Appendix E1

SEM/EDS Data for Test #3, Day-30 Aluminum Coupons

Figures

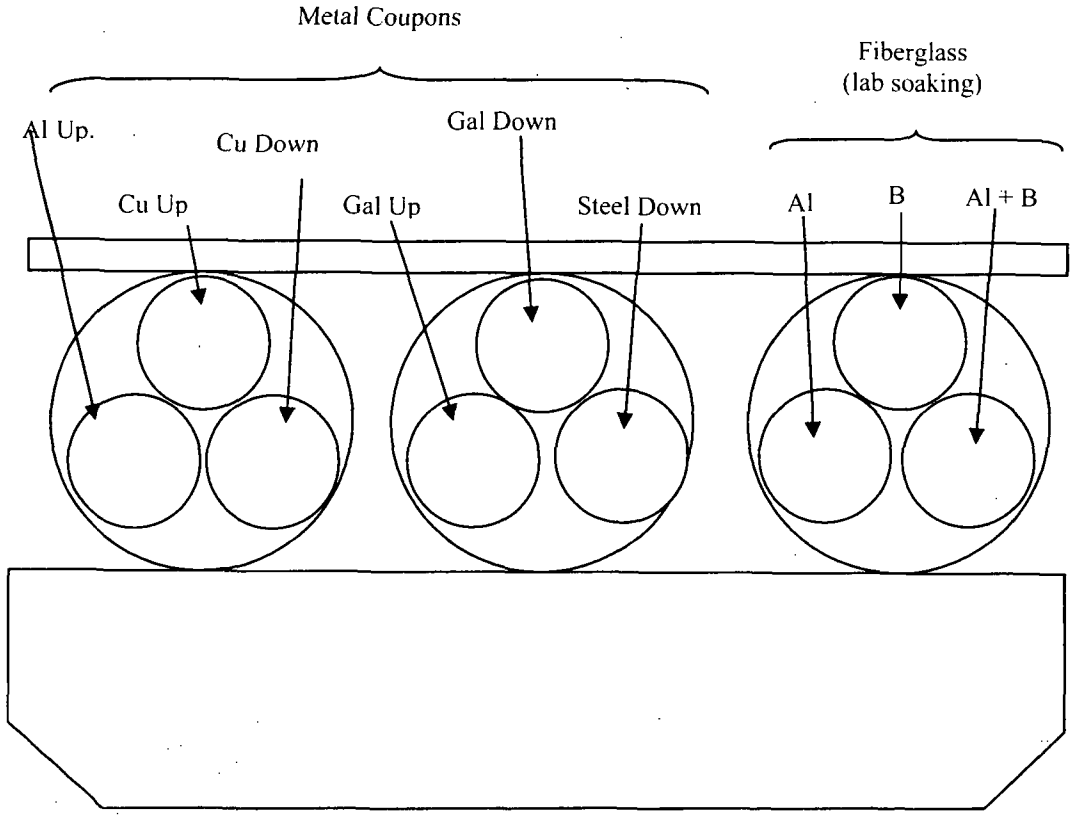
Tables

This appendix presents SEM/EDS results for metal aluminum coupons described by two different exposure categories: (1) suspended and (2) submerged. *Suspended* refers to the coupons located above the water level of the solution during the ICET tests. Suspended coupons were contacted by the solution only during the 4-hour spray period at initiation of the test. In addition, the surface of the suspended coupons may be affected by moisture in the test chamber vapor space. *Submerged* refers to coupons that were immersed in the solution for the duration of the test.

The coupon samples were collected on May 5, 2005 (the date Test #3 was shut down), and were later examined by SEM/EDS. The aluminum coupon samples were dried in air before coating with Au/Pd for SEM examination. SEM results present the surface condition of the aluminum coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Laboratory session from May 17, 2005.
Test #3, Day-30 Metal Coupons



**Coat with Gold

Suspended Al

Image:	T3D30AlSusp006	100 ×	SEM image	Figure E1-1
	T3D30AlSusp007	1000 ×	SEM image higher magnification	Figure E1-2
	T3D30AlSusp008	1000 ×	Backscattered image	Figure E1-3
EDS:	T3D30SuspAl05		Particles on 007	Figure E1-4
	T3D30SuspAl06		Surface on 007	Figure E1-5

Submerged Aluminum

Image:	T3D30AlSubm029	100 ×	SEM image	Figure E1-6
	T3D30AlSubm030	100 ×	Annotated backscatter SEM	Figure E1-7
EDS:	T3D30SubmAl17		Grey surface on 030	Figure E1-8
	T3D30SubmAl18		Dark spot on 030	Figure E1-9
Image:	T3D30AlSubm031	100 ×		Figure E1-10
	T3D30AlSubm032	500 ×		Figure E1-11

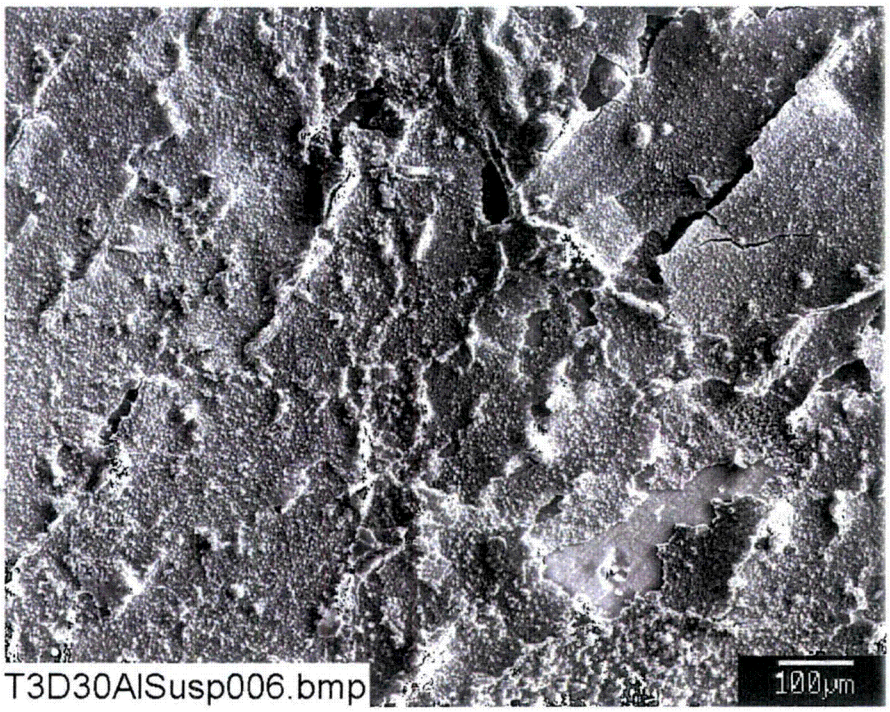


Figure E1-1. **SEM image magnified 100 times for a Test #3, Day-30 suspended aluminum coupon. (T3D30AlSusp006)**

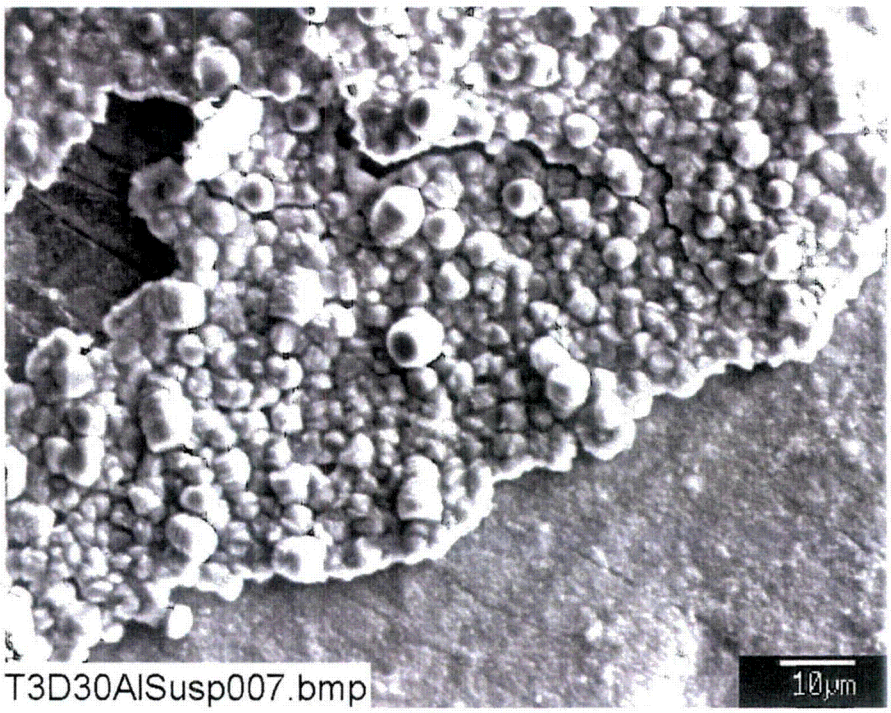


Figure E1-2. **SEM image magnified 1000 times for a Test #3, Day-30 suspended aluminum coupon. (T3D30AlSusp007)**

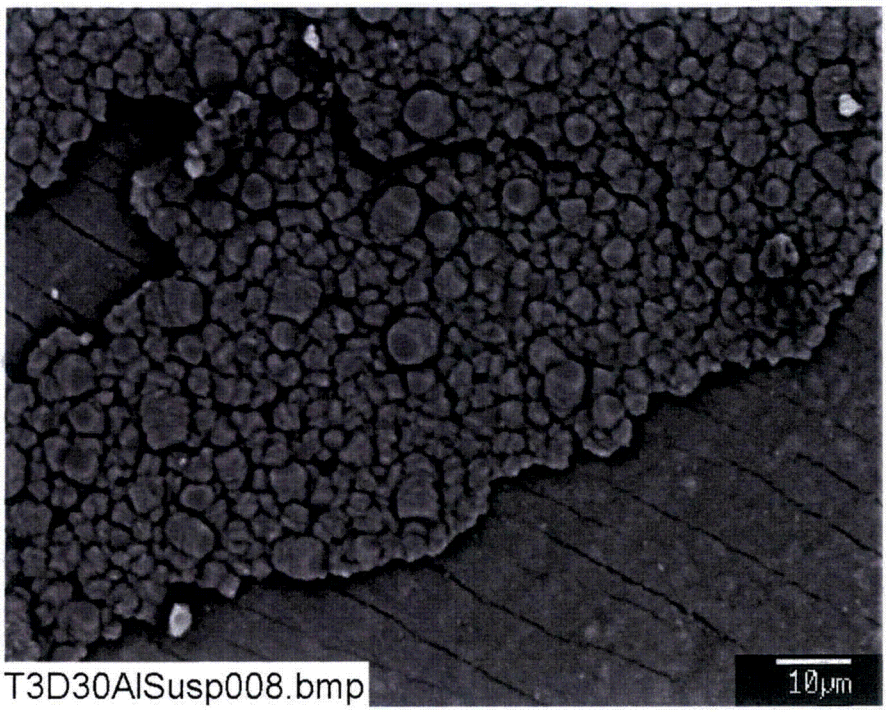


Figure E1-3. **Backscattered SEM image magnified 1000 times for a Test #3, Day-30 suspended aluminum coupon. (T3D30AlSusp008)**

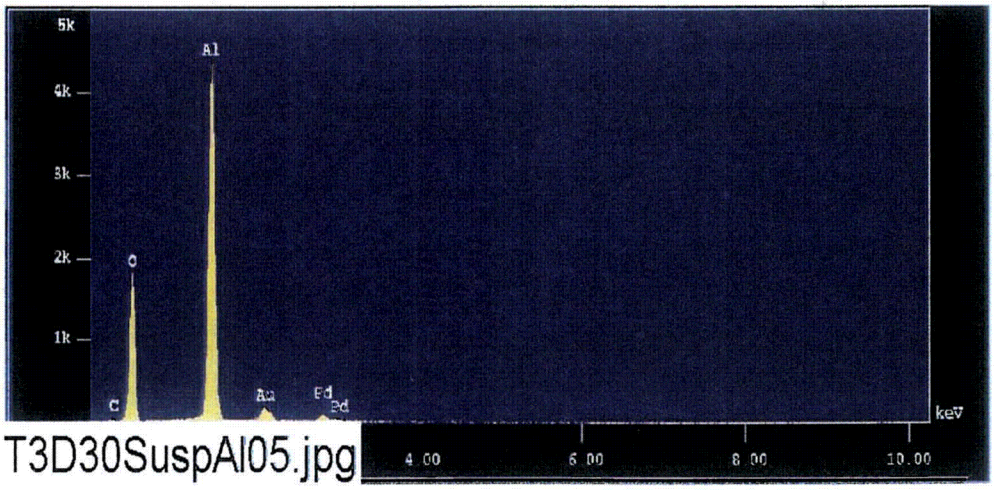


Figure E1-4. **EDS counting spectrum for the particles shown in Figure E1-2. (T3D30SuspAl05)**

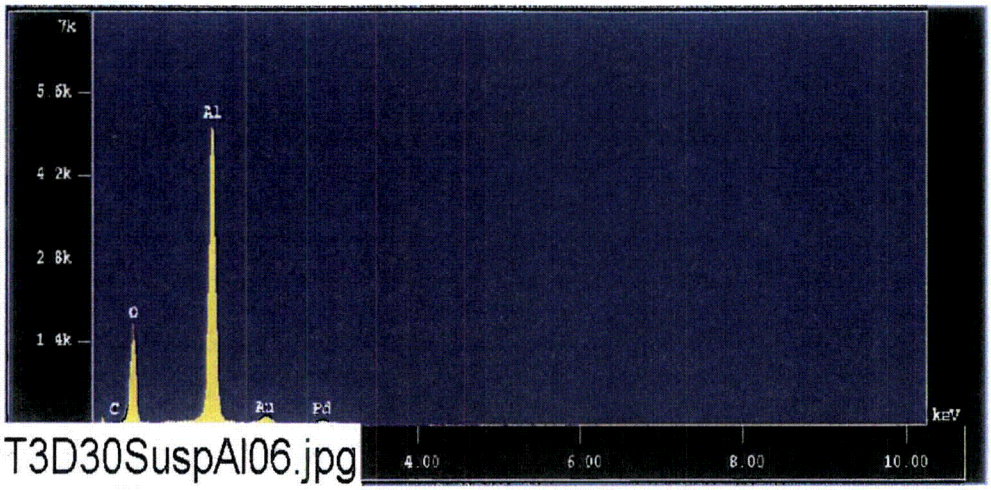


Figure E1-5. **EDS counting spectrum for the surface shown in Figure E1-2. (T3D30SuspAl06)**

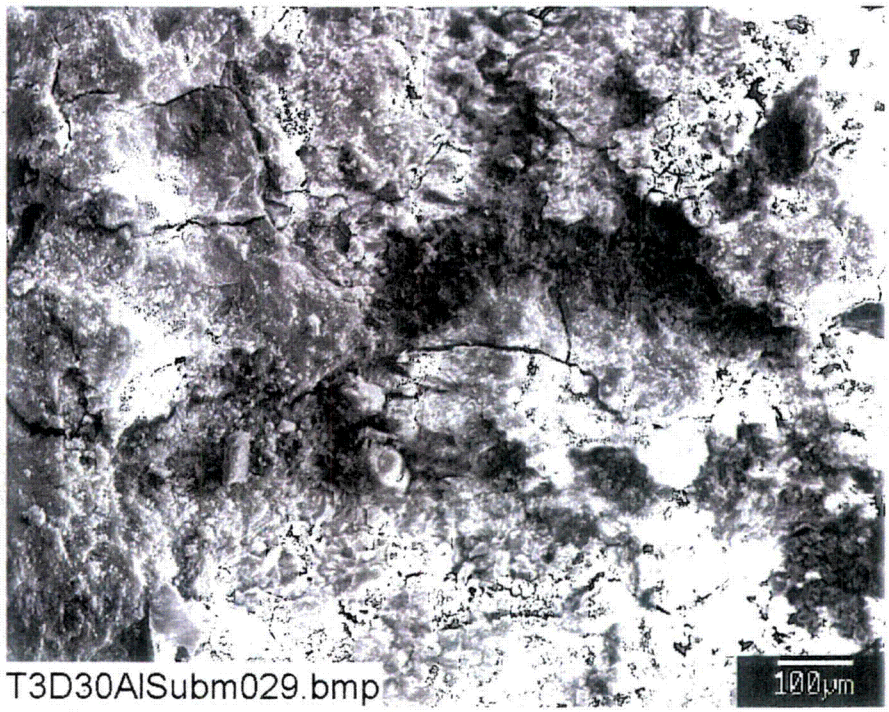


Figure E1-6. **SEM image magnified 100 times for a Test #3, Day-30 submerged aluminum coupon. (T3D30AlSubm029)**

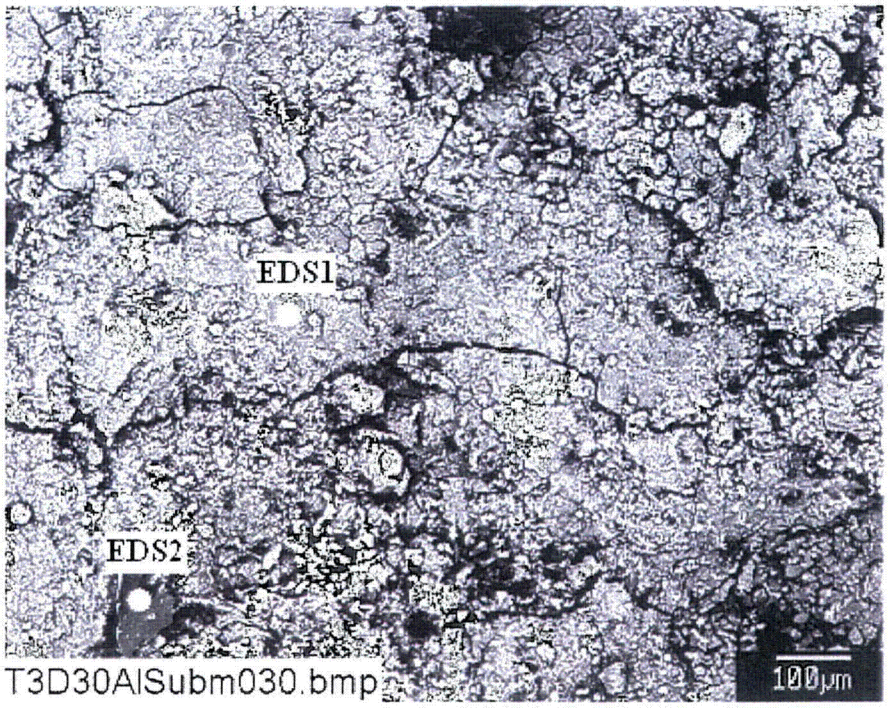


Figure E1-7. **Annotated backscattered SEM image magnified 100 times for a Test #3, Day-30 submerged aluminum coupon. (T3D30AlSubm030)**

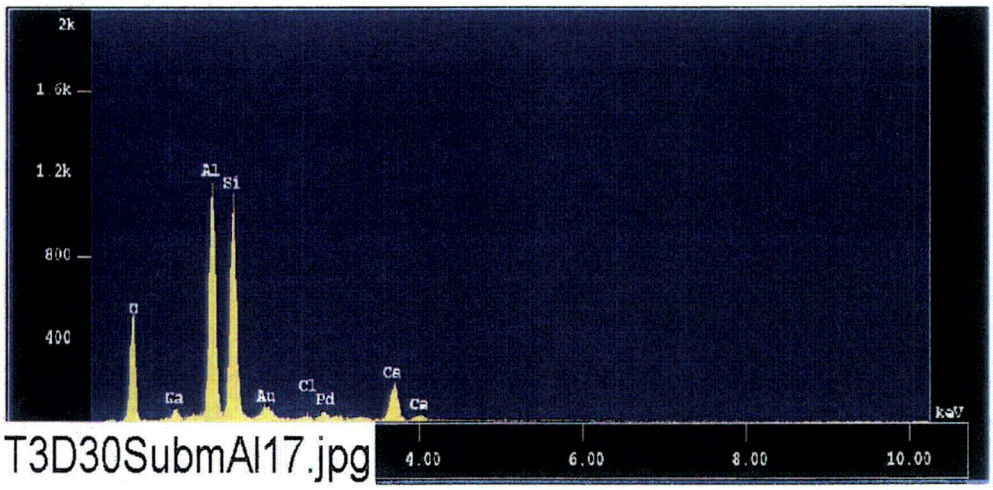


Figure E1-8. **EDS counting spectrum for the grey surface (EDS 1) shown in Figure E1-7. (T3D30SubmAl17)**

The results from the chemical composition analysis for T3D30SubmAl17 are given in Table E1-1.

Table E1-1. Chemical Compositions for T3D30SubmAl17, Figure E1-8

May 17 2005

Group : NRC
Sample : T3D30 ID# : 20
Comment : Submerged Al grey surface
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 7.746E-09 A
Stage Point : X=14.568 Y=53.830 Z=11.000
Acq. Date : Tue May 17 15:13:52 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	14.6772	0.0027	3531 / 18
Na K	Normal	0.81- 1.27	0.5575	0.0008	385 / 26
Al K	Normal	1.26- 1.78	9.9625	0.0017	9945 / 200
Si K	Normal	1.50- 2.05	9.6207	0.0009	8956 / 486
Ca K	Normal	3.40- 4.30	3.5591	0.0080	1610 / 18
Cl K	Normal	2.34- 3.06	0.4826	0.0007	308 / 28

Chi_square = 38.5655

Element	Mass%	Atomic%	ZAF	Z	A	F
O	39.310	53.7531	1.2511	0.9885	1.2656	1.0000
Na	1.470	1.3989	1.2317	1.0429	1.1833	0.9980
Al	22.707	18.4115	1.0647	1.0041	1.0675	0.9934
Si	27.544	21.4549	1.3373	0.9921	1.3483	0.9998
Ca	7.761	4.2363	1.0186	1.0000	1.0185	1.0001
Cl	1.208	0.7453	1.1692	1.0450	1.1209	0.9982
Total	100.000	100.0000				

Normalization factor = 2.1408

Fe	2.399	1.5375	0.9896	1.0503	1.0060	0.9366
Total	100.000	100.0000				

Normalization factor = 3.2486

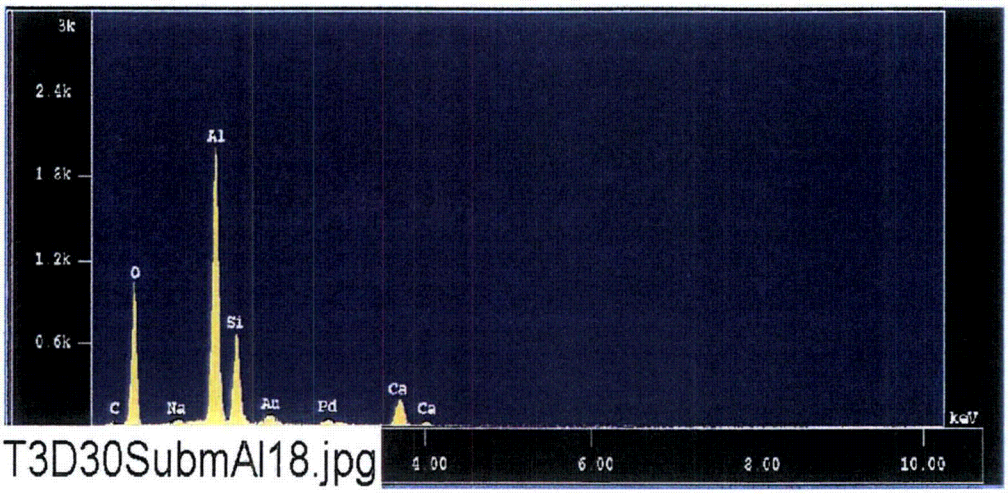


Figure E1-9. **EDS counting spectrum for the dark spot (EDS2) shown in Figure E1-7. (T3D30SubmAl18)**

The results from the chemical composition analysis for T3D30SubmAl18 are given in Table E1-2.

Table E1-2. Chemical Compositions for T3D30SubmAl18, Figure E1-9

May 17 2005

Group : NRC
Sample : T3D30 ID# : 21
Comment : Submerged Al dark surface
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 7.728E-09 A
Stage Point : X=14.568 Y=53.830 Z=11.000
Acq. Date : Tue May 17 15:18:41 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	27.8853	0.0036	6693 / 39
Al K	Normal	1.26- 1.78	16.9485	0.0022	16880 / 180
Si K	Normal	1.50- 2.05	6.0037	0.0008	5576 / 814
Ca K	Normal	3.40- 4.30	4.1252	0.0085	1862 / 16
Na K	Normal	0.81- 1.27	0.2751	0.0008	190 / 40
C K	Normal	0.09- 0.46	0.0000	0.0000	0 / 154

Chi_square = 70.9798

Element	Mass%	Atomic%	ZAF	Z	A	F
O	49.003	63.0911	1.0832	0.9889	1.0954	1.0000
Al	29.742	22.7054	1.0817	1.0048	1.0798	0.9970
Si	13.942	10.2255	1.4315	0.9929	1.4419	0.9999
Ca	6.739	3.4632	1.0069	1.0015	1.0053	1.0001
Na	0.575	0.5148	1.2875	1.0436	1.2361	0.9980
C	0.000	0.0000	6.5476	1.0370	6.3144	1.0000
Total	100.000	100.0000				

Normalization factor = 1.6223

Fe	2.599	1.5375	0.9896	1.0503	1.0060	0.9366
Total	100.000	100.0000				

Normalization factor = 3.2486

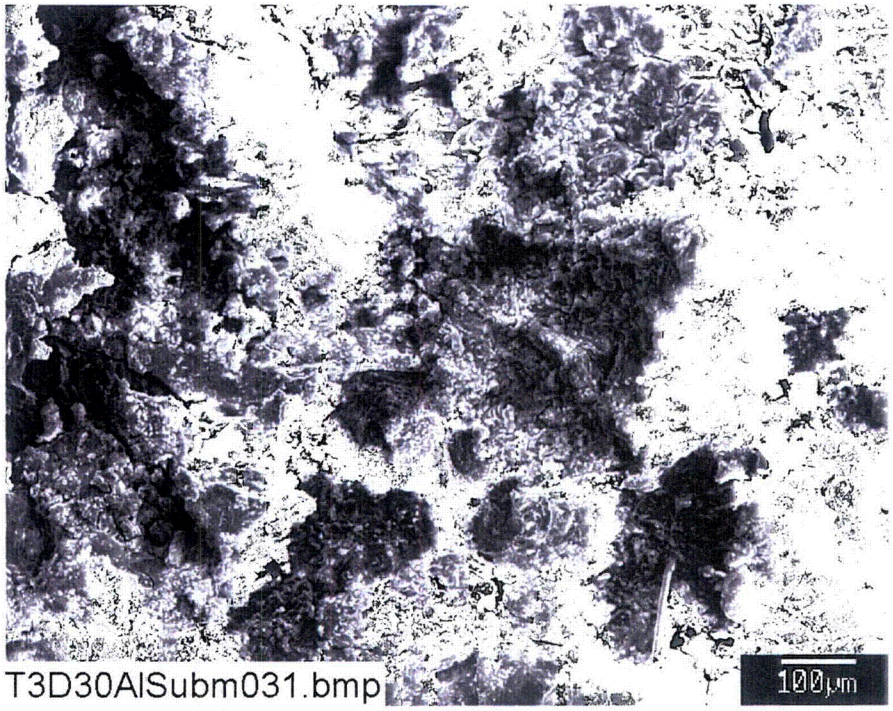


Figure E1-10. **SEM image magnified 100 times for a Test #3, Day-30 submerged aluminum coupon. (T3D30AlSubm031)**

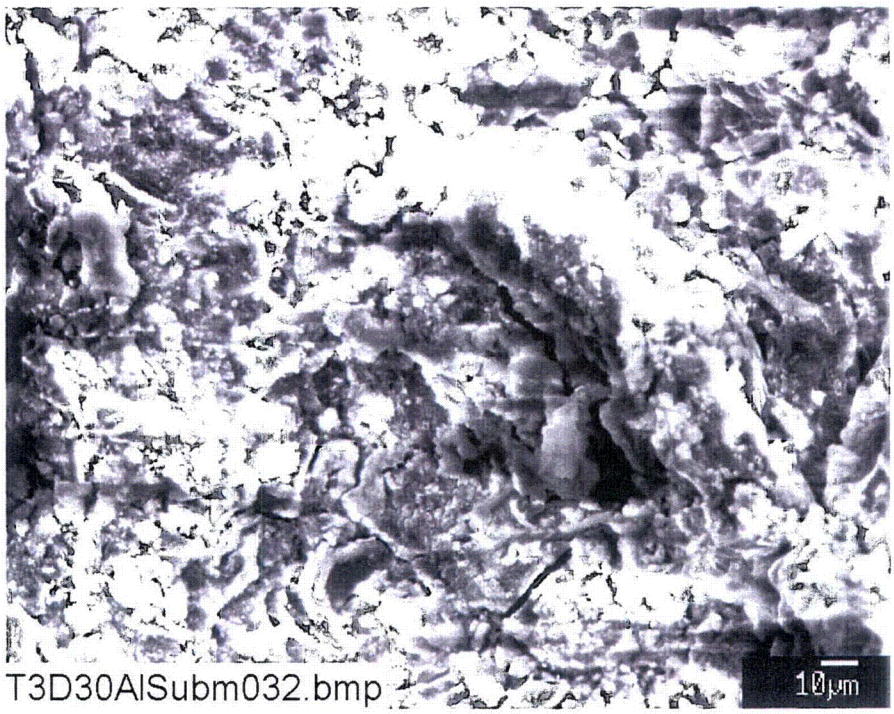


Figure E1-11. **SEM image magnified 500 times for a Test #3, Day-30 submerged aluminum coupon. (T3D30AlSubm032)**

Appendix E2

SEM/EDS Data for Test #3, Day-30 Copper Coupons

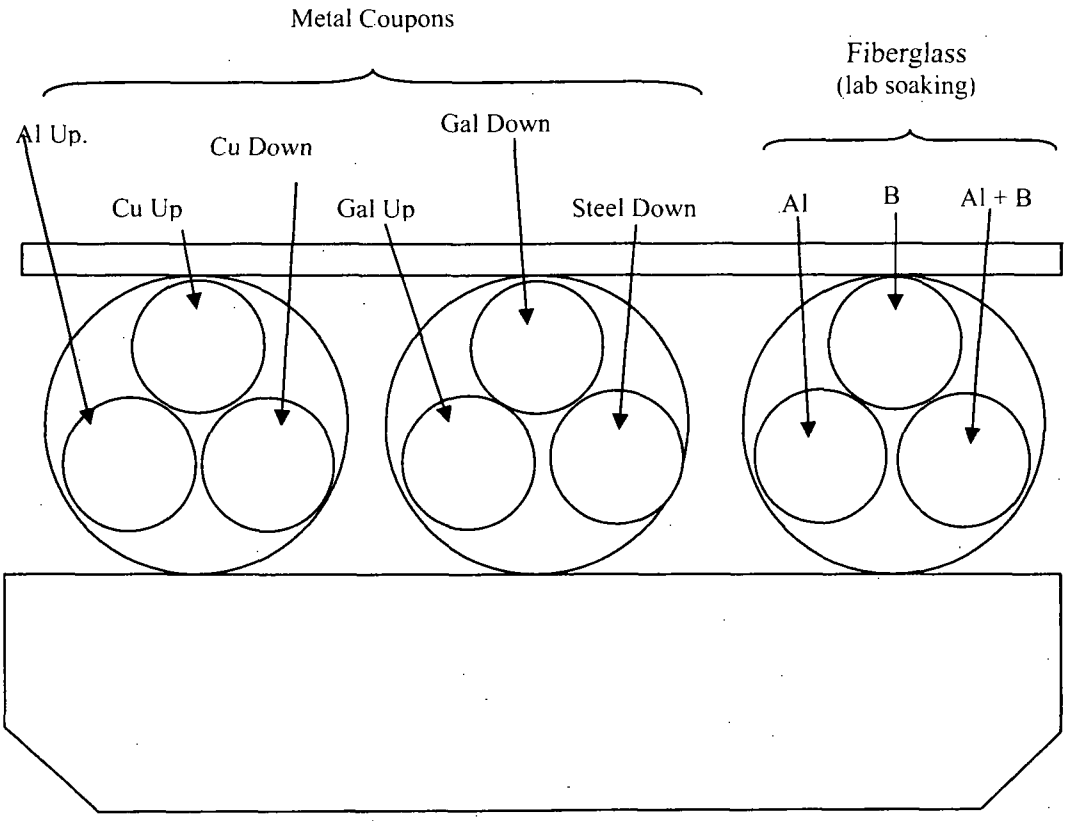
Figures

This appendix shows the SEM/EDS results for the metal copper coupons under two different catalogs: (1) suspended and (2) submerged. *Suspended* refers to coupons located above the water level of the solution during ICET tests. Suspended coupons were contacted with the solution only during the 4 hour spraying period at the initial date of the test. In addition, the surface of the suspended coupons may also be affected by the moisture in the test chamber gas space during the test. *Submerged* refers to the coupons that were submerged in the solution during the test.

The coupon samples were collected on May 5, 2005 (the date Test #3 was shut down), and subsequently examined by SEM/EDS. The copper coupon samples were dried in air before being coating 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. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Laboratory session from May 17, 2005.
Test #3, Day-30 Metal Coupons



**Coat with Gold

Suspended Cu

Image:	T3D30CuSusp001	100 ×	SEM image	Figure E2-1
	T3D30CuSusp002	1000 ×	Annotated SEM image	Figure E2-2
	T3D30CuSusp003	100 ×	Backscattered image	Figure E2-3
EDS:	T3D30SuspCu01		Medium (convex) dark spot on 002	Figure E2-4
	T3D30SuspCu02		Light spot (concave) on 002	Figure E2-5

Submerged Cu

Image:	T3D30CuSubm004	100 ×	SEM image	Figure E2-6
	T3D30CuSubm005	1000 ×	Annotated SEM image	Figure E2-7
EDS:	T3D30SubmCu03		White spot on 005	Figure E2-8
	T3D30SubmCu04		Dark surface on 005	Figure E2-9

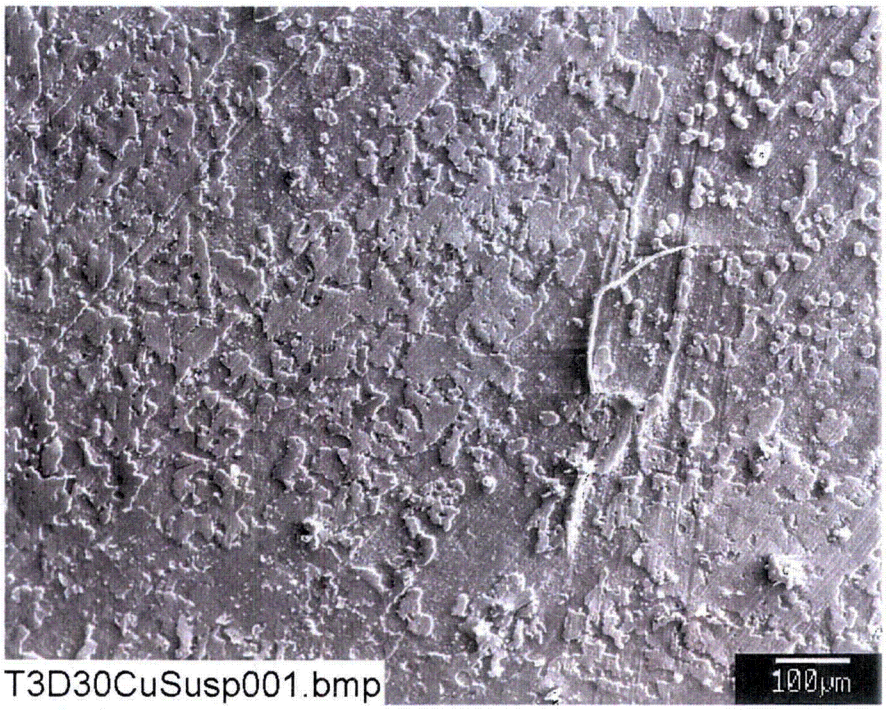


Figure E2-1. **SEM image magnified 100 times for a Test #3, Day-30 suspended copper coupon. (T3D30CuSusp001)**

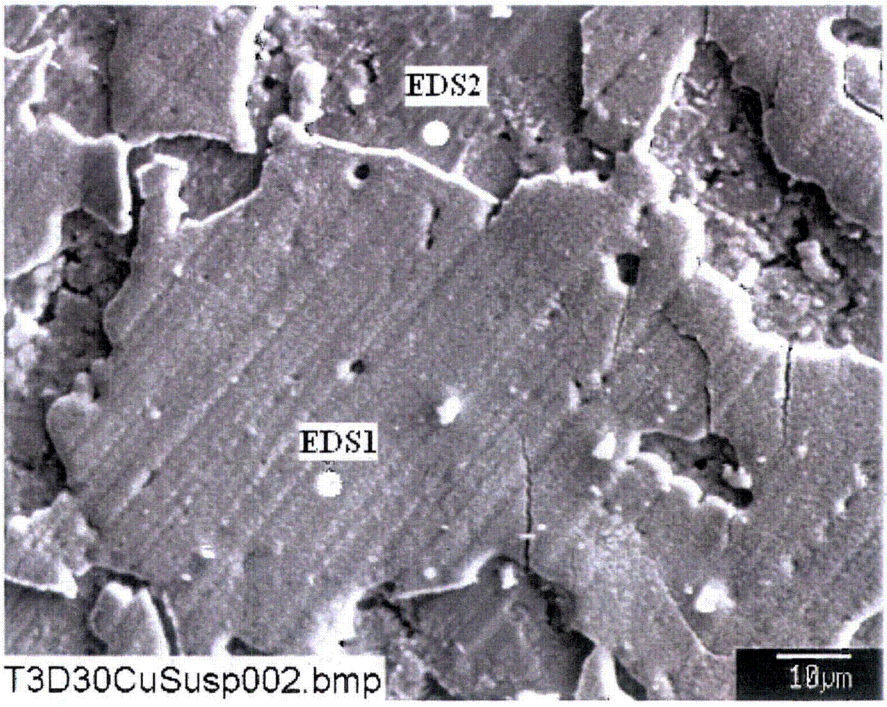


Figure E2-2. **Annotated SEM image magnified 1000 times for a Test #3, Day-30 suspended copper coupon. (T3D30CuSusp002)**

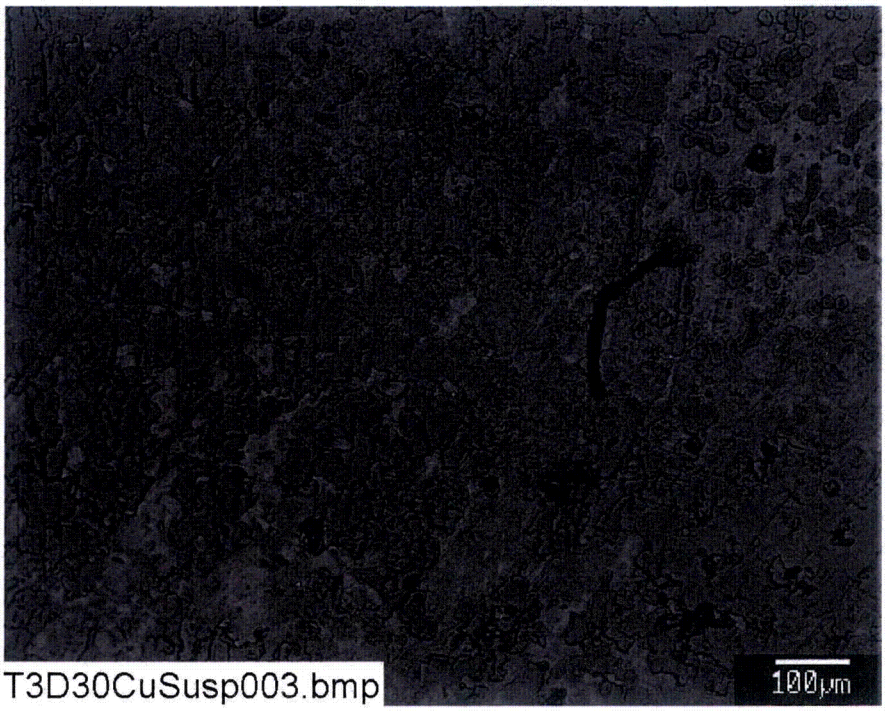


Figure E2-3. **Backscattered SEM image magnified 100 times for a Test #3, Day-30 suspended copper coupon. (T3D30CuSusp003)**

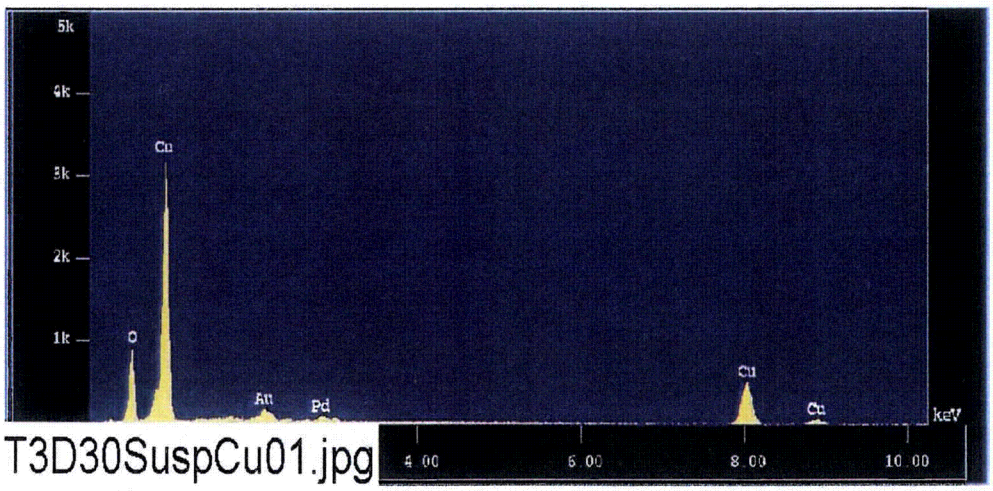


Figure E2-4. **EDS counting spectrum for the convex surface (EDS1) shown in Figure E2-32. (T3D30SuspCu01)**

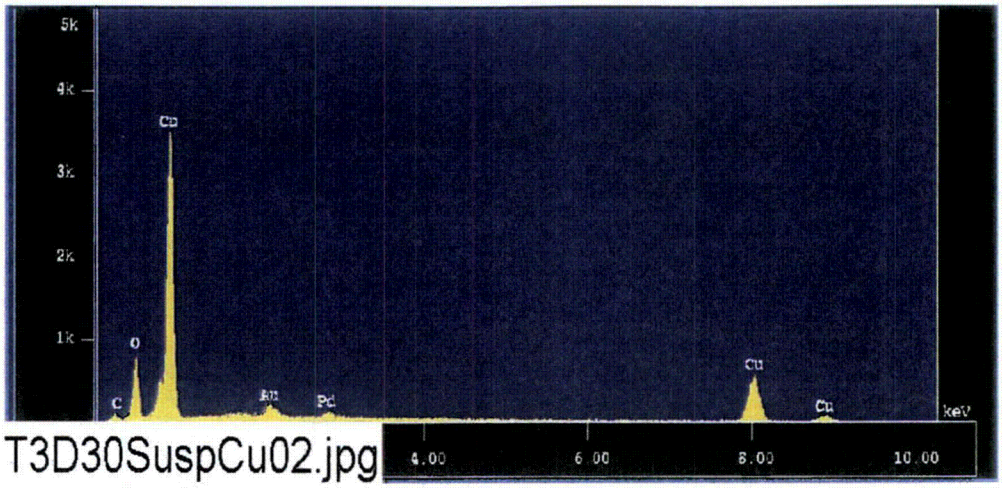


Figure E2-5. **EDS counting spectrum for the concave surface (EDS2) shown in Figure E2-32. (T3D30SuspCu02)**

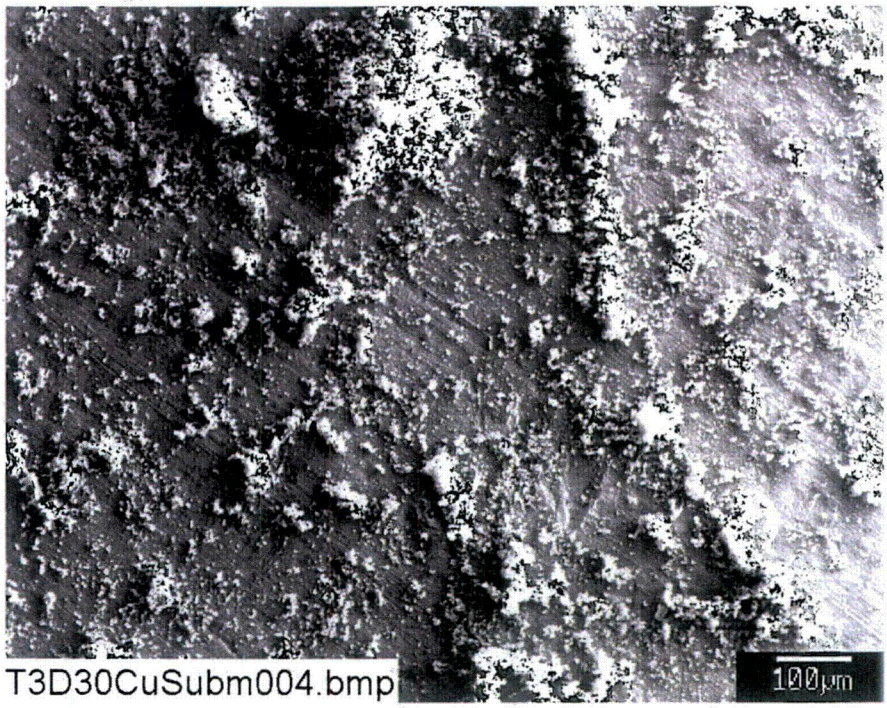


Figure E2-6. **SEM image magnified 100 times for a Test #3, Day-30 submerged copper coupon. (T3D30CuSubm004)**

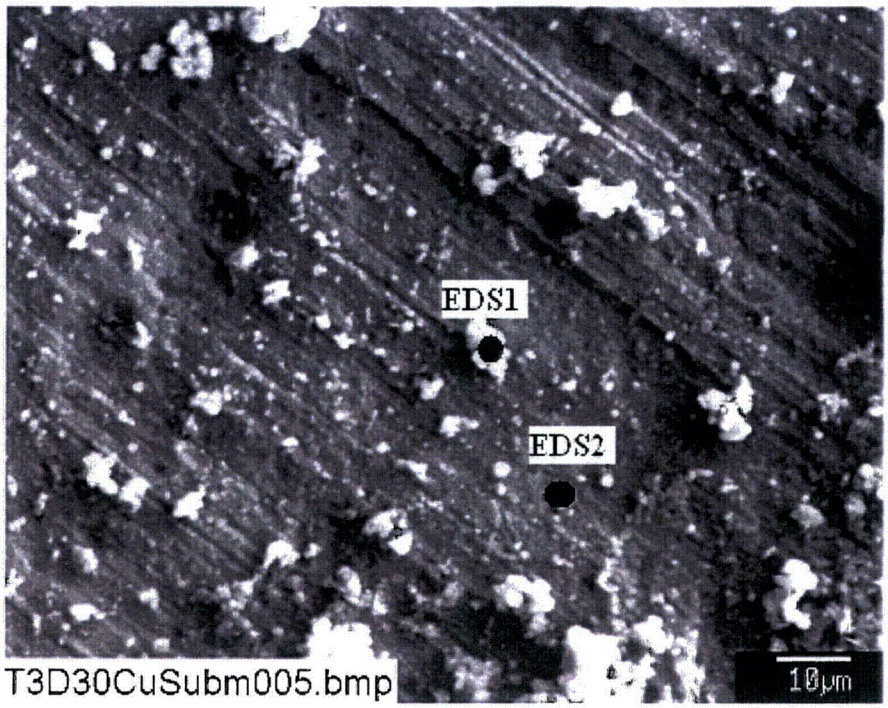


Figure E2-7. Annotated SEM image magnified 1000 times for a Test #3, Day-30 submerged copper coupon. (T3D30CuSubm005)

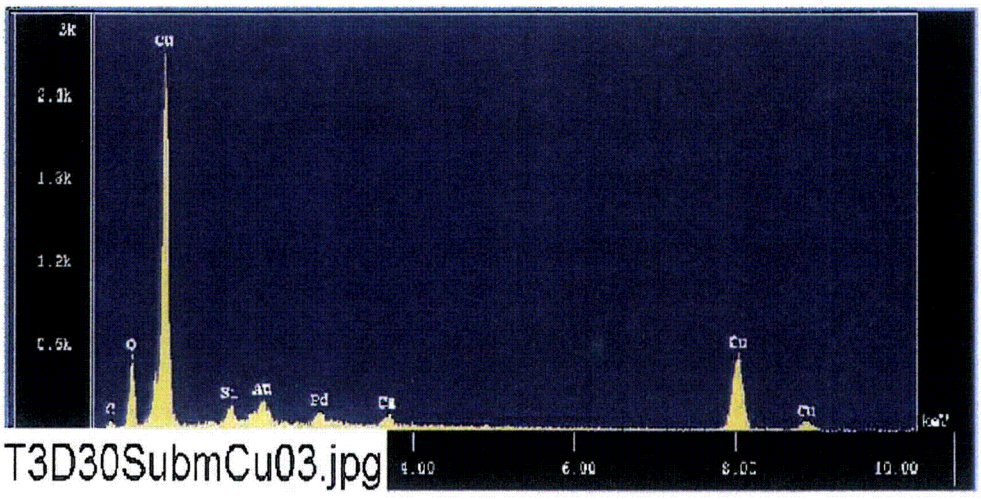


Figure E2-8. EDS counting spectrum for the white particles (EDS1) shown in Figure E2-7. (T3D30SubmCu03)

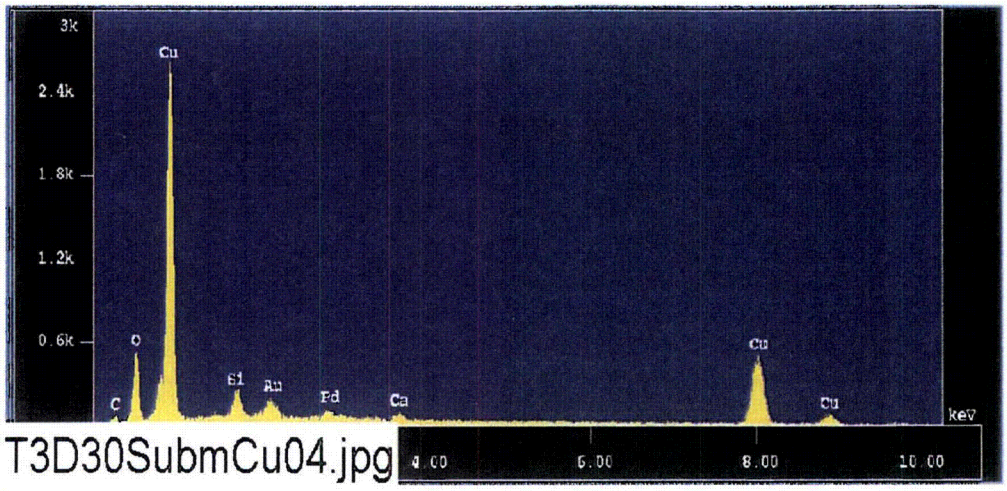


Figure E2-9. **EDS counting spectrum for the dark surface (EDS2) shown in Figure E2-7. (T3D30SubmCu04)**

Appendix E3

SEM/EDS Data for Test #3, Day-30 Galvanized Steel Coupons

Figures

Tables

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

The coupon samples were collected on May 5, 2005 (the date Test #3 was shut down), and subsequently examined by SEM/EDS. The galvanized steel coupon samples were dried in air before coating 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. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

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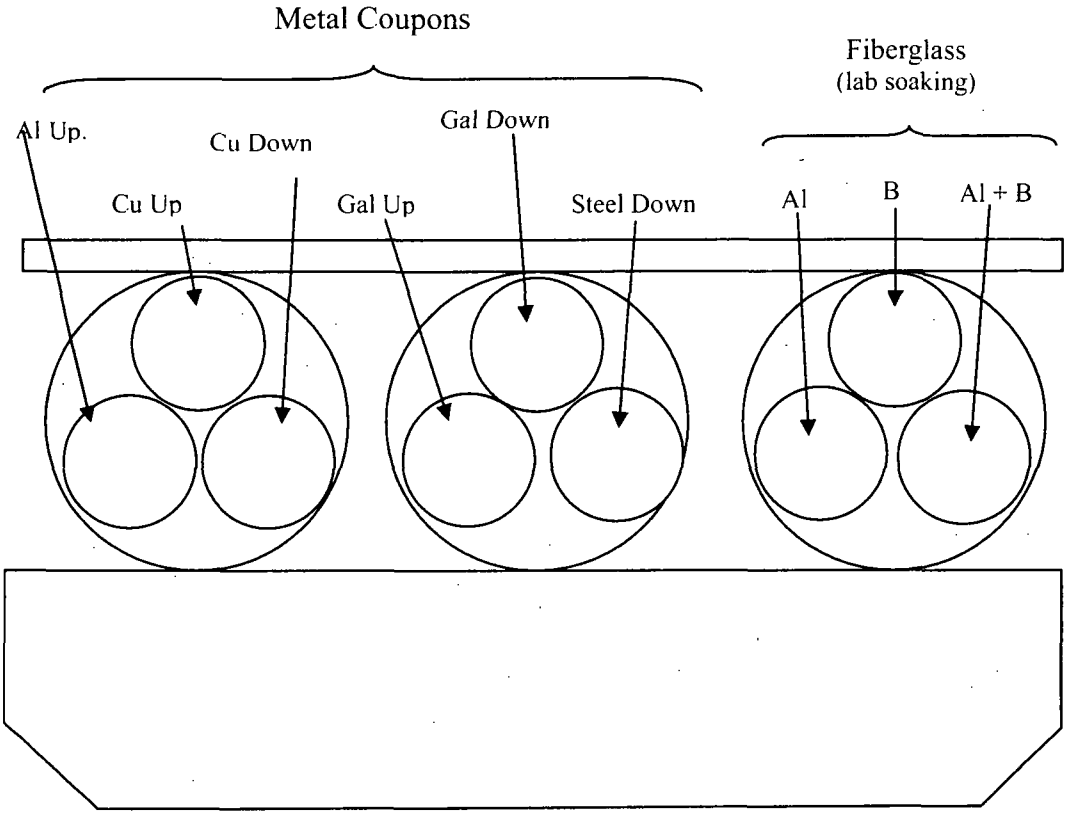
Test #3, Day-30 Metal Coupons



**Coat with Gold

Suspended Gal-Steel

Image:	T3D30GalSteelSusp012	100 ×		Figure E3-1
	T3D30GalSteelSusp013	1000 ×		Figure E3-2
	T3D30GalSteelSusp014	250 ×	Backscattered image	Figure E3-3
	T3D30GalSteelSusp014	250 ×	Annotated backscatter SEM	Figure E3-4
EDS:	T3D30SuspGal09		White spot on 014	Figure E3-5
Image:	T3D30GalSteelSusp012	100 ×	Annotated SEM image	Figure E3-6
EDS:	T3D30SuspGal10		Egg shaped particle on 014	Figure E3-7
	T3D30SuspGal12		Smooth surface on 012	Figure E3-8
	T3D30SuspGal11		Porous irregular shape on 014	Figure E3-9

Submerged Gal-Steel

Image:	T3D30GalSteelSubm009	100 ×		Figure E3-10
	T3D30GalSteelSubm010	1000 ×		Figure E3-11
	T3D30GalSteelSubm011	100 ×	Backscattered image	Figure E3-12
	T3D30GalSteelSubm009	100 ×	Annotated SEM image	Figure E3-13
EDS:	T3D30SubmGal07		White surface shown in 009	Figure E3-14
	T3D30SubmGal08		Dark surface shown in 009	Figure E3-15

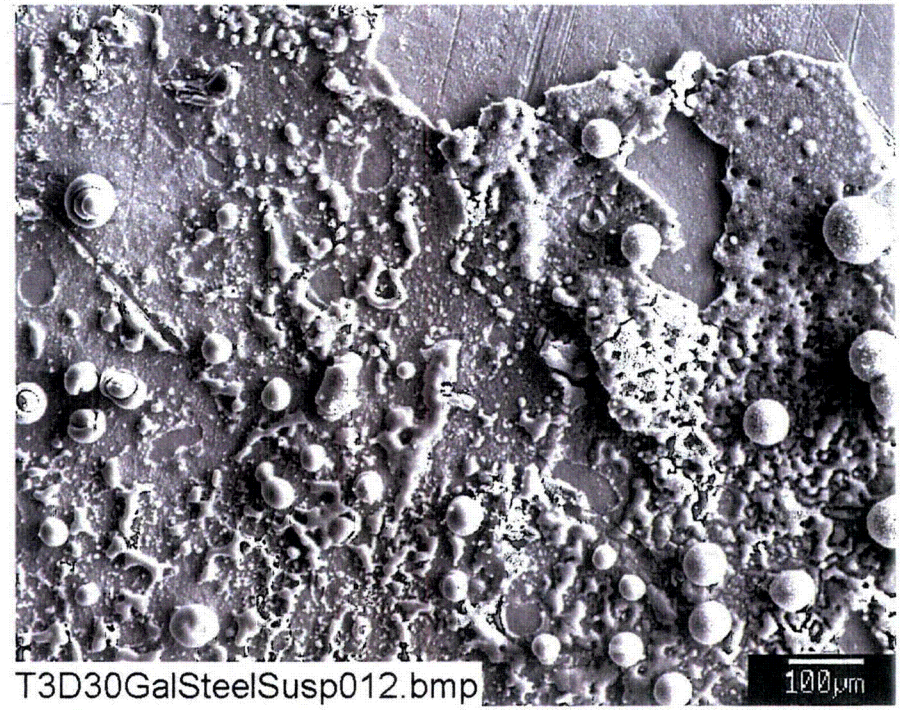


Figure E3-1. **SEM image magnified 100 times for a Test #3, Day-30 suspended galvanized steel coupon. (T3D30GalSteelSusp012)**

Figure E3-2. **SEM image magnified 1000 times for a Test #3, Day-30 suspended galvanized steel coupon. (T3D30GalSteelSusp013)**

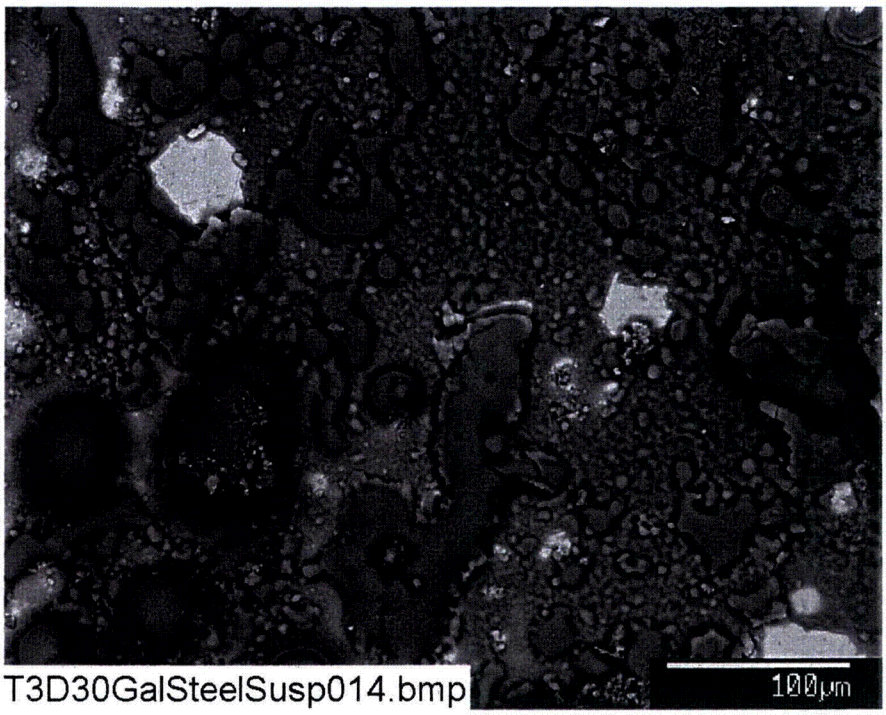


Figure E3-3. **Backscattered SEM image magnified 250 times for a Test #3, Day-30 suspended galvanized steel coupon. (T3D30GalSteelSusp014)**

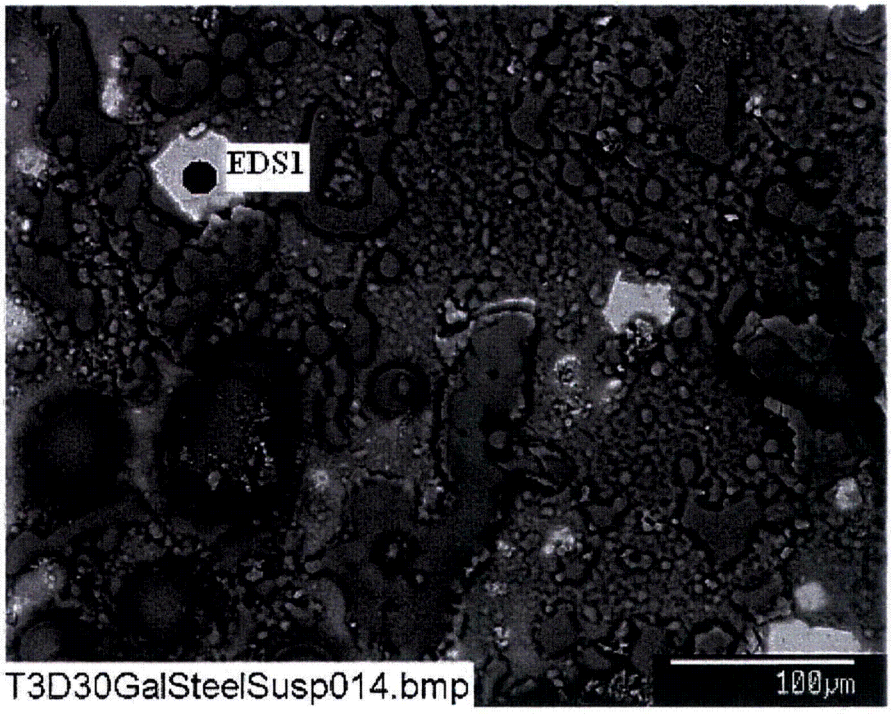


Figure E3-4. **Annotated backscattered SEM image magnified 250 times for a Test #3, Day-30 suspended galvanized steel coupon. (T3D30GalSteelSusp014)**

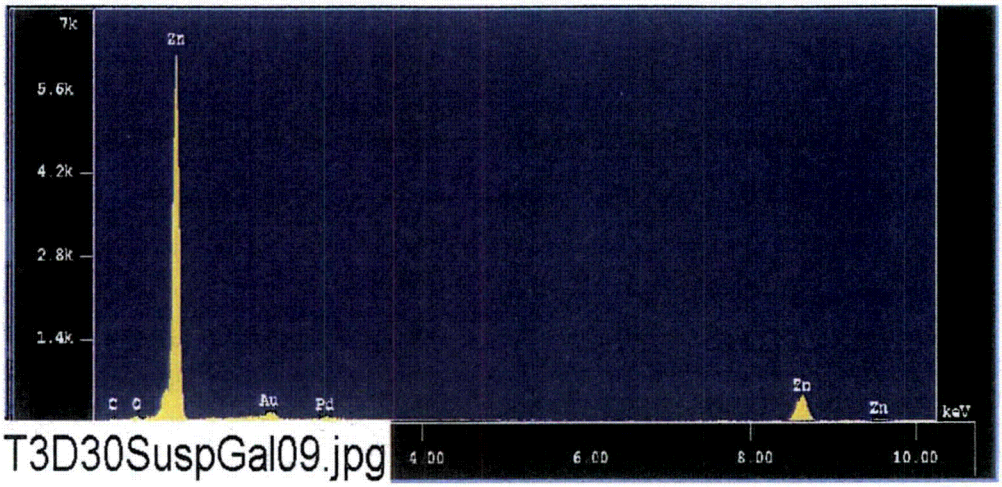


Figure E3-5. **EDS counting spectrum for the white spot (EDS1) shown in Figure E3-4. (T3D30SuspGal09)**

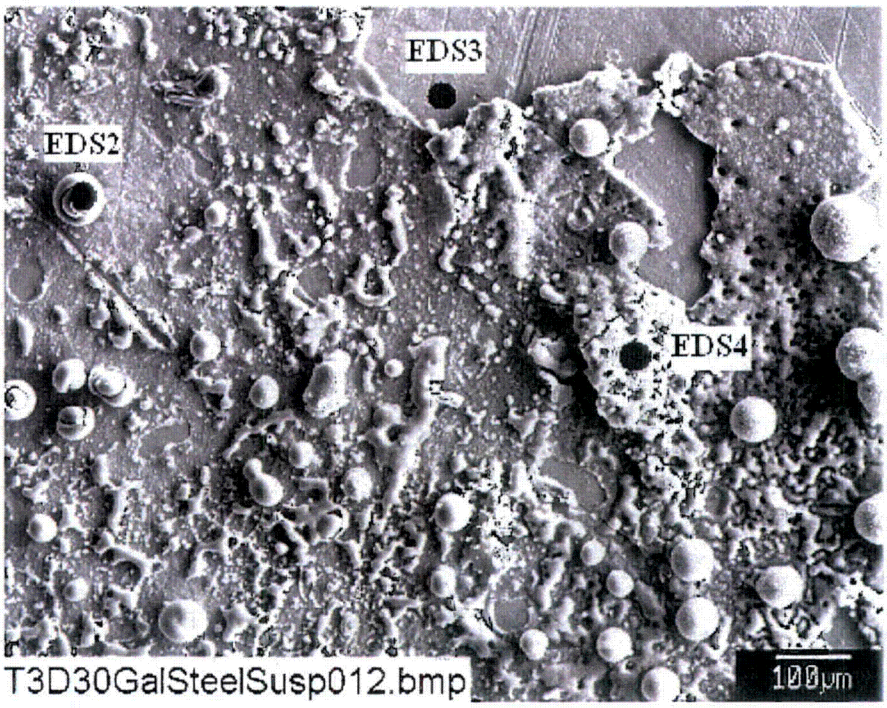


Figure E3-6. **Annotated SEM image magnified 100 times for a Test #3, Day-30 suspended galvanized steel coupon. (T3D30GalSteelSusp012)**

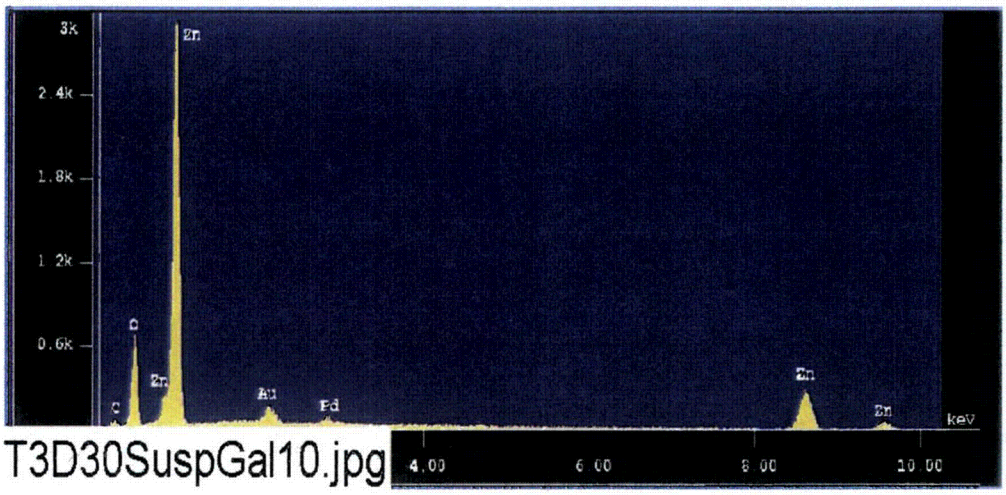


Figure E3-7. **EDS counting spectrum for an egg shaped particle (EDS2) shown in Figure E3-6 (T3D30SuspGal10)**

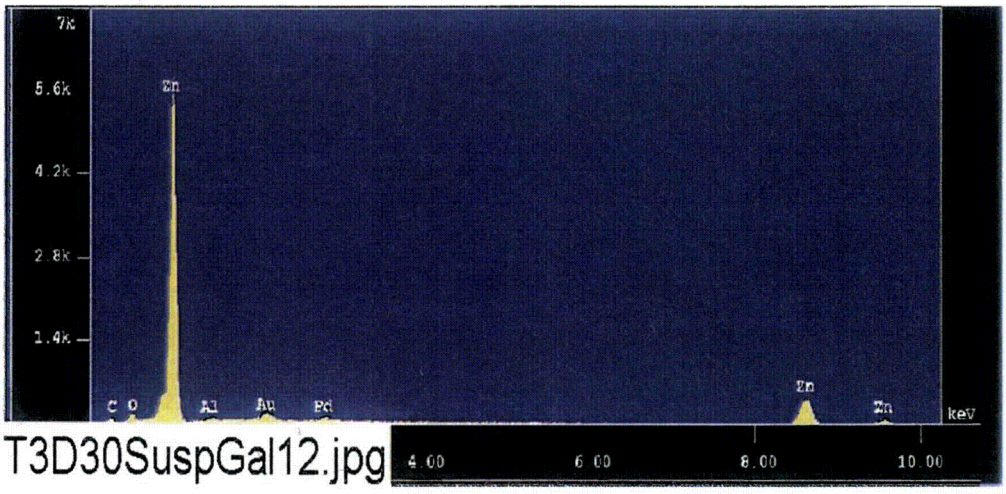


Figure E3-8. **EDS counting spectrum for the smooth surface (EDS3) shown in Figure E3-6. (T3D30SuspGal12)**

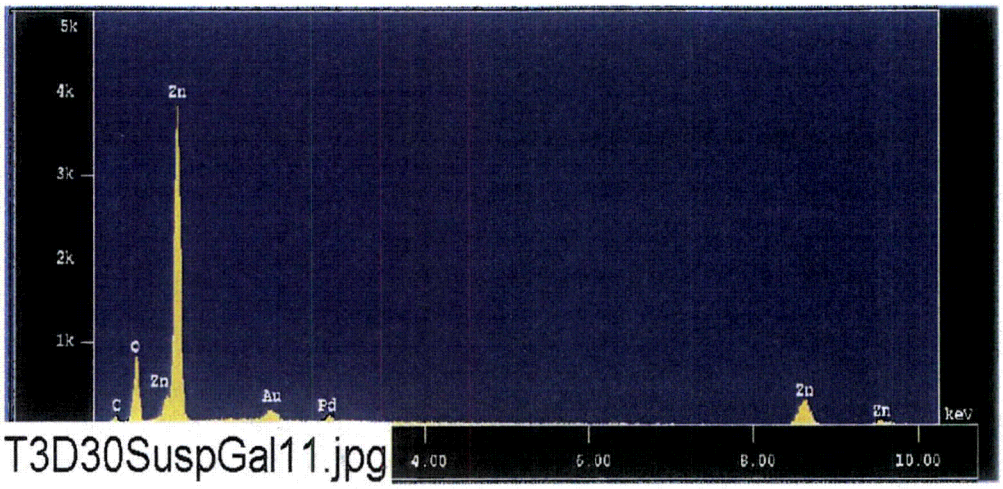


Figure E3-9. **EDS counting spectrum for a porous irregular particle (EDS4) shown in Figure E3-6. (T3D30SuspGal11)**

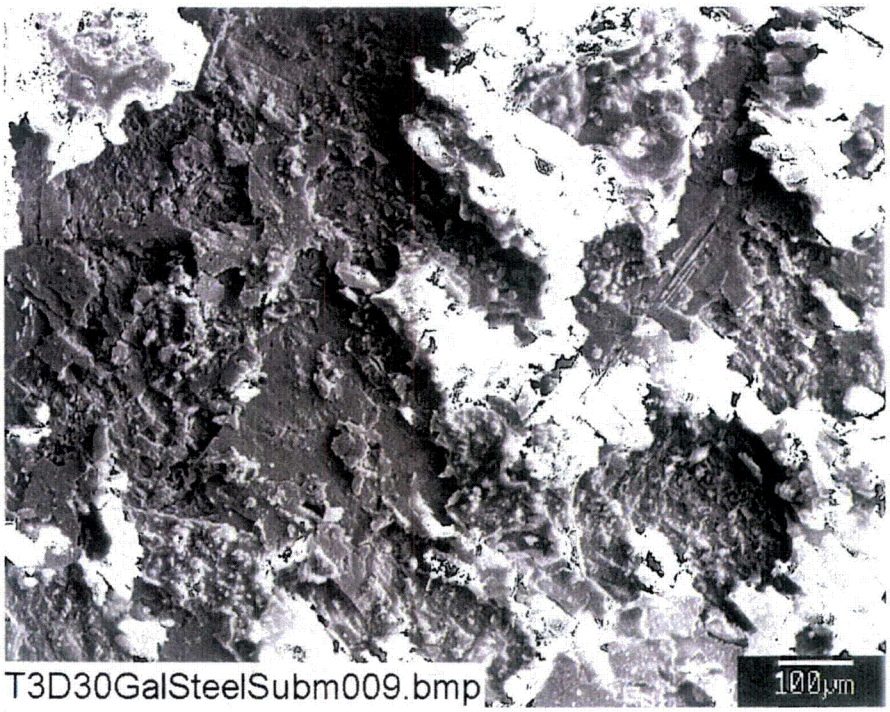


Figure E3-10. **SEM image magnified 100 times for a Test #3, Day-30 submerged galvanized steel coupon. (T3D30GalSteelSubm009)**

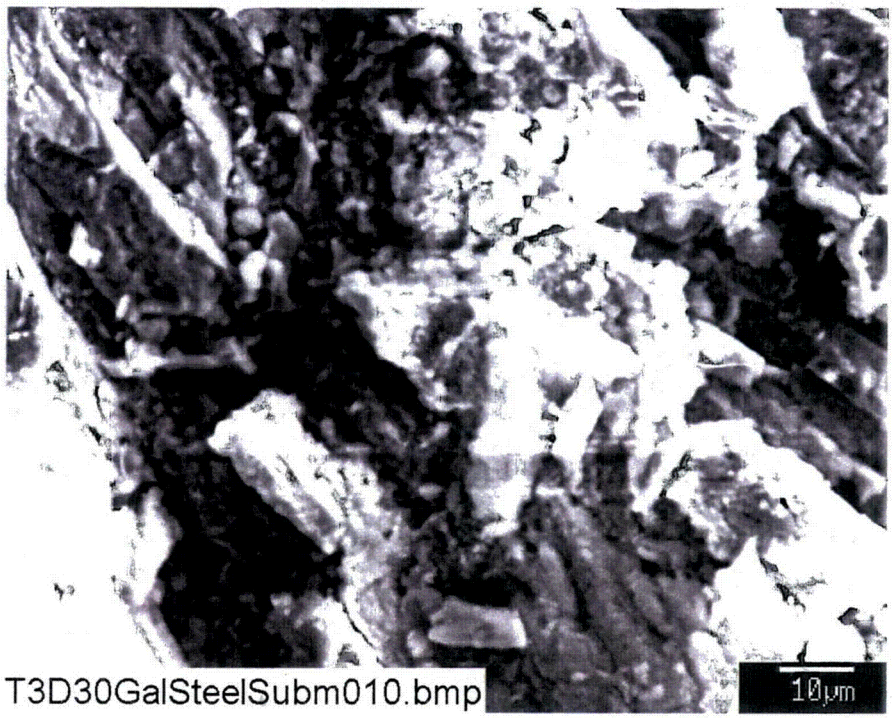


Figure E3-11. **SEM image magnified 1000 times for a Test #3, Day-30 submerged galvanized steel coupon. (T3D30GalSteelSubm010)**

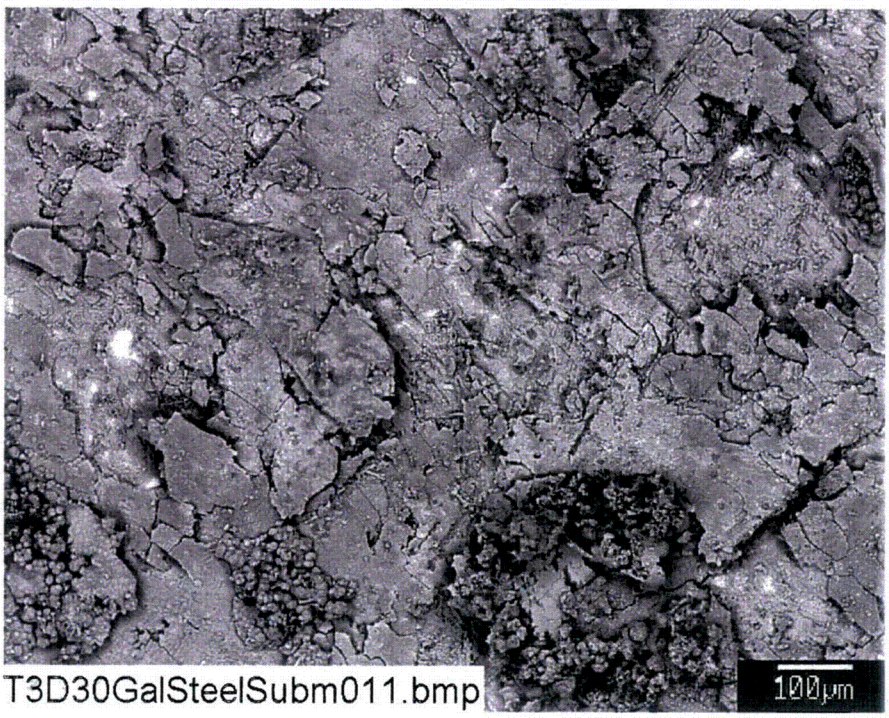


Figure E3-12. **Backscattered SEM image magnified 100 times for a Test #3, Day-30 submerged galvanized steel coupon. (T3D30GalSteelSubm011)**

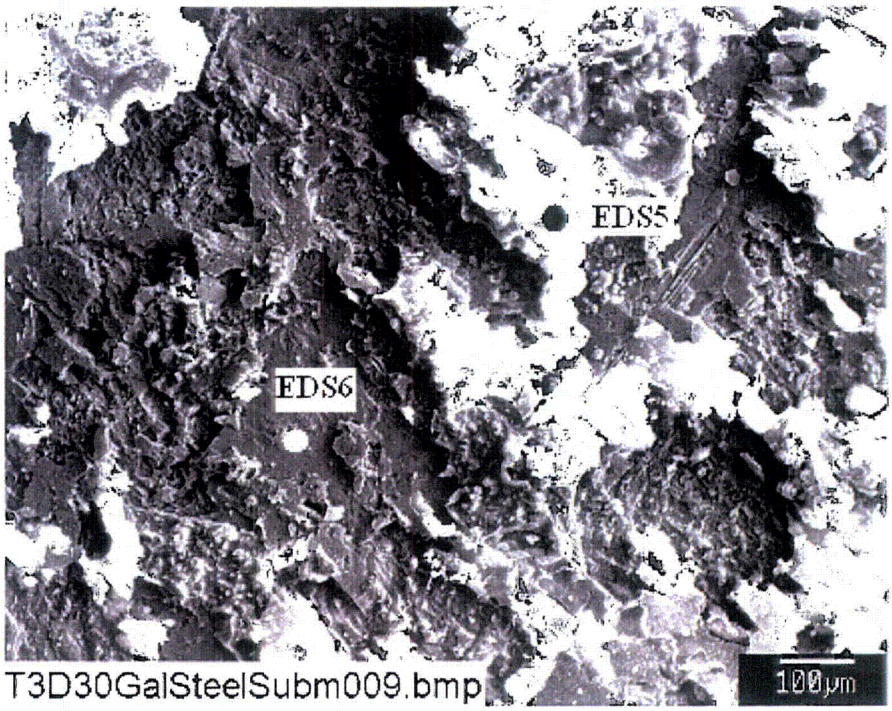


Figure E3-13. **Annotated SEM image magnified 100 times for a Test #3, Day-30 submerged galvanized steel coupon. (T3D30GalSteelSubm009)**

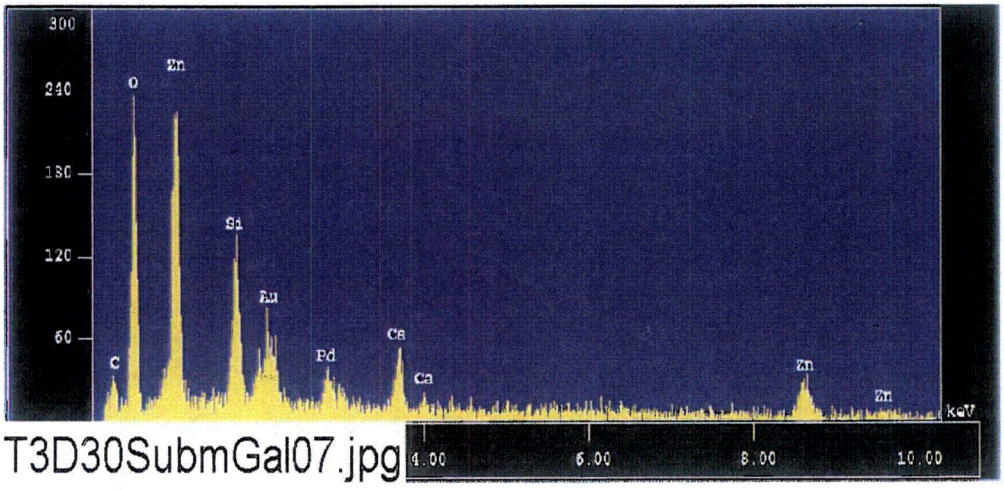


Figure E3-14. **EDS counting spectrum for the white surface (EDS5) shown in Figure E3-13. (T3D30SubmGal07)**

The results from the chemical composition analysis for T3D30SubmGal07 are given in Table E3-1.

Table E3-1. Chemical Compositions for T3D30SubmGal07, Figure E3-14

May 17 2005

Group : NRC
Sample : Test3 ID# : 10
Comment : Submerged Galsteel
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 7.359E-09 A
Stage Point : X=47.136 Y=56.832 Z=10.582
Acq. Date : Tue May 17 12:05:14 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	6.4123	0.0018	1466 / 24
Si K	Normal	1.50- 2.05	1.1657	0.0004	1031 / 33
Ca K	Normal	3.40- 4.30	0.9216	0.0044	396 / 16
Zn K	Normal	8.22-10.03	5.9921	0.0061	396 / 3
C K	Normal	0.09- 0.46	0.0000	0.0000	0 / 42

Chi_square = 7.3085

Element	Mass%	Atomic%	ZAF	Z	A	F
O	39.582	67.4180	0.9868	0.9165	1.0768	1.0000
Si	10.788	10.4670	1.4794	0.9181	1.6115	0.9999
Ca	5.422	3.6863	0.9405	0.9224	1.0205	0.9991
Zn	44.208	18.4286	1.1794	1.1810	0.9986	1.0000
C	0.000	0.0000	5.1734	0.9614	5.3811	1.0000
Total	100.000	100.0000				

Normalization factor = 6.2556

C	1.633	4.8654	5.2838	0.9099	5.8071	1.0000
P	2.298	2.6548	1.0399	1.0402	0.9998	0.9999
Fe	2.399	1.5375	0.9896	1.0503	1.0060	0.9366
Total	100.000	100.0000				

Normalization factor = 3.2486

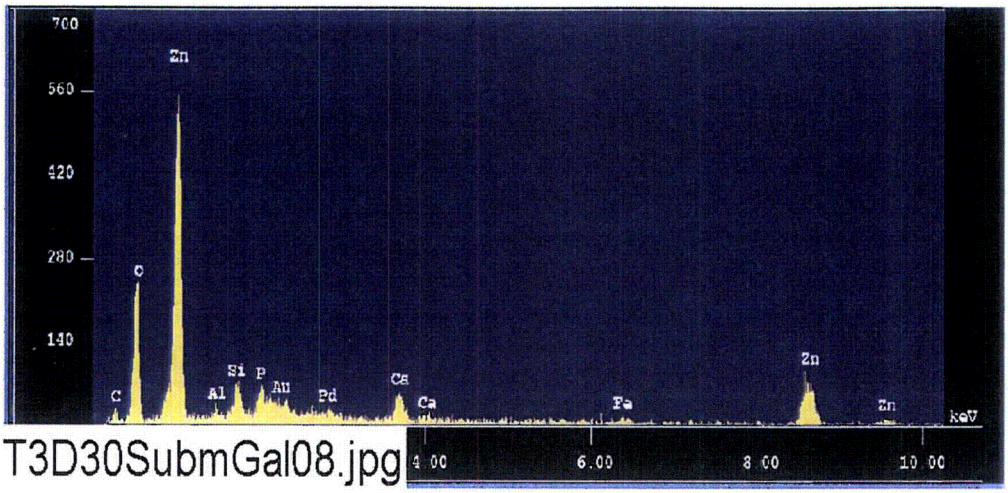


Figure E3-15. **EDS counting spectrum for the dark surface (EDS6) shown in Figure E3-13. (T3D30SubmGal08)**

The results from the chemical composition analysis for T3D30SubmGal08 are given in Table E3-2.

Table E3-2. Chemical Compositions for T3D30SubmGal08, Figure E3-15

May 17 2005

Group : NRC
Sample : Test3 ID# : 11
Comment : Submerged Galsteel dark surface
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 7.561E-09 A
Stage Point : X=47.136 Y=56.832 Z=10.582
Acq. Date : Tue May 17 12:11:13 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	6.9828	0.0019	1640 / 20
Al K	Normal	1.26- 1.78	0.0611	0.0004	60 / 29
Si K	Normal	1.50- 2.05	0.4753	0.0003	432 / 28
Ca K	Normal	3.40- 4.30	0.8741	0.0049	386 / 10
Zn K	Normal	8.22-10.03	18.5840	0.0084	1260 / 2
C K	Normal	0.09- 0.46	0.0951	0.0003	33 / 45
P K	Normal	1.75- 2.38	0.6802	0.0018	381 / 36
Fe K	Normal	6.00- 7.44	0.7464	0.0009	134 / 10

Chi_square = 5.2540

Element	Mass%	Atomic%	ZAF	Z	A	F
O	21.727	48.5961	0.9578	0.8671	1.1047	0.9999
Al	0.371	0.4925	1.8718	0.8784	2.1318	0.9996
Si	2.465	3.1407	1.5964	0.8673	1.8413	0.9996
Ca	2.557	2.2833	0.9006	0.8692	1.0376	0.9986
Zn	66.549	36.4297	1.1023	1.1023	1.0000	1.0000
C	1.633	4.8654	5.2838	0.9099	5.8071	1.0000
P	2.298	2.6548	1.0399	1.0402	0.9998	0.9999
Fe	2.399	1.5375	0.9896	1.0503	1.0060	0.9366
Total	100.000	100.0000				

Normalization factor = 3.2486

Appendix E4

SEM/EDS Data for Test #3, Day-30 Steel Coupons

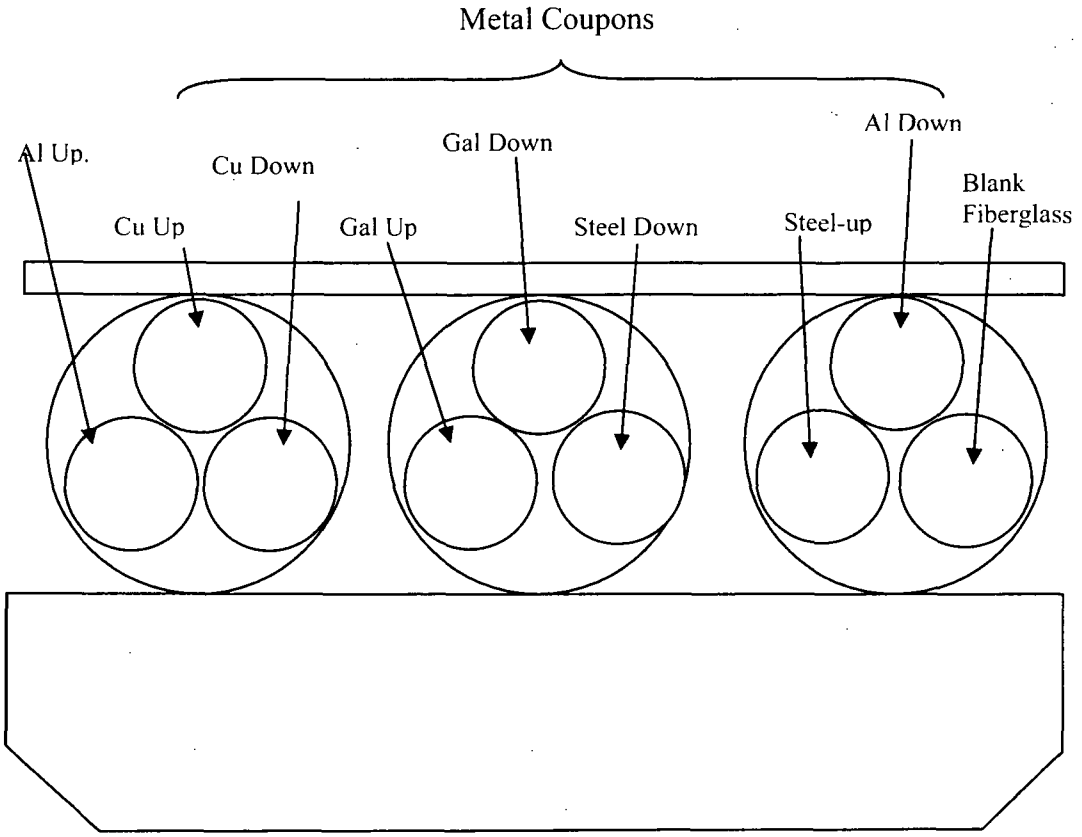
Figures

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

The coupon samples were collected on the date that Test #3 was shut down, May 5, 2005. The steel coupon samples were dried in air before coating with Au/Pd for SEM examination. SEM results present the surface condition of the steel coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Laboratory session from May 17, 2005.
Test #3, Day-30 Metal Coupons



**Coat with Gold

Suspended Steel

Image:	T3D30SteelSusp033	100 ×	SEM image	Figure E4-1
	T3D30SteelSusp033	100 ×	Annotated SEM image	Figure E4-2
EDS:	T3D30SuspSteel19		Convex on 033	Figure E4-3
	T3D30SuspSteel21		White flat surface on 033	Figure E4-4
Image:	T3D30SteelSusp034	100 ×	Backscattered	Figure E4-5
	T3D30SteelSusp034	100 ×	Annotated backscatter image	Figure E4-6
EDS:	T3D30SuspSteel20		Dark particle on 034	Figure E4-7
Image:	T3D30SteelSusp035	1000 ×		Figure E4-8

Submerged Steel

Image:	T3D30SteelSubm015	100 ×		Figure E4-9
	T3D30SteelSubm016	100 ×	Backscattered image	Figure E4-10
	T3D30SteelSubm016	100 ×	Annotated backscattered image	Figure E4-11
EDS:	T3D30SubmSteel13		Dark spot on 016	Figure E4-12
	T3D30SubmSteel14		White spot on 016	Figure E4-13
Image:	T3D30SteelSubm017	1000 ×		Figure E4-14

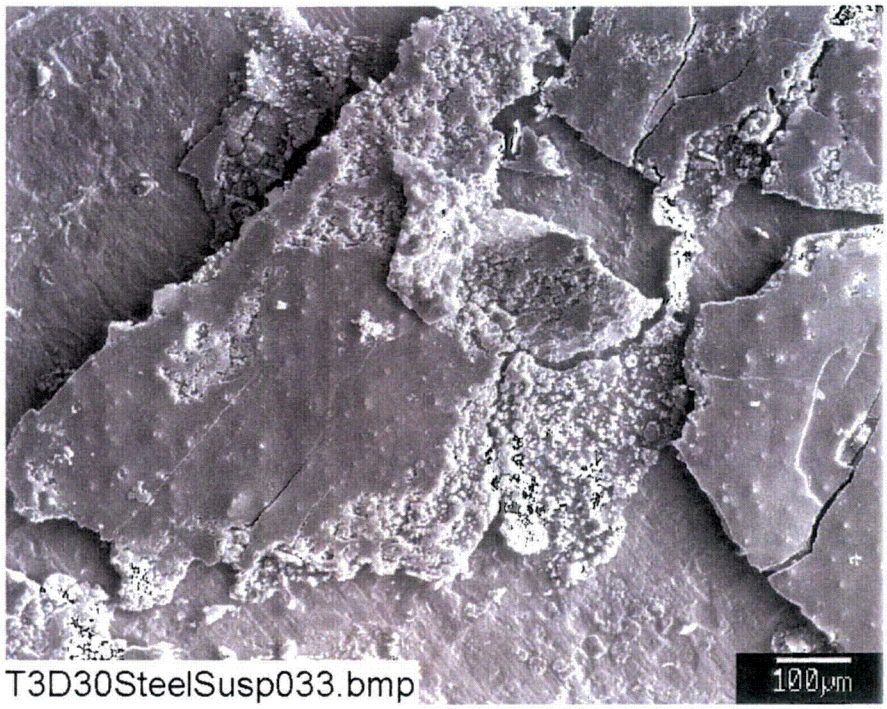


Figure E4-1. **SEM image magnified 100 times for a Test #3, Day-30 suspended carbon steel coupon. (T3D30SteelSusp033)**

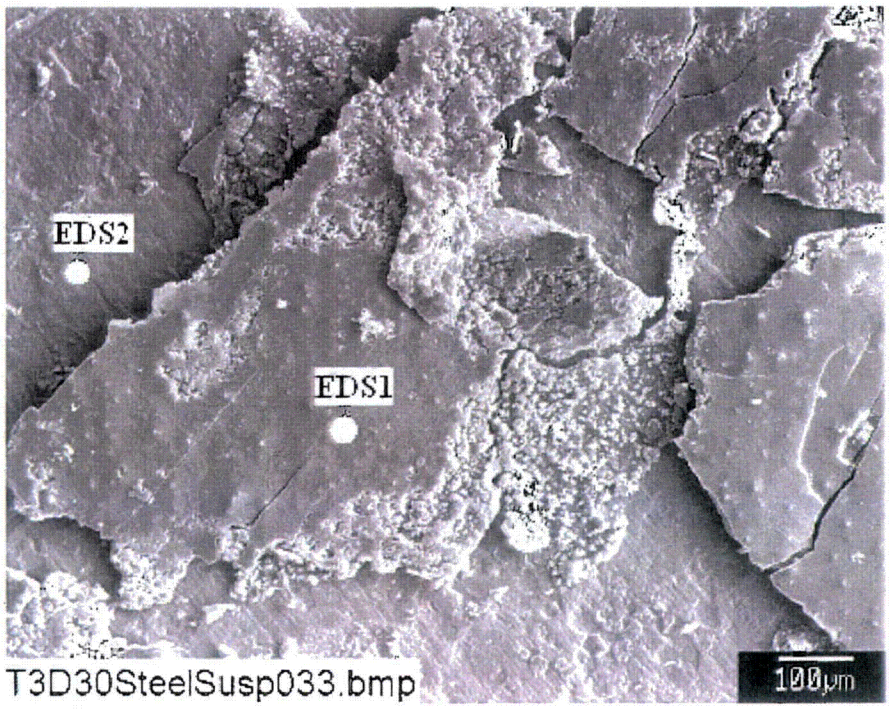


Figure E4-2. **Annotated SEM image magnified 100 times for a Test #3, Day-30 suspended carbon steel coupon. (T3D30SteelSusp033)**

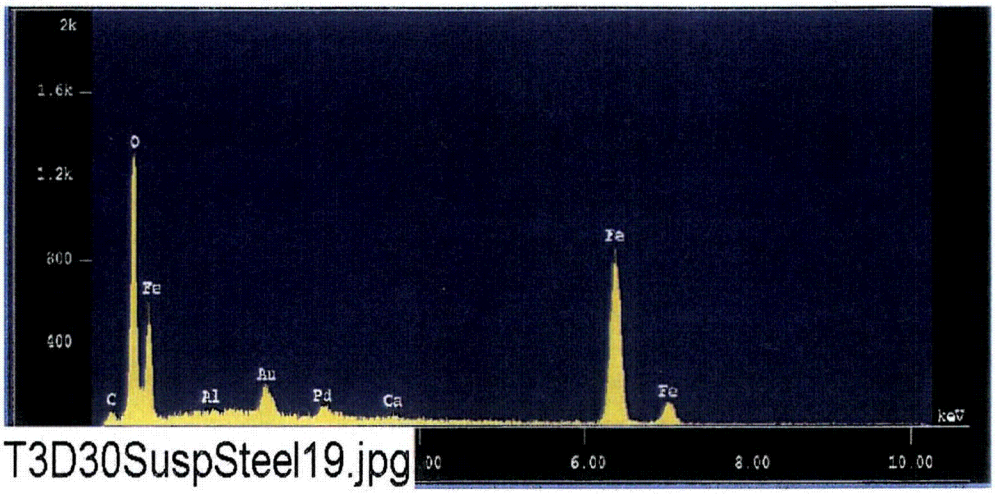


Figure E4-3. **EDS counting spectrum for the convex surface (EDS1) shown in Figure E4-2. (T3D30SuspSteel19)**

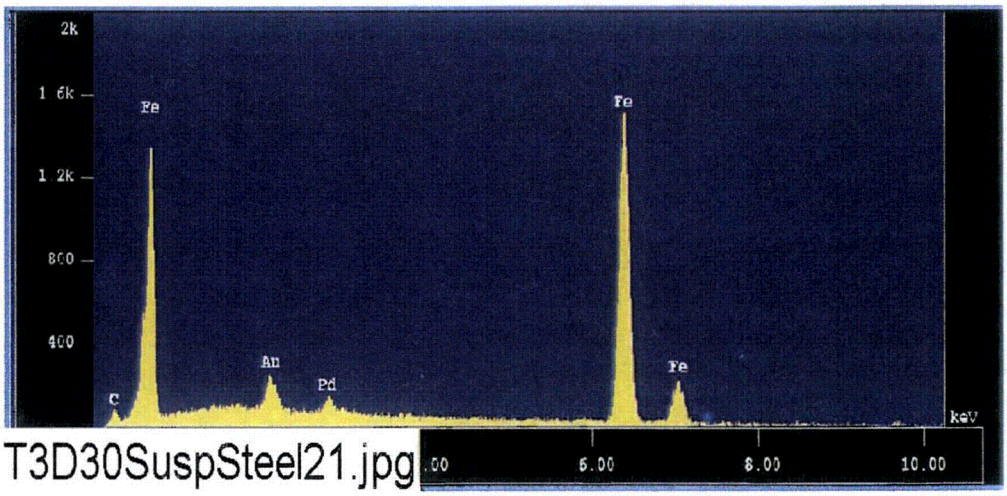


Figure E4-4. **EDS counting spectrum for the lower surface (EDS2) shown in Figure E4-2. (T3D30SuspSteel21)**

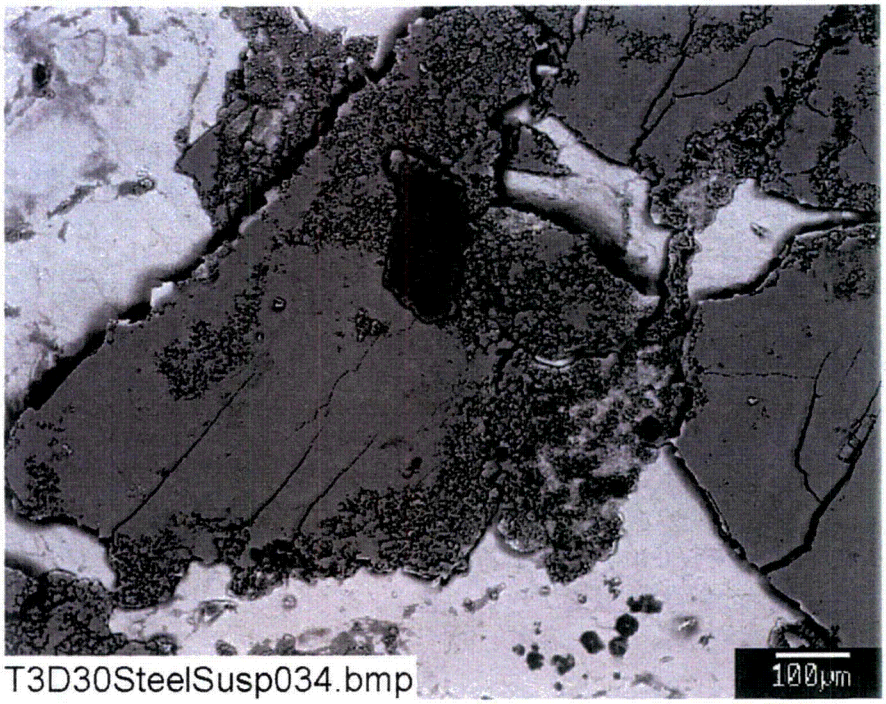


Figure E4-5. **Backscattered SEM image magnified 100 times for a Test #3, Day-30 suspended steel coupon. (T3D30SteelSusp034)**

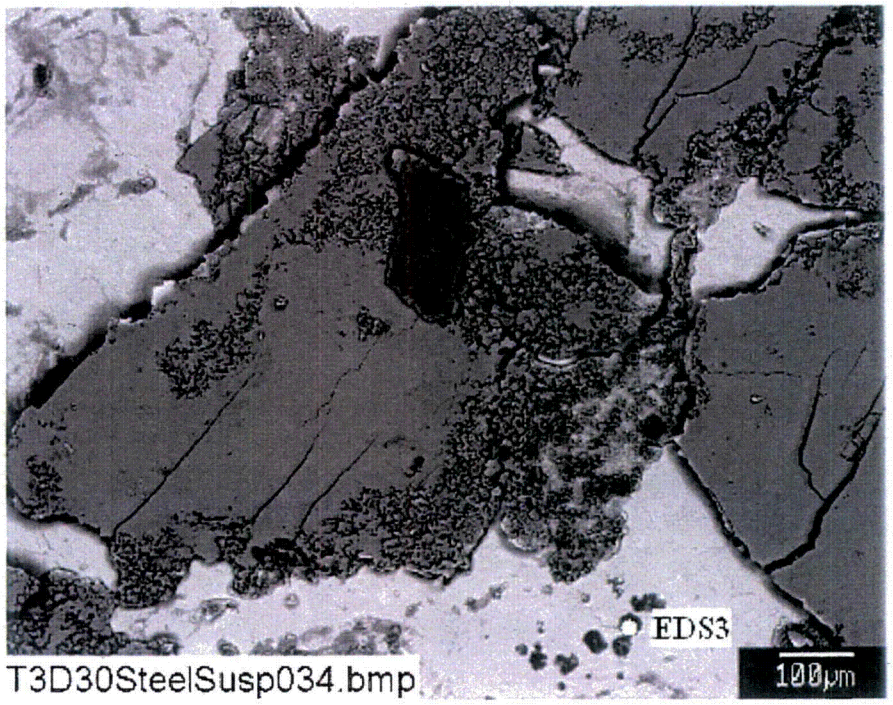


Figure E4-6. **Annotated backscattered SEM image magnified 100 times for a Test #3, Day-30 suspended steel coupon. (T3D30SteelSusp034)**

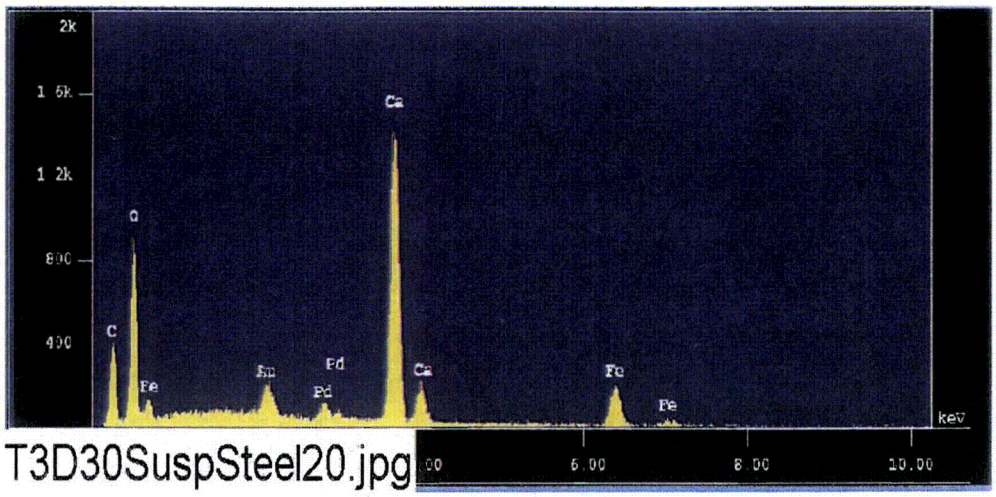


Figure E4-7. **EDS counting spectrum for a dark particle close to the lower edge (EDS3) of Figure E4-6. (T3D30SuspSteel20)**

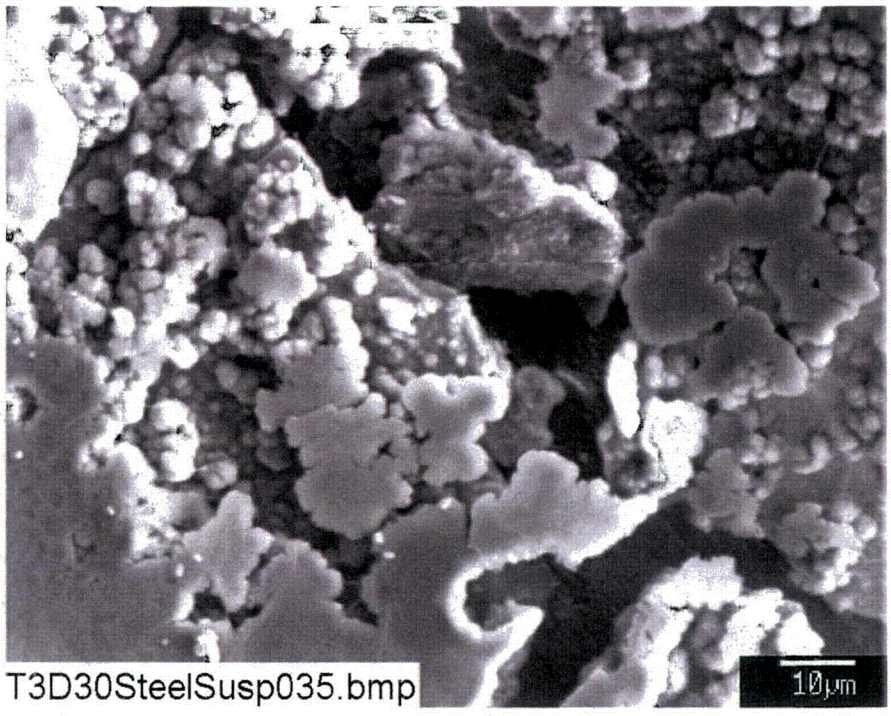


Figure E4-8. **SEM image magnified 1000 times for a Test #3, Day-30 suspended carbon steel coupon. (T3D30SteelSusp035)**

Figure E4-9. **SEM image magnified 100 times for a Test #3, Day-30 submerged carbon steel coupon. (T3D30SteelSubm015)**

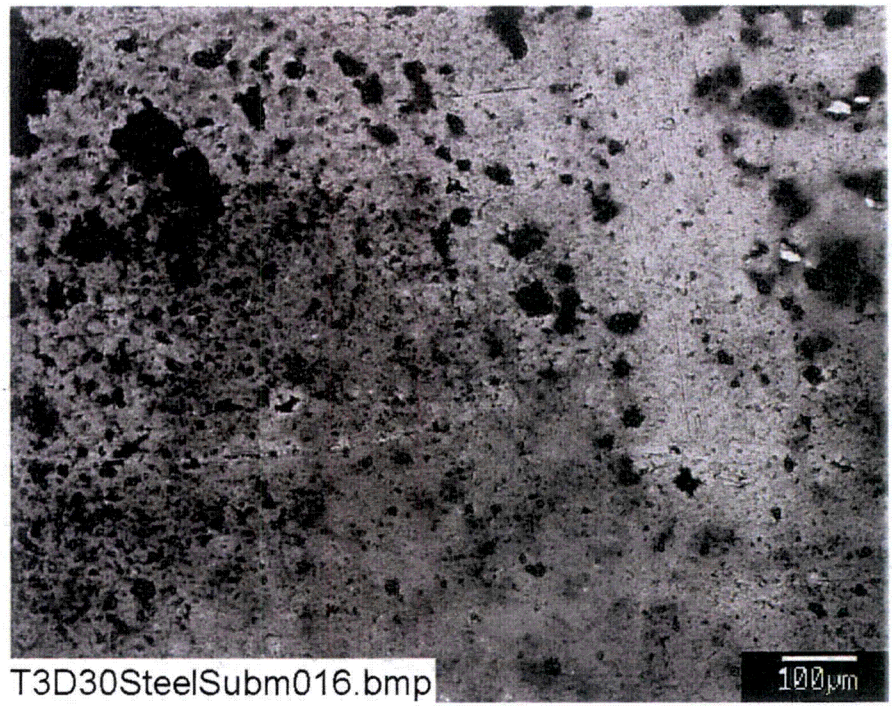


Figure E4-10. **Backscattered SEM image magnified 100 times for a Test #3, Day-30 submerged carbon steel coupon. (T3D30SteelSubm016)**

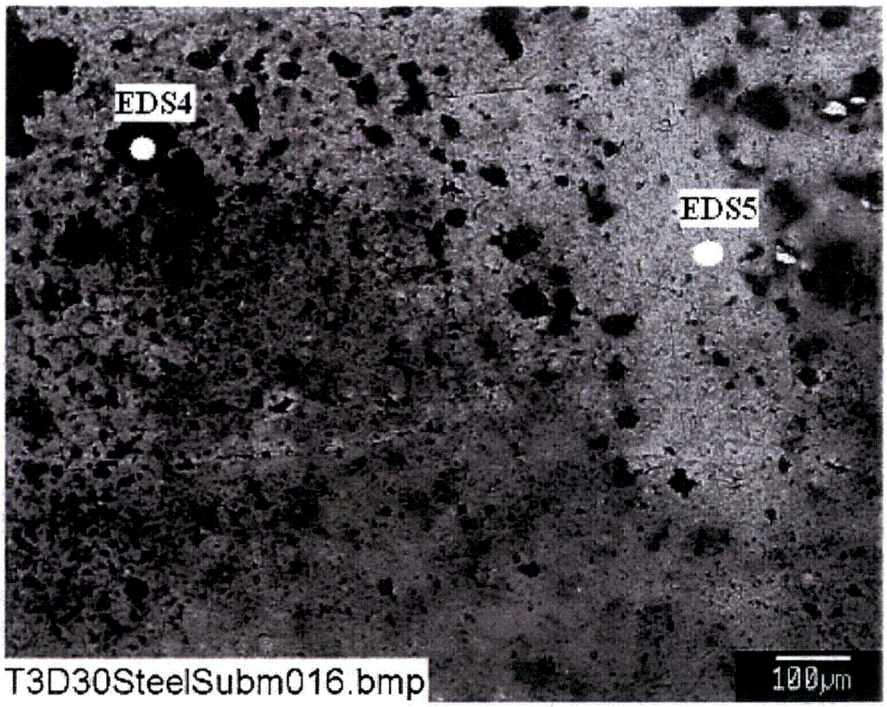


Figure E4-11. **Annotated backscattered SEM image magnified 100 times for a Test #3, Day-30 submerged carbon steel coupon. (T3D30SteelSubm016)**

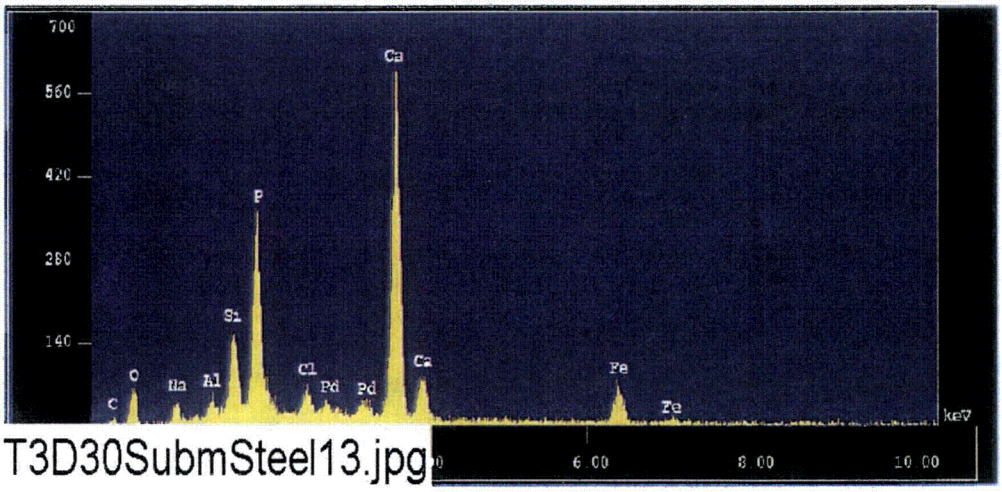


Figure E4-12. **EDS counting spectrum for a dark spot (EDS4) shown in Figure E4-11. (T3D30SubmSteel13)**

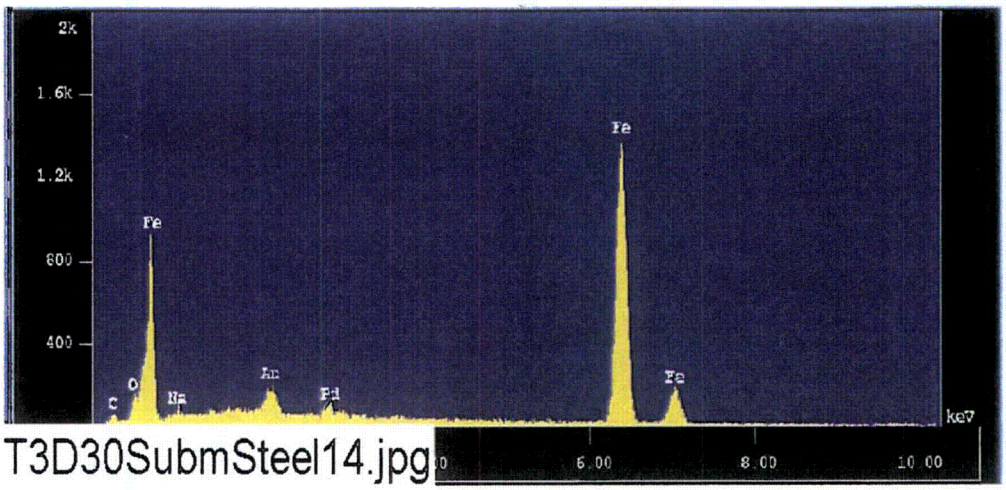


Figure E4-13. **EDS counting spectrum for a light spot (EDS5) shown in Figure E4-11. (T3D30SubmSteel14)**

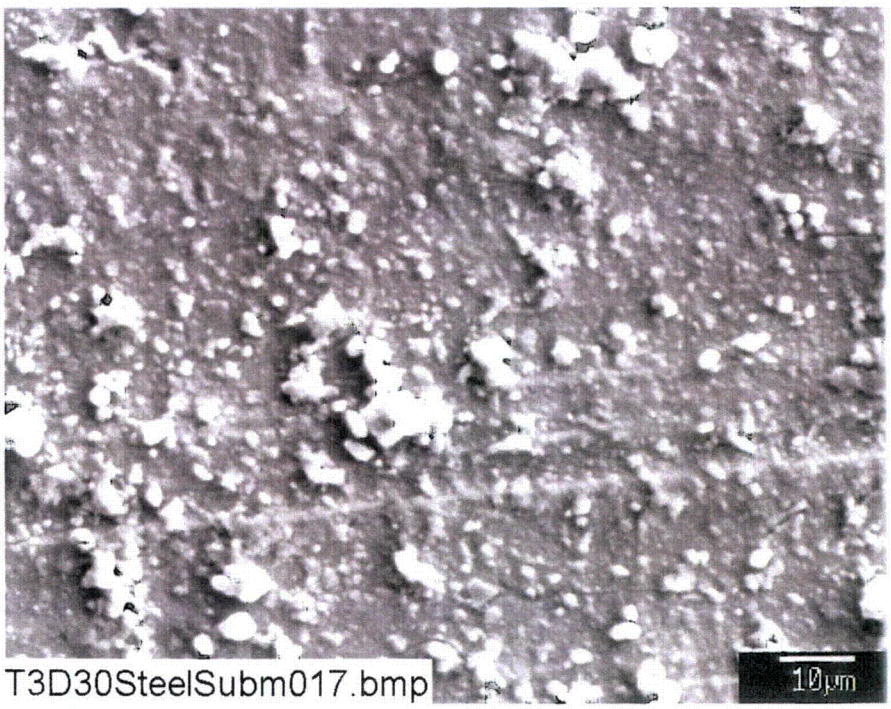


Figure E4-14. **SEM image magnified 1000 times for a Test #3, Day-30 submerged carbon steel coupon. (T3D30SteelSubm017)**

Appendix F

SEM/EDS Data for Test #3, Day-30 Flow Meter

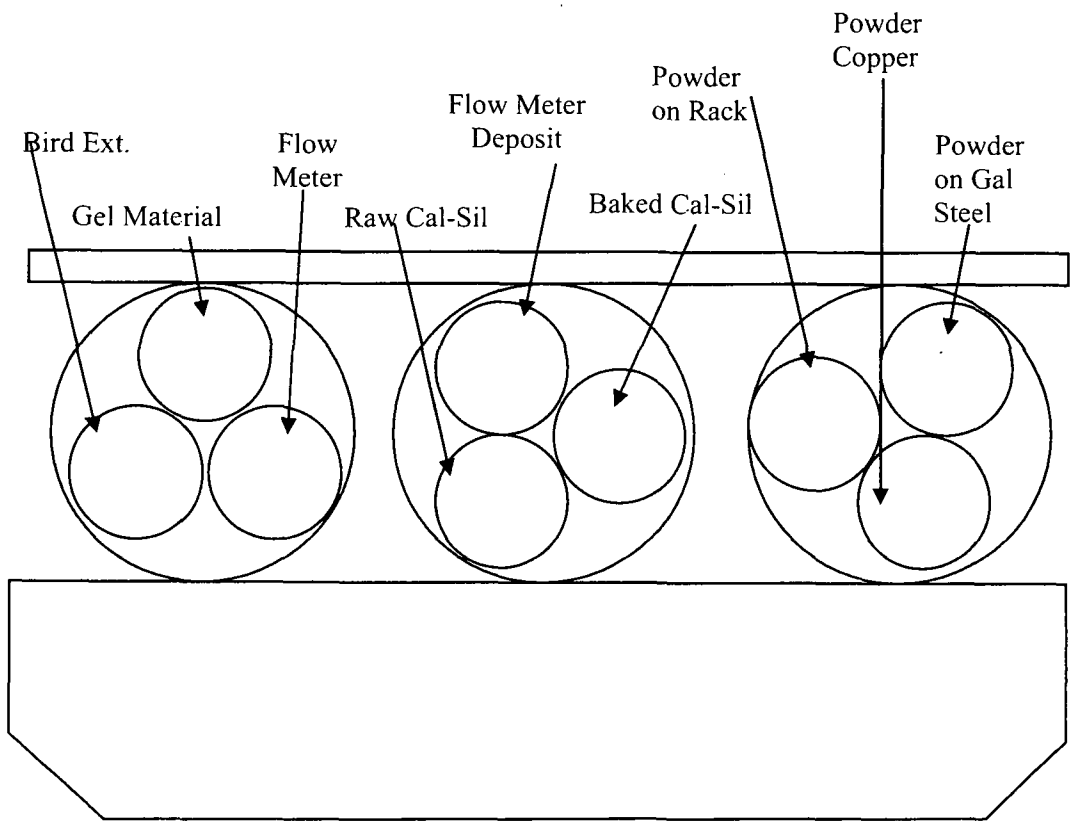
Figures

Tables

In ICET Test #3, significant amounts of debris and precipitates were found within the flow meter. SEM/EDS analysis was performed to examine the composition of the debris trapped in the flow meter, as well as the white precipitates deposited on the inner wall of the flow meter. The debris and the precipitates were collected on May 5, 2005, the date Test #3 was shut down. The samples were dried in air before being coated with Au/Pd for SEM examination. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Laboratory session from May 9, 2005.
Test #3, Day-30 Flow Meter



Flow Meter Debris

Image:	T3D30FlwMetrDebris005	80 ×	Figure F-1
	T3D30FlwMetrDebris006	600 ×	Figure F-2
EDS:	T3D30FlwDebris03		Figure F-3

Flow Meter Deposits

Image:	T3~Flow Meter	200 ×	Figure F-4
EDS:	T3Deposits08		Figure F-5

Figure F-1. **SEM image magnified 80 times for Test #3, Day-30 debris within the flow meter. (T3D30FlwMetrDebris005)**

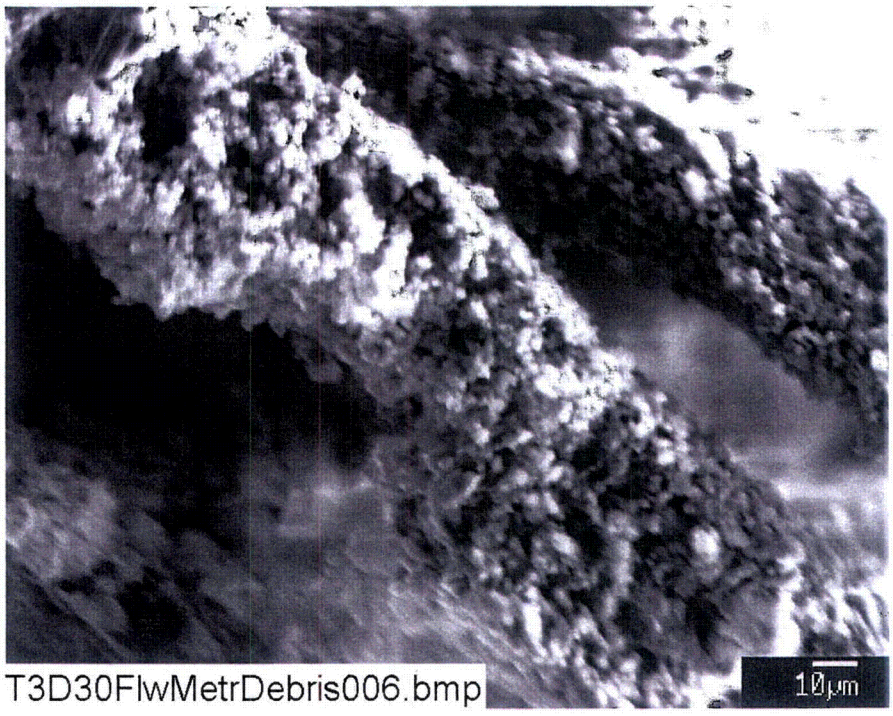


Figure F-2. **SEM image magnified 600 times for Test #3, Day-30 debris within the flow meter. (T3D30FlwMetrDebris006)**

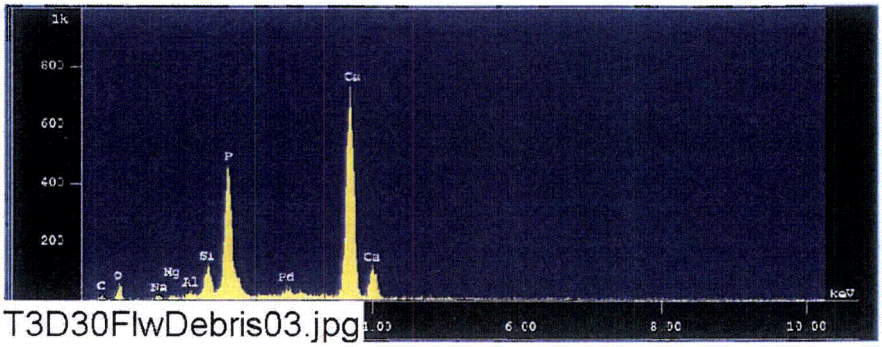


Figure F-3. **EDS counting spectrum for the coatings on the fibers shown in Figure F-2. (T3D30FlwDebris03)**

The results from the chemical composition analysis for T3D30FlwDebris03 are given in Table F-1.

Table F-1. Chemical Compositions for T3D30FlwDebris03, Figure F-3

May 9 2005

Group : NRC
Sample : T3D30 ID# : 3
Comment : Flowmeter Debris
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.596E-09 A
Stage Point : X=77.422 Y=68.992 Z=12.516
Acq. Date : Mon May 9 12:10:19 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	1.1291	0.0010	422 / 12
Si K	Normal	1.50- 2.05	0.6220	0.0004	899 / 142
P K	Normal	1.75- 2.38	4.6152	0.0040	4117 / 58
Ca K	Normal	3.39- 4.30	12.9947	0.0033	9129 / 16
C K	Normal	0.09- 0.46	0.1025	0.0001	57 / 9
Pd L	Normal	2.22- 3.81	0.5748	0.0010	419 / 35
Al K	Normal	1.19- 1.83	0.0834	0.0002	129 / 24
Na K	Normal	0.81- 1.27	0.0806	0.0004	86 / 10
Mg K	Normal	0.97- 1.57	0.0536	0.0001	84 / 15

Chi_square = 8.9413

Element	Mass%	Atomic%	ZAF	Z	A	F
O	15.028	28.4942	2.9323	0.9460	3.0996	1.0000
Si	3.174	3.4286	1.1244	0.9475	1.1960	0.9922
P	17.384	17.0260	0.8298	1.1366	0.7330	0.9960
Ca	57.802	43.7487	0.9799	0.9630	1.0176	1.0000
C	1.833	4.6300	3.9385	0.9925	3.9689	0.9999
Pd	3.395	0.9679	1.3011	1.3345	0.9949	0.9799
Al	0.487	0.5473	1.2851	0.9696	1.3304	0.9962
Na	0.538	0.7099	1.4707	0.9973	1.4750	0.9999
Mg	0.359	0.4474	1.4751	0.9384	1.5740	0.9987
Total	100.000	100.0000				

Normalization factor = 4.5392

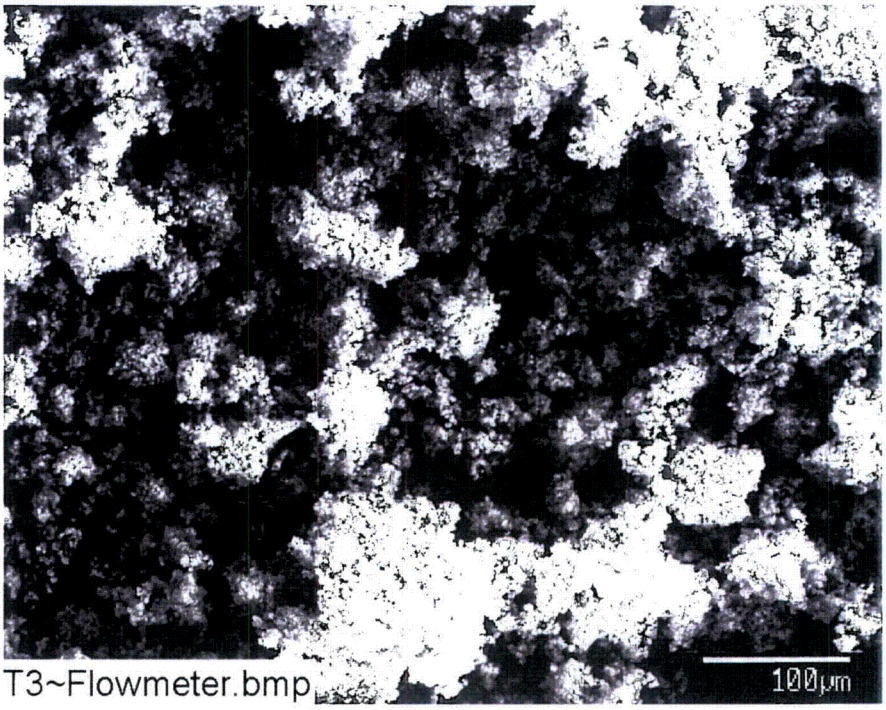


Figure F-4. SEM image magnified 200 times for Test #3, Day-30 deposits on the inner wall of the flow meter. (T3~Flowmeter)

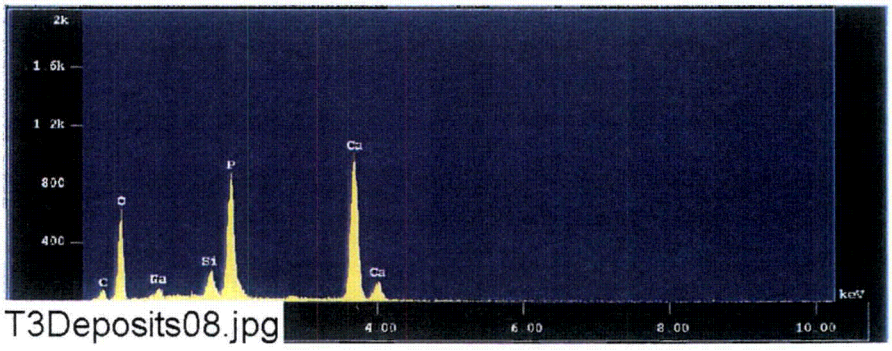


Figure F-5. EDS counting spectrum for the large masses of particulate deposits shown in Figure F-4. (T3Deposits08)

The results from the chemical composition analysis for T3Deposits08 are given in Table F-2.

Table F-2. Chemical Compositions for T3Deposits08, Figure F-5

May 9 2005

Group : NRC
Sample : T3D30 ID# : 8
Comment : Flowmeter Deposits
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.607E-09 A
Stage Point : X=47.897 Y=71.447 Z=12.516
Acq. Date : Mon May 9 14:45:11 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.5235	0.0004	292 / 100
O K	Normal	0.25- 0.77	10.8619	0.0030	4085 / 54
Na K	Normal	0.81- 1.27	0.2843	0.0009	307 / 44
Si K	Normal	1.50- 2.05	1.0559	0.0005	1537 / 258
P K	Normal	1.75- 2.38	8.7448	0.0054	7854 / 123
Ca K	Normal	3.39- 4.30	17.8554	0.0039	12630 / 21

Chi_square = 35.5886

Element	Mass%	Atomic%	ZAF	Z	A	F
C	3.939	7.2812	3.8083	1.0178	3.7418	0.9999
O	43.460	60.3090	2.0251	0.9706	2.0865	1.0000
Na	0.842	0.8134	1.4997	1.0240	1.4645	1.0000
Si	2.376	1.8782	1.1389	0.9739	1.1772	0.9933
P	14.513	10.4028	0.8400	1.1688	0.7203	0.9976
Ca	34.870	19.3155	0.9884	0.9928	0.9956	1.0000

Total 100.000 100.0000
Normalization factor = 1.9758
Total 100.000 100.0000
Normalization factor = 2.1120

Appendix G

SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Gel

Figures

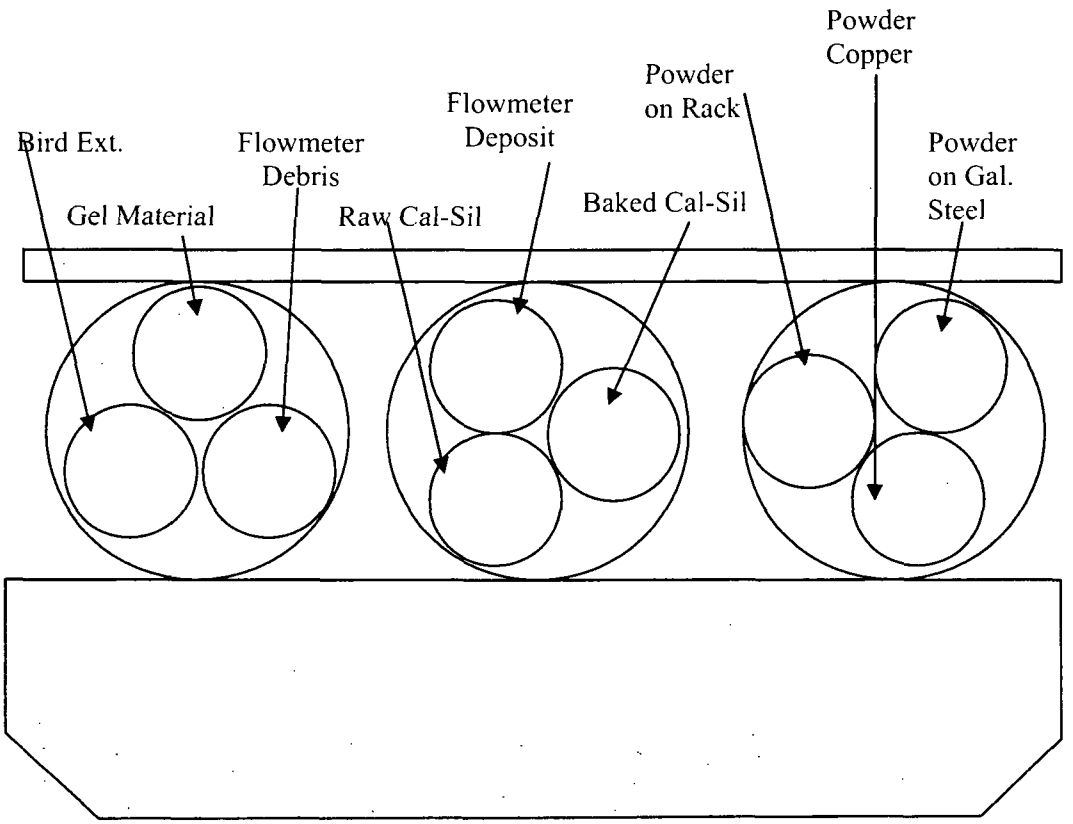
Tables

In ICET Test #3, one distinguished phenomenon is the presence of white gel-like precipitates in the testing solution. On the shutdown date of Test #3, deposits of the white gel-like precipitates were observed on the top of the birdcage. These precipitates may increase the containment sump screen head loss during a LOCA; therefore, it is necessary to investigate the morphology and composition of the white gel-like precipitate.

This appendix shows the ESEM/SEM/EDS and XRD/XRF results of the white gel-like precipitates. The precipitates were collected on the date Test #3 was shut down (May 5, 2005). For the SEM examination, the samples were dried in air before being coated with Au/Pd. EDS results provide a semi-quantitative elemental analysis of the sample compositions. Also, XRD results show the crystal structure of the white gel-like precipitates. Based on the XRD results, the composition of the white gel-like precipitates contained crystalline substances of sodium calcium hydrogen carbonate phosphate hydrate [$Ca_8H_2(PO_4)_6 \cdot H_2O \cdot NaHCO_3 \cdot H_2O$] and lithium calcium hydrogen carbonate phosphate hydrate [$Ca_8H_2(PO_4)_6 \cdot H_2O \cdot Li_2CO_3 \cdot H_2O$]. In addition, XRF results indicate the chemical composition of the precipitates. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Laboratory session from May 9, 2005.
Test #3, Day-30 Gel Material

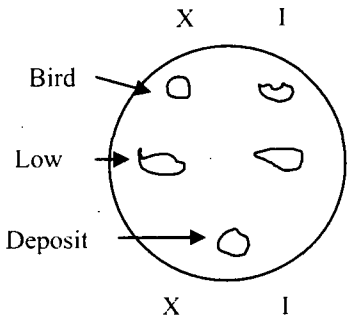


Gel Material

Image:	T3D30GelMaterial003	100 ×		Figure G-1
	T3D30GelMaterial004	1000 ×		Figure G-2
EDS:	T3D30Gel02		Whole screen of image 004	Figure G-3

Transcribed Laboratory Log

Laboratory session from May 6, 2005.
Test #3, Day-30 Gel Material



Gel-Like Material on Top of Birdcage

Image:	t3Gel08	1000 ×		Figure G-4
EDS:	t3GelED4		White gel has high C and low Si	Figure G-5
	t3geled5		Comparing t3bcexe2 from Appendix C4 & t3GelED4	Figure G-6
	t3geled6		EDS of gel material	Figure G-7
	t3geled7		Comparing t3bcexe2 from Appendix C4 & EDS6	Figure G-8

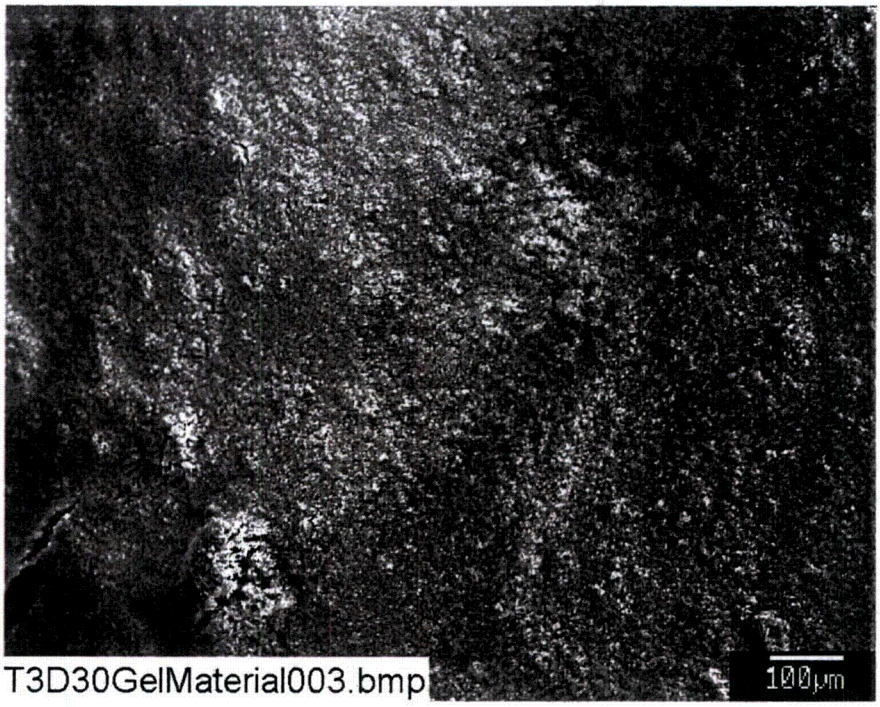


Figure G-1. **SEM image magnified 100 times for a Test #3, Day-30 white gel-like material on the top of the birdcage. (T3D30GelMaterial003)**

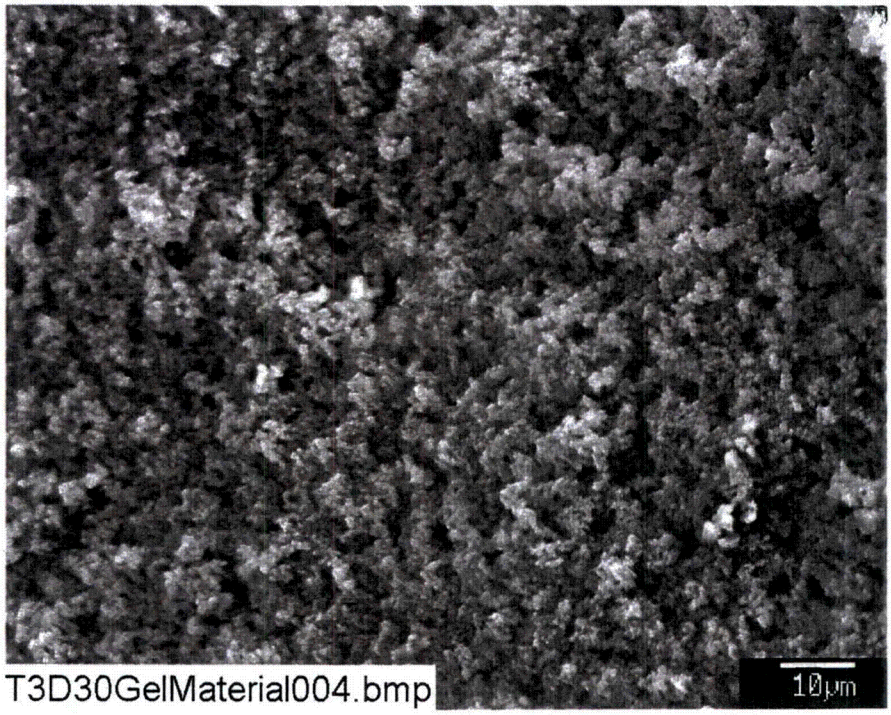


Figure G-2. **SEM image magnified 1000 times for a Test #3, Day-30 white gel-like material on the top of the birdcage. (T3D30GelMaterial004)**

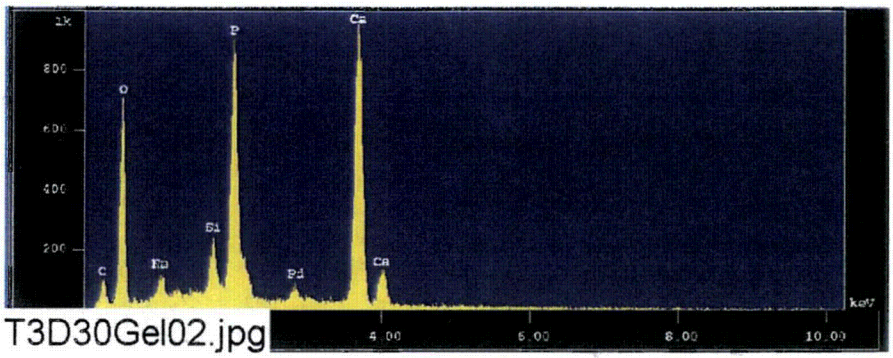


Figure G-3. **EDS counting spectrum for the white gel-like material (whole image) shown in Figure G-2. (T3D30Gel02)**

The results from the chemical composition analysis for T3D30Gel02 are given in Table G-1.

Table G-1. Chemical Compositions for T3D30Gel02, Figure G-3

May 9 2005

Group : NRC
Sample : T3D30 ID# : 2
Comment : GelMaterial
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.606E-09 A
Stage Point : X=79.625 Y=59.260 Z=11.424
Acq. Date : Mon May 9 11:42:11 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.6057	0.0005	338 / 119
O K	Normal	0.25- 0.77	12.2043	0.0032	4587 / 68
Na K	Normal	0.81- 1.27	0.5675	0.0010	613 / 50
Si K	Normal	1.50- 2.05	0.9391	0.0005	1366 / 271
P K	Normal	1.75- 2.38	8.4975	0.0055	7628 / 107
Ca K	Normal	3.39- 4.30	17.1295	0.0038	12109 / 26

Chi_square = 42.7915

Element	Mass%	Atomic%	ZAF	Z	A	F
C	4.355	7.8616	3.7318	1.0194	3.6611	0.9999
O	45.521	61.6928	1.9361	0.9721	1.9917	1.0000
Na	1.639	1.5456	1.4989	1.0256	1.4614	1.0000
Si	2.072	1.5994	1.1451	0.9756	1.1812	0.9937
P	13.776	9.6435	0.8415	1.1708	0.7203	0.9978
Ca	32.638	17.6571	0.9890	0.9947	0.9943	1.0000

Total 100.000 100.0000
Normalization factor = 1.9265
Total 100.000 100.0000
Normalization factor = 2.1120

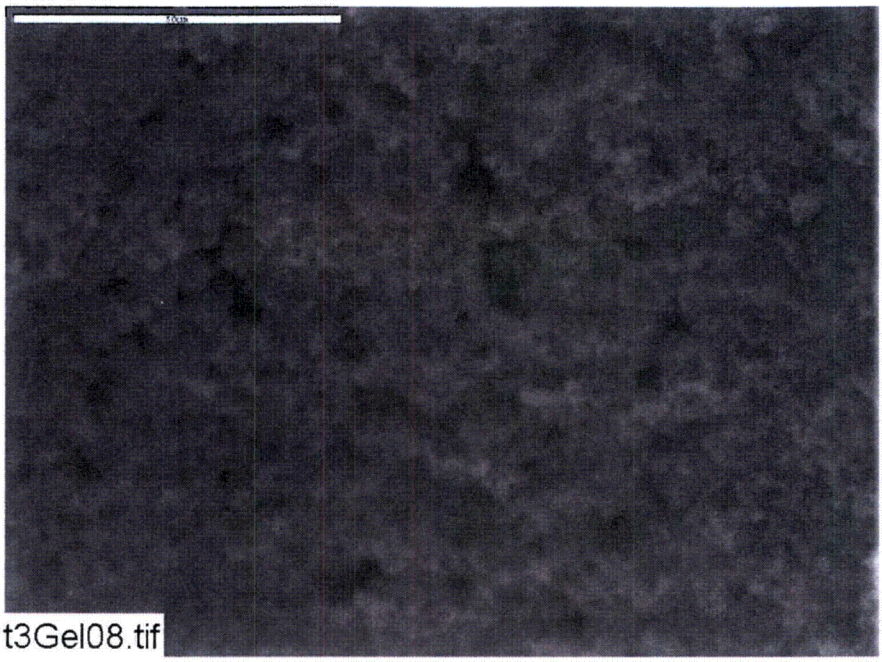


Figure G-4. ESEM image magnified 1000 times for a Test #3, Day-30 white gel-like material on the top of the birdcage. (t3Gel08)

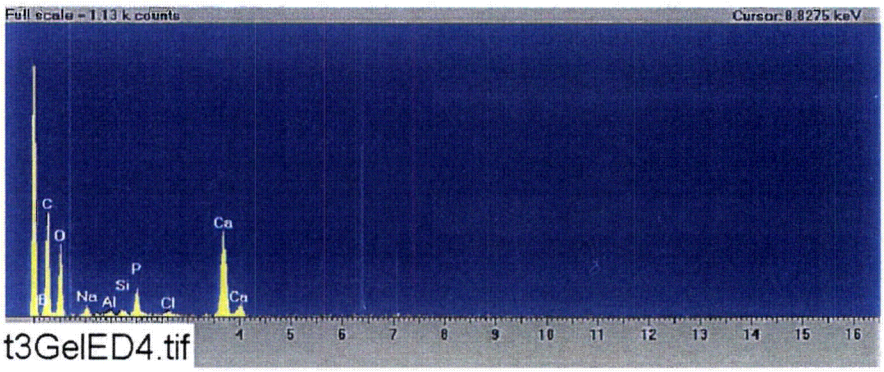


Figure G-5. EDS counting spectrum for the white gel-like material shown in Figure G-4. (t3GelED4)

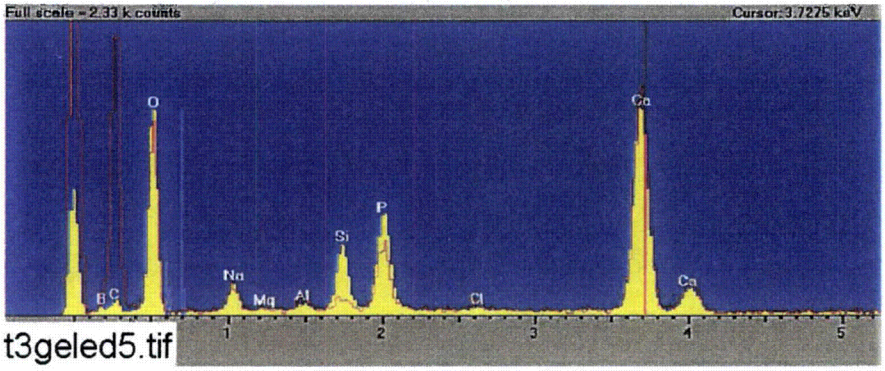


Figure G-6. **Comparison of EDS counting spectra for Figure G-5 (red, the gel-like materials shown in Figure G-4) and Figure C4-5 (yellow, the large deposits taken from the birdcage exterior shown in Figure C4-4). (t3geled5)**

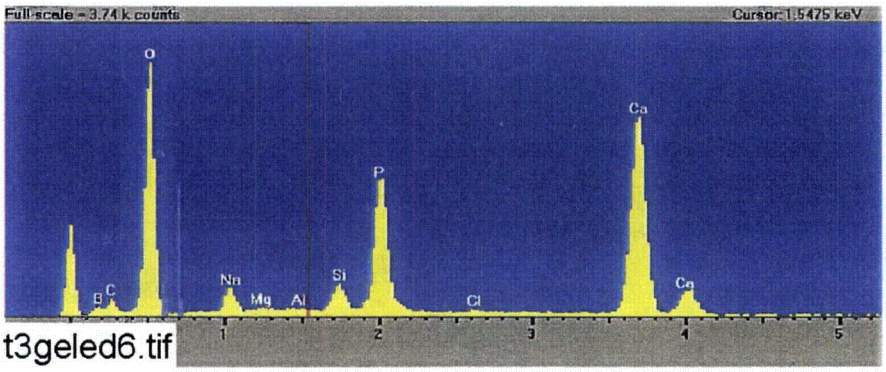


Figure G-7. **Another EDS counting spectrum for the white gel-like material shown in Figure G-4. (t3geled6)**

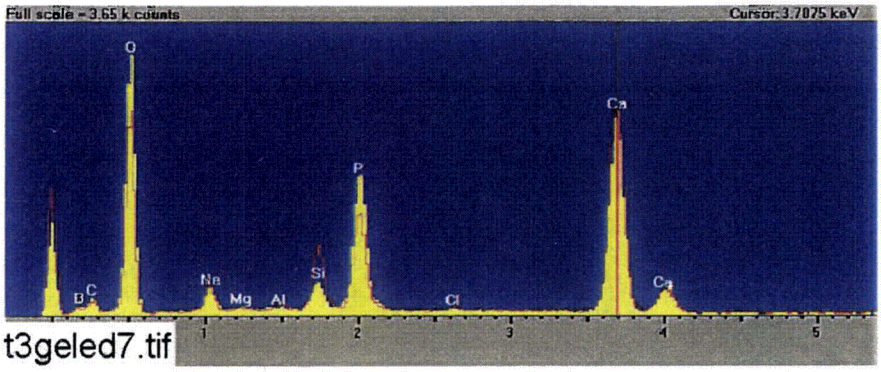


Figure G-8. **Comparison of EDS counting spectra for Figure G-7 (yellow, the gel-like materials shown in Figure G-4) and Figure C4-5 (red, the large deposits taken from the birdcage exterior shown in Figure C4-4). (t3geled7)**

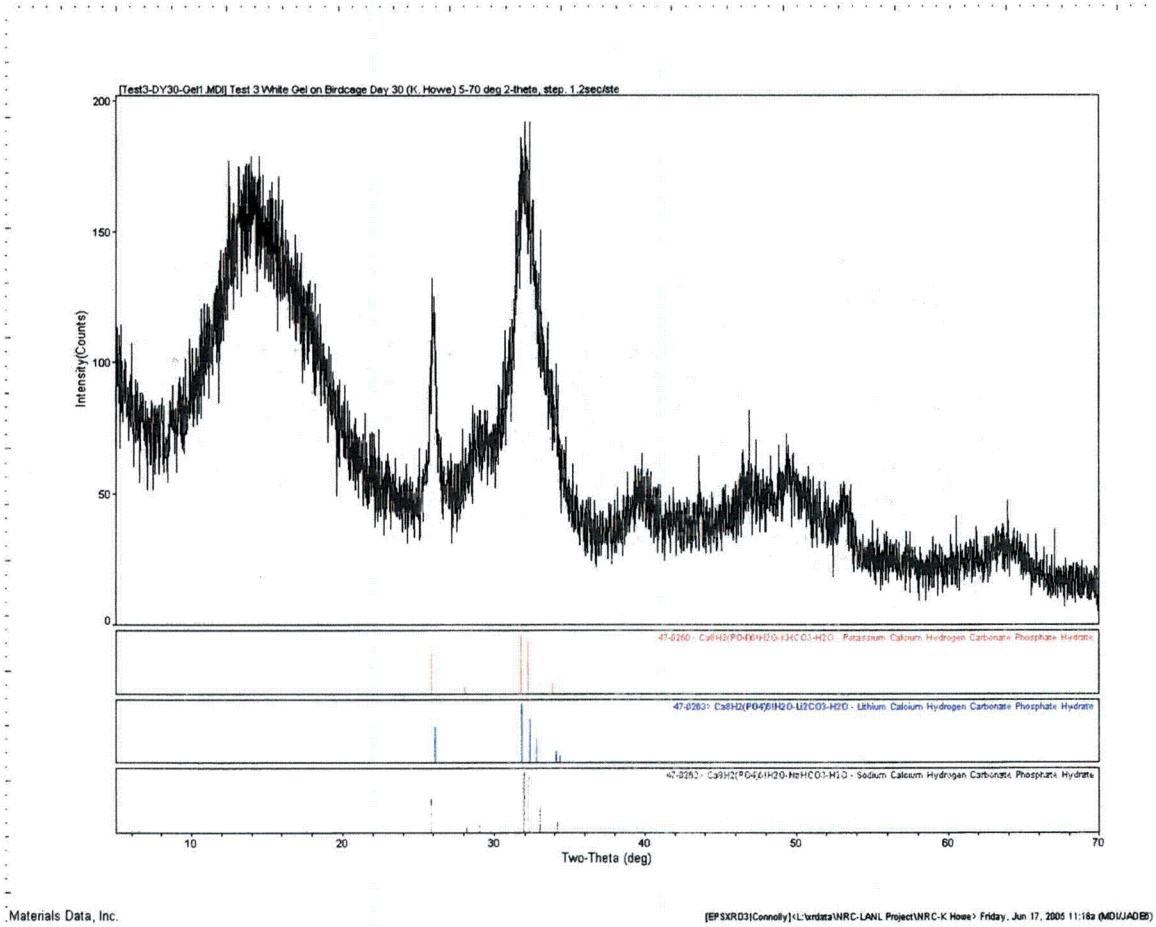


Figure G-9. **XRD results for Test #3, Day-30 white gel-like material.**

Table G-2. Dry Mass Composition of a Test #3, Day 30 White Gel-Like Sample by XRF Analysis

Sample ID	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	$H_20(-)$	$H_2O(+)CO_2$	P_2O_5	Total	H2O(+)CO2 /DF (10) & Cover. To %
Test #3, Day 30 White Gel	5.26	0.02	0.63	0.07	0.00	0.00	0.25	35.01	2.39	0.06	4.75	19.24	27.09	94.77	1.0196

Appendix H

SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Cal-Sil

Figures

Tables

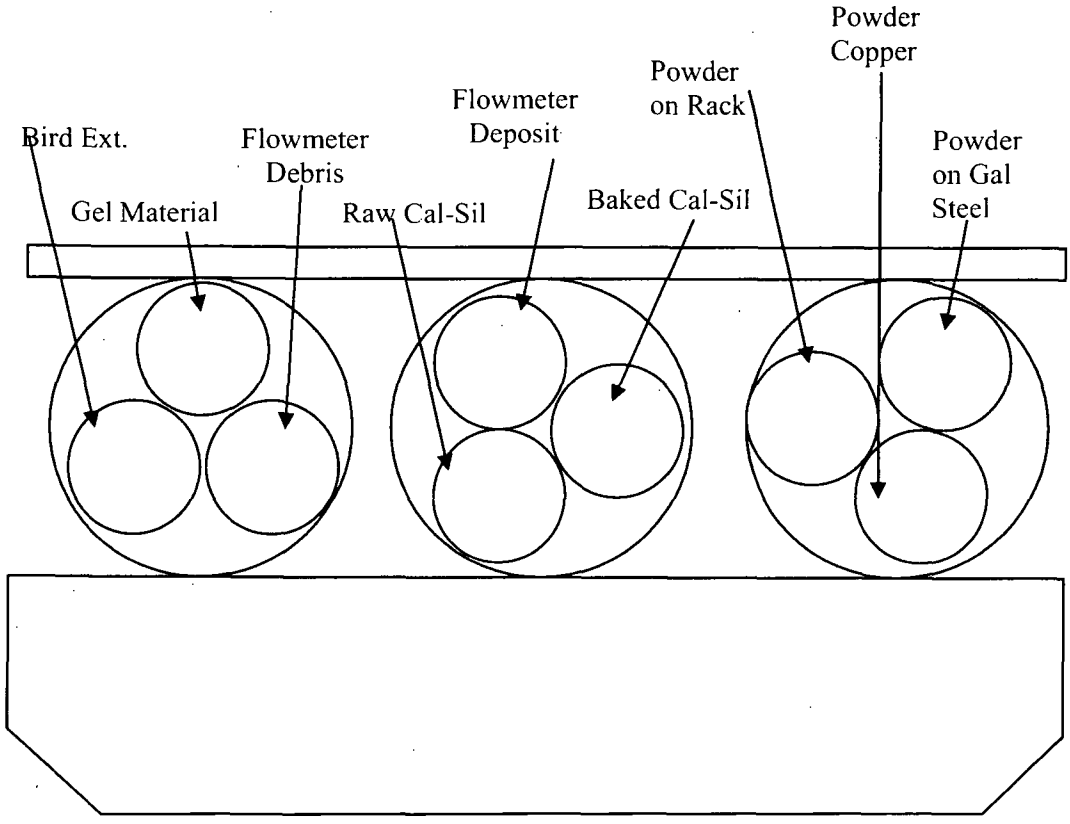
ICET #3 is the first ICET that incorporates calcium silicate (cal-sil) to represent insulation materials, along with fiberglass, in a power plant. This appendix presents the ESEM/SEM/EDS and XRD/XRF results of different cal-sil samples, including unused raw and unused baked cal-sil samples. The Test #3, Day-30 raw cal-sil samples were submerged in the birdcage, whereas the Test #3, Day-30 baked cal-sil sample were submerged in the high-flow zone.

The submerged cal-sil samples were collected on the date Test #3 was shut down (May 5, 2005). For the SEM examination, the samples were dried in air before being coated with Au/Pd. EDS results provide a semi-quantitative elemental analysis of the sample compositions.

In addition, XRD/XRF results show the crystal structure and the chemical composition of the unused raw and unused baked cal-sil samples. Based on XRD results, both unused raw and unused baked cal-sil samples contained crystalline substances of tobermorite [$Ca_{2.25}(Si_3O_{7.5}(OH)_{1.5})(H_2O)$] and calcite ($CaCO_3$). Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Laboratory session from May 9, 2005.
Test #3, Day-30 Cal-Sil



Raw Cal-Sil

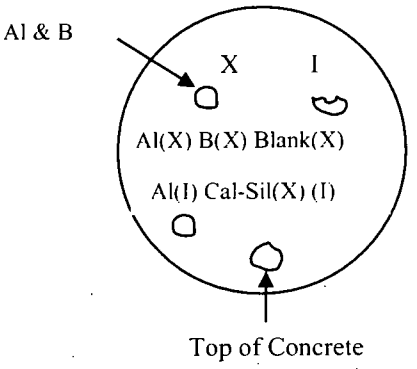
Image:	T3~RawCal-Sil007	100 ×		Figure H-1
	T3~RawCal-Sil008	1000 ×		Figure H-2
EDS:	T3Prtcle04		On particles of image 008	Figure H-3
	T3fiber05		On fiber of image 008	Figure H-4
	EDS06		Whole image of image 008	Figure H-5
Image:	T3~RawCal-Sil009	100 ×		Figure H-6

Baked Cal-Sil

Image:	T3~BakedCal-Sil010	200 ×		Figure H-7
EDS:	EDS07		EDS of image 010	Figure H-8
Image:	T3~BakedCal-Sil011	1000 ×		Figure H-9

Transcribed Laboratory Log

Laboratory session from May 10, 2005.
Test #3, Day-30 ESEM Birdcage.



Cal-Sil Exterior in Birdcage

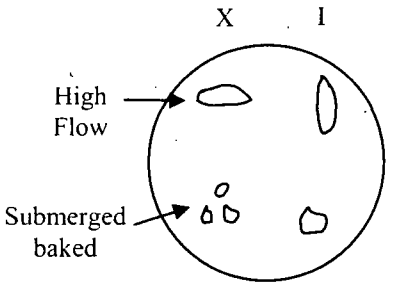
Image:	t3calx21	500 ×		Figure H-10
	T3Calx22	500 ×		Figure H-11
EDS:	t3calx23		EDS on T3Calx22	Figure H-12

Cal-Sil Interior in Birdcage

Image:	T3Call24	100 ×		Figure H-13
	T3Call25	500 ×		Figure H-14
EDS:	T3cali26		EDS on T3Call25	Figure H-15
Image:	t3cali27	500 ×		Figure H-16

Transcribed Laboratory Log

Laboratory session from May 11, 2005.
Test #3, Day-30 Baked Cal-Sil



Submerged Baked Cal-Sil Exterior

Image:	T3bcal39	100 ×		Figure H-17
	t3bcal40	1000 ×	Particles	Figure H-18
EDS	t3bcal41		EDS on white particles shown in t3bcal40	Figure H-19
	t3bcal42		EDS on dark particles shown in t3bcal40	Figure H-20
Image:	T3Bcal43	100 ×		Figure H-21
	t3bcal44	1000 ×		Figure H-22
	t3bcal45	1000 ×		Figure H-23
EDS:	t3bcal46		EDS on whole screen of image 45	Figure H-24

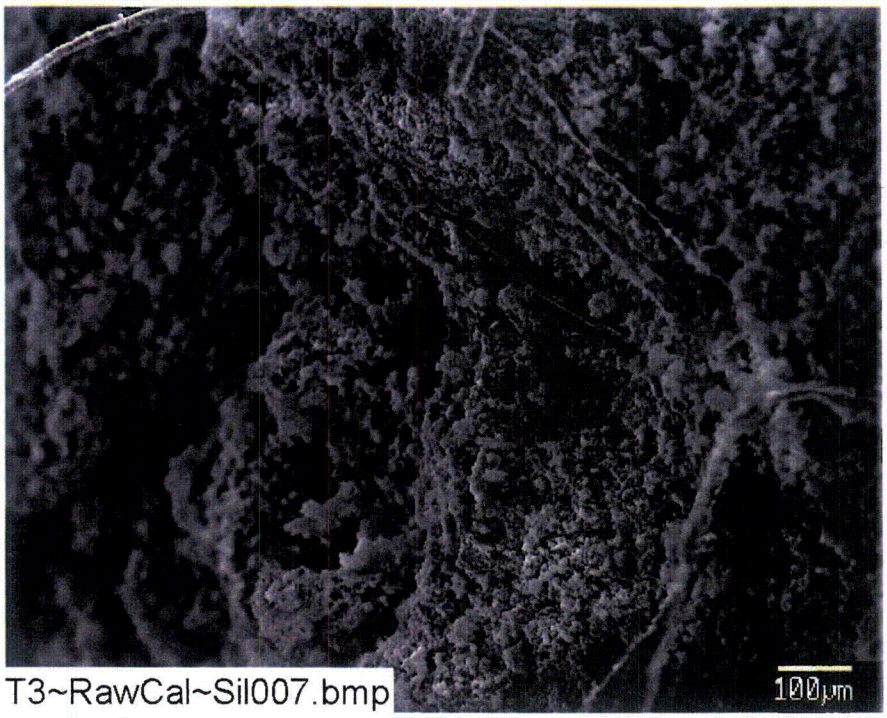


Figure H-1. **SEM image magnified 100 times for a raw cal-sil sample. (T3~RawCal~Sil007)**

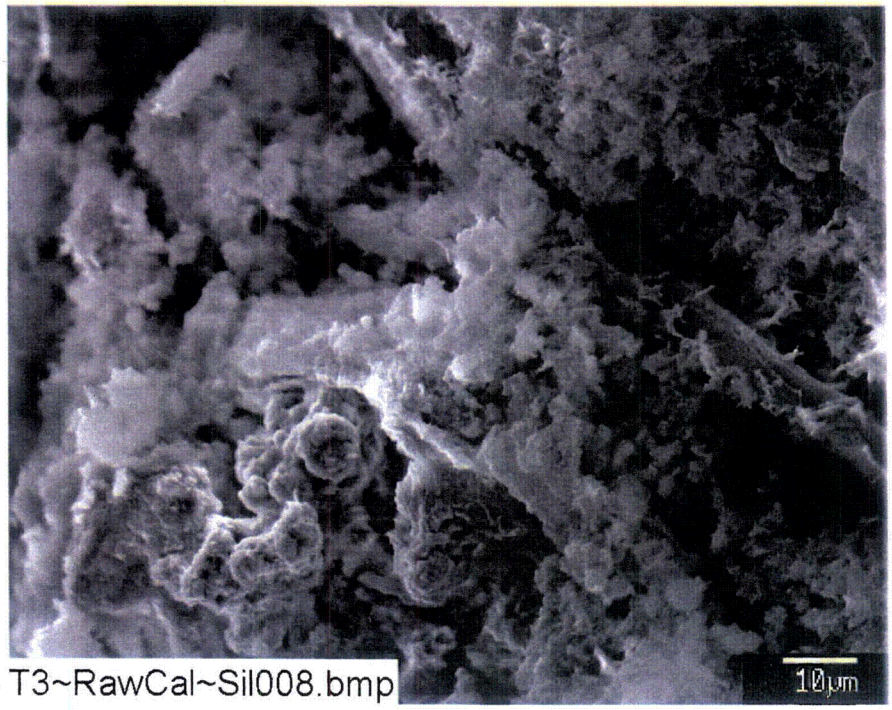


Figure H-2. **SEM image magnified 1000 times for a raw cal-sil sample. (T3~RawCal~Sil008)**

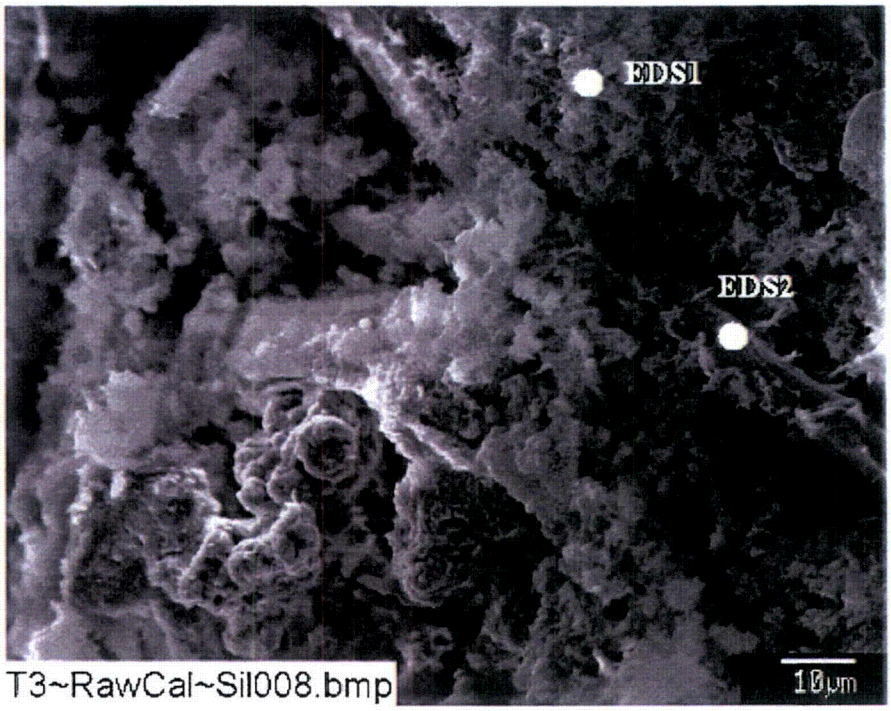


Figure H-3. **Annotated SEM image magnified 1000 times for a raw cal-sil sample. (T3~RawCal~Sil008)**

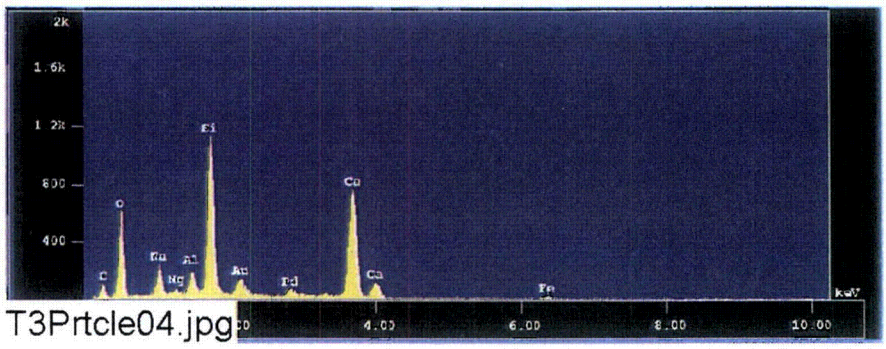


Figure H-4. **EDS counting spectrum for the particles (EDS1) shown in Figure H-3. (T3Prtcle04)**

The results from the chemical composition analysis for T3Prtcle04 are given in Table H-1.

Table H-1. Chemical Compositions for T3Prtcle04, Figure H-4

May 9 2005

Group : NRC
Sample : T3D30 ID# : 4
Comment : Raw Cal-Sil Particle
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.604E-09 A
Stage Point : X=58.349 Y=61.668 Z=12.516
Acq. Date : Mon May 9 14:05:35 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.9637	0.0005	537 / 119
O K	Normal	0.25- 0.77	10.7009	0.0031	4017 / 65
Na K	Normal	0.81- 1.27	1.1322	0.0012	1221 / 43
Mg K	Normal	0.97- 1.57	0.1062	0.0003	166 / 80
Al K	Normal	1.19- 1.83	0.9046	0.0004	1409 / 80
Si K	Normal	1.50- 2.05	6.6718	0.0010	9692 / 107
Ca K	Normal	3.39- 4.30	13.6411	0.0036	9631 / 25
Pd L	Normal	2.22- 3.81	1.1981	0.0013	878 / 68
Fe K	Normal	6.00- 7.44	0.8951	0.0012	257 / 18

Chi_square = 21.7880

Element	Mass%	Atomic%	ZAF	Z	A	F
C	7.204	13.1026	3.7872	1.0155	3.7297	0.9999
O	38.993	53.2408	1.8460	0.9683	1.9064	1.0000
Na	3.324	3.1581	1.4872	1.0216	1.4562	0.9997
Mg	0.323	0.2898	1.5385	0.9616	1.6029	0.9982
Al	2.373	1.9210	1.3288	0.9940	1.3428	0.9956
Si	15.630	12.1570	1.1868	0.9716	1.2226	0.9991
Ca	26.877	14.6492	0.9982	0.9904	1.0081	0.9997
Pd	3.135	0.6437	1.3257	1.3709	0.9776	0.9891
Fe	2.142	0.8379	1.2123	1.1945	1.0148	1.0000
Total	100.000	100.0000				

Normalization factor = 1.9740

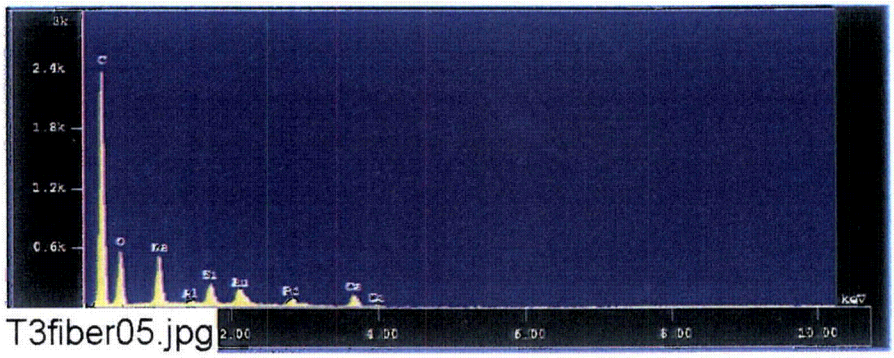


Figure H-5. EDS counting spectrum for the fiber (EDS2) shown in Figure H-3. (T3fiber05)

The results from the chemical composition analysis for T3fiber05 are given in Table H-2.

Table H-2. Chemical Compositions for T3fiber05, Figure H-5

May 9 2005

Group : NRC
Sample : T3D30 ID# : 5
Comment : Raw Cal-Sil Fiber
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.603E-09 A
Stage Point : X=58.273 Y=61.617 Z=12.516
Acq. Date : Mon May 9 13:56:08 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	33.8965	0.0016	18866 / 99
O K	Normal	0.25- 0.77	10.3921	0.0032	3899 / 1192
Na K	Normal	0.81- 1.27	3.1096	0.0017	3352 / 52
Al K	Normal	1.19- 1.83	0.1520	0.0003	237 / 64
Si K	Normal	1.50- 2.05	1.1022	0.0005	1600 / 74
Ca K	Normal	3.39- 4.30	2.0189	0.0016	1425 / 17

Chi_square = 50.8203

Element	Mass%	Atomic%	ZAF	Z	A	F
C	65.848	74.0755	1.6099	1.0143	1.5873	1.0000
O	25.742	21.7397	2.0528	0.9679	2.1208	1.0000
Na	4.419	2.5969	1.1775	1.0228	1.1510	1.0002
Al	0.219	0.1098	1.1954	0.9966	1.2002	0.9994
Si	1.434	0.6899	1.0783	0.9750	1.1061	0.9999
Ca	2.338	0.7882	0.9598	0.9997	0.9601	1.0000
Total	100.000	100.0000				

Normalization factor = 1.2067

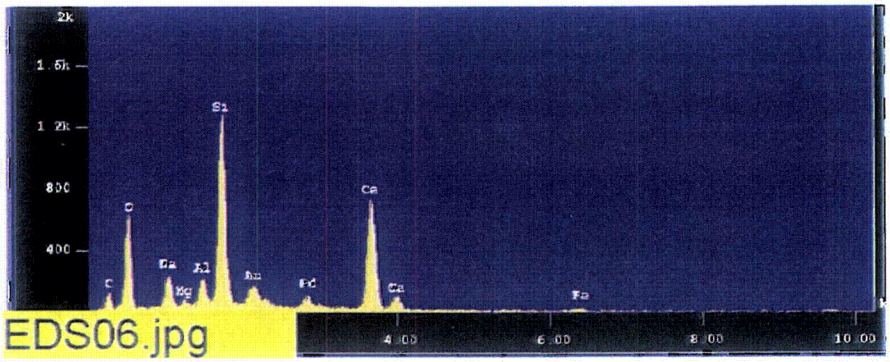


Figure H-6. **EDS counting spectrum for the whole image shown in Figure H-3. (EDS06)**

The results from the chemical composition analysis for EDS06 are given in Table H-3.

Table H-3. Chemical Compositions for EDS06, Figure H-6

May 9 2005

Group : NRC
Sample : T3D30 ID# : 6
Comment : Raw Cal-Sil
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.606E-09 A
Stage Point : X=57.876 Y=61.138 Z=12.516
Acq. Date : Mon May 9 14:28:12 2005

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	1.1389	0.0005	635 / 112
O K	Normal	0.25- 0.77	11.5668	0.0033	4348 / 73
Na K	Normal	0.81- 1.27	1.5352	0.0013	1658 / 56
Mg K	Normal	0.97- 1.57	0.1356	0.0003	213 / 79
Al K	Normal	1.19- 1.83	1.1066	0.0005	1726 / 98
Si K	Normal	1.50- 2.05	7.1883	0.0010	10455 / 146
Ca K	Normal	3.39- 4.30	12.0212	0.0034	8498 / 24
Fe K	Normal	6.00- 7.44	0.9544	0.0012	274 / 11

Chi_square = 26.8019

Element	Mass%	Atomic%	ZAF	Z	A	F
C	9.300	16.0597	4.1007	1.0233	4.0075	0.9999
O	39.825	51.6305	1.7291	0.9759	1.7719	1.0000
Na	4.382	3.9538	1.4336	1.0297	1.3927	0.9996
Mg	0.411	0.3503	1.5204	0.9694	1.5717	0.9979
Al	2.909	2.2365	1.3203	1.0021	1.3240	0.9952
Si	17.041	12.5848	1.1905	0.9797	1.2160	0.9994
Ca	23.814	12.3235	0.9948	0.9993	0.9959	0.9996
Fe	2.318	0.8608	1.2196	1.2068	1.0106	1.0000
Total	100.000	100.0000				

Normalization factor = 1.9913

Figure H-7. **SEM image magnified 100 times for a raw cal-sil sample. (T3~RawCal~Sil009)**

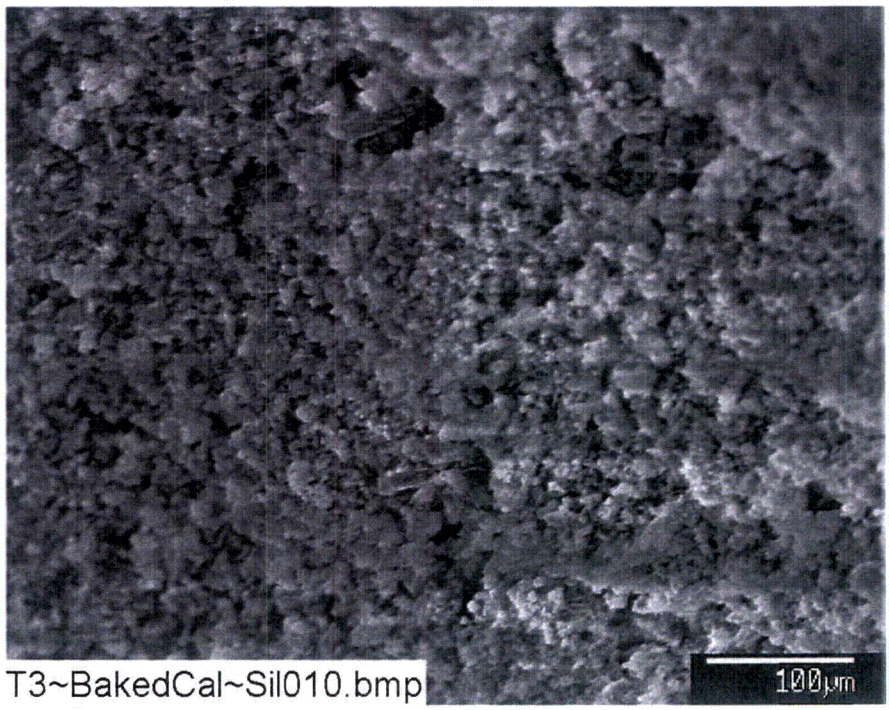


Figure H-8. **SEM image magnified 200 times for a baked cal-sil sample (T3~BakedCal~Sil010)**

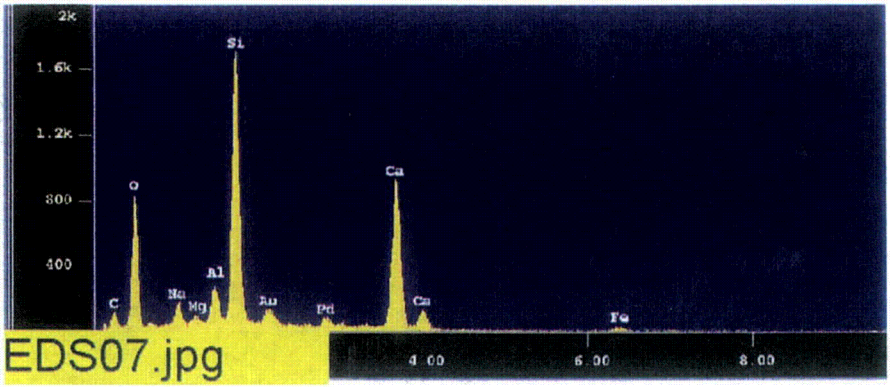


Figure H-9. **EDS counting spectrum for the baked cal-sil sample (whole image) shown in Figure H-7. (EDS07)**

The results from the chemical composition analysis for EDS07 are given in Table H-4.

Table H-4. Chemical Compositions for EDS07, Figure H-9

May 9 2005

Group : NRC
Sample : T3D30 ID# : 7
Comment : Baked Cal-Sil
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.604E-09 A
Stage Point : X=49.364 Y=57.751 Z=12.516
Acq. Date : Mon May 9 14:20:15 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.9259	0.0006	516 / 161
O K	Normal	0.25- 0.77	14.9326	0.0037	5606 / 77
Na K	Normal	0.81- 1.27	1.0025	0.0012	1081 / 65
Mg K	Normal	0.97- 1.57	0.1609	0.0003	252 / 79
Al K	Normal	1.19- 1.83	1.4816	0.0005	2308 / 124
Si K	Normal	1.50- 2.05	9.5017	0.0012	13802 / 172
Ca K	Normal	3.39- 4.30	15.8398	0.0038	11184 / 30
Fe K	Normal	6.00- 7.44	0.7928	0.0013	227 / 10

Chi_square = 33.5587

Element	Mass%	Atomic%	ZAF	Z	A	F
C	6.304	11.1172	4.1881	1.0251	4.0858	0.9999
O	42.250	55.9339	1.7404	0.9776	1.7803	1.0000
Na	2.361	2.1752	1.4487	1.0315	1.4051	0.9996
Mg	0.391	0.3407	1.4950	0.9710	1.5432	0.9977
Al	3.143	2.4673	1.3049	1.0037	1.3069	0.9947
Si	18.294	13.7966	1.1843	0.9812	1.2077	0.9994
Ca	25.681	13.5714	0.9973	1.0007	0.9968	0.9998
Fe	1.576	0.5977	1.2226	1.2082	1.0119	1.0000
Total	100.000	100.0000				

Normalization factor = 1.6257

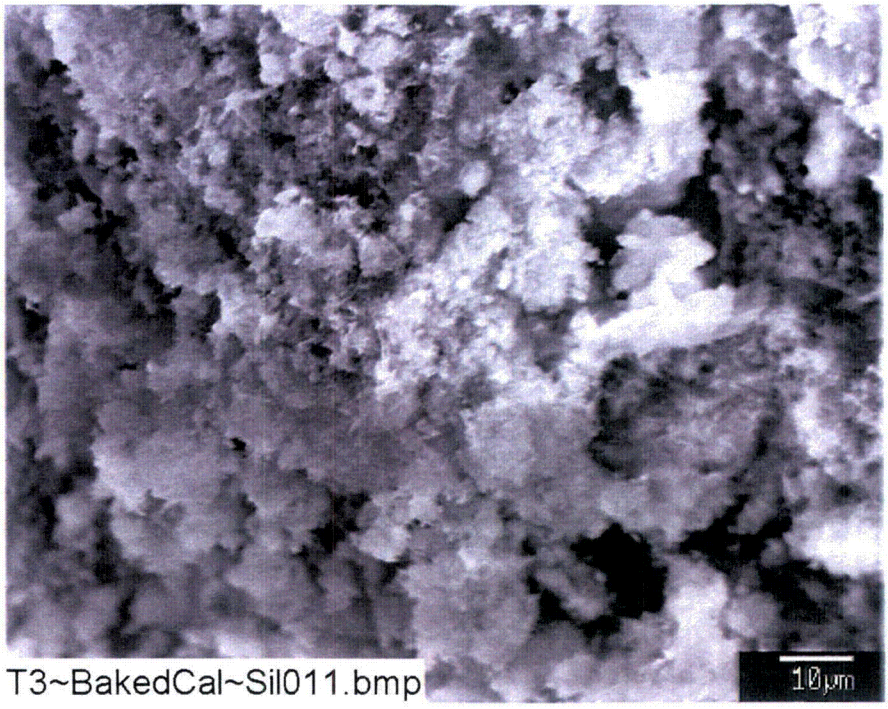


Figure H-10. **SEM image magnified 1000 times for a baked cal-sil sample. (T3~BakedCal~Sil011)**

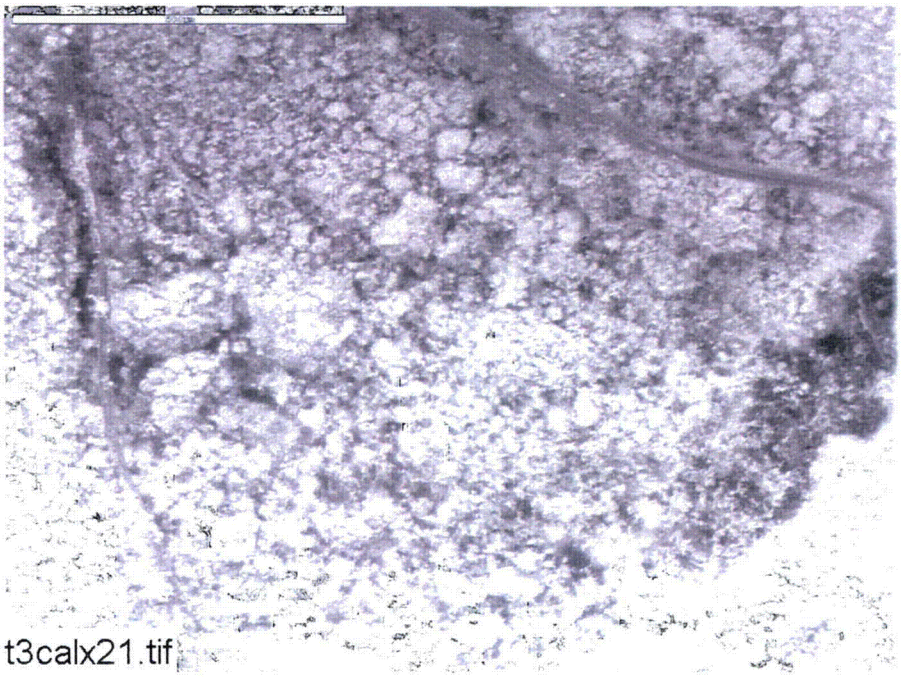


Figure H-11. **ESEM image magnified 100 times for the exterior of a Test#3, Day-30 raw cal-sil sample submerged in the birdcage. (t3calx21)**

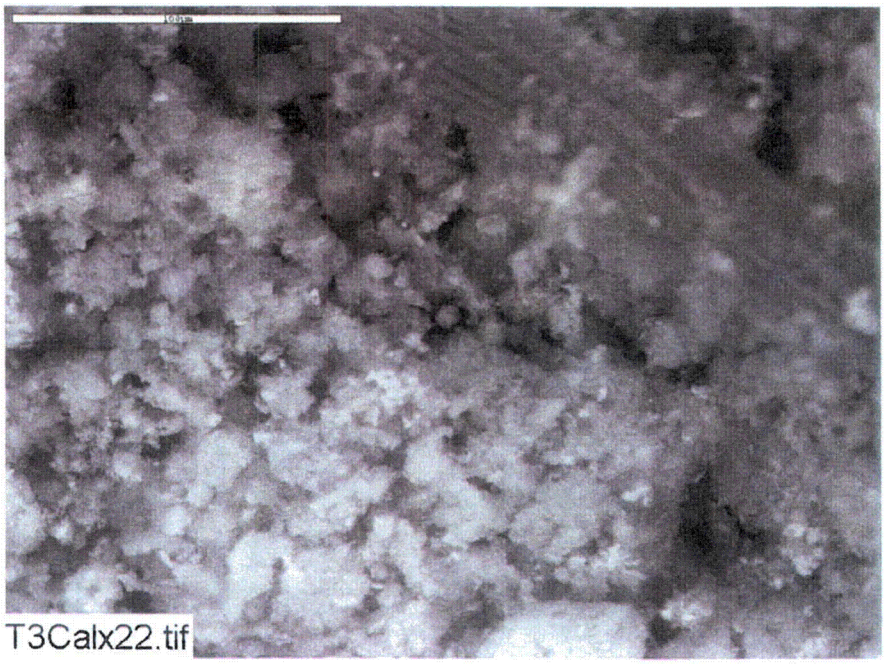


Figure H-12. **ESEM image magnified 500 times for the exterior of a Test#3, Day-30 raw cal-sil sample submerged in the birdcage. (T3Calx22)**

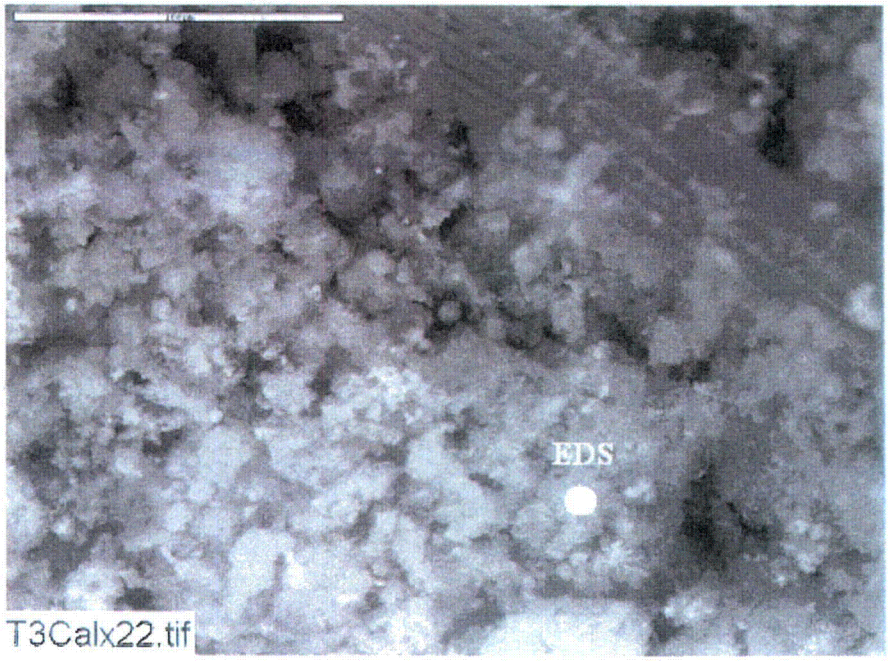


Figure H-13. **Annotated ESEM image magnified 500 times for the exterior of a Test#3, Day-30 raw cal-sil sample submerged in the birdcage. (T3Calx22)**

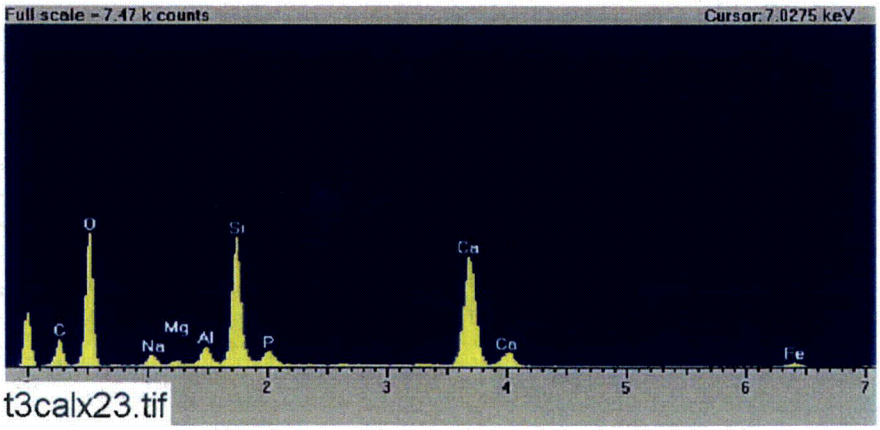


Figure H-14. EDS counting spectrum for the particles shown in Figure H-13. (t3calx23)

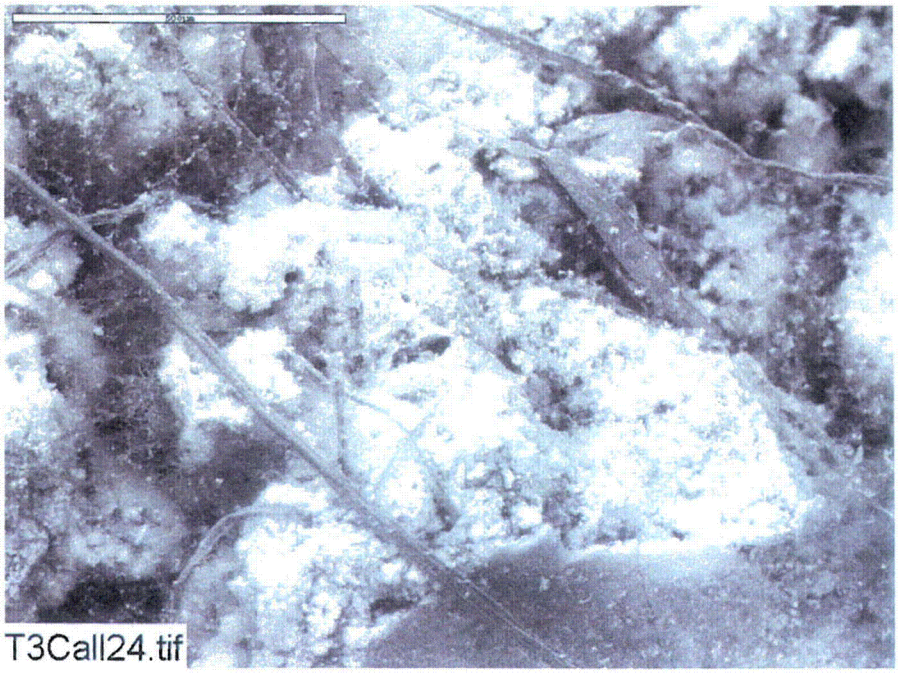


Figure H-15. ESEM image magnified 100 times for the interior of a Test#3, Day-30 raw cal-sil sample submerged in the birdcage. (T3Call24)

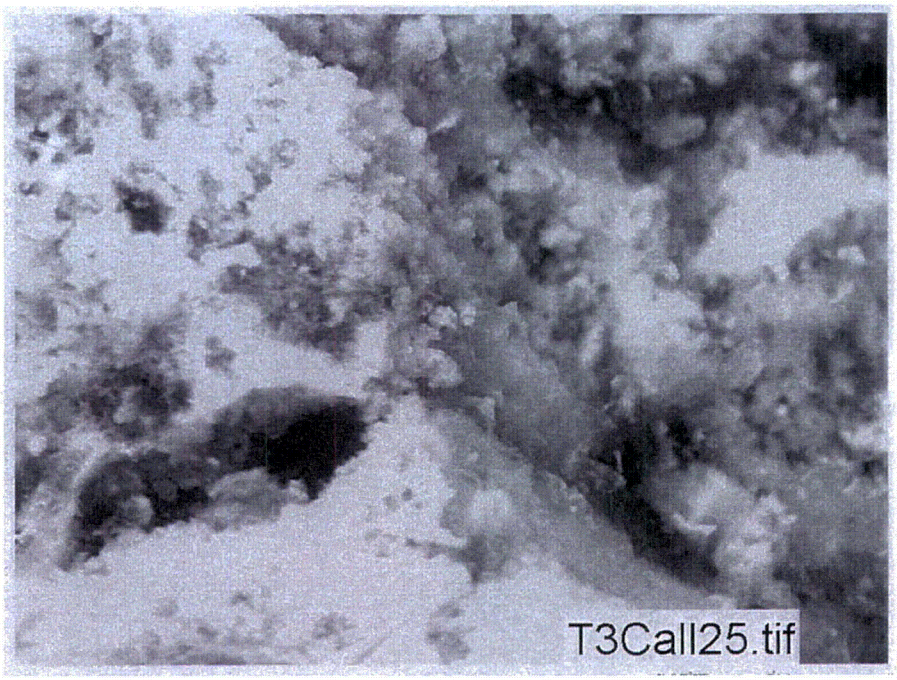


Figure H-16. **ESEM image magnified 500 times for the interior of a Test#3, Day-30 raw cal-sil sample submerged in the birdcage. (T3CalI25)**

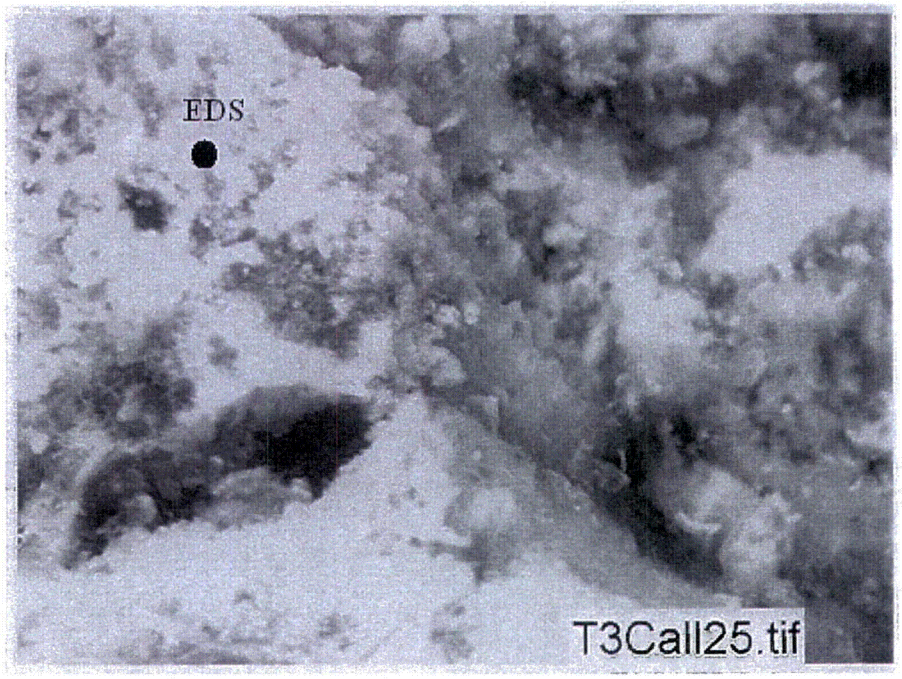


Figure H-17. **Annotated ESEM image magnified 500 times for the interior of a Test#3, Day-30 raw cal-sil sample submerged in the birdcage. (T3CalI25)**

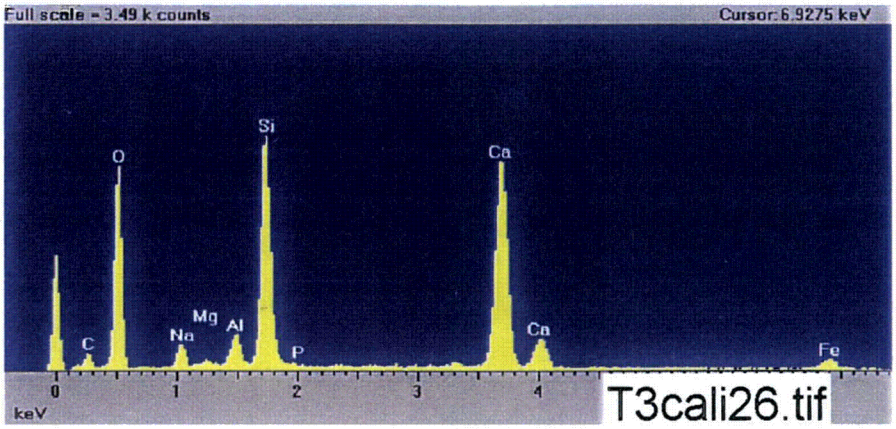


Figure H-18. EDS counting spectrum for the particles shown in Figure H-17. (T3cali26)

Figure H-19. ESEM image magnified 500 times for the interior of a Test#3, Day-30 raw cal-sil sample submerged in the birdcage. (T3Call27)

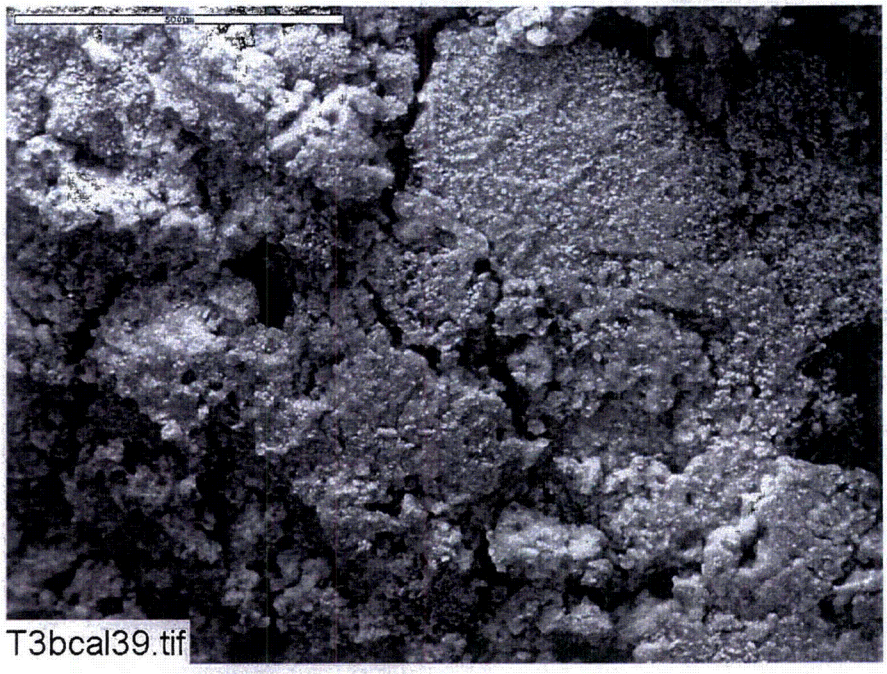


Figure H-20. **ESEM image magnified 100 times for the exterior of a Test#3, Day-30 submerged high-flow baked cal-sil sample. (T3bcal39)**

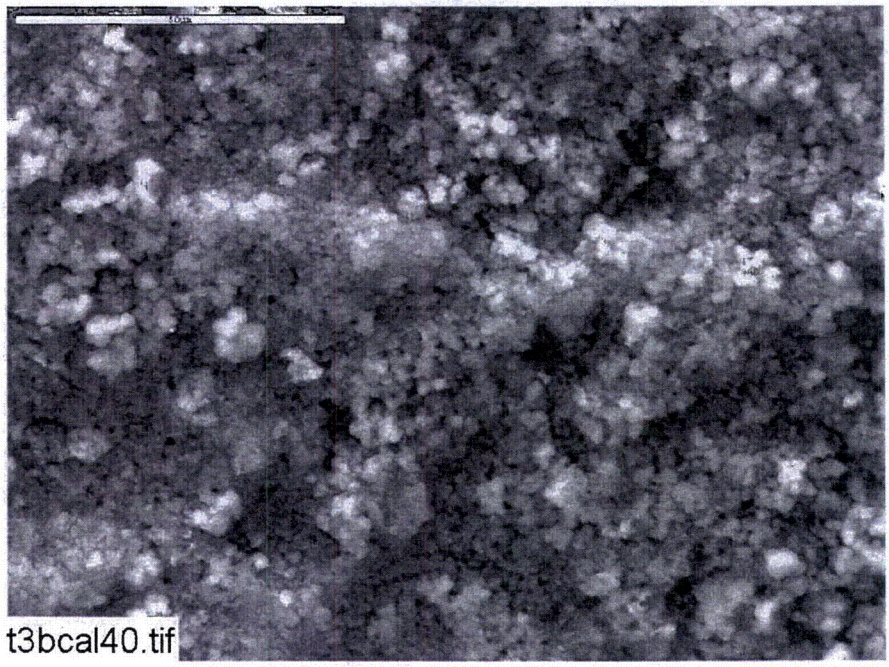


Figure H-21. **ESEM image magnified 1000 times for the exterior of a Test#3, Day-30 submerged high-flow baked cal-sil sample. (t3bcal40)**

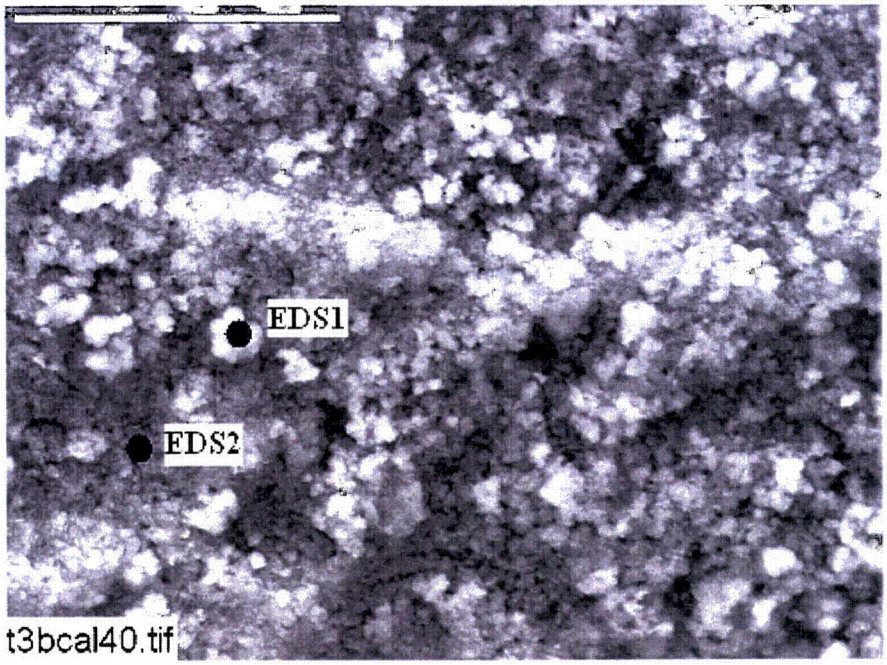


Figure H-22. **Annotated ESEM image magnified 1000 times for the exterior of a Test#3, Day-30 submerged high-flow baked cal-sil sample. (t3bcal40)**

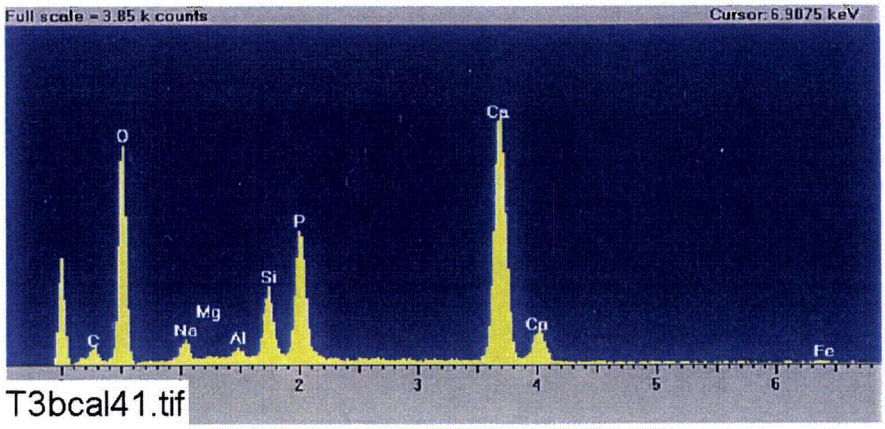


Figure H-23. **EDS counting spectrum for the light particles (EDS1) shown in Figure H-22. (T3bcal41)**

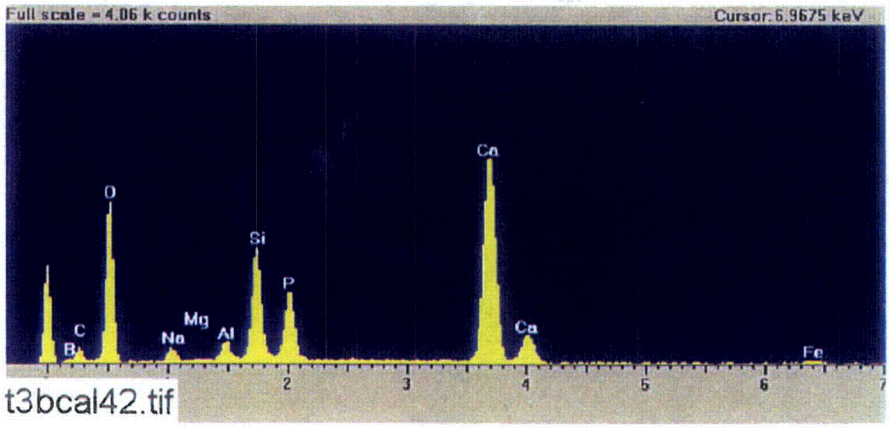


Figure H-24. **EDS counting spectrum for the dark particles (EDS2) shown in Figure H-22. (t3bcal42)**

Figure H-25. **ESEM image magnified 100 times for the interior of a Test#3, Day-30 submerged high-flow baked cal-sil sample. (T3Bcal43)**

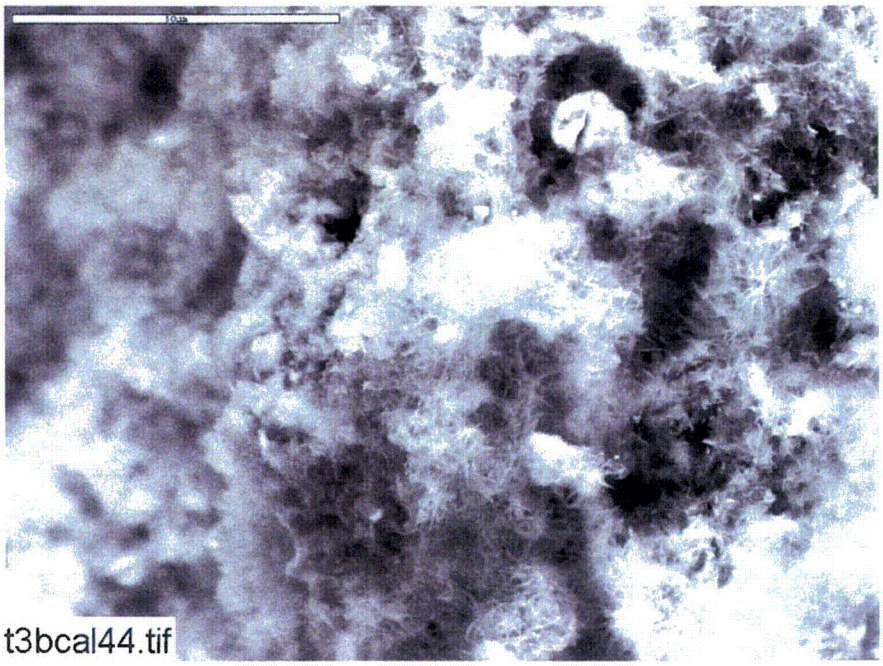


Figure H-26. **ESEM image magnified 1000 times for the interior of a Test#3, Day-30 submerged high-flow baked cal-sil sample. (t3bcal44)**

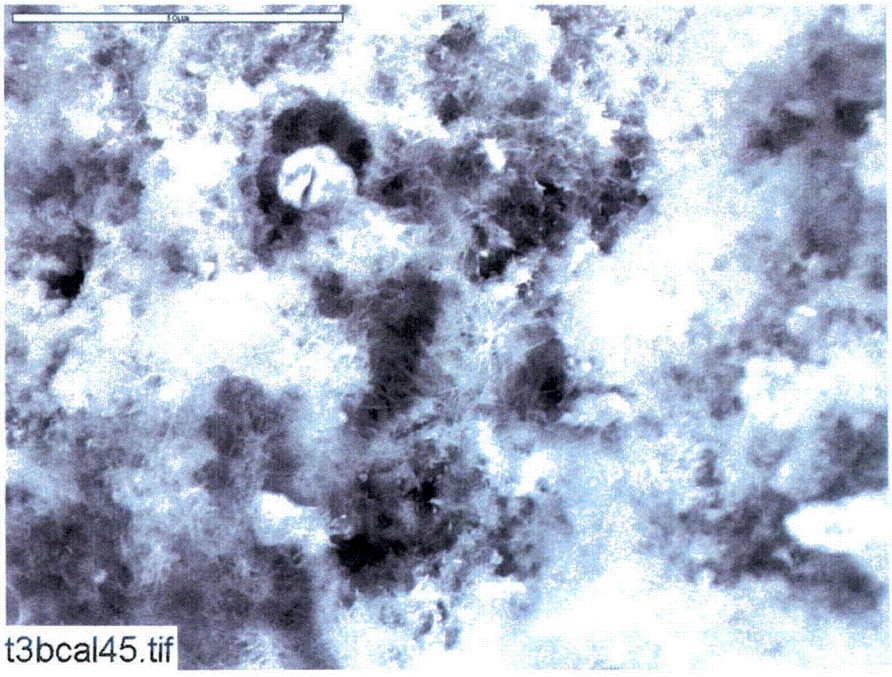


Figure H-27. **ESEM image magnified 1000 times for the interior of a Test#3, Day-30 submerged high-flow baked cal-sil sample. (t3bcal45)**

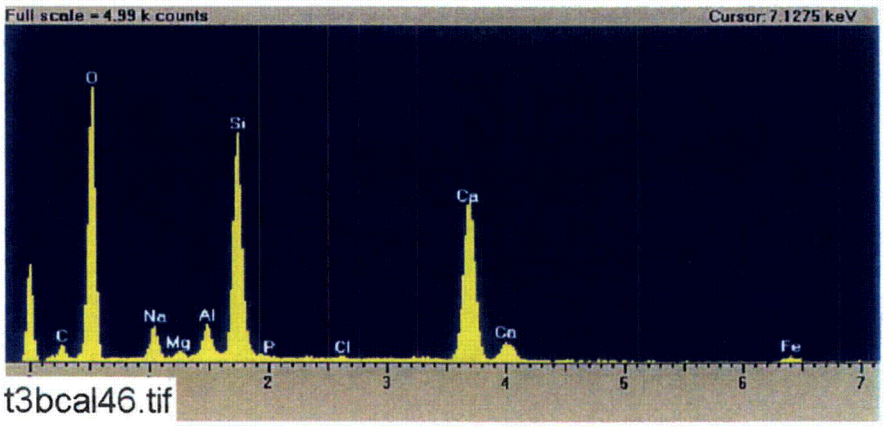


Figure H-28. EDS counting spectrum for the whole image shown in Figure H-27. (t3bcal46)

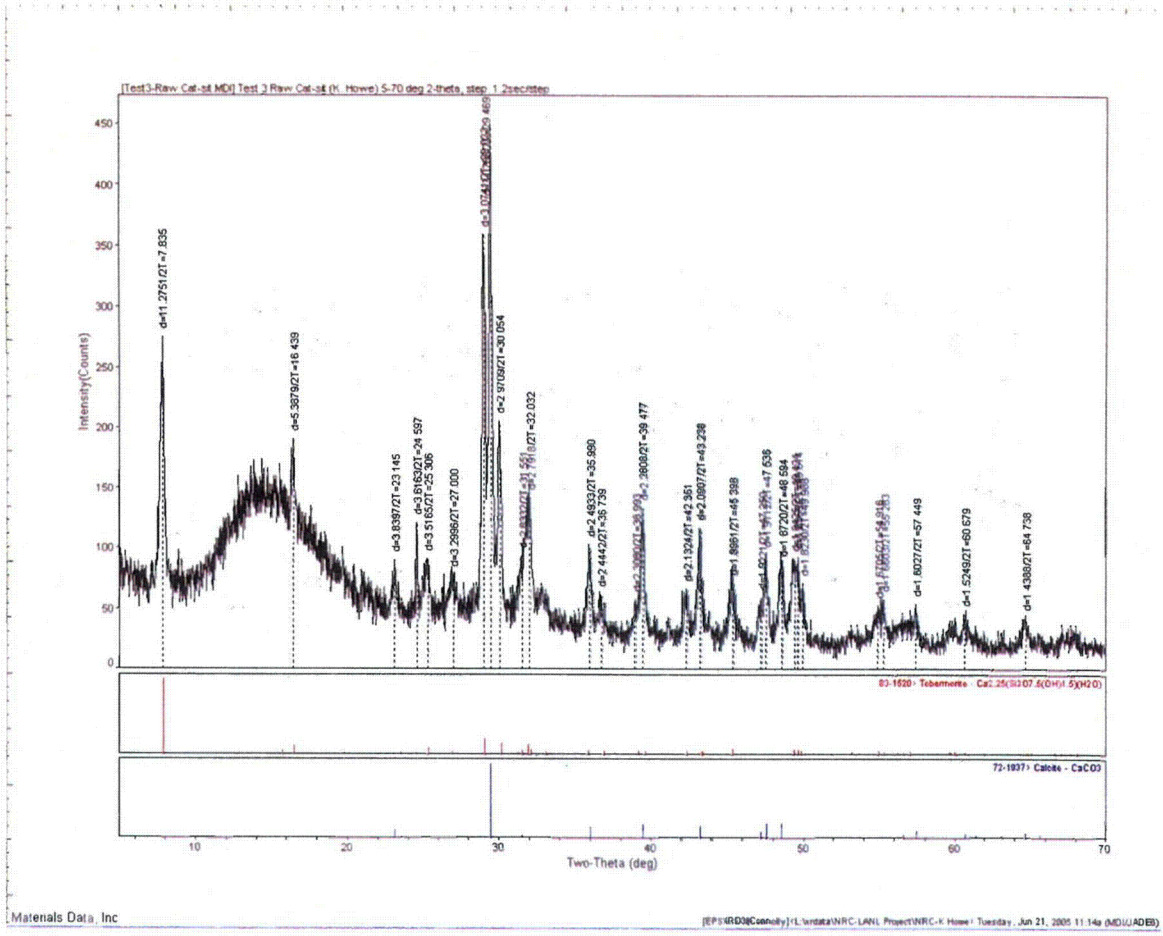


Figure H-29. XRD results for the unused raw cal-sil sample.

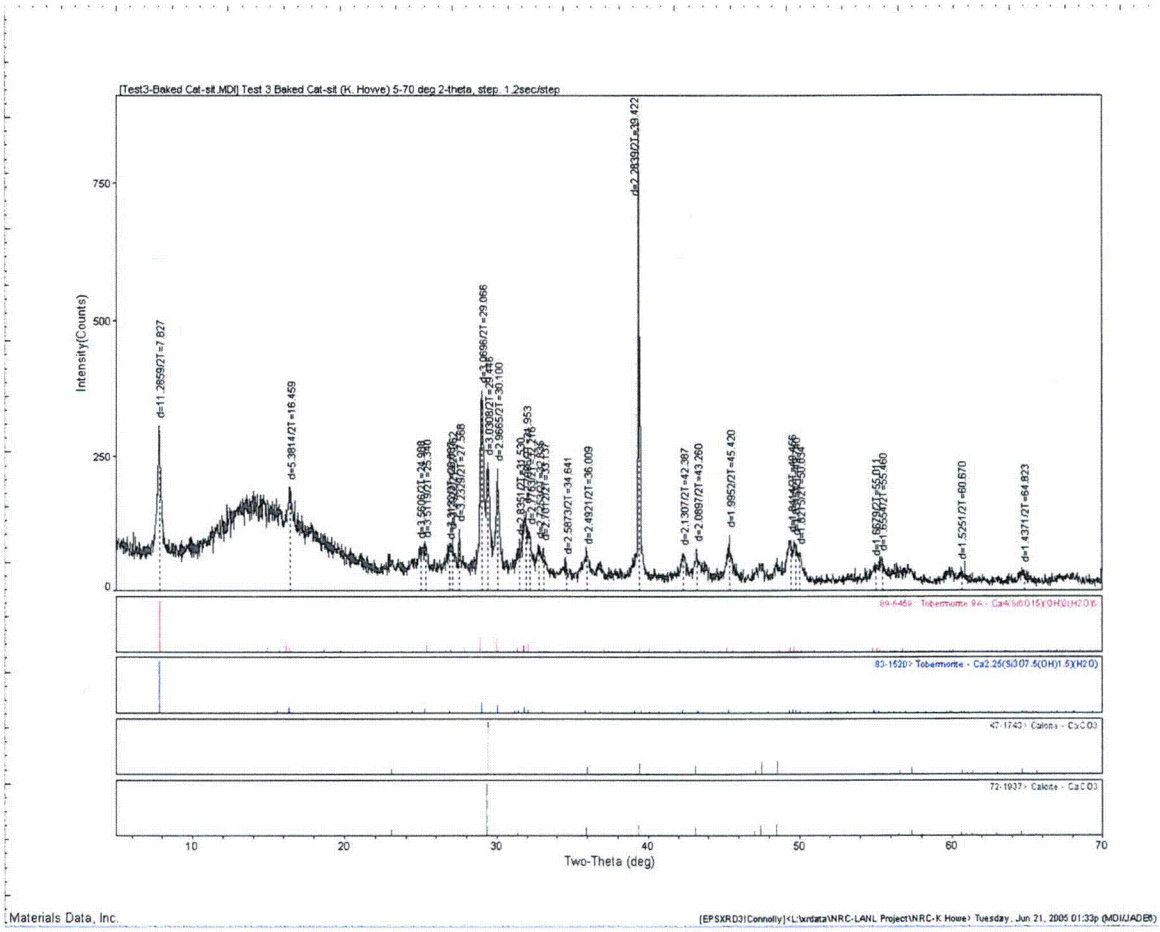


Figure H-30. XRD results for the unused baked cal-sil sample.

Table H-5. Dry Mass Composition of Unused Cal Sil Samples by XRF Analysis

Sample ID	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	$H_20(-)$	$H_2O(+)$ CO_2	P_2O_5	Total	H2O(+)CO2 /DF (10) & Cover. To %
Baked Cal Sil	38.34	0.18	5.02	2.54	0.00	0.06	0.79	34.76	2.32	0.42	0.35	18.75	0.15	103.67	1.0191
Raw Cal Sil	33.87	0.36	4.27	2.07	0.00	0.05	1.35	34.66	2.27	0.35	0.56	1.59	0.12	81.50	1.0016

Appendix I

ESEM/EDS Data for Test #3, Day-30 Sediment

Figures

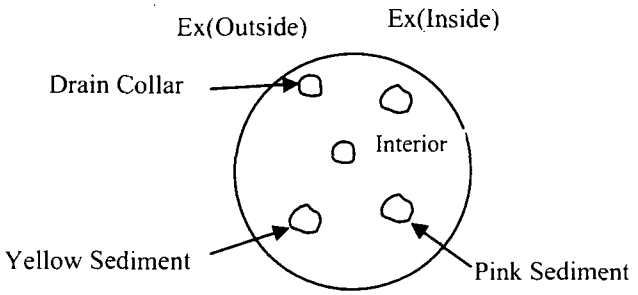
Tables

Particulate sediments at the bottom of the tank directly relate to the corrosion products and debris generated during ICET. This appendix lists the ESEM/EDS and XRD/XRF results for the sediment samples collected from the bottom of the tank on the date Test #3 was shut down (May 5, 2005). The sediment samples were identified and classified by their pink or yellow color. The purpose of these analyses is to provide information on the morphology and composition of these sediments.

ESEM was used to examine the sediment samples without any coating. EDS results provide an elemental composition of the sediment. Based on XRD results, the sediment sample contained crystalline substances of tobermorite [$Ca_{2.25}(Si_3O_{7.5}(OH)_{1.5})(H_2O)$] and calcite ($CaCO_3$), the same as unused raw or unused baked cal-sil samples. XRF results show the chemical composition of the sediment. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Laboratory session from May 6, 2005.
Test #3, Day-30 Sediment.



Pink Sediment

Image:	t3PNKP29	1000 ×	ESEM image	Figure I-1
	t3PNKP29	1000 ×	Annotated ESEM image	Figure I-2
EDS:	t3pnkp30		EDS on image 29	Figure I-3
Image:	t3pnkp31	100 ×	ESEM image	Figure I-4
	t3Pnkp32	100 ×	ESEM image	Figure I-5

Yellow Sediment

Image	t3ylwp34	100 ×	ESEM image	Figure I-6
	t3ylwp34	100 ×	Annotated ESEM image	Figure I-7
EDS:	t3ylwp35		EDS on image 34	Figure I-8
	t3ylwp36		Comparing EDS image t3ylwp35 with pink sediment EDS image t3pnkp30	Figure I-9
Image:	t3ylwp37	100 ×		Figure I-10

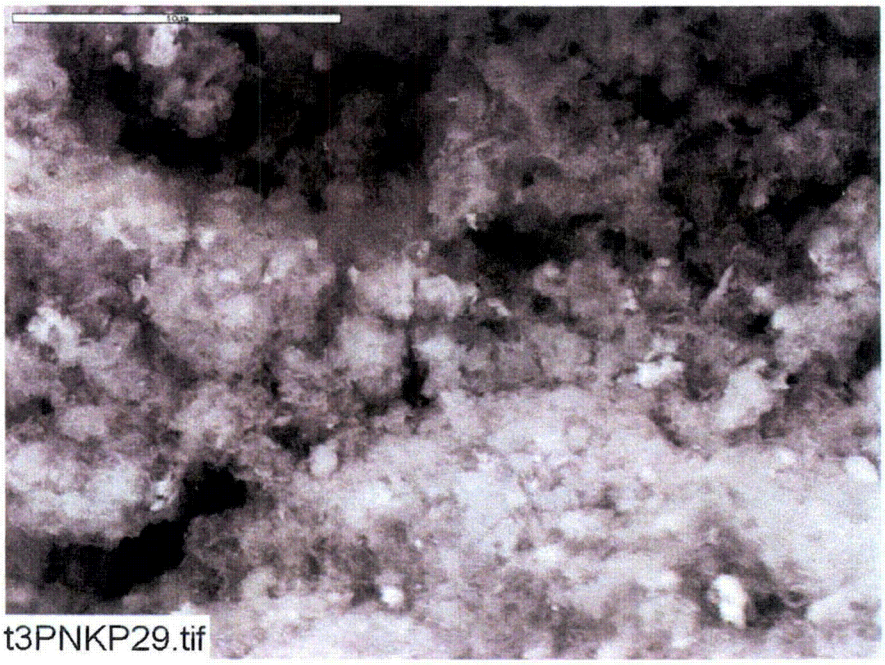


Figure I-1. **ESEM image magnified 1000 times for Test #3, Day-30 pink sediment. (t3PNKP29)**

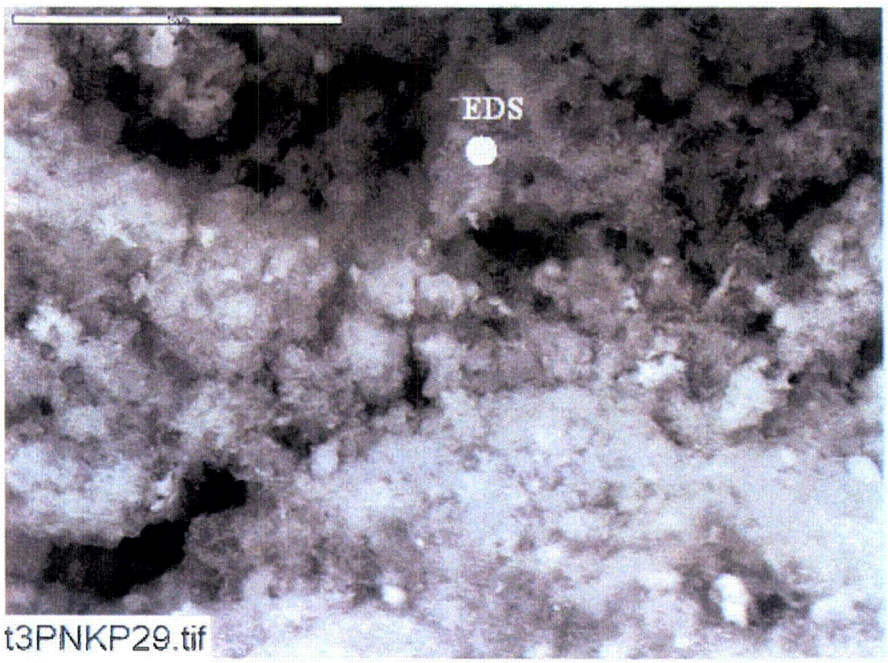


Figure I-2. **Annotated ESEM image magnified 1000 times for Test #3, Day-30 pink sediment. (t3PNKP29)**

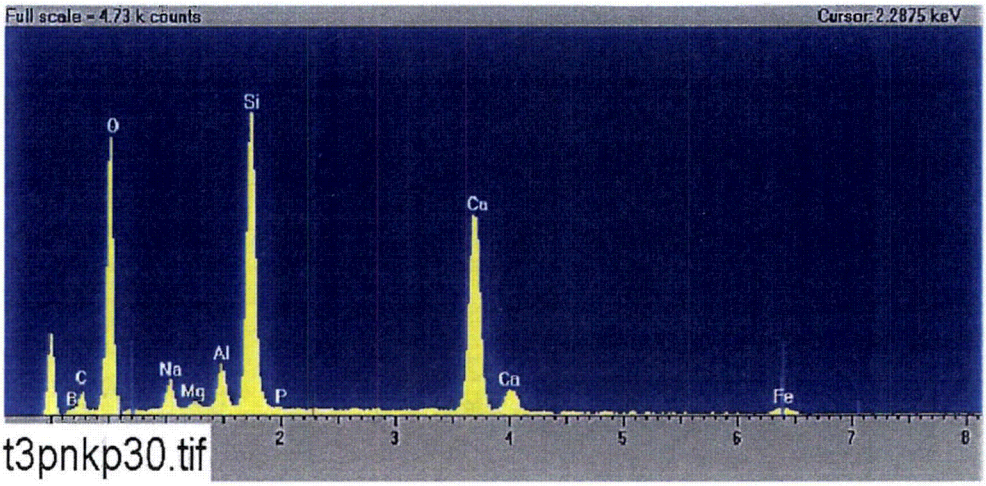


Figure I-3. **EDS counting spectrum for the particles shown in Figure I-2. (t3pnkp30)**

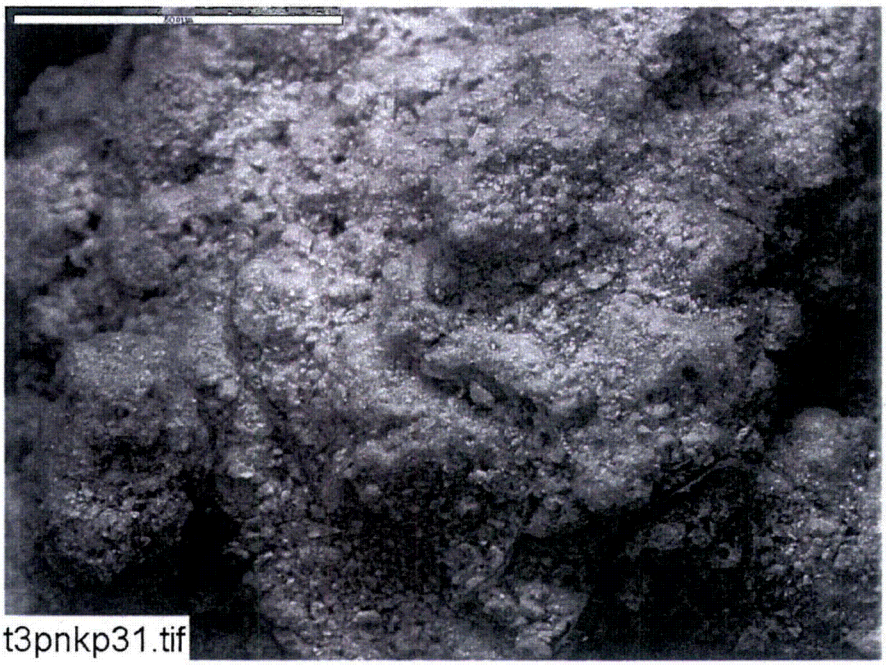


Figure I-4. **ESEM image magnified 100 times for Test #3, Day-30 pink sediment. (t3pnkp31)**

Figure I-5. **ESEM image magnified 100 times for Test #3, Day-30 pink sediment. (t3pnkp32)**

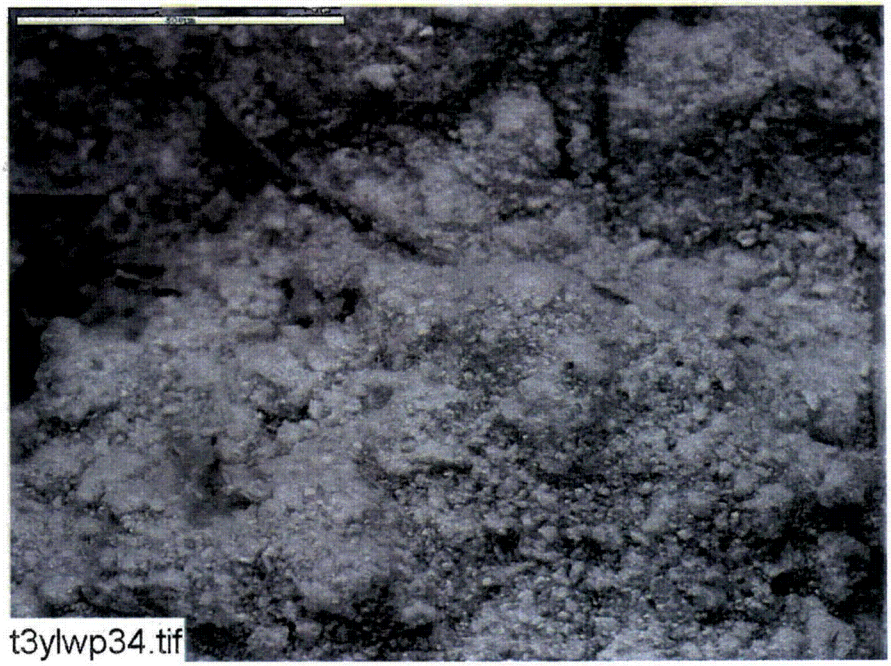


Figure I-6. **ESEM image magnified 100 times for Test #3, Day-30 yellow sediment. (t3ylwp34)**

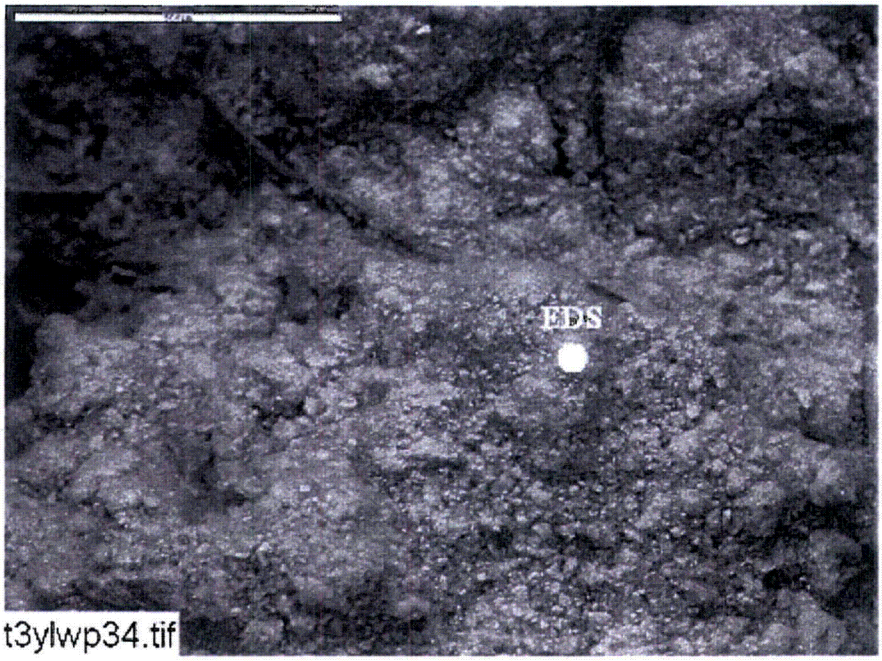


Figure I-7. Annotated ESEM image magnified 100 times for Test #3, Day-30 yellow sediment. (t3ylwp34)

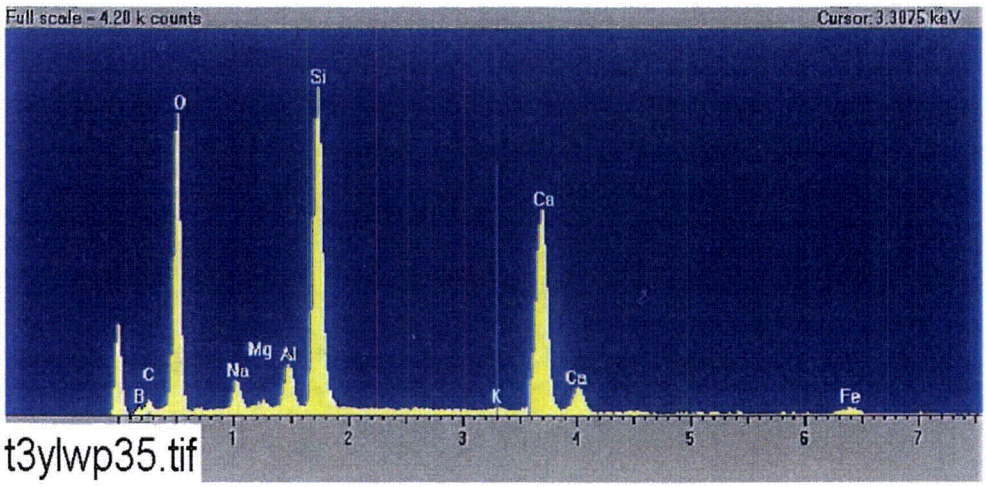


Figure I-8. EDS counting spectrum for the particles shown in Figure I-7. (t3ylwp35)

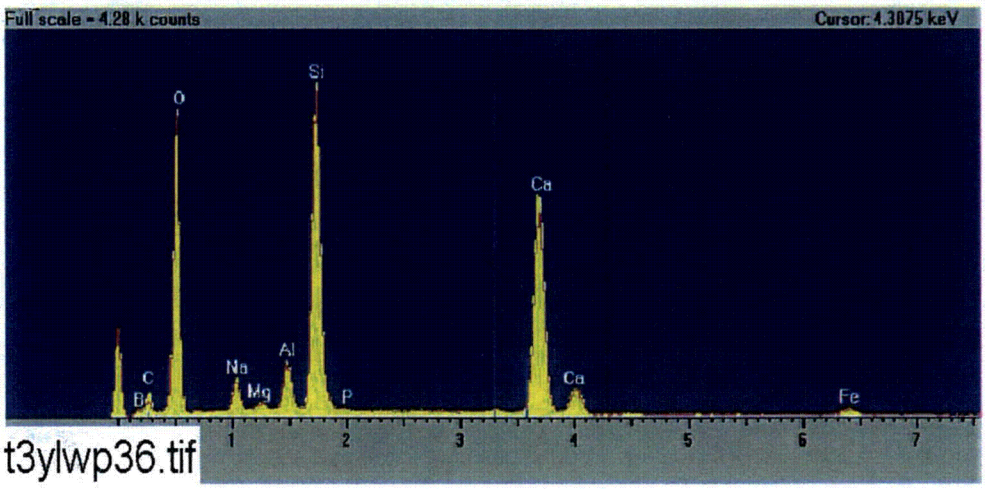


Figure I-9. **Comparison of EDS counting spectra for pink sediment (yellow, t3pnkp30) and yellow sediment (red, t3ylwp35). (t3ylwp36)**

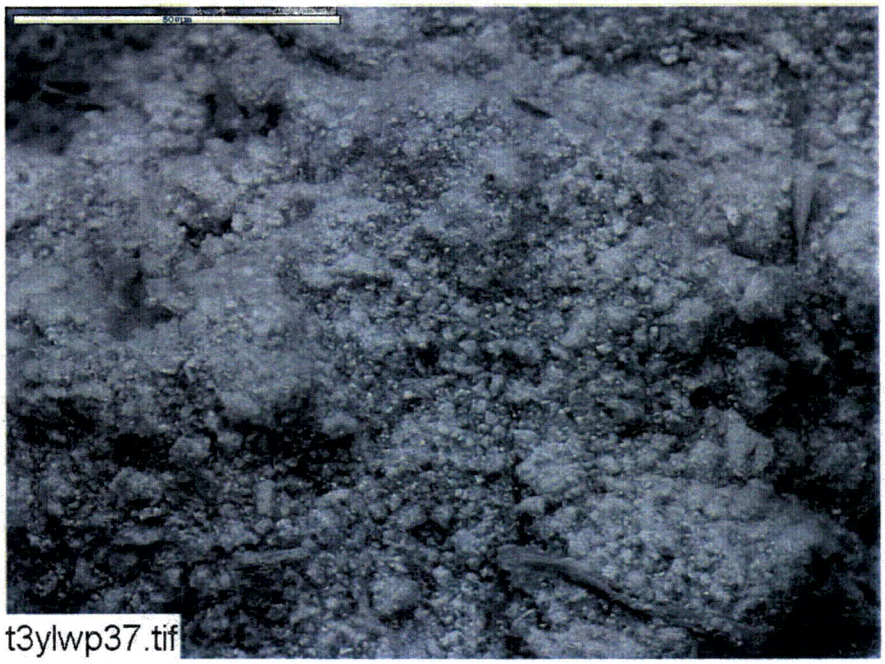


Figure I-10. **ESEM image magnified 100 times for Test #3, Day-30 yellow sediment. (t3ylwp37)**

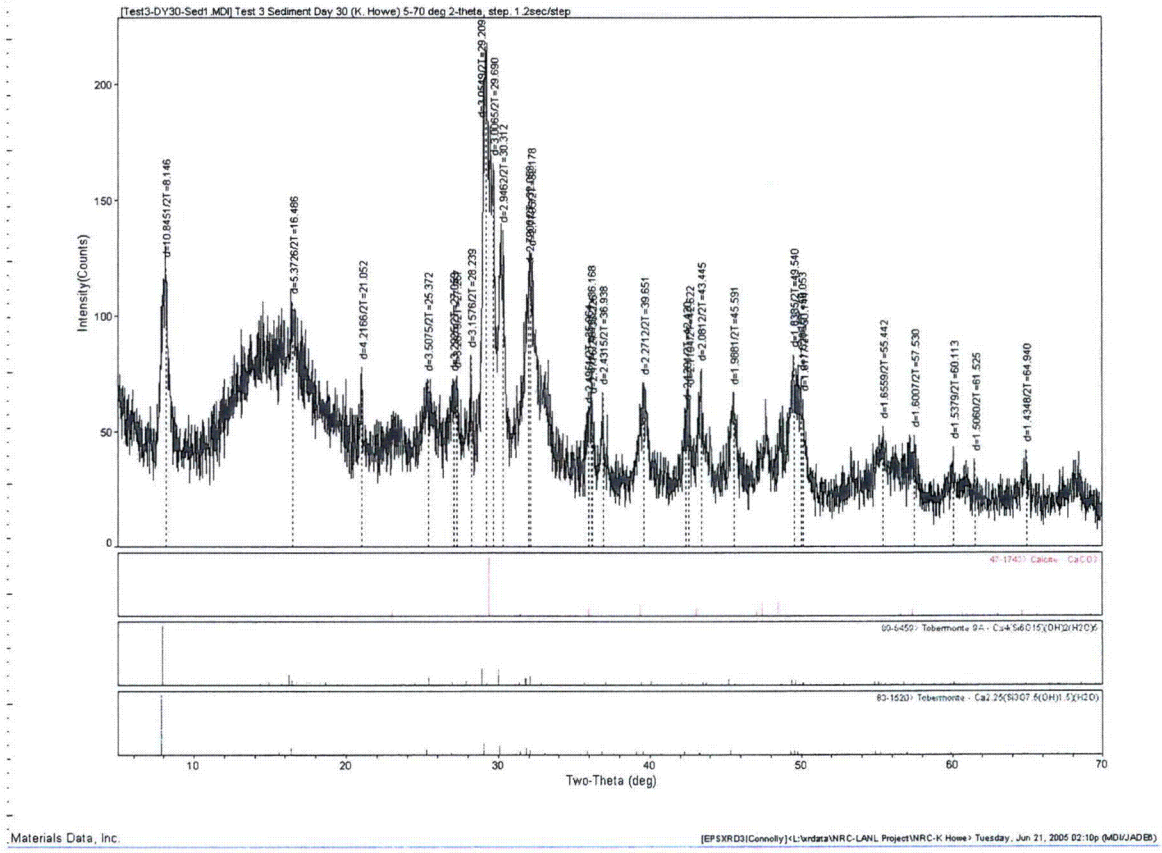


Figure I-11. XRD results forTest #3, Day-30 sediment.

Table I-1. Dry Mass Composition of a Test #3, Day-30 Sediment by XRF Analysis

Sample ID	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	$H_20(-)$	$H_2O(+)CO_2$	P_2O_5	Total	H2O(+)CO2 /DF (10) & Cover. To %
Test #3, Day 30 Sedi-ment	36.29	0.20	4.92	2.24	0.00	0.06	0.62	27.16	2.19	0.45	0.58	20.65	3.05	98.42	1.0211

Appendix J

TEM Data for Test #3 Solution Samples

Figures

This appendix presents TEM images, EDS, and diffraction patterns for Test #3, Day-4, Day-15, and Day-30 filtered and unfiltered solution samples. The filtered solution samples were passed through a 0.7-μm fiberglass filter at 60°C. The unfiltered solution samples were extracted from the tank directly. Based on the results, no significant diffraction pattern was observed because of the amorphous nature of the samples. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Laboratory session from May 6, 2005.

TEM Test #3, Day-4 solution samples
Test #3, Day-15 filtered and unfiltered solution
Test #3, Day 30 unfiltered solution

TEM Filtered Day-4 Solution Samples

Image:	T3D4F1	2000 ×		Figure J-1
EDS:	T3D4FEDS		Spectrum for image T3D4F1	Figure J-2
Image:	T3D4F2	2000 ×		Figure J-3

TEM Unfiltered Day-4 Solution Samples

Image:	T3D4UF2	2000 ×		Figure J-4
EDS:	T3D4UFEDS		Spectrum on image T3D4UF2	Figure J-5
Image:	T3D4UF	2000 ×		Figure J-6

TEM Unfiltered Day-15 Solution Samples

Image:	T3D15U-4k-01	4000 ×		Figure J-7
EDS:	T3D15UFEDS		Spectrum on image T3D15U-4k-01	Figure J-8
Image:	T3D15U-SAD20cm-01	2000 ×		Figure J-9

TEM Unfiltered Day-30 Solution Samples

Image:	T3D30U-4k-01	4000 ×		Figure J-10
EDS:	T3D30UFEDS		Spectrum on image T3D30U-4k-01	Figure J-11
Image:	T3D30U-SAD_	2000 ×		Figure J-12

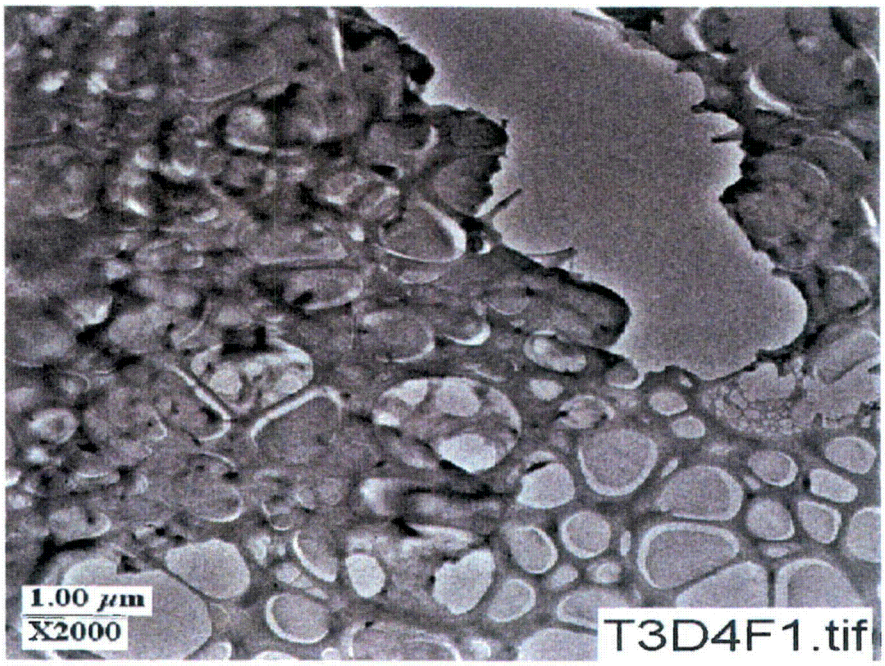


Figure J-1. **Electron micrograph magnified 2000 times for one Test #3, Day-4 filtered sample location. (T3D4F1)**

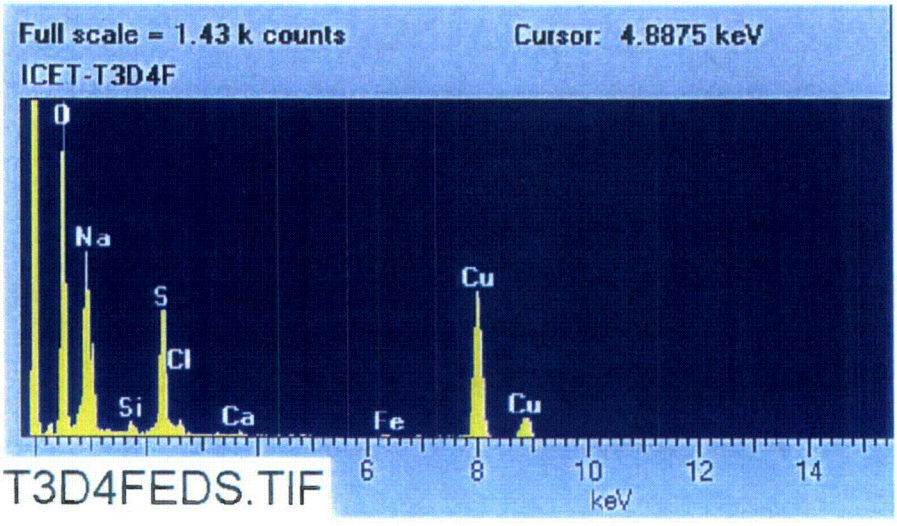


Figure J-2. **TEM energy-dispersive x-ray spectrum for the Day-4 filtered test sample as shown in Figure J-1. (T3D4FEDS)**

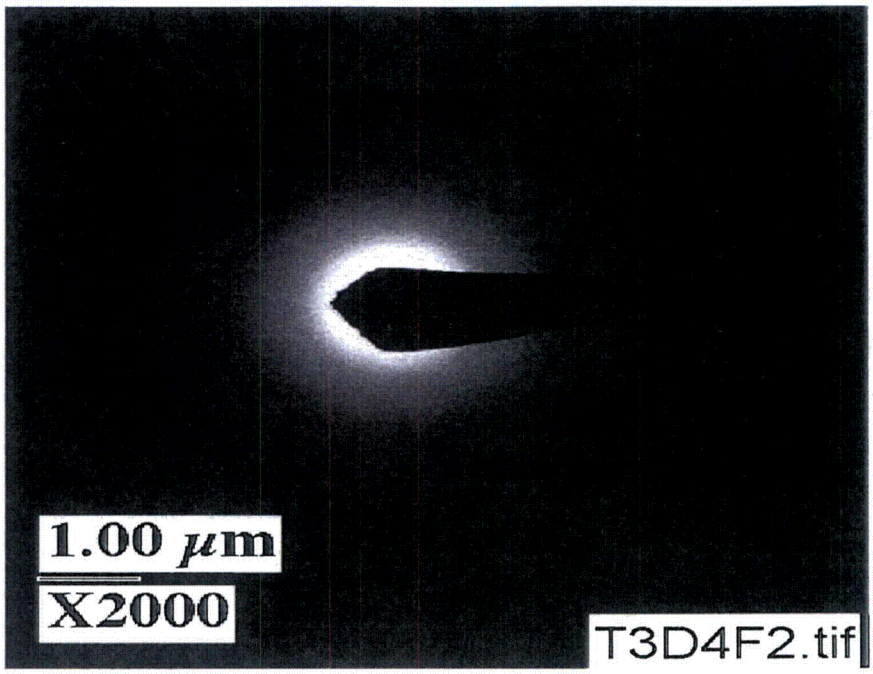


Figure J-3. TEM image for Test #3, Day-4 filtered sample solution (T3D4F2)

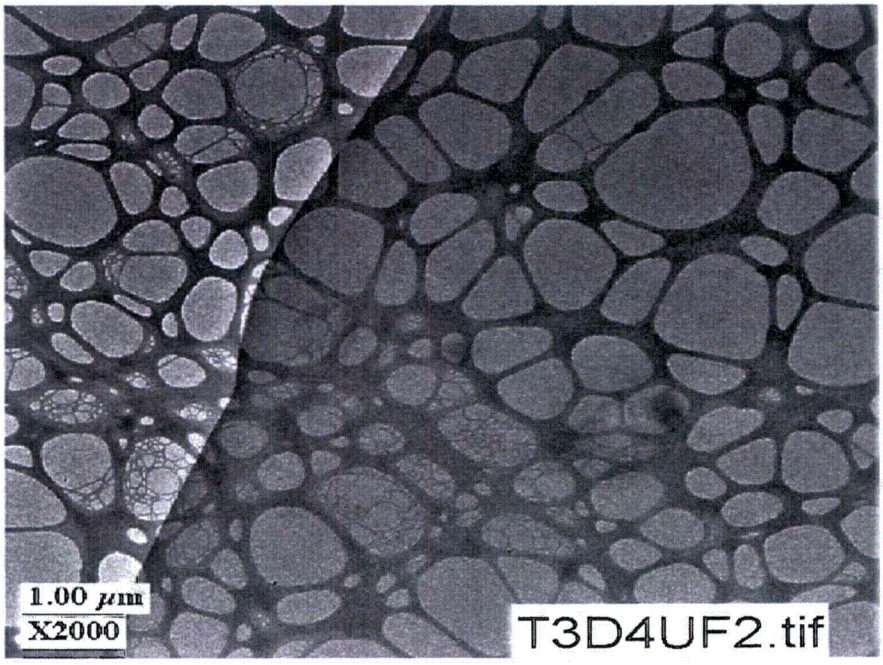


Figure J-4. Electron micrograph magnified 2000 times for one Test #3, Day-4 unfiltered sample location. (T3D4UF2)

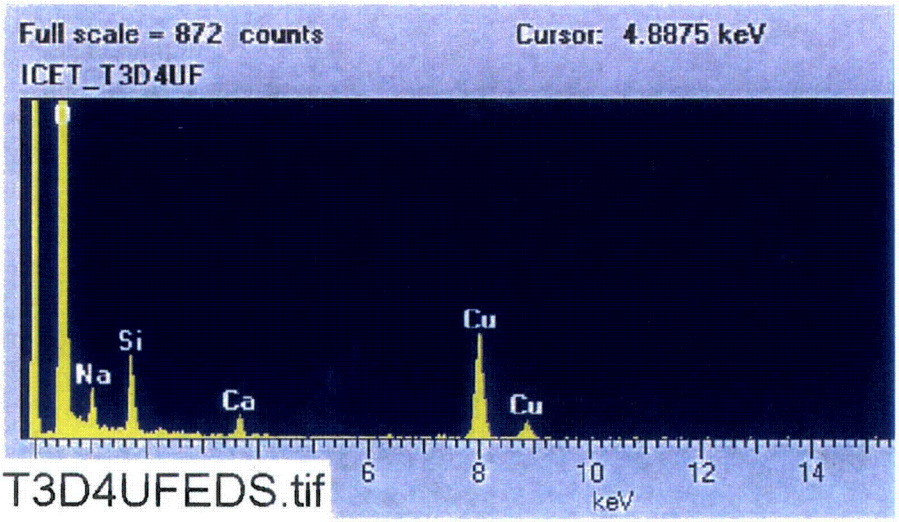


Figure J-5. **TEM energy-dispersive x-ray spectrum for the Day-4 unfiltered test sample as shown in Figure J-4. (T3D4UFEDS)**

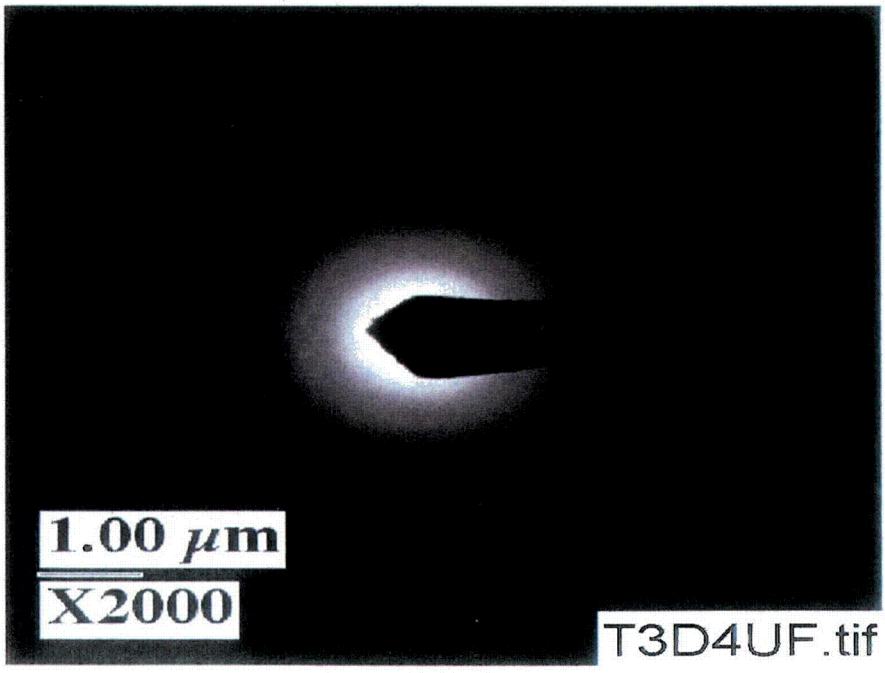


Figure J-6. **TEM image for Test #3, Day-4 unfiltered sample solution (T3D4UF)**

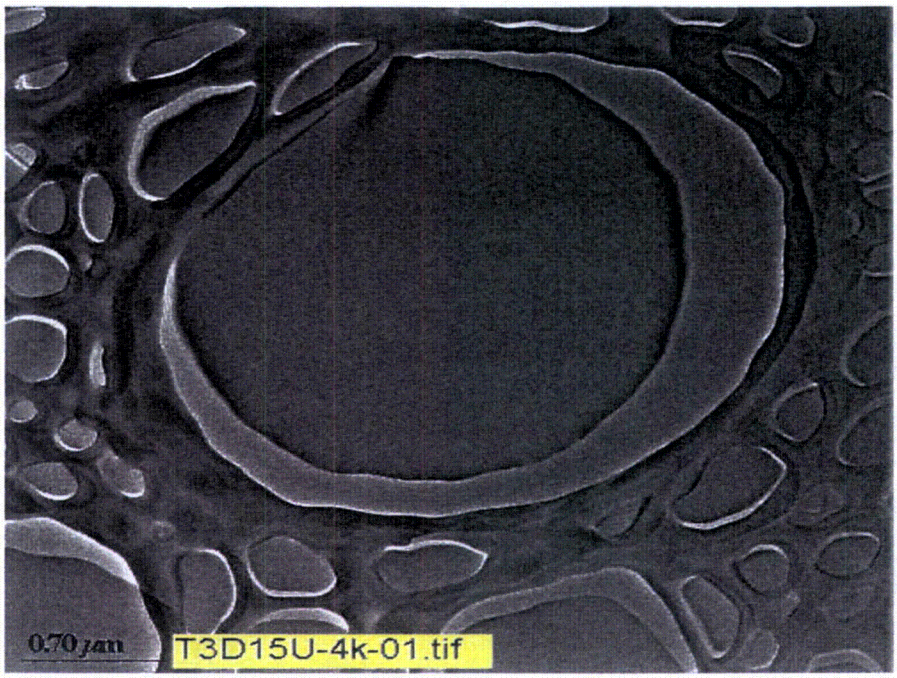


Figure J-7. Electron micrograph magnified 4000 times for one Test #3, Day-15 unfiltered sample location. (T3D15U-4k-01)

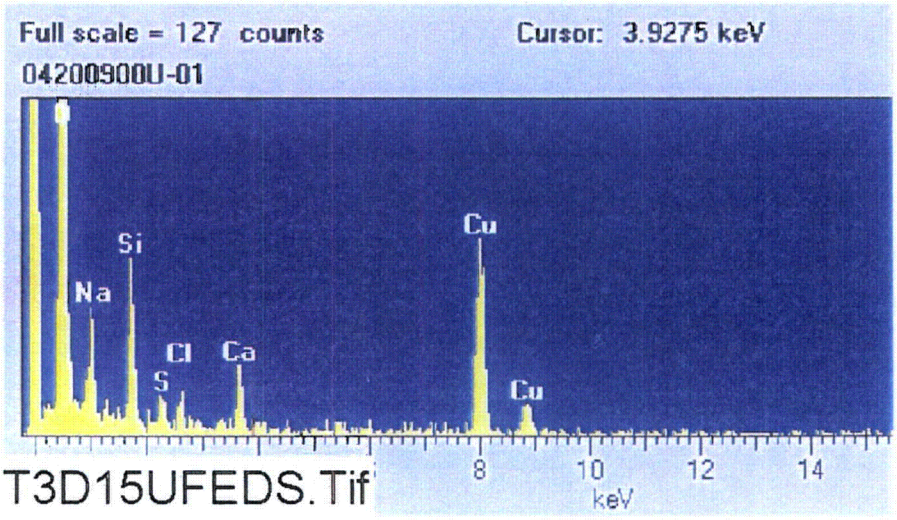


Figure J-8. TEM energy-dispersive x-ray spectrum for Test #3, Day-15 unfiltered test sample as shown in Figure J-7. (T3D15UFEDS)

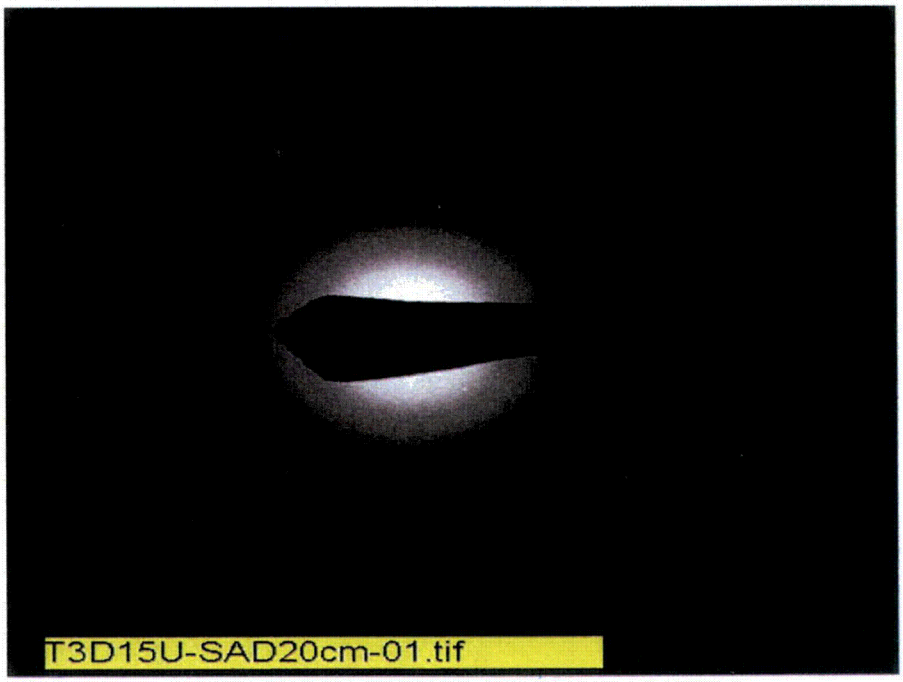


Figure J-9. TEM image magnified 2000 times for a Test #3, Day-15 unfiltered sample solution (T3D15U-SAD20cm-01)

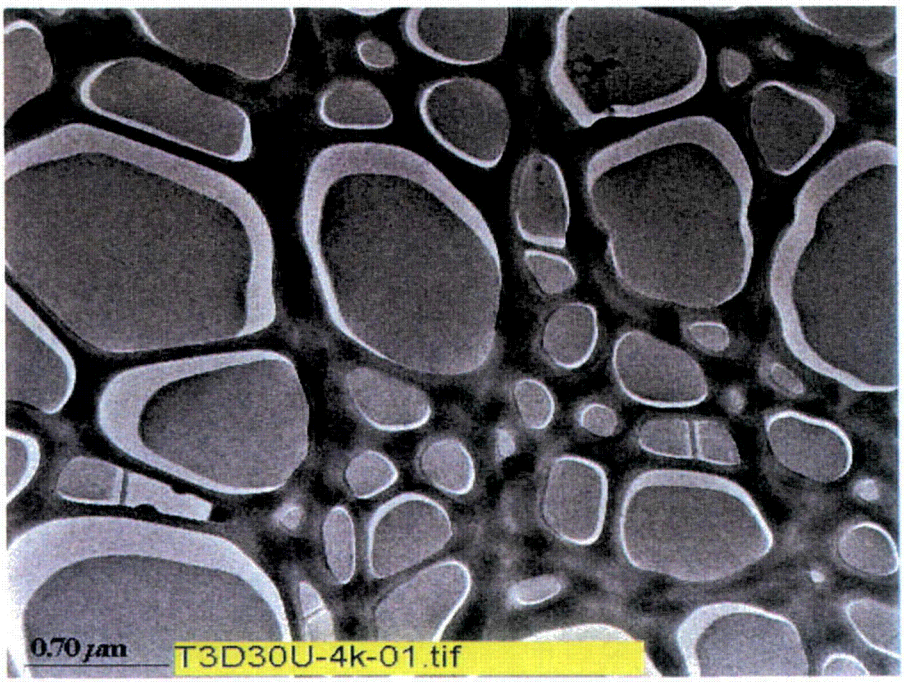


Figure J-10. Electron micrograph magnified 4000 times for one Test #3, Day-30 unfiltered sample location. (T3D30U-4k-01)

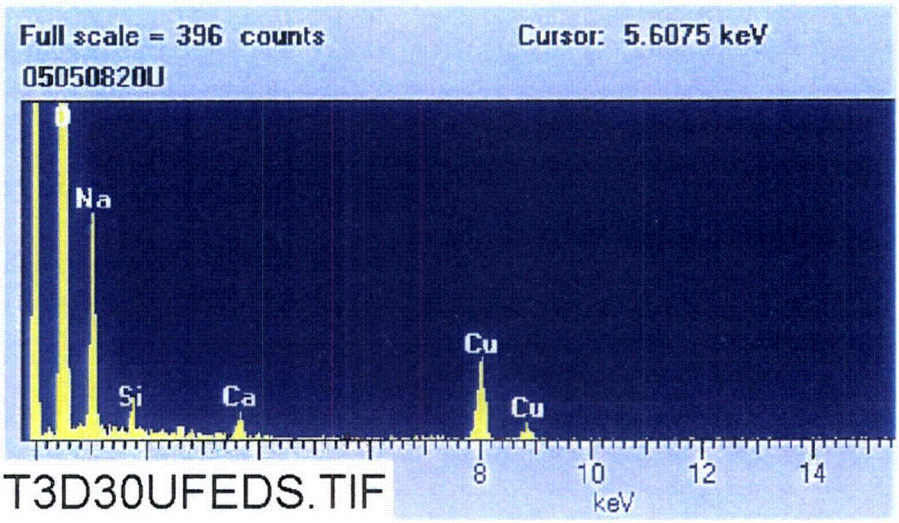


Figure J-11. TEM energy-dispersive x-ray spectrum for Test #3, Day-30 unfiltered test sample as shown in Figure J-10. (T3D30UFEDS)

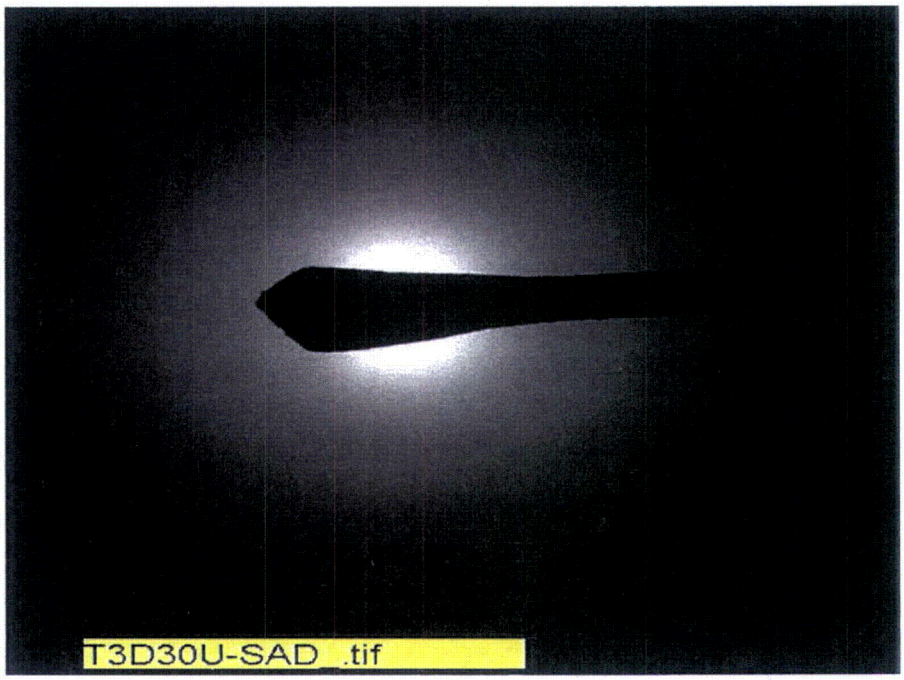


Figure J-12. TEM image magnified 2000 times for a Test #3, Day-30 unfiltered sample solution. (T3D30U-SAD)

Appendix K

UV Absorbance Spectrum—Day-30 Solution Samples

Figures

Tables

This appendix presents the ultraviolet (UV) absorbance result of the Test #3, Day-30 solution sample. The purpose of this analysis was to find any distinguishing absorbance peaks to identify the organics present in the solution. The solution sample at 60°C was collected through a 0.7-μm fiberglass filter to remove particulate impurities, followed by being scanned over the wavelength ranging from 200 to 800 nm by a UV-visible spectrophotometer. The spectrum of deionized water was used as background subtraction. From the result, the test solution did not exhibit any distinguished absorbance peaks. No logbook entries are available for this laboratory session, so, no transcribed notes are provided in this appendix.

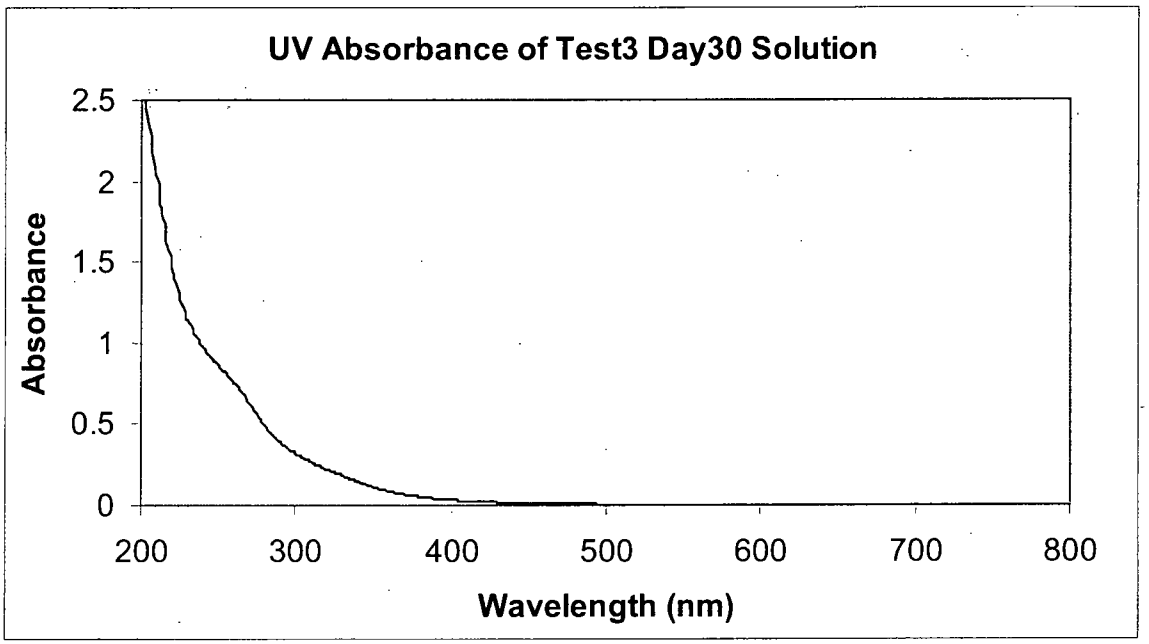


Figure K-1. **UV absorbance spectrum for Test #3, Day-30 solution samples.**

Table K-1. Test #3, Day-30 Solution Sample Laboratory Settings

Test #3, Day-30 Samples	
Collection Time:	5/5/2005 11:33:48 AM
Operator Name:	
Scan Software Version:	3.00(182)
Parameter List :	
Instrument:	Cary 50
Instrument Version:	3.00
Start (nm):	800.0
Stop (nm):	200.0
X Mode:	Nanometers
Y Mode:	Abs
UV-Vis Scan Rate (nm/min):	600.00
UV-Vis Data Interval (nm):	1.00
UV-Vis Ave. Time (s):	0.1000
Beam Mode:	Dual beam
Baseline Correction:	On
Baseline Type:	Baseline correction
Baseline File Name:	
Baseline Std Ref File Name:	
Cycle Mode:	Off
Comments:	
Method Log:	
Method Name:	Default
Date/Time Stamp:	5/5/2005 11:28:54 AM
Method Modifications:	
Cell Changer 6 × 6 Changed:	5/5/2005 11:28:58 AM / Old:1 / New:0
UVVIS SAT Changed:	5/5/2005 11:29:22 AM / Old:0.0125 / New:0.1000
NIR SAT Changed:	5/5/2005 11:29:22 AM / Old:0.0125 / New:0.1000
Common SAT Changed:	5/5/2005 11:29:22 AM / Old:0.0125 / New:0.1000
Baseline Correction Changed:	5/5/2005 11:29:22 AM / Old:0 / New:1
Temp Controller Changed:	5/5/2005 11:29:26 AM / Old:0 / New:2

Sipper Type Changed:	5/5/2005 11:30:11 AM / Old:Internal RSA / New:External sipper
End Method Modifications	
<Current Wavelength>	200.1

Appendix L

ICET Test #3: Pre-Test, Test, and Post-Test Project Instructions

The ICET series is conducted under the guidance of PIs, which identify the steps to follow for certain activities. These PIs are revised or rewritten as needed for each test. For Test #3, a new PI was written to address test operations. The PIs that address pre-test and post-test operations were revised for Test #3. These three PIs are included in this appendix to describe more completely the test apparatus and chemical solution preparations, the test startup and daily sampling, and the steps followed after test shutdown.

1.0 INTRODUCTION

1.1 PURPOSE

The purpose of this instruction is to ensure that all data acquisition, test samples, testing supplies, chemicals, and related materials are ready and accounted for prior to testing. In addition, this instruction provides instructions on preparing the chemical test apparatus for testing.

1.2 SCOPE

The pre-test operations preparation will ensure that successful initiation of the testing activity is achieved.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- Test 2 Chemical Additive Analysis – ICET-CALC-011
- Laboratory Safety Guidelines
- ASTM A 380 – 99, Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems
- Material Safety Data Sheets (MSDS) for all chemicals involved

2.0 PREREQUISITES

The data acquisition setup and inspection; instrument calibration; and the coupon receipt, preparation, inspection, and storage tasks must be completed in full prior to the completion of this activity. Fiberglass and calcium silicate (cal-sil) samples must be weighed and their planned locations in the tank identified. That data must be recorded.

2.1 Training Requirements

The following personnel training is required for this task:

1) LabVIEW and computer data acquisition training
2) Chemical handling training, specifically for ethyl alcohol, ammonium hydroxide, and lithium hydroxide.
3) Safe lift execution training

2.2 Equipment Requirements

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, eye wash station.

3.0 DOCUMENTATION REQUIRED

MSDSs must be available for all chemicals used.

A lab notebook must be maintained throughout the pre-test operations instruction. Contained within the lab notebook will be the date, times, description of activities, and quantities of chemicals added, number of cleanings, and physical observations of the tank cleaning and preparation procedures.

4.0 HAZARDS

The hazards associated with this activity include potential injuries associated with chemical handling.

5.0 INSTRUCTIONS

1. Ensure that all testing materials and supplies are ready and on-site. See checklist at the end of this document. Verify that eye wash station is operational. Note: The following solutions are not used in this instruction, but are to be prepared in advance of entering ICET-PI-014, "Test Operations, Test #3 (cal-sil and fiberglass with TSP)." After preparation, clearly label the containers with the solutions and place in an area restricted for ICET Project test use.
2. Prepare 21.2 g of concrete dust and 63.7 g of latent debris.
3. Prepare LiOH solution: dissolve 0.663 g of lithium hydroxide (LiOH) into about 100 mL water in a 250-mL sample container.
4. Prepare TSP Solution Batch 1.
   a. Heat about 1.5 gallons of demineralized water, add 300 g of boric acid (H_3BO_3), and stir until the boric acid is dissolved. Pour the solution into a 5-gallon plastic container. Dissolve the boric acid in multiple batches if necessary.
   b. Add additional demineralized water to the 5-gallon plastic container until it contains about 4 gallons.
   c. Dissolve 1893 g of TSP (Na_3PO_4-$12H_2O$) into the water in the container.
   d. Dilute with additional demineralized water until the volume is 5 gallons.
   e. Label container as "TSP Solution Batch 1."
5. Prepare TSP Solution Batch 2.
   a. Heat about 1.5 gallons of demineralized water, add 300 g of boric acid (H_3BO_3), and stir until the boric acid is dissolved. Pour the solution into a 5-gallon plastic container. Dissolve the boric acid in multiple batches if necessary.

b. Add additional demineralized water to the 5-gallon plastic container until it contains about 4 gallons.
c. Dissolve 1893 g of TSP (Na_3PO_4-$12H_2O$) into the water in the container.
d. Add 211 mL of 12.29 N hydrochloric acid (HCl) to the water in the container.
e. Dilute with additional demineralized water until the volume is 5 gallons.
f. Label container as "TSP Solution Batch 2."

6. Prepare laboratory control sample (LCS). See ICET-PI-005, "Chemical Sampling and Analysis," for details on the laboratory control sample.
7. Start the data acquisition system. Verify that the data acquisition system is monitoring flow rate, pump speed, temperature, and pH.
8. Clean the tank and piping.
   a. Cleaning should commence as soon after a test is completed as possible, to prevent material from hardening in the tank or piping and to maximize the time available for cleaning.
   b. Cleaning chemicals may consist of weak acids (e.g., acetic acid, citric acid, or dilute mineral acids), weak bases (e.g., ammonium hydroxide), week organic solvents (e.g., ethanol), or detergents/surfactants (e.g., trisodium phosphate, sodium dodecyl sulfate), as necessary. Cleaning solutions can be heated if necessary. Note that the discharge limit to the sanitary sewer is a maximum temperature of 140 °F and pH between 5.0 and 11.5. Cleaning solutions that are not within this range should be neutralized before discharge.
   c. During cleaning, the pump should be run and water directed through both recirculation lines (through the spray nozzles and lower headers)
   d. The sample line should be removed from the piping, physically cleaned, and carefully inspected. If the sample line cannot be adequately cleaned, it should be replaced.
   e. After each cleaning step, the tank and piping should be thoroughly rinsed with tap water or demineralized water.
   f. After each cleaning step, a segment of pipe should be removed, and the interior of the pipe visually inspected.
   g. Cleanliness criteria: When the tank visually appears to be satisfactorily cleaned, the tank and piping should be thoroughly rinsed with demineralized water. The interior surfaces of the tank and piping shall be free of any deposits that can be removed by vigorous scrubbing. Demineralized water drained from the tank should have turbidity less than 0.3 NTU and conductivity less than 50 uS/cm.
9. Tank is now ready for testing. Proceed immediately to Instruction No. ICET-PI-014, "Test Operations, Test #3 (cal-sil and fiberglass with TSP).

6.0 ATTACHMENTS

No forms are attached to this document.

7.0 Materials Checklist

_____ lithium hydroxide, 0.663 g

_____ TSP, 3.785 kg

_____ 211 mL of 12.29 N HCl

_____ Boric acid, 600 g

_____ tap water supply

_____ demineralized water production system

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ analytical balance

_____ top loading balance

_____ chemical spatula

_____ chemical scoop

_____ weigh boats

_____ two 5-gallon plastic containers

_____ 250 mL graduated cylinder

_____ 250-mL HDPE or PP bottle

_____ 2.5 gallons ethanol

_____ 2.5 gallons ammonium hydroxide

_____ turbidimeter and associated equipment

_____ conductivity meter and associated equipment

1.0 INTRODUCTION

1.1 PURPOSE

The intent of the instruction is to outline the steps that are to be followed during testing.

1.2 SCOPE

This activity forms the core of the entire Chemical Effects Testing project. All activities involved in this project affect and are affected by this activity.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- ASTM Standard G 4-01
- ASTM Standard D 3370-95a
- ASTM Standard G 31-72
- Material Safety Data Sheets (MSDS) for all chemicals involved
- LabVIEW operation manual
- Laboratory Safety Guidelines
- Test 2 Chemical Additive Analysis – ICET-CALC-011
- John Gisclon email to Bhagwat Jain, Cal-sil Information Used in Test #3, March 31, 2005

2.0 PREREQUISITES

All sample coupons must be placed in their corresponding racks. Also, the pre-operation test preparation activity must be completed in full.

2.1 Training Requirements

The following personnel training is required for this task:
1) LabVIEW and computer data acquisition training.
2) Chemical handling training for all chemicals involved.

2.2 Equipment Requirements

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, hard hats, steel-toed shoes, eye wash station, hydrogen detector and hydrogen removal system.

3.0 DOCUMENTATION REQUIRED

A lab notebook must be maintained throughout the testing procedure. In addition, a binder will be maintained that includes pertinent test instructions and the completed daily log sheets (see Attachment A). The daily log sheet contains the date, times, physical description, and quantity of fiberglass and water samples obtained each day. In addition, the daily log sheet contains information from the data acquisition system (DAS), the water samples taken, and other test information.

The electronic data that are acquired are backed up daily and stored in a separate location each testing day. Refer to ICET-PI-001, Data Acquisition Setup and Inspection.

4.0 HAZARDS

The hazards associated with this activity include tipping of the chemical tank assembly, ingestion and/or respiration of any chemicals involved, and scalding and/or burning hazards involved in daily tank venting, and possible hydrogen gas generation from corrosion reactions. Appropriate measures to control hydrogen gas must be in place before operations commence.

Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

5.0 INSTRUCTIONS

1. Because of the time required for heating the tank contents and dissolving chemicals, this sequence should be started at least 48 hours before the scheduled time t = 0. Pre-Test Operations preparation should be complete before proceeding with this sequence.
2. Ensure that all testing materials and supplies are ready and on-site (see checklist at end of this instruction).
3. Add 240 gallons of RO water to the tank by pumping water from the RO skid through the totalizing flow meter. Record flow to the nearest 0.5 gallon.
4. Verify valves are positioned as follows:

Valve	Description	Position
V-1	tank drain	closed
V-2	pump isolation	open
V-3	instrument loop supply	open
V-4	instrument loop discharge	open
V-5	instrument loop bypass	closed
V-6	in-line filter isolation	open
V-7	tank spray supply	closed
V-8	recirculation supply	open
V-9	sample line	closed
V-10	loop drain	closed
V-11	recirculation line injection	closed

5. Start pump and adjust to flow rate of approximately 25 gpm.
6. Start computer, start LabVIEW, verify that flow rate, pump speed, temperature, and pH are being recorded properly.
7. Turn on heater and allow water in tank to heat to 60 °C ± 2 °C. (This may take up to 20 hours.)
8. Add the pre-mixed LiOH solution.
9. Add 14.54 kg of boric acid (H_3BO_3), weighing in approx. 2 kg increments, recording the weight of each increment to the nearest 10 g.
10. Allow the water to circulate until the solution is visibly clear, indicating that the boric acid is completely dissolved.
11. Allow water in tank to heat to 65 °C ± 2 °C.
12. Take grab water sample for analysis for the parameters identified in steps a – h below. Also record physical appearance of the sample (clarity, presence of gelatinous material, etc). All Day 1 and subsequent samples will be analyzed by Assaigai Analytical Laboratory. In addition, periodic test samples and laboratory control samples (LCSs) will also be analyzed by the UNM laboratory.
    a. pH
    b. temperature
    c. turbidity
    d. viscosity
    e. total suspended solids (TSS)
    f. dissolved oxygen (DO)
    g. chloride
    h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved
13. Add 21.2 g of concrete dust and 63.7 g of latent debris samples (prepared earlier), wait 10 minutes, take 100 mL water sample for particulate size distribution, density, and TSS.
14. Stop pump.
15. Add the pre-determined amount of cal-sil dust. This will be approximately 43.5 lb.

16. Place coupon racks, fiberglass holders, and cal-sil holders into tank. This is done in accordance with previously determined quantities, size distributions, and locations. (Details of the cal-sil preparation and size distributions are given in the referenced email.)
17. Verify locations of coupon racks, fiberglass holders, and cal-sil holders.
18. Verify the tank temperature is 62 °C. (Because the tank lid will be off, the test will be started with the water temperature at its upper limit.)
19. Start pump and adjust pump speed to 25 gpm.
20. Open valve V-7 (tank spray supply) to direct water to nozzles and adjust valves V-7 (tank spray supply) and V-8 (recirculation supply) until nozzle flowmeter is reading 3.5 gpm. Verify total flow is still 25 gpm and adjust variable frequency drive (VFD) if necessary.
21. Record date and time at which nozzle flow started. This is time t = 0 for the test.
22. The spray phase will begin with the tank lid off. The objective is to be able to carefully monitor possible nozzle blockage and take immediate action to prevent it. At the first sign that a nozzle may be starting to plug, the spray flow rate should be increased rapidly to 5-10 gpm for approximately 5 s. (As long as the nozzle spray pattern is not affected however, the spray flow should remain at 3.5 gpm.) If a nozzle should block in spite of the increased flow rate, a stainless steel wire should be used to clear the nozzle exit.
23. If the spray through the nozzles is not affected by the cal-sil and shows no signs of blocking for a period of 15 minutes, the tank lid should be put in place. Flow through the nozzles should continue to be monitored every 5 minutes by looking through the tank view windows. In addition, every 15 minutes, the spray flow should be bumped to 5-10 gpm for approximately 5 s.
24. At 30 minutes, open valve V-11 (recirculation line injection) and start chemical metering pump from TSP Solution Batch 1 at a rate of 0.0476 gpm (180 mL/min). The objective here and in step #25 is to add a total of 10 gallons of TSP solution in 3.5 hours.
25. After 2 hours, switch the chemical metering pump to TSP Solution Batch 2.
26. Take a measurement of hydrogen concentration. At 2-hour increments, repeat the hydrogen concentration measurement. If the concentration reaches 10% of the flammability limit, purge the tank atmosphere. This needs to be repeated until the hydrogen concentration has been determined to be below 10% of the flammability limit, and then the frequency of hydrogen concentration measurements is to be re-evaluated.
27. At t = 4 hours, stop the chemical metering pump and close valves V-7 and V-11.
28. Immediately after closing valves V-7 and V-11 (at t = 4 hours), take water grab sample for analysis for the parameters listed below. Record the time of sample collection.
    a. pH
    b. temperature
    c. turbidity

d. viscosity
e. chloride
f. total suspended solids (TSS)
g. dissolved oxygen (DO)
h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved

29. At t = 24 hours, and daily thereafter, take water grab sample for analysis for the parameters listed below. (The LANL PI will propose a different sampling frequency to the project sponsors if test data support it.) Record the time of sample collection.
    a. pH
    b. turbidity
    c. viscosity
    d. temperature
    e. total suspended solids (TSS)
    f. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved. An exception is that B, Li, K, Pb, and chloride analyses will be performed only at t = days 15 and 30. Also, dissolved oxygen will be measured at day 30.
30. During each daily water sample collection, look inside tank (through windows) and record observations. If the tank water level indicates that the water volume is 245 gallons or less, add RO water to bring the volume up to 250 gallons and record the amount added.
31. At t = 24 hours, weekly thereafter, and at the end of the test, collect 100 mL water sample for particulate size distribution and density analysis, to be performed at AALI. The particulate size ranges to be used will be as close as possible to those called out in the test plan: (in microns), 1-10, 11-25, 26-50, 51-75, 76-100, and > 100 microns.
32. At t = 24 hours, weekly thereafter, and at the end of the test, collect water samples for strain rate viscosity measurements (see PI-010 for sample details.)
33. At 3 days $\leq$ t $\leq$ 5 days, 14 days $\leq$ t $\leq$ 16 days and at the end of the test, collect a sacrificial fiberglass sample to be inspected and examined with SEM.
34. At 24 hours, at 14 days $\leq$ t $\leq$ 16 days and at the end of the test, run 1L of water through a nucleopore filter. The filter will be taken for SEM analysis as specified in ICET-PI-007. (Note that depending on the solution, some filter material will not work well for this operation. If possible, use a nucleopore filter for SEM analysis, and then collect a second sample on nitrocellulose filter for later digestion and ICP analysis.)
35. Shut down pump
36. Indicate end of test on the data acquisition system and shut down the data acquisition software.
37. Proceed directly to PI-008 Post-Test Operations.

6.0 ATTACHMENTS

Attachment A. Daily Log Sheet.

7.0 MATERIAL CHECKLIST

_____ boric acid, 14.54 kg

_____ pre-mixed lithium hydroxide solution

_____ concrete dust, 21.2 g

_____ latent debris, 63.7 g

_____ Nucleopore filter

_____ TSP, 3786 g evenly mixed in two 5-gallon containers

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ top-loading balance

_____ weigh pan for 2 kg aliquots of boric acid

_____ stainless steel filter paper holder

_____ 500 mL graduated cylinder (for TSS)

_____ totalizing flow meter

_____ sample containers (see Chemical Sampling Instruction)

_____ analytical equipment (see Chemical Sampling Instruction)

_____ pre-assembled coupon racks

_____ pre-assembled fiberglass baskets, total of 2.2 lb of fiberglass

_____ pre-assembled cal-sil baskets, total of 26.7 lb of cal-sil

_____ pre-measured cal-sil dust, 43.5 lb

_____ coupon handling safety equipment (hard hat, leather gloves, boots)

_____ computer disks for backup of Labview data

_____ Masterflex peristaltic pump and tubing

_____ demineralized water production system

Attachment A. Daily Log Sheet

Daily Log Sheet
Integrated Chemical Effects Test (Test # 2)

Date: _____ Time of sample collection: _____
Sample taking and data reduction by _____ and _____

Sample bottle identification:
Assaigai (total): _____
Assaigai (filtered): _____
UNM (total): _____
UNM (filtered): _____

Control system readings:
Temperature: _____ Flow: _____ pH: _____

Analyses:
Volume filtered for TSS: _____ pH: _____
Temperature: _____ Dissolved oxygen: _____
Turbidity (at 60 °C): _____ (at 23 °C; and 10 min.)_____
Viscosity, unfiltered (60 °C): _____ (at 23 °C) _____
Viscosity, filtered (60 °C): _____ (at 23 °C) _____
Water Level: _____ Water Added: _____
Hydrogen: _____ Other: _____
Fiberglass or other samples taken: _____
TSS filter #: _____ TSS (mg/L): _____

Comments:

Observations written in lab notebook by _____
☐ Continued on back

1.0 INTRODUCTION

1.1 PURPOSE

The intent of this instruction is to ensure that the experimental samples are removed from the test apparatus, the test apparatus is rinsed and inspected, and the test apparatus is made ready for subsequent pre-test operations.

1.2 SCOPE

This activity marks the end of one chemical effects test run. Experimental sample removals and inspections, test apparatus rinsing, and preparations for cleaning and subsequent tests are addressed here.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- ASTM Standard G 4-01
- ASTM Standard G 31-72
- ICET-PI-002, Coupon Receipt, Preparation, Inspection, and Storage, November 19, 2004
- ICET-PI-014, Rev. 0, Test Operations, Test #3 (cal-sil and fiberglass, with TSP, April 5, 2005
- ICET-PI-005, Rev. 1, Chemical Sampling and Analysis, February 3, 2005
- Laboratory safety guidelines
- ICET Project Safety Plan

2.0 PREREQUISITES

All test operation PI criteria must be completed prior to conducting this task.

2.1 Training Requirements

- Laboratory Safety Guidelines
- ICET Project Safety Plan

2.2 Equipment Requirements

A city tap water supply outlet is required for this activity and chemical handling and lifting safety equipment. A reverse osmosis unit is required for the final flush.

3.0 DOCUMENTATION REQUIRED

Documentation related to test parameters, chemical water analyses, coupon and fiberglass examinations, and daily test operations are outlined elsewhere. In this instruction, the steps required to remove samples from the test apparatus and to make it ready for the next test are outlined. In addition, observations as to the test apparatus' condition are obtained and recorded here.

4.0 HAZARDS

The hazards associated with this activity include ingestion/respiration and/or dermal and eye contact with residual chemicals. Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

5.0 INSTRUCTIONS

1) On the last day of testing, collect water samples and perform analyses as outlined in ICET-PI-014 and ICET-PI-005.
2) Remove 10L of water from the test apparatus and store at test temperature, for future analyses
3) Shut off the recirculation pump.
4) Remove the small fiberglass samples for SEM examination.
5) Leave one heater on and continue to monitor tank water temperature.
6) Isolate and drain the test apparatus piping.
7) Remove the tank lid.
8) Before removing coupon racks or insulation samples, examine and take photographs and notes of the inside of the tank, the coupons and racks, and the insulation samples.
9) Remove the six non-submerged coupon racks to a staging area for drying and post-test examinations (refer to ICET-PI-002).
10) Take additional photographs of the inside of the tank.
11) Drain the tank slowly, down to the level that uncovers the submerged rack, but keeping the water level above the heater.
12) Remove the submerged coupon rack to the staging area.
13) Repeat step # 10.
14) Turn off the heater.
15) Completely drain the tank, taking precautions so that the sediment on the bottom of the tank is not disturbed any more than necessary.
16) Store water that was drained from the test apparatus until it is cleared for disposal or shipment. (This step was just moved from later in the PI – the old step #26.)
17) When the tank is drained, repeat step # 10. Note especially the locations and orientations of the remaining samples.
18) Remove the remaining insulation samples to the staging area to dry.

19) Ensure that all samples removed from the tank are clearly marked as to their location and orientation within the tank.
20) After all samples have been removed, repeat step # 10.
21) Inspect the interior of the tank, noting any observations.
22) Note the presence of any sediment. Carefully remove as much sediment as possible, noting any unique aspects of it, such as location. Place the sediment in plastic containers with lids, marking the location of the sediment in the tank.
23) Remove the tank drain screen and remove the insulation sample for future analysis.
24) Remove the flow meter from the loop and take pictures of the flow meter interior.
25) Remove any deposits within the flow meter and place the deposits in plastic containers with lids. This is to keep the samples hydrated.
26) Remove a section of pipe, take pictures of the pipe interior, and remove and store any deposits there.
27) Replace the flow meter and piping section.
28) Rinse the tank with tap water and drain the water.
29) Fill the system with 250 gallons of tap water and circulate water through the spray nozzles and recirculation headers for at least 60 minutes. Repeat with de-mineralized water.
30) If any signs of deterioration are observed on the inside of the test apparatus tank, remove selected insulation on the tank. Inspect the stainless steel tank for any abnormalities.

6.0 ATTACHMENTS

No forms are attached to this document.

NRC FORM 335 (9-2004) NRCMD 3.7

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10. SUPPLEMENTARY NOTES

B. P. Jain, NRC Project Manager; prepared in cooperation with Electric Power Research Institute

11. ABSTRACT *(200 words or less)*

A 30-day test was conducted in the Integrated Chemical Effects Test (ICET) project test apparatus. The test simulated the chemical environment present inside a pressurized water reactor containment water pool after a loss-of-coolant-accident. The initial chemical environment contained 14.54 kg of boric acid and 0.663 g of lithium hydroxide. Trisodium phosphate (3.786 kg), hydrochloric acid (211 mL), and additional boric acid (600 g) were added beginning at 30 minutes and lasting until 4 hours into the test. The test was conducted at a constant temperature of 60°C (140°F). The materials tested within this environment included representative amounts of submerged and unsubmerged aluminum, copper, concrete, zinc, carbon steel, and insulation samples (80% calcium silicate and 20% fiberglass). Representative amounts of concrete dust and latent debris were also added to the test solution. The test solution reached a pH of 7.9 by Day 3, and the test solution turbidity decreased to less than 1 NTU after 24 hours. During the introduction of trisodium phosphate at the beginning of the test, a white flocculence was observed through the submerged observation window. Turbidity and TSS initially rose, but dropped after chemical addition was complete, and the white flocculence was no longer visible in the water after the first day. Observations of the test solution indicated similar behavior of the solution at both room temperature and test temperature. No chemical byproducts were visible in the water after Day 1, and no precipitation occurred as samples cooled from test temperature to room temperature. Large amounts of white deposits of varying size were observed on the submerged galvanized steel, aluminum, copper, and inorganic zinc coated steel coupons. The bottom of the tank was filled with sediment that had a pinkish-white deposit on top. The test solution remained clearly Newtonian for the entire test. Aluminum was detectable in the solution, but only in trace amounts.

12. KEY WORDS/DESCRIPTORS *(List words or phrases that will assist researchers in locating the report.)*

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