Appendix F2

SEM/EDS Data for Test #4, Day-30 Copper Coupons

Figures

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Table F2-2.	Chemical Compositions for T4D30CuSubm06.jpg, Figure F2-8	F2-9
Table F2-3.	Chemical Compositions for T4D30CuSubm07.jpg, Figure F2-9	F2-11

This appendix shows the SEM/EDS results for the metal copper coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged refers to coupons held in the test tank gas space above the water level of the solution during ICET. Unsubmerged coupons were contacted by the solution only during the 4-hour spraying period at the initial date of the test. In addition, the surface of the unsubmerged coupons may also be affected by the moisture in the gas space during the test. Submerged refers to the coupons that were under the solution during the test.

The coupon samples were collected on June 23, 2005 (the date Test #4 was shut down), and by SEM/EDS on June 29, 2005. The copper coupon samples were dried in air before being coated with Au/Pd for SEM examination. SEM results present the surface condition of the copper coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

Transcribed Laboratory Log

Laboratory session from June 29, 2005. SEM Test #4, Day-30 Copper Coupons

1. Unsubmerged Al	3. Sus. Cu	5. Sus. Gal Steel	7. Sus. Steel
2. Submerged Al	4. Sub. Cu	6. Sub. Gal Steel	8. Sub. Steel
9. Sediment	10. Powder on	Sub. Rack	



Unsubmerged Copper Coupon

Image:	T4D30CuSusp008	$100 \times$	SEM image	Figure F2-1
	T4D30CuSusp009	$500 \times$	SEM image higher magnification	Figure F2-2
	T4D30CuSusp010	$1500 \times$	SEM annotated image	Figure F2-3
EDS:	T4D30CuSusp05		On particles in T4D30CuSusp010	Figure F2-4

Submerged Copper Coupon

Image:	T4D30CuSubm011	$100 \times$	SEM image of fiberglass	Figure F2-5
	T4D30CuSubm012	$500 \times$	SEM image higher magnification	Figure F2-6
	T4D30CuSubm013	$1800 \times$	SEM annotated image	Figure F2-7
EDS:	T4D30CuSubm06		On bright particles shown in 013	Figure F2-8
	T4D30CuSubm07		Surface shown in 013	Figure F2-9



Figure F2-1. SEM image magnified 100 times for a Test #4, Day-30 unsubmerged copper coupon sample. (T4D30CuSusp008.bmp)



Figure F2-2. SEM image magnified 500 times for a Test #4, Day-30 unsubmerged copper coupon sample. (T4D30CuSusp009.bmp)



Figure F2-3. Annotated SEM image magnified 1500 times for a Test #4, Day-30 unsubmerged copper coupon sample. (T4D30CuSusp010.bmp)



Figure F2-4. EDS counting spectrum for the deposits (EDS1) on the coupon surface shown in Figure F2-3. (T4D30CuSusp05.jpg)

The results from the chemical composition analysis for T4D30CuSusp05.jpg are given in Table F2-1.

Table F2-1. Chemical Compositions for T4D30CuSusp05.jpg, Figure F2-4

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Group Sample Comment Condition	: NRC : T4D30 ID# : 5 : particles on suspended Cu : Full Scale : 20KeV(10eV/ch,2Kch) Live Time : 60.000 sec Aperture # : 2 Acc. Volt : 15.0 KV Probe Current : 1.065E-09 A Stage Point : X=81.233 Y=71.962 Z=10.786 Acq. Date : Wed Jun 29 13:41:34 2005
Element OK CuK	Mode ROI(KeV) K-ratio(%) +/- Net/Background Normal 0.25-0.77 16.7471 0.0012 554 / 13 Normal 7.63-9.27 105.3640 0.0057 1327 / 2
	Chi_square = 1.3920
Element Ma O 1 Cu 8	Atomic* ZAF Z A F 1.102 33.1531 0.8154 0.8265 0.9868 0.9998 8.898 66.8469 1.0378 1.0386 0.9993 1.0000
Total 10 Normalizat	0.000 100.0000 on factor = 0.8130



Figure F2-5. SEM image magnified 100 times for a Test #4, Day-30 submerged copper coupon sample. (T4D30CuSubm011.bmp)



Figure F2-6. SEM image magnified 500 times for a Test #4, Day-30 submerged copper coupon sample. (T4D30CuSubm012.bmp)



Figure F2-7. Annotated SEM image magnified 1800 times for a Test #4, Day-30 submerged copper coupon sample. (T4D30CuSubm013.bmp)



Figure F2-8. EDS counting spectrum for the deposit (EDS2) on the coupon surface shown in Figure F2-7. (T4D30CuSubm06.jpg)

The results from the chemical composition analysis for T4D30CuSubm06.jpg are given in Table F2-2.

Table F2-2. Chemical Compositions for T4D30CuSubm06.jpg, Figure F2-8

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Group Sample Comment Condition	: NRC : T4D30 ID# : light parti : Full Scale Live Time Acc. Volt Stage Point Acq. Date	: 6 cles on : 20KeV : 60.0 : 15.0 : X=57. : Wed J	submerg (10eV/c) 000 sec KV 699 Y=5 Jun 29 1	ed Cu h,2Kch) Aper Prob 8.376 Z 3:54:39	ture # e Curre =10.786 2005	: 2 nt : 1	.065E-09 A
Element	Mode RO	(KeV)	K-rati	a (%)	/- N	et /Back	-
OK	Normal 0.2	5- 0.77	55.29	61 A	0018	1920	/ 10
Na K	Normal 0.8	- 1.27	11.08	27 0	0010	1053	/ 10
Al K	Normal 1.2	5- 1.78	1.15	53 0.	0003	159	/ 13
Si K	Normal 1.5	- 2.07	4.08	96 0.	0005	523	/ 17
CaK	Normal 3.4	- 4.30	4.29	31 0.	0035	267	/ 6
Cu K	Normal 7.63	3- 9.27	9.70	52 0.	0023	122	1 2
CK	Normal 0.09	9- 0.46	0.00	00 0.	0000		1 16
Mg K	Normal 0.9	7- 1.57	0.07	18 0.	0001	10	/ 36
		Ch	i_square	e = 2.	6388		
Element Ma	ass% Atomic	ZAF	z	A	F		
0 5	52.039 67.6839	0.8346	0.9692	0.8612	0.9999		
Na 2	20.992 19.0006	1.6798	1.0227	1.6423	1.0001		
Al	1.925 1.4848	1.4779	0.9846	1.5028	0.9988		
Si	6.377 4.7251	1.3829	0.9729	1.4216	0.9999		
Ca	4.710 2.4453	0.9729	0.9809	0.9925	0.9994		
Cu 1	3.788 4.5154	1.2597	1.2639	0.9967	1.0000		
c	0.000 0.0000	4.0970	1.0163	4.0314	0.9999		
Mg	0.169 0.1449	2.0917	0.9627	2.1740	0.9994		
Total 10 Normalizat	0.000 100.0000 ion factor =	1.1276					



Figure F2-9.EDS counting spectrum for the flat coupon surface (EDS3) shown in Figure F2-7.
(T4D30CuSubm07.jpg)

The results from the chemical composition analysis for T4D30CuSubm07.jpg are given in Table F2-3.

Table F2-3. Chemical Compositions for T4D30CuSubm07.jpg, Figure F2-9

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Group	NRC	
Sample	T4D30 ID# : 7	
Comment	surface of submerged Cu	
Condition	Full Scale : 20KeV(10eV/ch 2Keh)	
	Live Time : 60 000 cog Aporturo # 0	
	Acc Volt , 15 0 W Buch Common 2 occ as	-
	Acc. Volt : 15.0 KV Probe Current : 1.065E-09	А
	Stage Point : X=57.699 Y=58.376 Z=10.786	
	Acq. Date : Wed Jun 29 13:59:37 2005	
D1 amount		
Element	Mode ROI(KeV) K-ratio(%) +/- Net/Background	
OK	Normal 0.25-0.77 12.2183 0.0011 404 / 2	21
Cu K	Normal 7.63-9.27 113.6477 0.0061 1432 /	0
СК	Normal 0.09-0.46 2.1513 0.0001 106 /	4
		-
	Chi_square = 0.9631	
Element Ma	s% Atomic% ZAF Z A F	
0	.923 20.9480 0.8970 0.8301 1.0807 0.9998	
Cu 8	.861 57.1606 1.0450 1.0460 0.9991 1.0000	
С	.216 21.8914 3.9968 0.8712 4.5876 1.0000	
Total 10	.000 100.0000	
Normalizat	on factor = 0.7229	

Appendix F3

SEM/EDS Data for Test #4, Day-30 Galvanized Steel Coupons

Figures

Figure F3-1.	SEM image magnified 100 times for a Test #4, Day-30 unsubmerged
	galvanized steel coupon sample. (T4D30GalSusp014.bmp)F3-4
Figure F3-2.	SEM image magnified 500 times for a Test #4, Day-30 unsubmerged
	galvanized steel coupon sample. (T4D30GalSusp015.bmp)F3-4
Figure F3-3.	Annotated SEM image magnified 1800 times for a Test #4, Day-30
	unsubmerged galvanized steel coupon sample. (T4D30GalSusp016.bmp)F3-5
Figure F3-4.	EDS counting spectrum for the deposits (EDS1) on the coupon surface shown
	in Figure F3-3. (T4D30GalSusp08.jpg)F3-5
Figure F3-5.	EDS counting spectrum for the flat coupon surface (EDS2) shown in Figure
	F3-3. (T4D30Galsusp09.jpg)
Figure F3-6.	SEM image magnified 100 times for a Test #4, Day-30 submerged galvanized
	steel coupon sample. (T4D30GalSubm017.bmp)F3-7
Figure F3-7.	SEM image magnified 500 times for a Test #4, Day-30 submerged galvanized
	steel coupon sample. (T4D30GalSubm018.bmp)F3-8
Figure F3-8.	Annotated SEM image magnified 1800 times for a Test #4, Day-30
	submerged galvanized steel coupon sample. (T4D30GalSubm019.bmp)
Figure F3-9.	EDS counting spectrum for the deposits (EDS3) on the coupon surface shown
	in Figure F3-8. (T4D30GalSubm10.jpg)
Figure F3-10.	EDS counting spectrum for the flat coupon surface (EDS4) shown in Figure
	F3-8. (T4D30GalSubm11.jpg)F3-11

Tables

Table F3-1.	Chemical Compositions for T4D30GalSusp08.jpg, Figure F3-4	F3-6
Table F3-2.	Chemical Compositions for T4D30GalSubm10.jpg, Figure F3-9	F3-10
Table F3-3.	Chemical Compositions for T4D30GalSubm11.jpg, Figure F3-10	F3-11

This appendix shows the SEM/EDS results for the metal galvanized steel coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged refers to coupons held in the test tank gas space above the water level of the solution during ICET. Unsubmerged coupons were contacted by the solution only during the 4-hour spraying period at the initial date of the test. In addition, the surface of the unsubmerged coupons may also be affected by the moisture in the gas space during the test. Submerged refers to the coupons that were under the solution during the test.

The coupon samples were collected on June 23, 2005 (the date Test #4 was shut down), and examined by SEM/EDS on June 29, 2005. The galvanized steel coupon samples were dried in air before being coated with Au/Pd for SEM examination. SEM results present the surface condition of the galvanized steel coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

Transcribed Laboratory Log

Laboratory session from June 29, 2005. SEM Test #4, Day-30 Galvanized Steel Coupons

1. Unsubmerged Al	3. Sus. Cu	5. Sus. Gal Steel	7. Sus. Steel
2. Submerged Al	4. Sub. Cu	6. Sub. Gal Steel	8. Sub. Steel
9. Sediment	10. Powder on	Sub. Rack	



Unsubmerged Galvanized Steel Coupon

T4D30GalSusp014	$100 \times$	SEM image	Figure F3-1
T4D30GalSusp015	$500 \times$	SEM image	Figure F3-2
T4D30GalSusp016	$1800 \times$	SEM annotated image	Figure F3-3
T4D30GalSusp08		Particles shown in 016	Figure F3-4
T4D30Galsusp09		Surface shown in 016	Figure F3-5
	T4D30GalSusp014 T4D30GalSusp015 T4D30GalSusp016 T4D30GalSusp08 T4D30Galsusp09	T4D30GalSusp014 $100 \times$ T4D30GalSusp015 $500 \times$ T4D30GalSusp016 $1800 \times$ T4D30GalSusp08T4D30GalSusp09	T4D30GalSusp014 $100 \times$ SEM imageT4D30GalSusp015 $500 \times$ SEM imageT4D30GalSusp016 $1800 \times$ SEM annotated imageT4D30GalSusp08Particles shown in 016T4D30Galsusp09Surface shown in 016

Submerged Galvanized Steel Coupon

Image:	T4D30GalSubm017	$100 \times$	SEM image	Figure F3-6
	T4D30GalSubm018	$500 \times$	SEM image	Figure F3-7
	T4D30GalSubm019	$1800 \times$	SEM annotated image	Figure F3-8
	T4D30GalSubm10		Particles shown in 019	Figure F3-9
	T4D30GalSubm11		Surface shown in 019	Figure F3-10



Figure F3-1. SEM image magnified 100 times for a Test #4, Day-30 unsubmerged galvanized steel coupon sample. (T4D30GalSusp014.bmp)



Figure F3-2. SEM image magnified 500 times for a Test #4, Day-30 unsubmerged galvanized steel coupon sample. (T4D30GalSusp015.bmp)



Figure F3-3. Annotated SEM image magnified 1800 times for a Test #4, Day-30 unsubmerged galvanized steel coupon sample. (T4D30GalSusp016.bmp)



Figure F3-4. EDS counting spectrum for the deposits (EDS1) on the coupon surface shown in Figure F3-3. (T4D30GalSusp08.jpg)

The results from the chemical composition analysis for T4D30GalSusp08.jpg are given in Table F3-1.

 Table F3-1.
 Chemical Compositions for T4D30GalSusp08.jpg, Figure F3-4

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Group	: NRC								
Sample	: T4D3	0 ID# :	8						
Comment	: part	icle on	suspend	ed Gal-:	steel				
Condition	: Full	Scale	: 20KeV	(10eV/c)	2Kc	h)			
	Live	Time	60.0	00 880	an	arture	# .	2	
	Acc	Volt	. 15 0	WV BEC	200	che Cur	T i	1 064	17 00 N
	Stag	e Boint	- X-44	143 V-E	P 014	Z-10 Z	rent :	1.004	12-03 A
	Aca	Date	. Mod .T	143 1=5	1.12.	2=10.7	00		
	Acq.	Date	: wea J	un 29 14	.:12:	20 2005			
Element	Mode	ROI	(KeV)	K-ratio	o (₩)	+/-	Net/B	ackory	bund
CK	Norma	1 0.09	- 0.46	17.710	08	0.0003	8	68 /	7
OK	Norma	1 0.25	- 0.77	7.99	33	0.0009	ž	64 /	43
Na K	Norma	1 0.81	- 1.27	4.11	79	0.0007	3	91 /	
Si K	Norma	1 1.50	- 2.07	1.211	3	0.0004	1	55 /	14
Cl K	Norma	1 2.34	- 3.06	3.70	51	0.0005	3	24 /	14
CaK	Norma	1 3.40	- 4.30	5.18	18	0.0037	3	22 /	5
			Ch	i_square	9 = (6.7756			
Discort Ma				_			_		
Element Ma	1888	Atomic*	ZAF	z		A	F		
0 5	7.624	69.8633	2.4583	1.0116	2.43	03 1.00	00		
.0 2	2.840	20.7886	2.1589	0.9652	2.23	58 1.00	00		
Na	6.485	4.1080	1.1900	1.0197	1.16	58 1.00	02		
S1	1.750	0.9073	1.0916	0.9716	1.124	17 0.99	89		
CI	4.786	1.9657	0.9759	1.0255	0.95	52 0.99	63		
Ca	6.515	2.3671	0.9500	0.9837	0.96	57 1.00	01		
m									
Total 10	0.000 :	100.0000							
Normalizat	ion fac	ctor = 1	1.3235						



Figure F3-5. EDS counting spectrum for the flat coupon surface (EDS2) shown in Figure F3-3. (T4D30Galsusp09.jpg)



Figure F3-6. SEM image magnified 100 times for a Test #4, Day-30 submerged galvanized steel coupon sample. (T4D30GalSubm017.bmp)



Figure F3-7. SEM image magnified 500 times for a Test #4, Day-30 submerged galvanized steel coupon sample. (T4D30GalSubm018.bmp)



Figure F3-8. Annotated SEM image magnified 1800 times for a Test #4, Day-30 submerged galvanized steel coupon sample. (T4D30GalSubm019.bmp)



Figure F3-9. EDS counting spectrum for the deposits (EDS3) on the coupon surface shown in Figure F3-8. (T4D30GalSubm10.jpg)

The results from the chemical composition analysis for T4D30GalSubm10.jpg are given in Table F3-2.

 Table F3-2.
 Chemical Compositions for T4D30GalSubm10.jpg, Figure F3-9

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Group	NRC	
Sample	T4D30 ID# : 10	
Comment	Particle on submerged Gal-steel	
Condition	Full Scale : 20KeV(10eV/ch.2Kch)	
	Live Time : 60,000 sec Aperture # . 2	
	Acc. Volt : 15.0 KV Probe Current : 1 0628-00	
	Stage Point - X=50 108 V=68 926 7-10 786	A
	Acg. Date . Wed Jup 29 14.20.24 2005	
	Mod: Date , Wed Dun 29 14:30:34 2005	
Element	Mode ROI(KeV) K-ratio(%) +/- Net/Background	
СК	Normal 0.09-0.46 4.3159 0.0001 212 /	6
ок	Normal 0.25-0.77 17.0145 0.0011 564 /	10
Na K	Normal 0.81-1.27 6.3047 0.0007 600 /	Ĩě
Al K	Normal 1.26-1.78 1.7139 0.0004 236 /	56
Si K	Normal 1.50-2.07 7.1958 0.0006 923 /	25
Ca K	Normal 3,40-4,30 8,1648 0,0042 509 /	- 2
		- °
	Chi_square = 2.5440	_
Element Ma	s% Atomic% ZAF Z A F	
C 2	.363 34.2953 3.7436 1.0268 3.6461 1.0000	
0 3	.077 41.9614 1.5476 0.9794 1.5802 1.0000	
Na 1	.659 8.9415 1.2789 1.0340 1.2371 0.9998	
Al	.933 1.9165 1.1835 0.9960 1.1924 0.9965	
Si 1	.465 7.8251 1.1980 0.9844 1.2173 0.9997	
Ca 1	.503 5.0602 0.9743 0.9944 0.9797 1.0001	
makel 1		-
Total 10	.000 100.0000	
Normalizat	on factor = 1.4460	



Figure F3-10. EDS counting spectrum for the flat coupon surface (EDS4) shown in Figure F3-8. (T4D30GalSubm11.jpg)

The results from the chemical composition analysis for T4D30GalSubm11.jpg are given in Table F3-3

Table F3-3.	Chemical Com	ositions for	T4D30GalSubm	11.jpg,	Figure	F3-1	10
	0		1.20004.04.04.04.04.04.04.04.04.04.04.04.04				

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<pre>Group : NRC Sample : T4D30 ID# : 11 Comment : Surface of submerged Gal-steel Condition : Full Scale : 20KeV(10eV/ch,2Kch) Live Time : 60.000 sec Aperture # : 2 Acc. Volt : 15.0 KV Probe Current : 1.066E-09 A Stage Point : X=50.108 Y=68.926 Z=10.786 Acq. Date : Wed Jun 29 14:35:16 2005</pre>
Element Mode ROI(KeV) K-ratio(%) +/- Net/Background O K Normal 0.25-0.77 7.3244 0.0010 243 / 18 Al K Normal 1.26-1.78 0.9436 0.0004 130 / 16 Si K Normal 1.50-2.07 0.4713 0.0003 60 / 22 Zn K Normal 8.22-10.03 118.8961 0.0077 1137 / 2 C K Normal 0.09-0.46 2.8301 0.0001 139 / 2
Chi_square = 0.5995
Element Mass% Atomic% ZAF Z A F O 4.987 12.5657 1.0235 0.8341 1.2272 0.9999 Al 1.235 1.8457 1.9679 0.8447 2.3300 0.9999 Si 0.520 0.7459 1.6575 0.8339 1.9877 1.0000 Zn 83.279 51.3537 1.0528 1.0538 0.9991 1.0000 C 9.979 33.4891 5.2998 0.8754 6.0542 1.0000
Total 100.000 100.0000 Normalization factor = 0.6653