998 1842 0.998 1298 0.999 2690 0.999 7558 0.999 0086 1.000	7 00 36 38 74 43 97 00	
Normalizat	ion facto	r = 1.5785				



Figure C2-14. EDS counting spectrum for the light patches shown in Figure C2-11. (CEDS32)

The results from the chemical composition analysis for CEDS32 are given in Table C2-2.

Table C2-2. The Chemical Composition for CEDS32 (Figure C2-14)

Apr 4 14:01 2005 /tmp/eds_pout.log Page 1

Group : NRC Sample : Metals ID# : 32 Comment : Light spot on T2D30 submged Cu Condition : Full Scale : 20KeV(10eV/ch,2Kch) Live Time : 60.000 sec Aperture # : 1 Acc. Volt : 15.0 KV Probe Current : 5.518E-10 A Stage Point : X=42.970 Y=70.696 Z=11.000 Acq. Date : Mon Apr 4 13:58:47 2005
ElementModeROI(KeV)K-ratio(%)+/-Net/BackgroundO KNormal0.25-0.7715.40640.00231990 /92P KNormal1.75-2.382.07450.0020640 /26Ca KNormal3.39-4.301.19100.0011289 /14Cu KNormal7.63-9.2761.86550.00863043 /4
Chi_square = 7.3941 Element Mass% Atomic% ZAF Z A F O 16.042 42.1700 0.8549 0.8436 1.0136 0.9998 P 2.528 3.4327 1.0005 1.0112 0.9894 1.0000 Ca 1.293 1.3565 0.8912 0.8542 1.0459 0.9975 Cu 80.137 53.0408 1.0634 1.0641 0.9994 1.0000
Total 100.000 100.0000 Normalization factor = 1.2181


Figure C2-15. EDS counting spectrum for the lathlike crystals shown in Figure C2-12. (CEDS33)



Appendix C3

SEM/EDS Data for Unused and Test #2, Day-30 Galvanized Steel Coupons

Figures

Figure C3-1.	SEM image for an unused GS coupon. (G002031) C3-5
Figure C3-2.	SEM image for an unused GS coupon. (G002032) C3-5
Figure C3-3.	SEM image for an unused GS coupon. (G002033) C3-6
Figure C3-4.	Backscattered SEM image for an unused GS coupon. (G002034) C3-6
Figure C3-5.	EDS counting spectrum for a smooth zinc surface. (GEDS20)
Figure C3-6.	EDS counting spectrum for the small dark circular spots shown in Figure
	C3-4. (GEDS21) C3-7
Figure C3-7.	EDS counting spectrum for the bright spots shown in Figure C3-4. (GEDS22) C3-7
Figure C3-8.	SEM image for a Test #2, Day-30 submerged galvanized steel coupon.
	(G334048)
Figure C3-9.	SEM image for a Test #2, Day-30 submerged GS coupon. (G334049) C3-8
Figure C3-10.	Backscattered SEM image for a Test #2, Day-30 submerged GS coupon.
	(G334050)
Figure C3-11.	EDS counting spectrum on fiberlike crystals shown in Figure C3-10.
	(GEDS29)
Figure C3-12.	SEM image for a Test #2, Day-30 submerged GS coupon. (G334051) C3-10
Figure C3-13.	SEM image for a Test #2, Day-30 unsubmerged GS coupon. (G390044) C3-10
Figure C3-14.	SEM image for a Test #2, Day-30 unsubmerged GS coupon. (G390045) C3-11
Figure C3-15.	Backscattered SEM image for a Test #2, Day-30 unsubmerged GS coupon.
r: 03.14	(G390046)
Figure C3-16.	(G390046)
Figure C3-16.	(G390046)
Figure C3-16. Figure C3-17.	(G390046)
Figure C3-16.	(G390046)C3-11EDS counting spectrum on the dark spots shown in Figure C3-15.C3-12(GEDS27)C3-12EDS counting spectrum on the light surface shown in Figure C3-15.C3-14
Figure C3-16. Figure C3-17. Figure C3-18.	 (G390046)

Tables

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This appendix shows SEM/EDS results for the metal GS coupons in three different categories: (1) unused, (2) suspended or unsubmerged, and (3) submerged. *Unused* refers to the coupon condition before being subjected to ICET test conditions. *Suspended* or *unsubmerged* refers to coupons located above the water level of the solution during ICET tests. Suspended coupons contacted the solution only during the 4-hour spraying period at the start of the test. In addition, the surface of the suspended coupons may also be affected by moisture in the test chamber gas space. *Submerged* refers to the coupons that were immersed in the solution for the entire test.

The coupon samples were collected on March 7, 2005 (the date Test 2 was shut down), and examined by SEM/EDS on March 23, 2005. The GS coupon samples were dried in air at room temperature before being coating with carbon for SEM examination. SEM results present the surface condition of the GS coupon. In addition, EDS results provide a semiquantitative elemental analysis of the coupon surface and the corrosion products that are present.

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Transcribed Laboratory Log

Laboratory session from March 21, 2005

Galvanized Steel Samples



Conditions: 15-kV, 1-nA beam current, Aperture = 2

Metal	Sample	Test
Galvanized Steel:	G002	Unused G002
	G241	Test 1 Unsubmerged G241
	G332	Test 1 Submerged G332
	G334	Test 2 Submerged G334
	G390	Test 2 Unsubmerged G390

Note: All samples were carbon coated.

Results for Test 1 samples G241 and G332 are provided in the Test #1 summary data report.

Sample G002 Unused Galvanized Steel

Image:	G002031	40 ×	SEI overview	Figure C3-1
	G002032	150 ×	SEI different area	Figure C3-2
	G002033	1000 ×	SEI same area	Figure C3-3
	G002034	1000 ×	BEI same area	Figure C3-4
EDS:	GEDS20		Smooth zinc surface	Figure C3-5
	GESD21		Small circular spots in zinc (image 034)	Figure C3-6
	GEDS22		Bright spots in zinc	Figure C3-7

Laboratory session from April 4, 2005

Galvanized Steel Samples (Continued)



Conditions: 15-kV, 1-nA beam current, Aperture = 2

Sample G334 Test #2 Submerged Galvanized Steel

Image:	G334048	40 ×	On corroded surface	Figure C3-8
	G334049	150 ×	SEI on corroded surface	Figure C3-9
	G334050	150 ×	BEI on corroded surface	Figure C3-10
EDS:	GEDS29		EDS on fiberlike crystals on T2D30 submerged G-Steel	Figure C3-11
Image:	G334051	$1000 \times$		Figure C3-12

Sample G390 Test #2 Unsubmerged Galvanized Steel

Image:	G390044	$40 \times$	overview of surface	Figure C3-13
	G390045	150 ×	SEI same as above	Figure C3-14
	G390046	150 ×	BEI same as above	Figure C3-15
EDS:	GEDS27		EDS on dark spot T2D30 unsubmerged G-steel	Figure C3-16
	GEDS28		EDS on light surface of T2D30 unsubmerged G- steel	Figure C3-17
Image:	G390047	$1000 \times$	SEI on corroded surface	Figure C3-18



Figure C3-1. SEM image for an unused GS coupon. (G002031)



Figure C3-2. SEM image for an unused GS coupon. (G002032)



Figure C3-3. SEM image for an unused GS coupon. (G002033)



Figure C3-4. Backscattered SEM image for an unused GS coupon. (G002034)



Figure C3-5. EDS counting spectrum for a smooth zinc surface. (GEDS20)



Figure C3-6. EDS counting spectrum for the small dark circular spots shown in Figure C3-4. (GEDS21)



Figure C3-7. EDS counting spectrum for the bright spots shown in Figure C3-4. (GEDS22)



Figure C3-8. SEM image for a Test #2, Day-30 submerged galvanized steel coupon. (G334048)



Figure C3-9. SEM image for a Test #2, Day-30 submerged GS coupon. (G334049)



Figure C3-10. Backscattered SEM image for a Test #2, Day-30 submerged GS coupon. (G334050)



Figure C3-11. EDS counting spectrum on fiberlike crystals shown in Figure C3-10. (GEDS29)



Figure C3-12. SEM image for a Test #2, Day-30 submerged GS coupon. (G334051)



Figure C3-13. SEM image for a Test #2, Day-30 unsubmerged GS coupon. (G390044)



Figure C3-14. SEM image for a Test #2, Day-30 unsubmerged GS coupon. (G390045)



Figure C3-15. Backscattered SEM image for a Test #2, Day-30 unsubmerged GS coupon. (G390046)



Figure C3-16. EDS counting spectrum on the dark spots shown in Figure C3-15. (GEDS27)

The results from the chemical composition analysis for GEDS27 are given in Table C3-1.

Table C3-1. The Chemical Compositions for GEDS27

Apr 4 11:16 2005 /tmp/eds_pout.log Page 1

	Group Sample Comment Conditic	: NRC : Meta : Dark on : Full Live Acc. Stag Acq.	ls ID# spot on 7 Scale Time Volt e Point Date	: 27 [2D30 ur : 20KeV : 60.00 : 15.0 F : X=70.8 : Mor Ap	nsubmged (10eV/ch 00 sec KV 816 Y=58 pr 4 11	d bsce (1,2Kch) Apert Probe 3.930 Z= .:09:30	G-Steel cure # e Curren =11.000 2005	: 1 t : 8	.078E-1	0 A
	Element O K Na K Si K P K Zn K	Mode Norma Norma Norma Norma	ROI 1 0.25- 1 0.81- 1 1.50- 1 1.75- 1 8.22-	(KeV) - 0.77 - 1.27 - 2.05 - 2.38 -10.03	K-ratic 21.252 0.000 0.688 5.942 45.860	b) (%) +/ 28 0.0 30 0.0 30 0.0 16 0.0 00 0.0	/- Ne 0030 0000 0003 0032 0111	t/Bac 4018 0 503 2683 2504	kground / / / /	68 50 96 57 3
1				Ch:	i_square	e = 49.4	1797			
	Element O Na Si P Zn	Mass% 25.047 0.000 1.403 7.881 65.668	Atomic% 54.4632 0.0000 1.7380 8.8519 34.9468	ZAF 0.9144 2.3046 1.5824 1.0292 1.1110	Z 0.8736 0.9205 0.8740 1.0482 1.1123	A 1.0468 2.5030 1.8129 0.9818 0.9988	F 0.9999 1.0002 0.9987 1.0000 1.0000			
9	Total Normaliz	100.000 zation fa	100.0000 actor = 3	1.2889						



Figure C3-17. EDS counting spectrum on the light surface shown in Figure C3-15. (GEDS28)



Figure C3-18. SEM image at 1000× magnification for a Test #2, Day-30 unsubmerged GS coupon. (G390047)

Appendix C4

SEM/EDS Data for Unused and Test #2, Day-30 Steel Coupons

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	(\$007029)
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This appendix shows SEM/EDS results for the metal steel coupons in three different categories: (1) unused, (2) suspended or unsubmerged, and (3) submerged. Unused refers to the coupon condition before being subjected to ICET test conditions. Suspended or unsubmerged refers to coupons located above the water level of the solution during ICET tests. Suspended coupons contacted the solution only during the 4-hour spray period at the start of the test. In addition, the surface of the suspended coupons may also be affected by moisture in the test chamber gas space. Submerged refers to the coupons that were immersed in the test solution for the entire test.

The coupon samples were collected on March 7, 2005 (the date Test #2 was shut down), and examined by SEM/EDS on March 23, 2005. (The unused steel coupon was examined on March 21, 2005.) The steel coupon samples were dried in air at room temperature before being coating with carbon for SEM examination. SEM results present the surface condition of the steel coupon. In addition, EDS results provide a semiquantitative elemental analysis of the coupon surface and the corrosion products that are present.

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Laboratory session from March 21, 2005

Steel Samples



Conditions: 15-kV, 1-nA beam current, Aperture = 2

Metal	Sample	Test
Steel:	S014	Unused S014
	S007	Test #2 Submerged S007
	S009	Test #2 Unsubmerged S009

Note: All samples were carbon coated.

Sample S-014 Unused Steel

Image:	S014019	$40 \times$	SEI overview of surface	Figure C4-1
	S014020	150 ×	SEI surface	Figure C4-2
	S014021	150 ×	BEI same as above	Figure C4-3
	S014022	1000 ×	SEI close-up	Figure C4-4
EDS:	SEDS10		Steel surface	Figure C4-5

Sample S007 Test #2 Submerged Steel

Image:	S007028	150 ×	SEI corroded surface	Figure C4-6
	S007028annotated	150 ×	EDS spots 17-19	Figure C4-7
	S007029	150 ×	BEI same place	Figure C4-8
EDS:	SEDS17		Light area in image 029	Figure C4-9
Image:	S007030	$1000 \times$	SEI on corrosion surface	Figure C4-10
EDS:	SEDS18		Crystals on corroded steel (see image 030)	Figure C4-11
	SEDS19		Clump of corrosion product	Figure C4-12

Sample S-009 Unsubmerged Steel

Image:	S009023	40 ×	SEI overview of corroded & noncorroded surface	Figure C4-13
	S009024	40 ×	BEI same area	Figure C4-14
	S009024annotated	40 ×	EDS spots 11-15	Figure C4-15
EDS:	SEDS11		Steel surface- light (in image 024)	Figure C4-16
	SEDS12		Steel—light gray in BSE	Figure C4-17
	SEDS13		Medium gray corrosion on steel	Figure C4-18
	SEDS14		Dark gray corrosion	Figure C4-19
	SEDS15		Dark gray corrosion	Figure C4-20
Image:	S009025	150 ×	SE at interface between badly corroded & less corroded surface	Figure C4-21
	S009026	150 ×	BSE same area	Figure C4-22
	S009027	1000 ×	SE corroded area	Figure C4-23
EDS:	SEDS16		Clump of rounded corrosion crystals	Figure C4-24



Figure C4-1. SEM image for an unused steel coupon. (S014019)



Figure C4-2. SEM image for an unused steel coupon. (S014020)



Figure C4-3. Backscattered SEM image for an unused steel coupon. (S014021)



Figure C4-4. SEM image for an unused steel coupon. (S014022)



Figure C4-5. EDS counting spectrum for the steel surface shown in Figure C4-4. (SEDS10)



Figure C4-6. SEM image for a Test #2, Day-30 submerged steel coupon. (S007028)



Figure C4-7. SEM image for the corrosion surface of a Test #2, Day-30 submerged steel coupon. (S007028annotated)



Figure C4-8. Backscattered SEM image for a Test #2, Day-30 submerged steel coupon. (S007029)



Figure C4-9. EDS counting spectrum for the light spot shown in Figure C4-8. (SEDS17)



Figure C4-10. SEM image for the corrosion surface of a Test #2, Day-30 submerged steel coupon. (S007030)



Figure C4-11. EDS counting spectrum for the thin crystals shown in Figure C4-10. (SEDS18)



Figure C4-12. EDS counting spectrum for the clumps surrounding crystals shown in Figure C4-10. (SEDS19)



Figure C4-13. SEM image for a Test #2, Day-30 unsubmerged steel coupon. (S009023)



Figure C4-14. Backscattered SEM image for a Test #2, Day-30 unsubmerged steel coupon. (S009024)



Figure C4-15. Annotated backscattered SEM image for a Test #2, Day-30 unsubmerged steel coupon. (S009024annotated)



Figure C4-16. EDS counting spectrum for the light streak (EDS-11) shown in Figure C4-15. (SEDS11)



Figure C4-17. EDS counting spectrum for the light grey streak (EDS-12) shown in Figure C4-15. (SEDS12)



Figure C4-18. EDS counting spectrum for the medium grey spot (EDS-13) shown in Figure C4-15. (SEDS13)



Figure C4-19. EDS counting spectrum for the dark grey spot (EDS-14) shown in Figure C4-15. (SEDS14)



Figure C4-20. EDS counting spectrum for the dark spot (EDS-15) shown in Figure C4-15. (SEDS15)



Figure C4-21. SEM image for a Test #2, Day-30 unsubmerged steel coupon (S009025). The image shows the interface between badly corroded (right) and less corroded surface (left).



Figure C4-22. Backscattered SEM image for a Test #2, Day-30 unsubmerged steel coupon (S009026). It shows the same area as shown in Figure C4-21.



Figure C4-23. SEM image for a corroded area of a Test #2, Day-30 unsubmerged steel coupon. (S009027)



Figure C4-24. EDS counting spectrum for a clump of round corrosion crystals shown in Figure C4-23. (SEDS16)
Appendix D1

ESEM and SEM/EDS Data for Test #2, Day-16 Fiberglass in High- and Low-Flow Zones

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The debris accumulated on fiberglass in the ICET tests is of great interest because it may contribute to additional head loss during recirculation of the coolant following a LOCA. To evaluate these potential debris accumulations, fiberglass samples submerged in high- and low-flow zones in the tank were examined by ESEM and SEM/EDS.

In this appendix, images and data are presented for fiberglass samples that were extracted on February 21, 2005 (Test #2, Day 16). Both exterior and interior locations on the fiberglass samples were examined for material that had been placed in both high- and low-flow locations. Microprobe SEM was used to examine the fiberglass samples after they were air dried at room temperature and then coated with gold/palladium. Low-vacuum ESEM examinations were performed on hydrated material without any required coating to avoid sample modifications that might occur during the drying process. Microprobe SEM/EDS and ESEM analyses of Test #2, Day-16 fiberglass samples were obtained on February 25 and March 4, 2005, respectively. Accompanying EDS results provide a semiquantitative elemental analysis of the debris deposited on the fiberglass under both low- and high-flow conditions.

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Transcribed Laboratory Log

ESEM laboratory session from March 4, 2005



Instrument Conditions: 20-kV, 12-mm Working Distance, Low-Vacuum Mode (80 Pa)

T2 High-Flow Exterior

Image:	T2HIX1	90 ×	BSE Image Overview	Figure D1-1
	T2HIX2	$350 \times$	On center of image T2HIX1 (above)	Figure D1-2
	T2HIX3	$1000 \times$		Figure D1-3
	T2HIX4	350 ×	Another sample area	Figure D1-4

T2 High-Flow Interior

Image:	T2HII5	90 ×	Overview	Figure D1-5
	T2HII6	$1000 \times$	Same area as image T2HII5 (above)	Figure D1-6
	T2HII7	550 ×	Another area	Figure D1-7

T2 Low-Flow Exterior

Image:	T2LOX8	90 ×	Overview	Figure D1-8
	T2LOX9	500 ×	Upper left of image T2LOX8 (above)	Figure D1-9

T2 Low-Flow Interior

Image:	T2LO110	90 ×	Overview	Figure D1-10
	T2LOI11	500 ×	On center of image T2LOI10 (above)	Figure D1-11

Transcribed Laboratory Log

Microprobe laboratory session from February 25, 2005

T2D16 Samples plus high-volume filter from T1D30



Conditions: 15-kV, 1 nA

High-Flow Exterior Crust

Image:	HI_flow_ex_crust_001	$40 \times$	Overview (Charging)	Figure D1-12
	HI_flow_ex_crust_002	$40 \times$	Overview the same as 001	Figure D1-13
	HI_flow_ex_crust_003	60 ×	Zoom in on crust	Figure D1-14
	Hl_flow_ex_crust_004	$150 \times$	Zoom in on crust	Figure D1-15
EDS:	Hiflow_excrust_EDS1		Area of picture 004—deposit	Figure D1-16
Image:	HI_flow_ex_crust_005	$270 \times$	Zoom in on different region	Figure D1-17
EDS:	Hiflow_excrust_EDS2		Area of picture 005—deposit	Figure D1-18
Image:	HI_flow_ex_crust_006	$40 \times$	Overview of another area	Figure D1-19

Laboratory Session March 6, 2005

High-Flow Interior (T2D16)—(after sputtering again to get better pictures)

Image:	Hiflow-interior001	40 ×	SE overview image	Figure D1-20
	Hiflow-interior002	80 ×	SE image	Figure D1-21
	Hiflow-interior003	65 ×	SE image-pretty clean fibers	Figure D1-22
	Hiflow-interior004	300 ×	SE image-deposits	Figure D1-23
	Hiflow-interior005	5000 ×	SE image—zoom in on deposits on fiberglass	Figure D1-24

Low-Flow Interior (T2D16)

		-		
Image:	Lowflow-interior001	55 ×	SE overview image	Figure D1-25
	Lowflow-interior002	120 ×	Close-up of Lowflow- interior001	Figure D1-26
	Lowflow-interior003	3500 ×	SE image zoom in on deposits	Figure D1-27
	Lowflow-interior004	370 ×	SE image zoom in on deposits	Figure D1-28
	Lowflow-interior005	85 ×	SE overview image	Figure D1-29
	Lowflow-interior006	370 ×	Zoom in	Figure D1-30
	Lowflow-interior007	100 ×	SE image	Figure D1-31

General Observations: Low-flow interior looks cleaner than high-flow interior. No crusts, only small particles. Images Lowflow-interior001 through 004 taken on the same region

Low-Flow Exterior (T2D16)

Image:	Lowflow-exterior001	43 ×	SE overview image	Figure D1-32
	Lowflow-exterior002	120 ×	Close-up (charging)	Figure D1-33
	Lowflow-exterior003	$600 \times$	Zooming on deposits	Figure D1-34
	Lowflow-exteriorEDS1		Particle on fiberglass (pic 003)	Figure D1-35
	Lowflow-exteriorEDS2		Particle on fiberglass (pic 003)	Figure D1-36

General Observations: Although sputtered again, some problems with charging remain.



Figure D1-1. Back-scattered electron (BSE) image overview. (T2HIX1)



Figure D1-2. BSE image on center of Figure D1-1. (T2HIX2)



Figure D1-3. BSE image at 1000 times magnification. (T2HIX3)



Figure D1-4. BSE image at another angle on the center of the image. (T2HIX4)



Figure D1-5. SEM image overview for interior of high flow. (T2HII5)



Figure D1-6. SEM image of Figure D1-5 at 1000 times magnification. (T2HII6)



Figure D1-7. SEM image of high-flow interior for another sample area. (T2HII7)



Figure D1-8. SEM image overview for exterior of the low-flow sample. (T2LOX8)



Figure D1-9. SEM image of the upper left portion of Figure D1-8, low-flow exterior. (T2LOX9)



Figure D1-10. SEM image overview for interior of the low-flow sample. (T2LOI10)



Figure D1-11. SEM image on the center of Figure D1-10, low-flow interior. (T2LO111)



Figure D1-12. SEM image overview of exterior crust. (HI_flow_ex_crust001)



Figure D1-13. SEM image overview of the same area of Figure D1-12. (HI_flow_ex_crust002)



Figure D1-14. SEM image close-up view of crust. (HI_flow_ex_crust003)



Figure D1-15. SEM image of crust at 150 times magnification. (HI_flow_ex_crust004)



Figure D1-16. Counting spectrum image of the deposit area in Figure D1-15. (hiflow~excrust~EDS1)

The results from the chemical composition analysis for hiflow~excrust~EDS1 are given Table D1-1.

Table D1-1. The Chemical Composition for hiflow~excrust~EDS1 (Figure D1-16)

Feb 25 11:07 2005 /tmp/eds_pout.log Page 1

Group Sample Comment Condition	: NRC : Hiflow_ : deposit : Full Sc Live Ti Acc. Vo Stage P Acq. Da	excrust ID# picture4 cale : 20KeV me : 60.0 plt : 15.0 point : X=55. te : Fri F	: 1 7(10eV/ch, 000 sec KV 412 Y=74. Seb 25 10:	2Kch) Aperture : Probe Cur: 945 Z=11.1 59:03 2005	∉ : 2 rent : 1. 17	.002E-0	18 A
Element	Mode	ROI (KeV)	K-ratio(웅) +/-	Net/Bac}	ground	l
ОК	Normal	0.25- 0.77	34.5490	0.0043	10752	1	46
Al K	Normal	1.26- 1.78	0.0857	0.0006	126	/	114
Si K	Normal	1.50- 2.07	1.0405	0.0012	1534	1	150
Ca K	Normal	3.40- 4.30	0.4471	0.0054	349	1	27
Na K	Normal	0.83- 1.28	3.7139	0.0097	3635	/	40
РK	Normal	1.75- 2.38	5.2367	0.0043	3892	1	132
Cl K	Normal	2.34- 3.06	0.9080	0.0010	749	/	48
		Cł	ni_square	= 15.4770			
Element M	Mass% At	omic% ZAF	Z	A	F		
0	69.992 80	.1268 0.7836	5 0.9917 0	.7902 1.00	00		
Al	0.265 0	.1798 1.1962	2 1.0046 1	.1939 0.99	74		
Si	2.944 1	.9201 1.0946	5 0.9924 1	.1082 0.99	53		
Ca	1.156 0	.5280 0.9997	7 1.0065 0	.9932 1.00	01		
Na	11.074 8	.8224 1.1534	1 0.9963 1	.1562 1.00	13		
P	11.995 7	.0932 0.8861	1.1964 0	.7411 0.99	93		
Cl	2.574 1	3297 1.0964	1.0507 1	.0440 0.99	96		
Total 1 Normaliza	100.000 100 ation facto	0.0000 or = 2.5852					

D1-14



Figure D1-17. SEM image of a new exterior crust area at 270 times magnification. (HI_flow_ex_ crust005)





The results from the chemical composition analysis for hiflow~excrust~EDS2 are given in Table D1-2.

Table D1-2. The Chemical Composition for hiflow~excrust~EDS2 (Figure D1-18)

Feb 25 11:18 2005 /tmp/eds_pout.log Page 1

Group Sample Comment Condition	: NRC : Hiflow_ : deposit : Full Sc Live Ti Acc. Vo Stage P Acq. Da	excrust ID# picture5 wale : 20KeV me : 60.0 lt : 15.0 voint : X=55. te : Fri F	: 2 (10eV/ch,2K 00 sec A KV P 001 Y=74.84 eb 25 11:13	ch) perture # robe Currer 5 Z=11.147 :54 2005	: 2 it : 1.001E-	08 A
Element O K	Mode Normal	ROI(KeV) 0.25- 0.77	K-ratio(%) 13.1976	+/- Ne 0.0028	t/Backgroun 4103 /	id 40
Al K	Normal	1.26- 1.78	0.1087	0.0005	160 /	122
Si K	Normal	1.50- 2.07	1.6773	0.0013	2470 /	108
Ca K	Normal	3.40-4.30	0.4860	0.0055	3/9 /	20
NAK	Normal	0.83-1.28	2.1936	0.0079	2145 /	162
Cl K	Normal	2.34-3.06	1.2020	0.0010	990 /	40
Chi_square = 13.2170						
Element Ma	ass% At	omic% ZAF	Z	A F		
0 6	53.140 74	.8335 0.8873	0.9904 0.89	959 1.0000		
Al	0.704 0	.4946 1.2005	1.0030 1.20	08 0.9967		
Si	9.996 6	.7486 1.1053	0.9908 1.1:	175 0.9982		
Ca	2.633 1	.2457 1.0047	1.0045 1.00	001 1.0001		
Na 1	.3.248 10	.9272 1.1201	0.9948 1.12	246 1.0012		
P	3.262 1	.9970 0.9380	1.1944 0.78	368 0.9981		
CL	7.017 3	.7533 1.0828	1.0488 1.03	332 0.9992		
Total 10 Normalizat	0.000 100 ion facto:	.0000 r = 5.3920	1999 - 1999 -		ten e o distribuir e e e e e e e e e	



Figure D1-19. SEM image overview of another area for the exterior crust. (HI_flow_ex_crust006)



Figure D1-20. Test #2, Day-16 SEM image overview. (Hiflow-interior001)



Figure D1-21. Test #2, Day-16 SEM image for high-flow interior. (Hiflow-interior002)



Figure D1-22. Test #2, Day-16 SEM image showing clean fibers. (Hiflow-interior003)



Figure D1-23. Test #2, Day-16 SEM image of deposits for high-flow interior. (Hiflow-interior004)



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Figure D1-24. Test #2, Day-4 SEM image zoomed in on deposits on fiberglass, magnification 5000 times. (Hiflow-interior005)



Figure D1-25. Test #2, Day-16 SEM image overview for low-flow interior. (Lowflow-interior001)



Figure D1-26. Test-2 Day-16 SEM close-up image of Figure D1-25. (Lowflow-interior002)



Figure D1-27. Test #2, Day-16 SEM image at 3500 times magnification of deposits shown in Figure D1-26. (Lowflow-interior003)



Figure D1-28. Test #2, Day-16 SEM image at 370 times magnification showing close-up of deposits in Figure D1-26. (Lowflow-interior004)



Figure D1-29. Test #2, Day-16 SEM image overview of a new sample area. (Lowflow-interior005)



Figure D1-30. Test #2, Day-16 SEM image zooming in on Figure D1-29. (Lowflow-interior006)



Figure D1-31. Test #2, Day-16 SEM image showing a cleaner appearance than seen in some high-flow interior samples. (Lowflow-interior007)



Figure D1-32. Test #2, Day-16 SEM image overview for low-flow exterior. (Lowflow-exterior001)



Figure D1-33. Test #2, Day-16 SEM image close-up (charging) of Figure D1-32. (Lowflow-exterior002)



Figure D1-34. Test #2, Day-16 SEM image zooming in on deposits at 600 times magnification. (Lowflow-exterior003)



Figure D1-35. Test #2, Day-16 counting spectrum image for a particle on the fiberglass shown in Figure D1-34. (Lowflow~exterior~EDS1)

The results from the chemical composition analysis for Lowflow~exterior~EDS1 are given in Table D1-3.

Table D1-3. The Chemical Composition for EDS1

Mar 6 21:48 2005 /tmp/eds_pout.log Page 1

Group Sample Comment Condition	: NRC : T2D16 L : Exterio : Full Sc Live Ti Acc. Vo Stage P Acq. Da	ow-flow ID r particle o ale : 20KeV me : 60.0 lt : 15.0 oint : X=52. te : Sun M	t : 1 200 fibergl 7(10eV/ch, 000 sec KV 122 Y=69. Mar 6 21:	ass 2Kch) Apertun Probe (909 Z=12 42:04 2(re # : Current : 2.467 005	3 2.003E	-08 A
Element O K Na K Mg K Al K Si K Ca K	Mode Normal Normal Normal Normal Normal	ROI(KeV) 0.25- 0.77 0.83- 1.28 1.03- 1.52 1.26- 1.78 1.50- 2.07 3.40- 4.30	K-ratio(21.1989 13.6362 0.4152 0.6657 11.9355 3.2837	(%) +/- 0.002 0.008 0.006 0.006 0.006 0.006 0.006 0.006	Net/Ba 17 149 32 300 07 10 04 22 14 390 42 50	ackgrou 91 / 16 / 16 / 22 / 76 / 79 /	nd 14 204 137 24 4
		Cł	ni_square	= 2.883	32		
Element Ma O 4 Na 2 Mg Al Si 2 Ca	SS% At 1.276 53 4.595 22 0.973 0 1.542 1 5.465 18 6.149 3	omic% ZAF .6697 1.0489 .2554 0.9716 .8323 1.2621 .1892 1.2481 .8616 1.1493 .1917 1.0088	Z 0.9914 1 5 0.9956 0 0.9981 1 1.0035 1 0.9912 1 3 1.0039 1	A .0580 0 .9752 1 .2653 0 .2512 0 .1597 0	F .9999 .0007 .9993 .9940 .9999		i ig
Total 10 Normalizat	0.000 100 ion facto	.0000 r = 1.8564					



Figure D1-36. Test #2, Day-16 counting spectrum for a particle on the fiberglass shown in Figure D1-34. (Lowflow-exterior~EDS2)

The results from the chemical composition analysis for Lowflow~exterior~EDS2 are given in Table D1-4.

Table D1-4. The Chemical Composition for EDS2

Mar 6 21:53 2005 /tmp/eds_pout.log Page 1

Group Sample Comment Condition	: NRC : T2D16 L : Exteric : Full Sc Live Ti Acc. Vc Stage P Acq. Da	ow-flow ID or film on fr cale : 20KeV .me : 60.0 olt : 15.0 Point : X=52. ate : Sun M	# : 2 iberglass V(10eV/ch,2 000 sec KV 133 Y=69.8 Mar 6 21:5	2Kch) Aperture # Probe Curr 84 Z=12.46 52:13 2005	: 3 ent : 2.003E 7	5-08 A
Element	Mode	ROI(KeV)	K-ratio(%	;) +/-	Net/Backgrou	ind
ОК	Normal	0.25- 0.77	49.3257	0.0025	3469 /	20
Na K	Normal	0.83- 1.28	19.2296	0.0098	4252 /	22
Mg K	Normal	1.03- 1.52	1.0859	0.0010	303 /	308
Al K	Normal	1.26- 1.78	1.2414	0.0005	414 /	240
Si K	Normal	1.50- 2.07	20.6078	0.0018	6865 /	48
Ca K	Normal	3.40- 4.30	4.1927	0.0049	739 /	10
		Ch	ni_square =	5.6127		iotic e
Element Ma	ss% At	omic% ZAF	Z	A F		
0 4	8.657 60	.9403 0.9606	0.9936 0.	9668 0.999	9	
Na 1	9.974 17	.4088 1.0115	0.9979 1.	0129 1.000	7	
Mg	1.370 1	.1292 1.2286	1.0005 1.	2288 0.999	3	
Al	1.568 1	.1644 1.2300	1.0060 1.	2299 0.994	1	
Si 2	4.091 17	.1875 1.1384	0.9937 1.	1458 0.999	9	
Ca	4.340 2	.1699 1.0081	1.0068 1.	0012 1.000	1	
Total 10 Normalizat	0.000 100 ion facto	.0000 r = 1.0269	. 			0 -6 10 10 10