Appendix B

SEM/EDS Data for Test-2 Day-30 Corrosion Products

List of Figures

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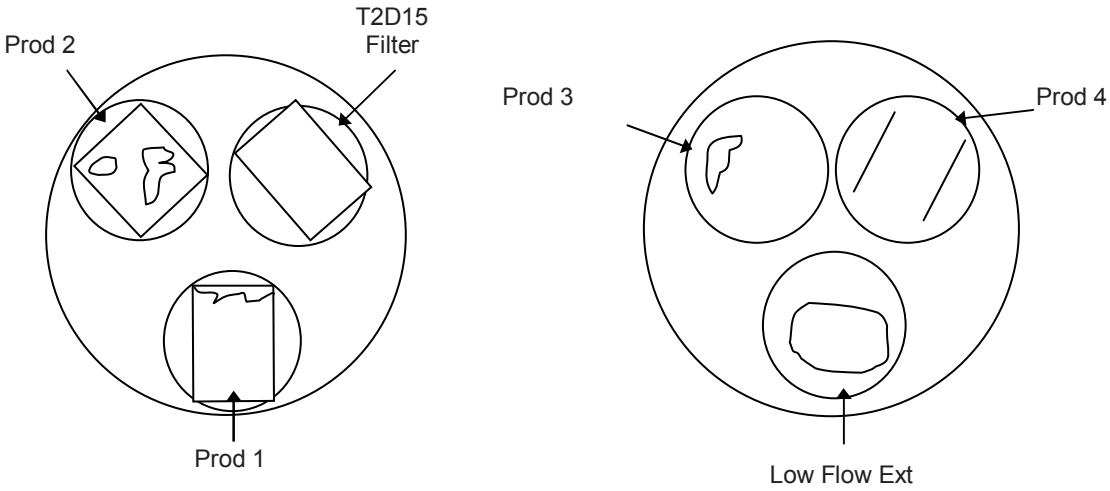
For ICET tests, one process of interest is the corrosion effect on metal and concrete coupons. One means of understanding the corrosion process is through direct examination of the corrosion products after the test is completed. For this purpose, corrosion products were collected when Test #2 was shut down (March 7, 2005). These corrosion products included (1) fine powders on a vertical piece of the submerged CPVC rack, (2) white residue on a horizontal piece of the submerged CPVC rack, (3) corrosion products on a submerged galvanized steel coupon, and (4) corrosion products on a submerged aluminum coupon.

Corrosion products were collected by directly adhering the sample onto double-sided carbon tape suitable for SEM/EDS examination. After the samples were dried in air, a Au/Pd coating was applied to enhance the surface conductivity of the samples and to prevent possible charging problems during the SEM examination. For many samples, semiquantitative elemental analysis was performed on the accompanying EDS spectra. This appendix presents the SEM/EDS data that were generated on March 7, 2005 for each of the sample types identified above. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Laboratory session from March 7, 2005

T2D30 Samples—NRC



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

Product 1. Corrosion Product (Powder) on CPVC

Image:	T2D30_Cor_Prod001	150 ×	SEI	Figure B-1
	T2D30_Cor_Prod002	1000 ×	SEI	Figure B-2
EDS:	T2D30EDS1		EDS of Prod002	Figure B-3
Image	T2D30_Cor_Prod003	650 ×	SEI	Figure B-4

Product 2. Corrosion Product Sediment on Rack

Image:	T2D30_Cor_Prod004	130 ×	SEI	Figure B-5
EDS:	T2D30EDS2		EDS of Prod004	Figure B-6
Image:	T2D30_Cor_Prod005	130 ×	BSE on different area EDS3 collected on bright particle in center right	Figure B-7
EDS:	T2D30EDS3		Bright particle	Figure B-8
Image:	T2D30_Cor_Prod006	1000 ×	SEI	Figure B-9
	T2D30_Cor_Prod007	40 ×	BSE overview	Figure B-10

Product 3. Corrosion Product on Galvanized Steel

Image:	T2D30_Cor_Prod008	95 ×	BSE image	Figure B-11
EDS:	T2D30EDS4		Center of agglomeration in image 008	Figure B-12
	T2D30EDS5		Above location of EDS4	Figure B-13
	T2D30EDS6		Same as EDS4 but using 25kV	Figure B-14
	T2D30EDS7		Small tubular crystals	Figure B-15
Image:	T2D30_Cor_Prod009	120 ×	BSE of crystal	Figure B-16
	T2D30_Cor_Prod010	55 ×	BSE lower part of sample	Figure B-17
EDS:	T2D30EDS9		Spot on right side of image 010	Figure B-18
	T2D30EDS10		Crystals at left of image 010	Figure B-19

Product 4. Corrosion Product on Aluminum

Image:	T2D30_Cor_Prod011	90 ×	BSE overview	Figure B-20
Image:	T2D30_Cor_Prod012	90 ×	SE image on another area	Figure B-21
EDS:	T2D30EDS11		Spot on agglomeration in upper right of image 012	Figure B-22
	T2D30_Cor_Prod013	1000 ×	SE same area	Figure B-23
EDS:	T2D30EDS12		EDS on center of agglomeration	Figure B-24

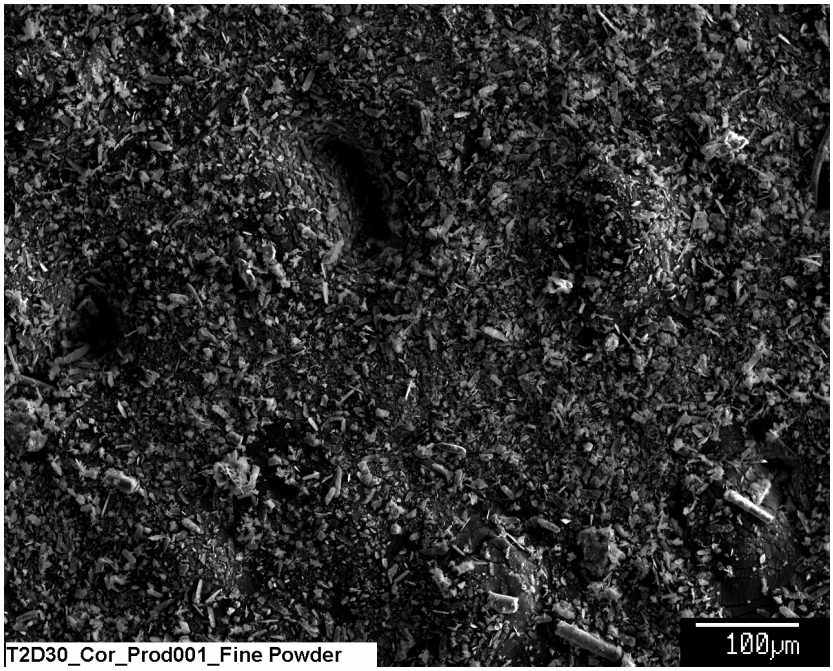


Figure B-1. SEM image (150×) for a Test-2 Day-30 sample of fine powder on a vertical piece of the submerged PVC rack (T2D30_Cor_Prod001_Fine Powder).

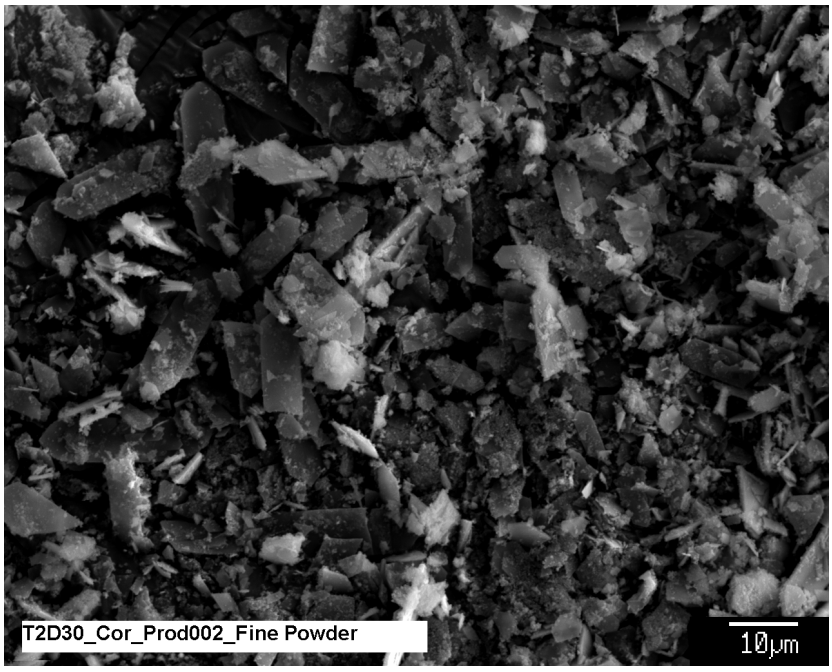


Figure B-2. SEM image at 1000× magnification for a Test-2 Day-30 sample of fine powder on a vertical piece of the submerged PVC rack (T2D30_Cor_Prod002_Fine Powder).

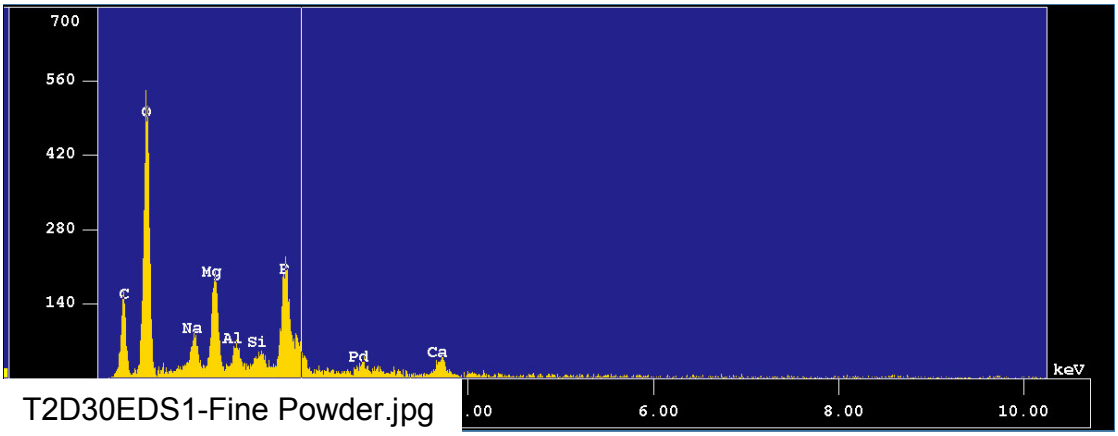


Figure B-3. EDS counting spectrum for the SEM image shown in Figure B-2 suggesting that the fine powder may contain Mg salts ($MgCO_3$ and/or $Mg_3(PO_4)_2$) (T2D30EDS1-Fine Powder)

Figure B-4. Another SEM image at 650× magnification for a Test-2 Day-30 sample of fine powder on a vertical piece of the submerged PVC rack. The cylinder shaped debris is likely to be fiberglass (T2D30_Cor_Prod003_Fine Powder).

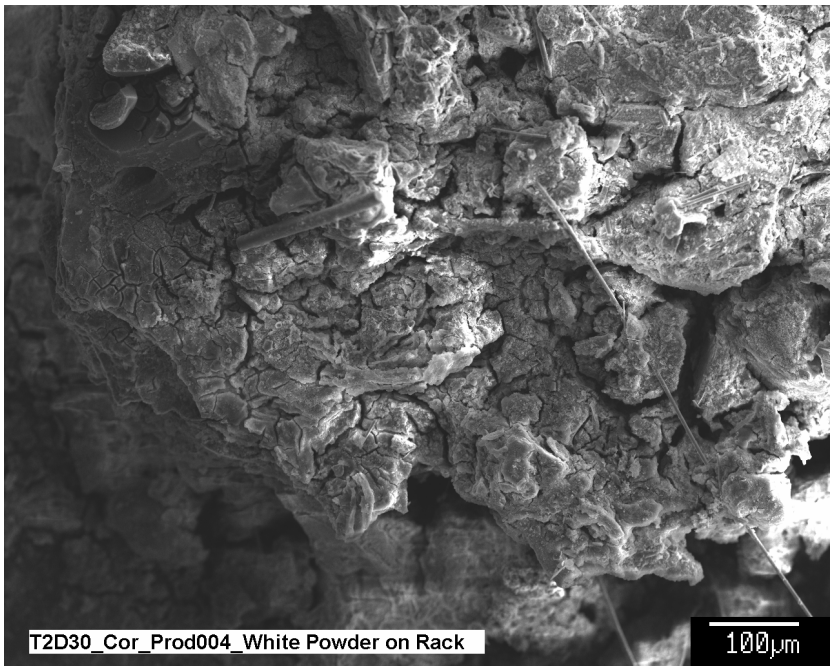


Figure B-5. SEM image (130×) for a Test-2 Day-30 sample of white residue on a horizontal piece of the submerged CPVC rack (T2D30_Cor_Prod004_White Powder on Rack).

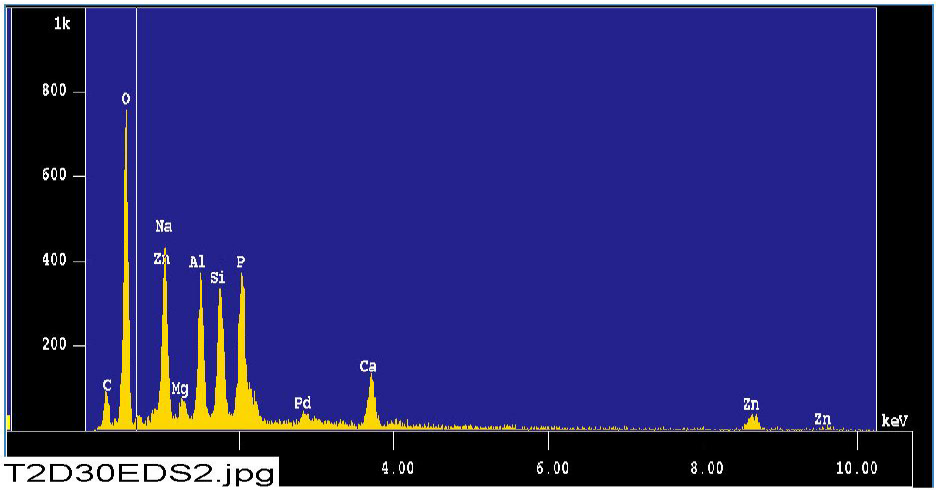


Figure B-6. EDS counting spectrum collected on the field of view at 130× magnification for the white residue shown in Figure B-5(T2D30EDS2).

The results from the chemical composition analysis for T2D30EDS2 are given in Table B-1.

Table B-1. The Chemical Composition for T2D30EDS2

Mar 7 15:38 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T2D30 ID# : 2
Comment : Corrosion product sediment on rack
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.071E-09 A
Stage Point : X=83.673 Y=63.044 Z=10.833
Acq. Date : Mon Mar 7 15:34:35 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.7465	0.0004	278 / 70
O K	Normal	0.25- 0.77	26.0444	0.0035	6529 / 62
Na K	Normal	0.83- 1.28	0.8606	0.0133	678 / 45
Al K	Normal	1.26- 1.78	2.5000	0.0010	2970 / 157
Si K	Normal	1.50- 2.07	2.5870	0.0014	3072 / 265
Ca K	Normal	3.40- 4.30	2.3973	0.0067	1506 / 10
Fe K	Normal	6.04- 7.40	0.0295	0.0279	6 / 8
Zn K	Normal	8.22-10.03	8.4662	0.0065	613 / 5

Chi_square = 6.9122

Element	Mass%	Atomic%	ZAF	Z	A	F
C	6.592	11.4056	4.4054	1.0018	4.3976	1.0000
O	51.610	67.0354	0.9885	0.9553	1.0347	1.0000
Na	2.469	2.2320	1.4313	0.9592	1.4903	1.0012
Al	6.884	5.3019	1.3736	0.9668	1.4225	0.9988
Si	6.586	4.8732	1.2700	0.9548	1.3302	0.9999
Ca	4.631	2.4012	0.9637	0.9664	0.9977	0.9995
Fe	0.055	0.0205	0.9310	0.9595	1.0014	0.9689
Zn	21.171	6.7302	1.2474	1.2511	0.9971	1.0000
Total	100.000	100.0000				

Normalization factor = 2.0047

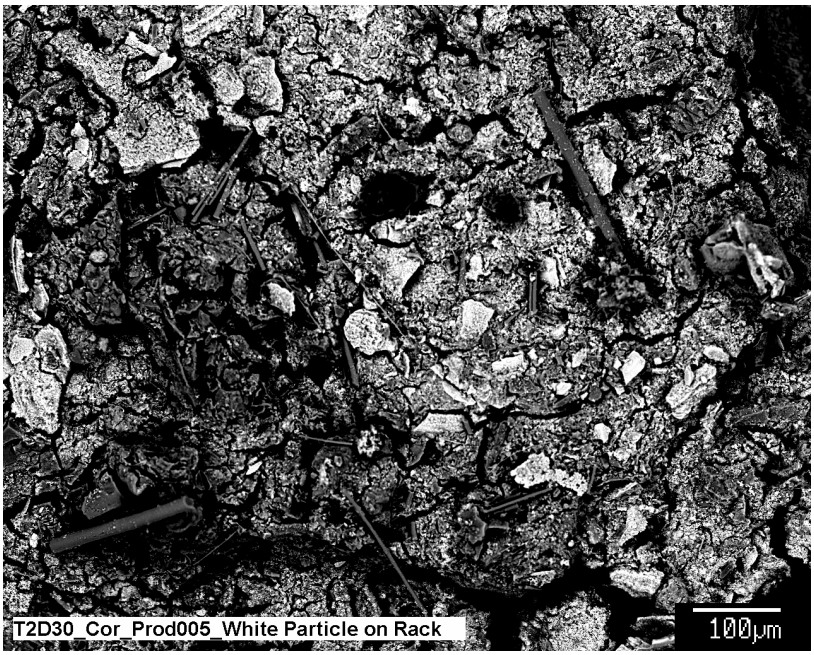


Figure B-7. Backscattered SEM image at 130× magnification for a Test-2 Day-30 sample of white residue on a horizontal piece of the submerged PVC rack (T2D30_Cor_Prod005_White Particle on Rack).

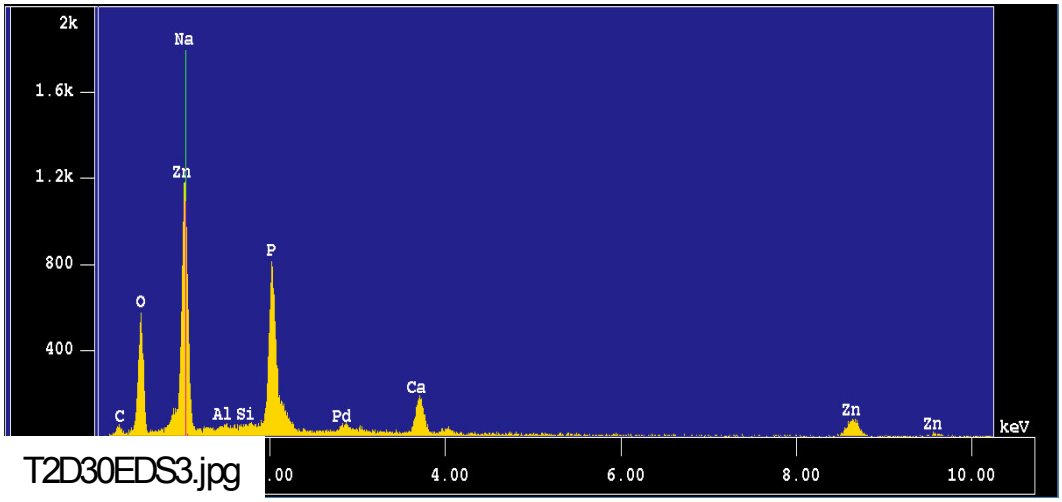


Figure B-8. EDS counting spectrum collected on the bright particles shown in Figure B-7 (T2D30EDS3).

The results from the chemical composition analysis for T2D30EDS2 are given in Table B-2.

Table B-2. The Chemical Composition for T2D30EDS3

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Mar  7 15:42 2005  /tmp/eds_pout.log Page 1


 Group     : NRC
 Sample    : T2D30  ID# : 3
 Comment   : Corrosion product sediment on rack
 Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   :  60.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 1.064E-09 A
             Stage Point : X=83.602 Y=62.915 Z=10.833
             Acq. Date   : Mon Mar  7 15:40:33 2005

 Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
   O K      Normal   0.25- 0.77   19.4279   0.0031     4838 /      46
   P K      Normal   1.75- 2.38   13.5910   0.0051     8082 /      50
  Ca K      Normal   3.40- 4.30    3.5982   0.0082     2245 /      20
  Zn K      Normal   8.22-10.03   18.9540   0.0093     1363 /       6
-------------------------------------------------------------------
                             Chi_square =  4.9345

 Element Mass%    Atomic%   ZAF      Z      A      F
     O   35.902  61.6543 1.1175 0.9205 1.2141 1.0000
     P   21.205  18.8094 0.9435 1.1063 0.8530 0.9998
    Ca    5.687   3.8987 0.9558 0.9266 1.0321 0.9994
    Zn   37.206  15.6376 1.1870 1.1878 0.9993 1.0000
-------------------------------------------------------------------
 Total   100.000 100.0000
 Normalization factor =  1.6537
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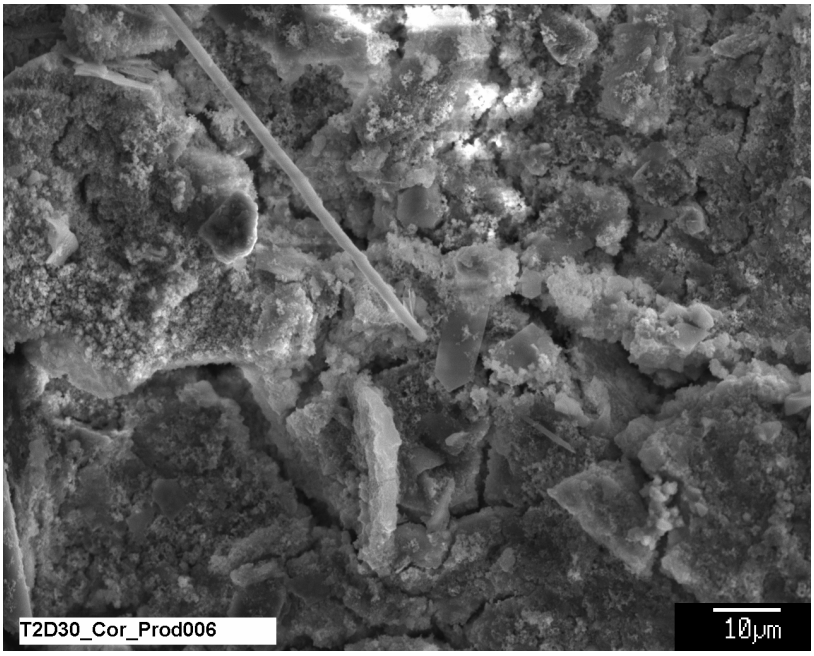

Figure B-9. SEM image at 1000× magnification for a Test-2 Day-30 sample of white residue on the horizontal piece of the submerged PVC rack (T2D30_Cor_Prod006).

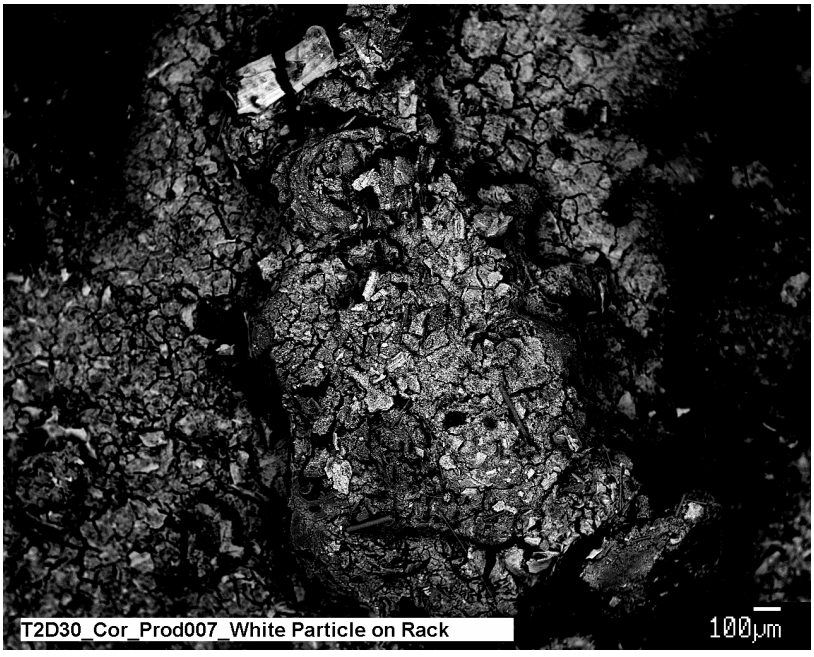

Figure B-10. Backscatter SEM overview at 40× magnification of a Test-2 Day-30 sample of white residue on the horizontal piece of the submerged CPVC rack. (Looks similar to the corrosion product on galvanized steel) (T2D30_Cor_Prod007_White Particle on Rack).

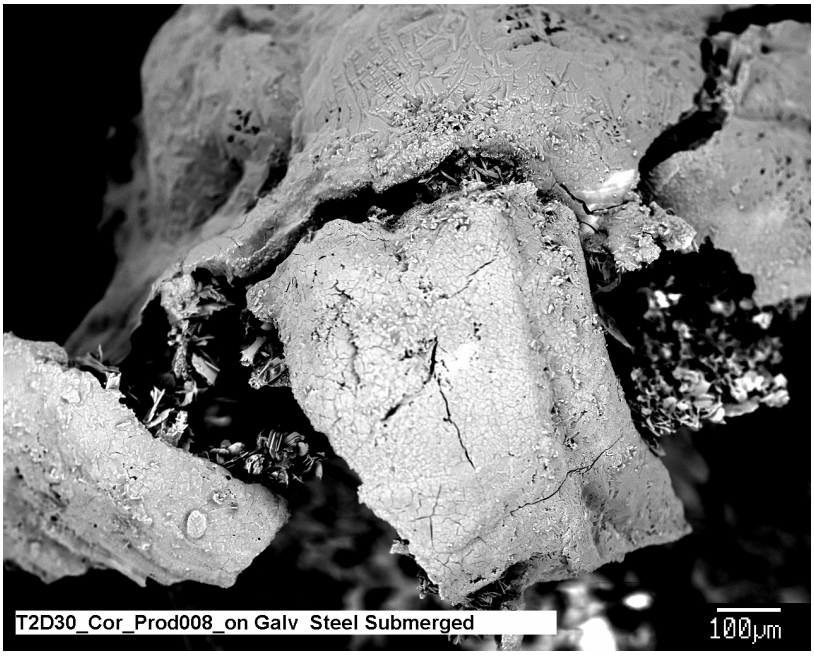

Figure B-11. Backscatter SEM image at 95× magnification of a Test-2 Day-30 sample of corrosion products on a submerged galvanized steel coupon (T2D30_Cor_Prod008_on Galv Steel Submerged).

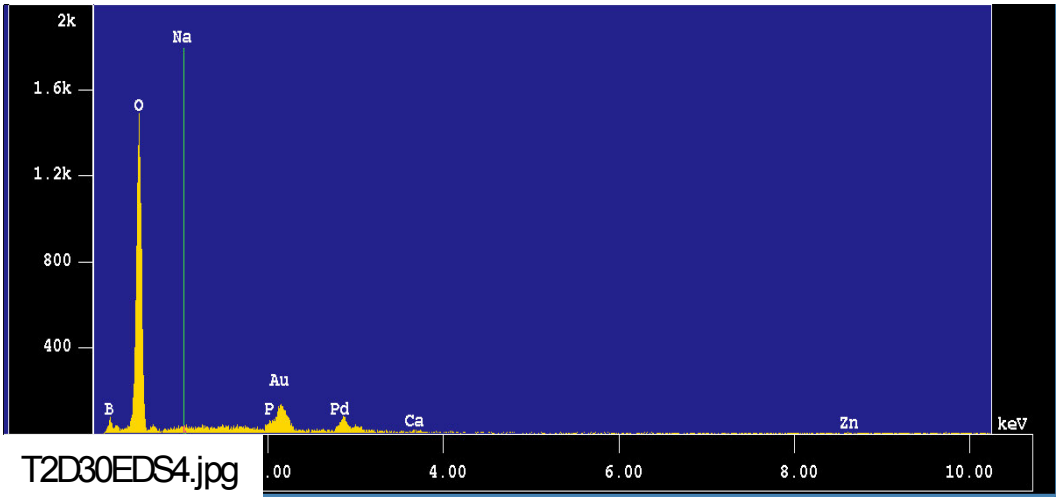

Figure B-12. EDS counting spectrum collected from the center of the SEM image shown in Figure B-11 (T2D30EDS4).

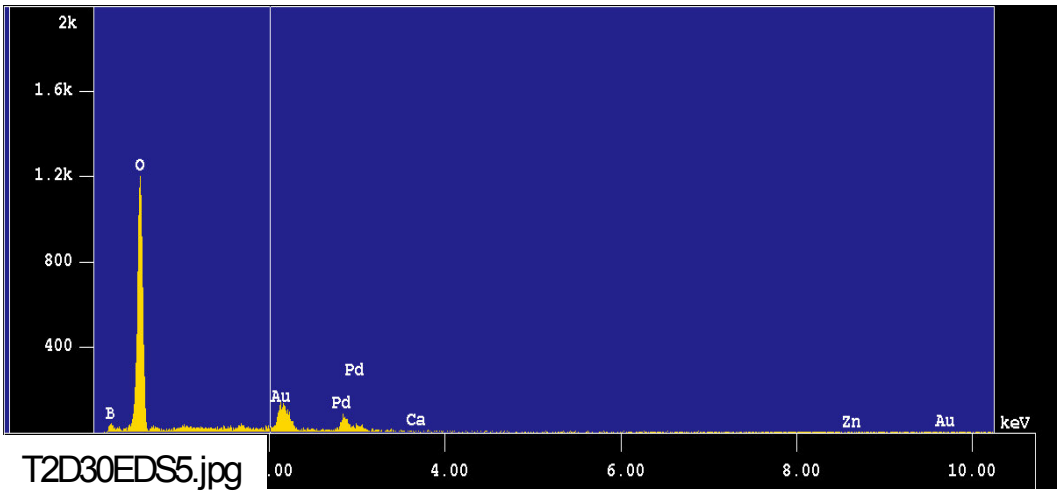

Figure B-13. EDS counting spectrum collected from the upper part of the SEM image shown in Figure B-11 (T2D30EDS5).

The results from the chemical composition analysis for T2D30EDS5 are given in Table B-3.

Table B-3. The Chemical Composition for T2D30EDS5

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Group : NRC
Sample : T2D30 ID# : 5
Comment : Corrosion product on galv steel
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.072E-09 A
Stage Point : X=20.619 Y=59.144 Z=10.833
Acq. Date : Mon Mar 7 15:59:51 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
B K	Normal	0.00- 0.36	1.4897	0.0002	156 / 13
O K	Normal	0.25- 0.77	40.4790	0.0042	10156 / 18
Zn K	Normal	8.22-10.03	0.0000	0.0000	0 / 2

Chi_square = 72.1961

Element	Mass%	Atomic%	ZAF	Z	A	F
B	20.212	27.2669	4.4701	1.1295	3.9577	1.0000
O	79.788	72.7331	0.6494	0.9731	0.6673	1.0000
Zn	0.000	0.0000	1.2952	1.3038	0.9934	1.0000
Total	100.000	100.0000				

Normalization factor = 3.0354

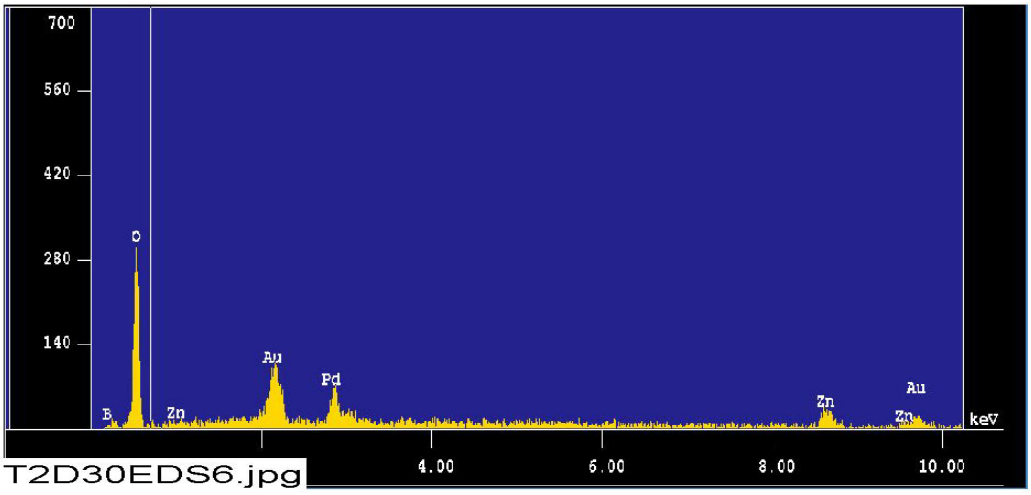

Figure B-14. EDS counting spectrum collected from the center of the SEM image shown in Figure B-11 but using a 25-kV beam voltage (T2D30EDS6).

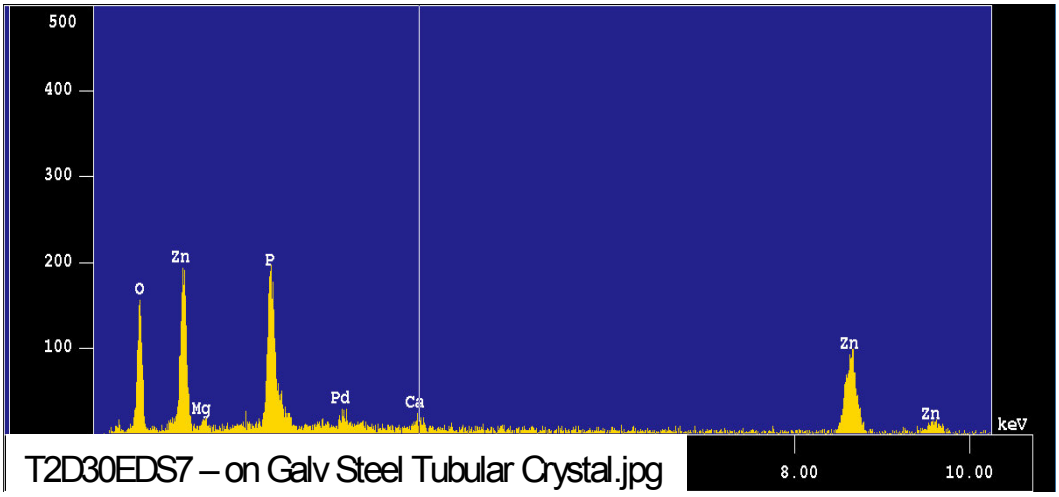

Figure B-15. EDS counting spectrum for small tubular crystals in Figure B-11 suggesting that the corrosion products of galvanized steel are rich in Zn, P, and O (possible presence of $Zn_3(PO_4)_2$).

The results from the chemical composition analysis for T2D30EDS7 are given in Table B-4.

Table B-4. The Chemical Composition for T2D30EDS7

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Mar  7 16:11 2005  /tmp/eds_pout.log Page 1


 Group     : NRC
 Sample    : T2D30  ID# : 7
 Comment   : corrosion product on galv steel
 Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   :  60.000 sec    Aperture #    : 1
             Acc. Volt   : 20.0 KV        Probe Current : 3.863E-10 A
             Stage Point : X=20.619 Y=59.144 Z=10.833
             Acq. Date   : Mon Mar  7 16:08:57 2005


 Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
   O K      Normal   0.25- 0.77   17.5032   0.0028     1299 /      12
  Mg K      Normal   0.97- 1.57    0.2027   0.0002      103 /      50
   P K      Normal   1.75- 2.38    7.7861   0.0032     2056 /      10
  Zn K      Normal   8.22-10.03   21.2758   0.0050     1615 /       4
-------------------------------------------------------------------
                              Chi_square =  2.8316

 Element Mass%    Atomic%   ZAF      Z      A      F
      O   33.841  61.7575 0.9431 0.9116 1.0346 0.9999
     Mg    1.528   1.8349 3.6762 0.9047 4.0654 0.9996
      P   15.204  14.3319 0.9525 1.0778 0.8837 1.0001
     Zn   49.426  22.0757 1.1331 1.1370 0.9966 1.0000
-------------------------------------------------------------------
 Total   100.000 100.0000
 Normalization factor =  2.0502
```

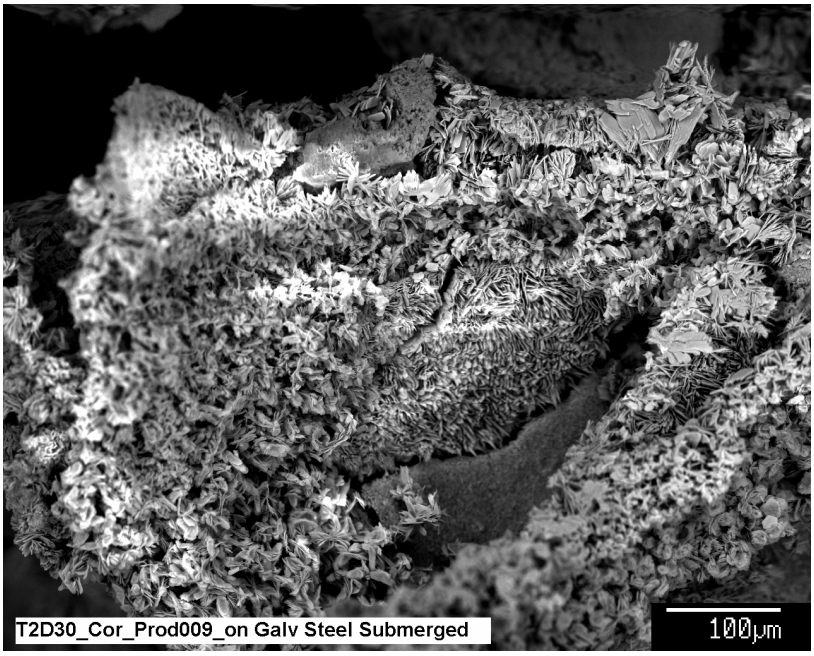

Figure B-16. Backscatter SEM image at 120× magnification for a Test-2 Day-30 sample of corrosion products on a submerged galvanized steel coupon (T2D30_Cor_Prod009_on Galv Steel Submerged).

Figure B-17. Backscatter SEM image at 55× magnification for a Test-2 Day-30 sample of corrosion products on a submerged galvanized steel coupon, annotated to show EDS sample locations (T2D30_Cor_Prod010_on Galv Steel Submerged).

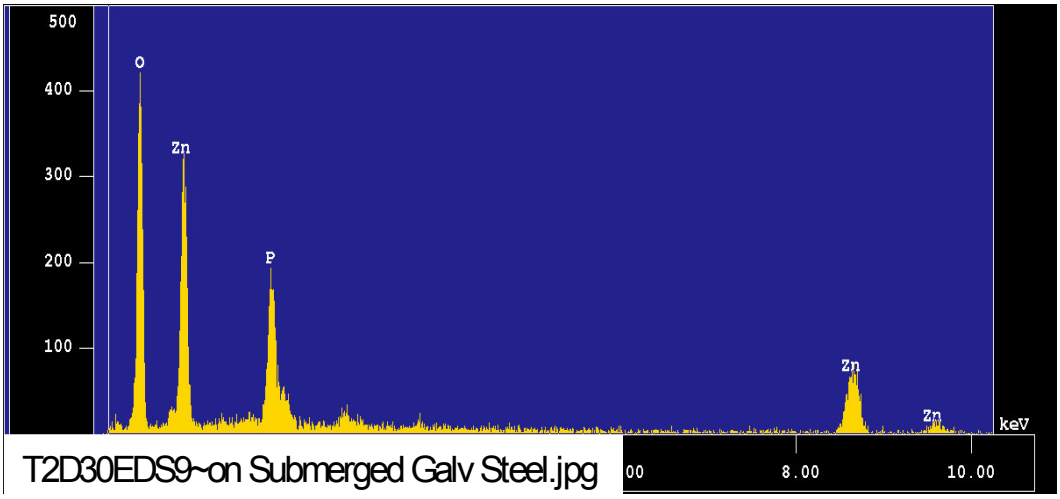

Figure B-18. EDS counting spectrum collected from the spot EDS9 indicated in Figure B-17 (T2D30EDS9~on Submerged Galv Steel).

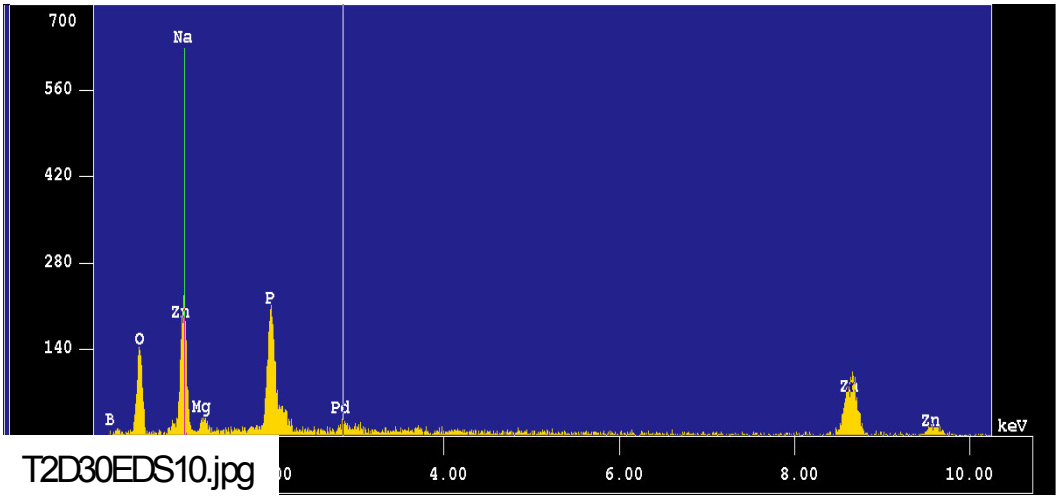

Figure B-19. EDS counting spectrum collected from the spot EDS10 indicated in Figure B-17 (T2D30EDS10).

The results from the chemical composition analysis for T2D30EDS10 are given in Table B-5.

Table B-5. The Chemical Composition for T2D30EDS10

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Group : NRC
Sample : T2D30 ID# : 10
Comment : Corrosion product on galv steel
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 20.0 KV Probe Current : 3.658E-10 A
Stage Point : X=20.619 Y=62.110 Z=10.558
Acq. Date : Mon Mar 7 16:29:13 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	17.7418	0.0029	1247 / 6
Zn K	Normal	8.22-10.03	24.1289	0.0053	1735 / 3
P K	Normal	1.75- 2.38	8.9417	0.0034	2236 / 12
Na K	Normal	0.81- 1.27	0.0000	0.0000	0 / 16
Mg K	Normal	0.97- 1.57	0.4076	0.0002	196 / 42

Chi_square = 2.3325

Element	Mass%	Atomic%	ZAF	Z	A	F
O	31.348	58.5209	0.9613	0.9101	1.0564	0.9999
Zn	50.152	22.9142	1.1308	1.1343	0.9969	1.0000
P	15.751	15.1883	0.9584	1.0759	0.8907	1.0001
Na	0.000	0.0000	2.6509	0.9564	2.7714	1.0001
Mg	2.749	3.3766	3.6685	0.9031	4.0639	0.9995
Total	100.000	100.0000				

Normalization factor = 1.8381

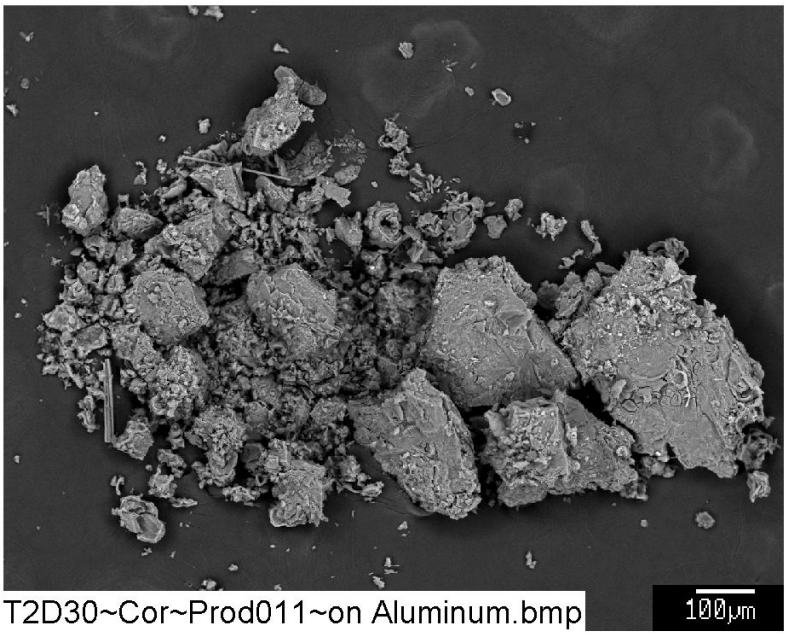

Figure B-20. Backscatter SEM image at 90× magnification for a Test-2 Day-30 sample of corrosion products on a submerged aluminum coupon (T2D30_Cor_Prod011_on Aluminum).

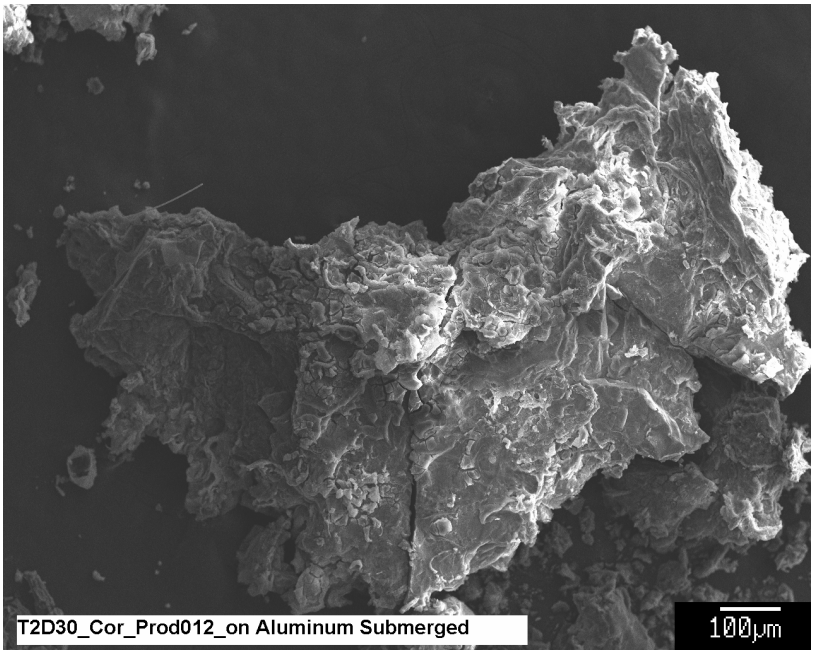

Figure B-21. SEM image at 90× magnification on another area of a Test-2 Day-30 sample of corrosion products on a submerged aluminum coupon (T2D30_Cor_Prod012_on Aluminum Submerged).

Figure B-22. EDS counting spectrum for upper right spot in Figure B-21suggesting that the corrosion products of aluminum are rich in Al, Si, P, and O (T2D30EDS11~on Aluminum spot glob).

The results from the chemical composition analysis for T2D30EDS11 are given in Table B-6.

Table B-6. Chemical Composition for T2D30EDS11

Mar 7 16:45 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T2D30 ID# : 11
Comment : Corrosion product on Aluminum
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 20.0 KV Probe Current : 3.506E-10 A
Stage Point : X= 5.779 Y=60.038 Z=10.558
Acq. Date : Mon Mar 7 16:43:34 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	21.1440	0.0030	1425 / 6
Na K	Normal	0.81- 1.27	0.9134	0.0007	238 / 12
Al K	Normal	1.19- 1.83	7.2047	0.0005	3383 / 20
Si K	Normal	1.50- 2.05	4.8796	0.0004	2204 / 220
Ca K	Normal	3.39- 4.30	1.9757	0.0010	520 / 6
P K	Normal	1.75- 2.38	4.9153	0.0028	1178 / 128

Chi_square = 4.0370

Element	Mass%	Atomic%	ZAF	Z	A	F
O	48.106	62.3021	1.1060	0.9888	1.1186	1.0000
Na	2.567	2.3135	1.3661	1.0402	1.3156	0.9983
Al	20.029	15.3810	1.3514	1.0145	1.3391	0.9947
Si	15.288	11.2788	1.5231	0.9905	1.5420	0.9972
Ca	4.262	2.2032	1.0486	1.0096	1.0387	1.0000
P	9.748	6.5213	0.9641	1.1723	0.8226	0.9999
Total	100.000	100.0000				

Normalization factor = 2.0571

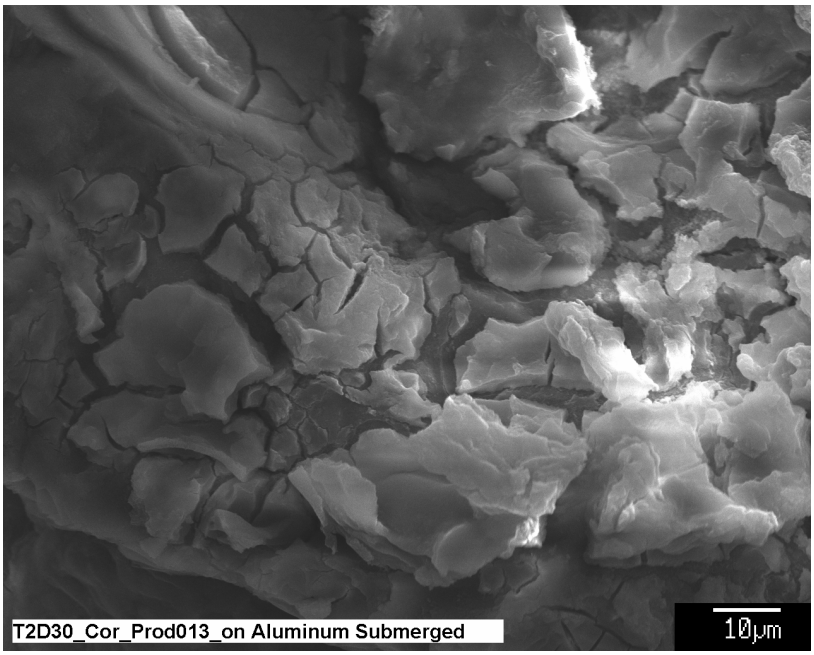

Figure B-23. SEM image at 1000× magnification of a Test-2 Day-30 sample of corrosion products on a submerged aluminum coupon (T2D30_Cor_Prod013_on Aluminum Submerged).

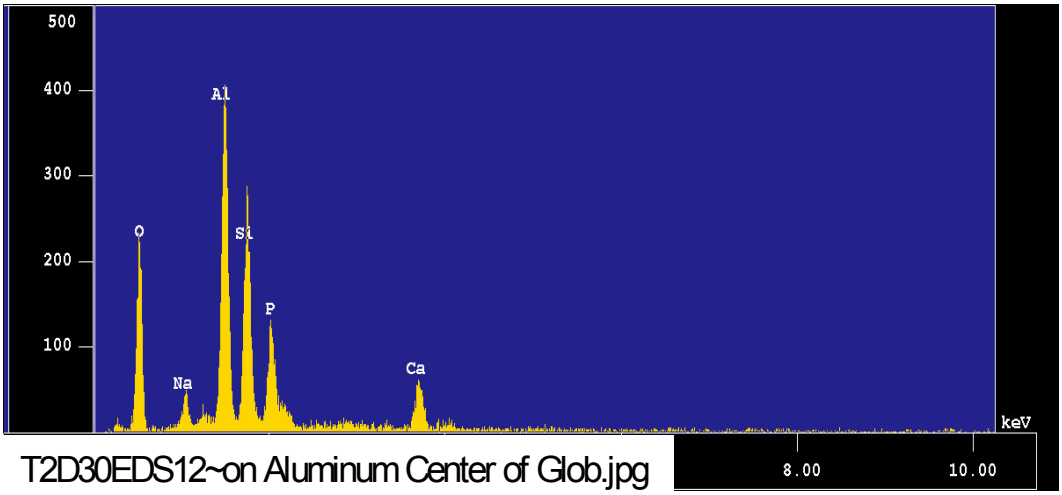

Figure B-24. EDS counting spectrum on the center of Figure B-23 suggesting that the corrosion products of aluminum are rich in Al, Si, P, and O (T2D30EDS12~on Aluminum Center of Glob).

The results from the chemical composition analysis for T2D30EDS12 are given in Table B-7.

Table B-7. The Chemical Composition for T2D30EDS12

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Group : NRC
Sample : T2D30 ID# : 12
Comment : Corrosion product on Aluminum
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 20.0 KV Probe Current : 3.388E-10 A
Stage Point : X= 4.958 Y=60.774 Z=10.558
Acq. Date : Mon Mar 7 16:56:37 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	28.8019	0.0033	1875 / 7
Na K	Normal	0.81- 1.27	1.4361	0.0008	362 / 12
Al K	Normal	1.19- 1.83	8.1233	0.0005	3686 / 24
Si K	Normal	1.50- 2.05	5.8349	0.0004	2547 / 230
Ca K	Normal	3.39- 4.30	3.0762	0.0010	782 / 3
P K	Normal	1.75- 2.38	5.3699	0.0027	1244 / 145

Chi_square = 3.9774

Element	Mass%	Atomic%	ZAF	Z	A	F
O	50.761	64.8096	1.0876	0.9891	1.0996	1.0000
Na	3.268	2.9041	1.4045	1.0406	1.3517	0.9985
Al	18.215	13.7895	1.3837	1.0150	1.3700	0.9951
Si	14.298	10.3989	1.5122	0.9909	1.5297	0.9976
Ca	5.190	2.6450	1.0411	1.0101	1.0306	1.0000
P	8.269	5.4530	0.9502	1.1728	0.8104	0.9998
Total	100.000	100.0000				

Normalization factor = 1.6205

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