Appendix D

ESEM and SEM/EDS Data for Test #3 Day-30 Corrosion Products

List of Figures

List of Tables

For ICET tests, one process of interest is the corrosion effect of 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 #3 was shut down (May 5, 2005). These corrosion products included (1) fine powders on a vertical piece of the submerged CPVC rack; (2) corrosion products on a submerged galvanized steel coupon; (3) corrosion products on a submerged copper coupon; (4) corrosion products on a submerged aluminum coupon; and (5) corrosion products on a submerged concrete 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 process. Based on EDS results, a semi-quantitative elemental analysis was performed after calibration of the x-ray signal using an internal standard of the microscopy. This appendix presents the SEM/EDS data that were generated on May 17, 2005. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

Transcribed Laboratory Log

Test #3 Day-30 Powder Deposits
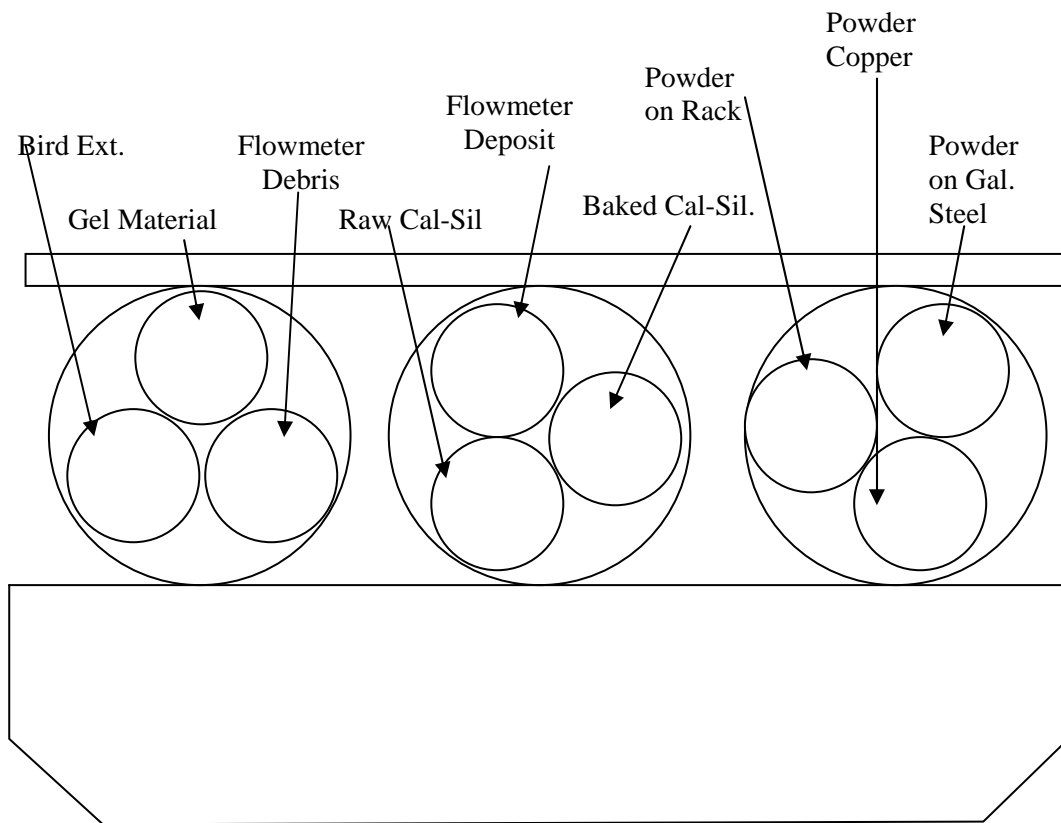
Powder on Rack

Image:	T3~RackPowder013	200 ×		Figure D-1
EDS:	T3RackPowder09		Powder on image 013	Figure D-2

Powder on Galvanized Steel

Image:	T3~Gal~Steel~Powder014	500 ×		Figure D-3
EDS:	CorPdct~GalStel10		White powder on image 014	Figure D-4
	CorPrdct~GalSteel11		Dark powder on image 014	Figure D-5

Powder on Copper

Image: T3~Copper~Powder015   500 ×   Figure D-6

EDS: CorPrdct_cu12   Powder on image 015   Figure D-7

Transcribed Laboratory Log

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

Metal Coupons

Fiberglass
(lab soaking)

Al Up.

Cu Down

Gal Down

Cu Up   Gal Up   Steel Down   Al   B   Al + B
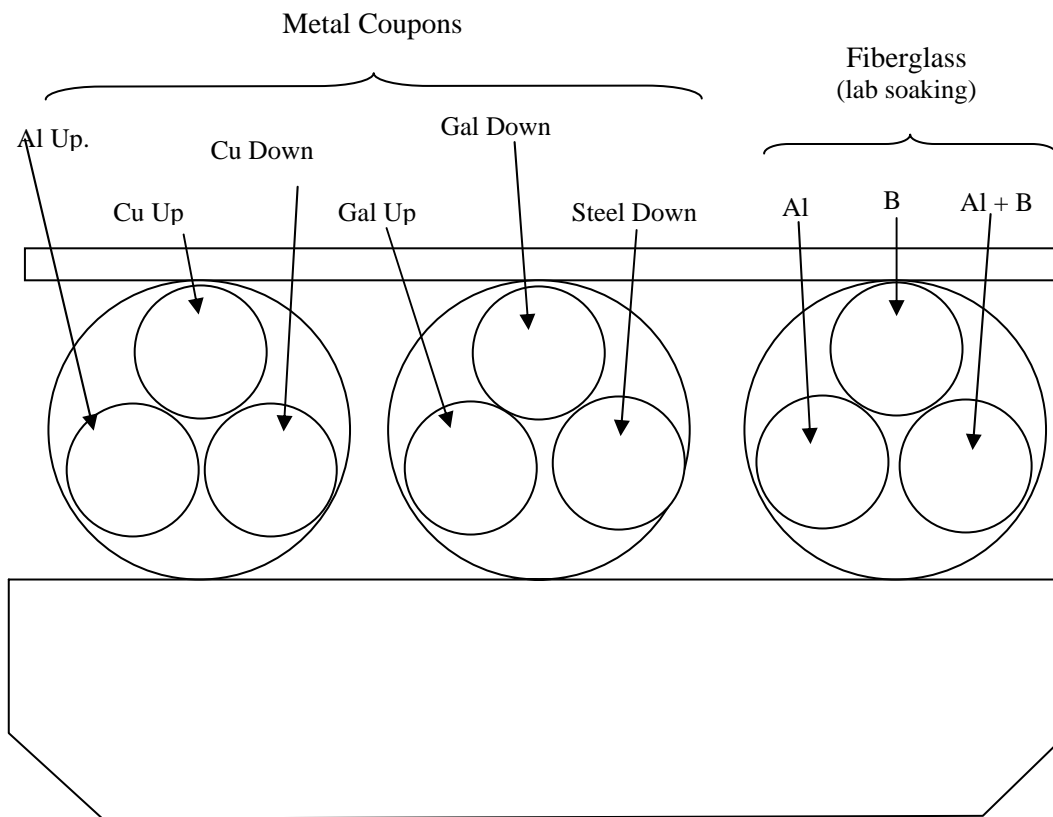
**Coat with Gold

Corrosion Product on Submerged Aluminum

Image:	T3D30CorrPrdctSubmAl039	100 ×		Figure D-8
	T3D30CorrPrdctSubmAl040	100 ×	Backscattering	Figure D-9
EDS:	T3D30CorrPrdtAl22		Particles on image 040	Figure D-10
Image:	T3D30CorrPrdctSubmAl041	1000 ×		Figure D-11

Transcribed Laboratory Log

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

Al & B

X   I

Al(X)   B(X)   Blank(X)

Al(I)   Cal-Sil(X)   (I)
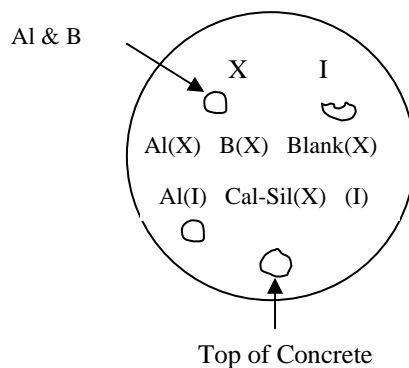
Top of Concrete

Deposits on the Top of Concrete

Image:	T3Cont28	1000 ×		Figure D-12
	t3cont29	100 ×		Figure D-13
EDS:	t3cont30		Particles on image 29	Figure D-14

Figure D-1: SEM image magnified 200 times for a Test #3 Day-30 powder on the submerged rack. (T3~RackPowder013)
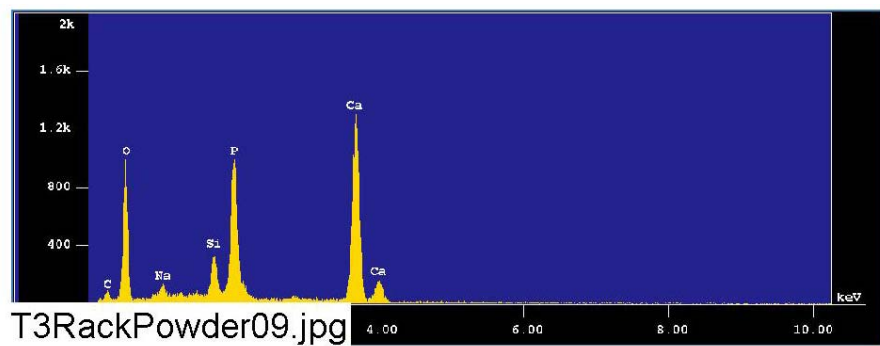
Figure D-2: EDS counting spectrum for the powder on the submerged rack shown in Figure D-1. (T3RackPowder09)

The results from the chemical composition analysis for T3RackPowder09 are given in Table D-1.

Table D-1. Chemical Compositions for T3RackPowder09, Figure D-2.

```
May  9       2005



Group      : NRC
Sample     : T3D30  ID# : 9
Comment    : Powder on submerged Rack
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.608E-09 A
            Stage Point : X=23.811 Y=58.398 Z= 9.938
            Acq. Date   : Mon May  9 15:02:46 2005


Element     Mode     ROI(KeV)    K-ratio(%)   +/-     Net/Background
  O K      Normal   0.25- 0.77    16.4417    0.0037      6188 /        66
 Na K      Normal   0.81- 1.27     0.4147    0.0011       448 /        64
 Si K      Normal   1.50- 2.05     1.8593    0.0006      2708 /       288
  P K      Normal   1.75- 2.38    10.0274    0.0058      9012 /       160
 Ca K      Normal   3.39- 4.30    22.3191    0.0043     15798 /        22
  C K      Normal   0.09- 0.46     0.0345    0.0005        19 /       166
-----------------------------------------------------------------
                    Chi_square = 55.4587

Element Mass%    Atomic%   ZAF      Z      A      F
    O    48.147  67.3988 1.8951 0.9734 1.9469 1.0000
   Na     0.977   0.9516 1.5243 1.0269 1.4843 1.0000
   Si     3.302   2.6332 1.1493 0.9767 1.1837 0.9941
    P    13.197   9.5423 0.8517 1.1722 0.7283 0.9977
   Ca    34.174  19.0962 0.9909 0.9956 0.9953 1.0000
    C     0.203   0.3778 3.7963 1.0208 3.7192 0.9999
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5452
 Total   100.000 100.0000
Normalization factor =  2.1120
```
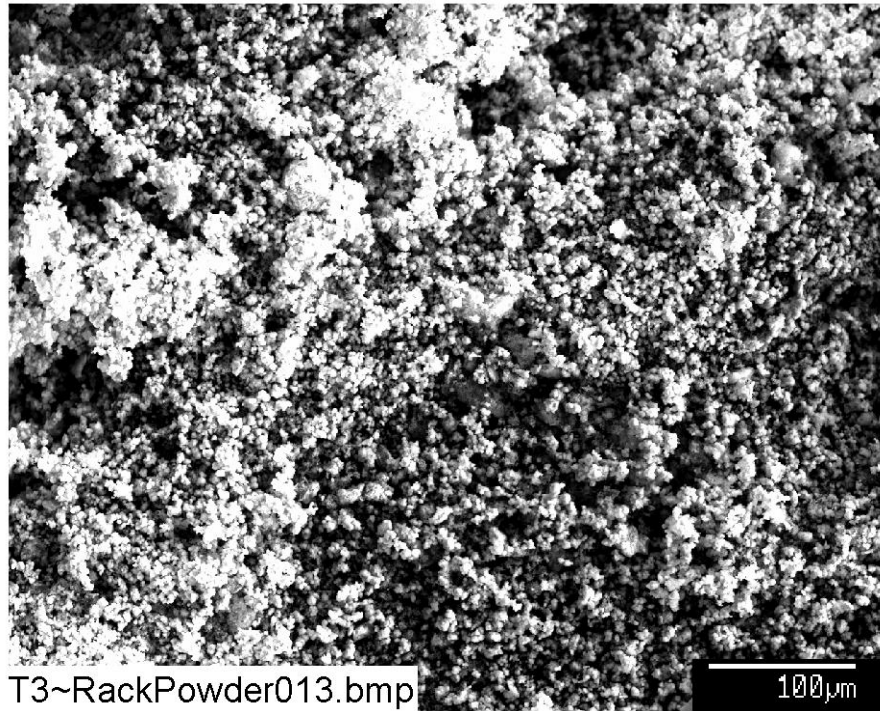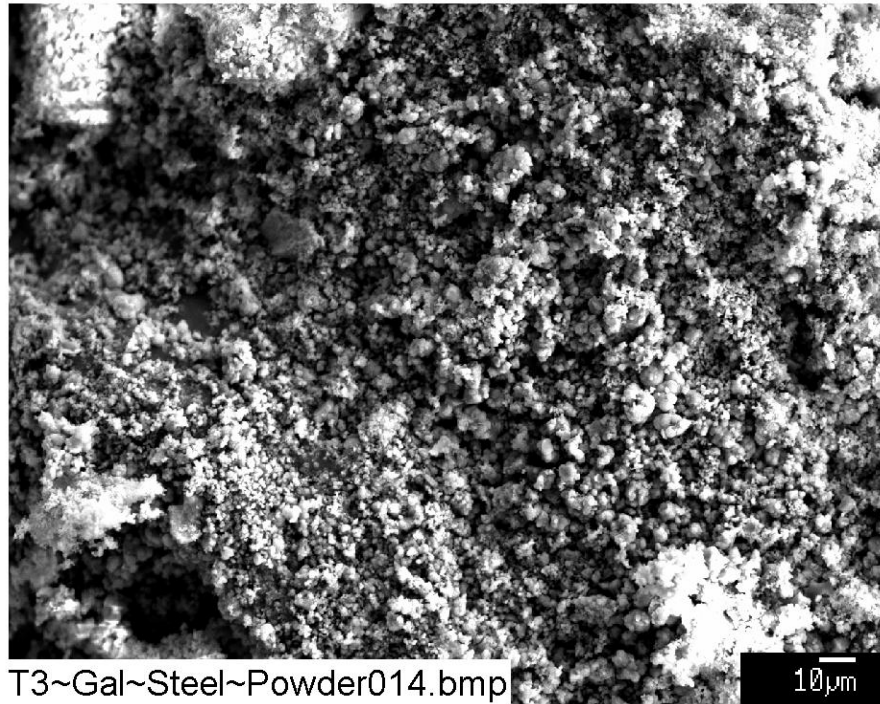
Figure D-3: SEM image magnified 500 times for a Test #3 Day-30 powder on a submerged galvanized steel coupon. (T3~Gal~Steel~Powder014)
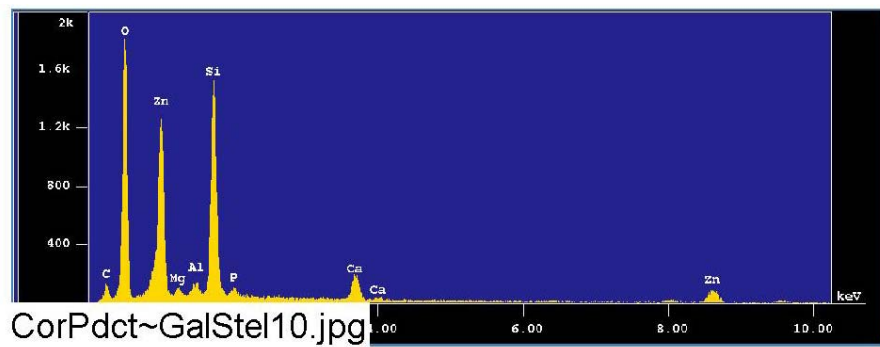

Figure D-4: EDS counting spectrum for the white powder on galvanized steel shown in Figure D-3. (CorPdct~GalStel10)
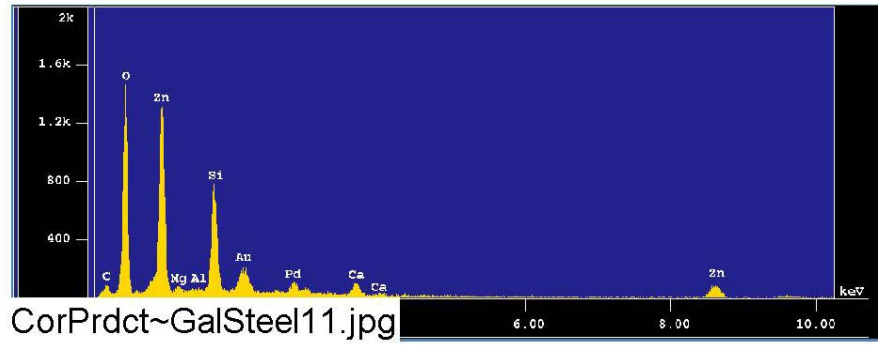
Figure D-5: EDS counting spectrum for the dark powder on galvanized steel shown in Figure D-3. (CorPrdct~GalSteel11)

The results from the chemical composition analysis for CorPrdct~GalSteel11 are given in Table D-2.

Table D-2. Chemical Compositions for CorPrdct~GalSteel11, Figure D-5.

```
May   9        2005



Group      : NRC
Sample     : T3D30   ID# : 11
Comment    : Corrosion product of Gal-Steel
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.608E-09 A
            Stage Point : X=12.508 Y=58.337 Z= 9.938
            Acq. Date   : Mon May  9 15:23:47 2005


Element    Mode     ROI(KeV)    K-ratio(%)   +/-     Net/Background
  O K      Normal   0.25- 0.77    24.6017    0.0044     9259 /     71
 Mg K      Normal   0.97- 1.57     0.1342    0.0003      211 /    424
 Si K      Normal   1.50- 2.05     4.3383    0.0008     6318 /     94
 Ca K      Normal   3.39- 4.30     1.4823    0.0016     1049 /     28
 Zn K      Normal   8.22-10.03    13.8053    0.0096     1501 /      6
 Al K      Normal   1.19- 1.83     0.1257    0.0003      196 /     77
  C K      Normal   0.09- 0.46     0.0000    0.0000        0 /    254
----------------------------------------------------------------------
                       Chi_square = 39.1864

Element Mass%     Atomic%    ZAF       Z      A      F
     O   47.383   72.4077  0.9119  0.9354 0.9750 1.0000
    Mg    0.655    0.6591  2.3126  0.9284 2.4928 0.9993
    Si   13.143   11.4404  1.4344  0.9377 1.5298 0.9999
    Ca    3.059    1.8657  0.9770  0.9542 1.0247 0.9991
    Zn   35.285   13.1966  1.2102  1.2129 0.9978 1.0000
    Al    0.475    0.4304  1.7897  0.9594 1.8692 0.9979
     C    0.000    0.0000  5.2279  0.9811 5.3287 1.0000
----------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.1120
```

T3~Copper~Powder015.bmp   100µm

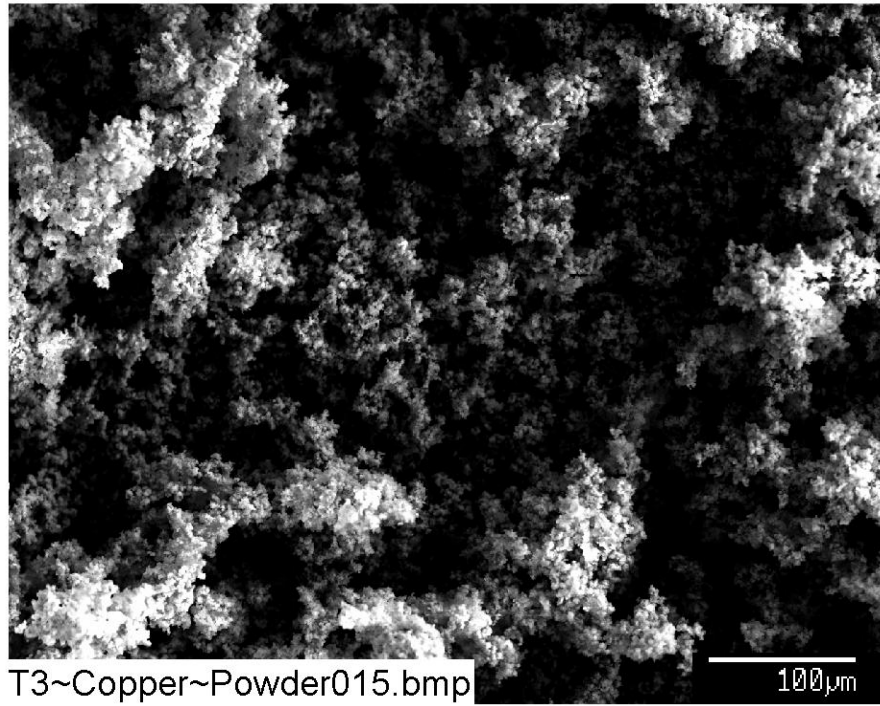**Figure D-6: SEM image magnified 500 times for a Test #3 Day-30 powder on a submerged copper coupon. (T3~Copper~Powder015)**
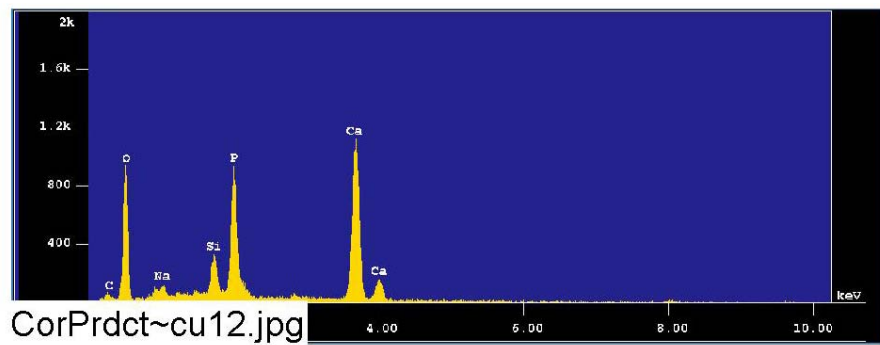

CorPrdct~cu12.jpg

Figure D-7: EDS counting spectrum for the powder on copper shown in Figure D-6. (CorPrdct~cu12)

The results from the chemical composition analysis for CorPrdct~cu12 are given in Table D-3.

Table D-3. Chemical Compositions for CorPrdct~cu12, Figure D-7.

```
May  9       2005



Group      : NRC
Sample     : T3D30  ID# : 112
Comment    : Corrosion product of Copper
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   :  60.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 1.609E-09 A
             Stage Point : X=17.909 Y=70.722 Z= 9.938
             Acq. Date   : Mon May  9 15:34:51 2005


Element      Mode      ROI(KeV)   K-ratio(%)   +/-     Net/Background
  O K       Normal    0.25- 0.77   16.9583    0.0036      6386 /      54
 Na K       Normal    0.81- 1.27    0.3712    0.0011       402 /      46
 Si K       Normal    1.50- 2.05    1.7009    0.0006      2479 /     254
  P K       Normal    1.75- 2.38    8.6109    0.0054      7744 /     164
 Ca K       Normal    3.39- 4.30   19.7450    0.0040     13984 /      28
  C K       Normal    0.09- 0.46    0.0000    0.0000         0 /     156
-------------------------------------------------------------------
                            Chi_square = 59.8005

Element Mass%    Atomic%   ZAF       Z       A      F
     O   51.054  70.0693  1.8119  0.9751  1.8582  1.0000
    Na    0.947   0.9045  1.5353  1.0287  1.4923  1.0000
    Si    3.260   2.5484  1.1533  0.9785  1.1852  0.9945
     P   12.216   8.6603  0.8538  1.1744  0.7286  0.9978
    Ca   32.523  17.8176  0.9913  0.9976  0.9936  1.0000
     C    0.000   0.0000  3.6900  1.0225  3.6090  0.9999
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.6616
```
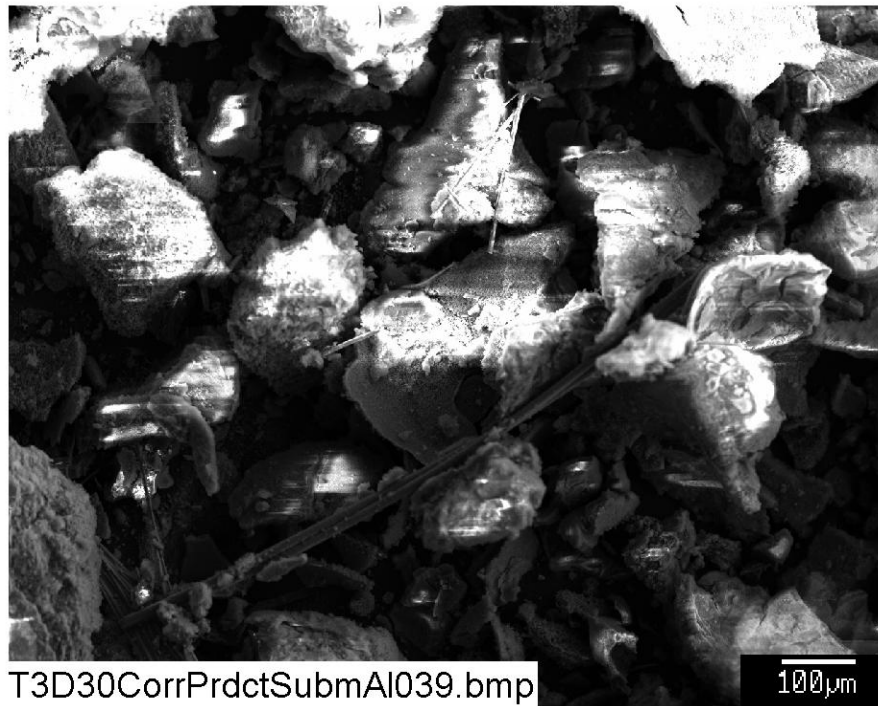
T3D30CorrPrdctSubmAl039.bmp 100μm

Figure D-8: SEM image magnified 100 times for Test #3 Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdctSubmAl039)
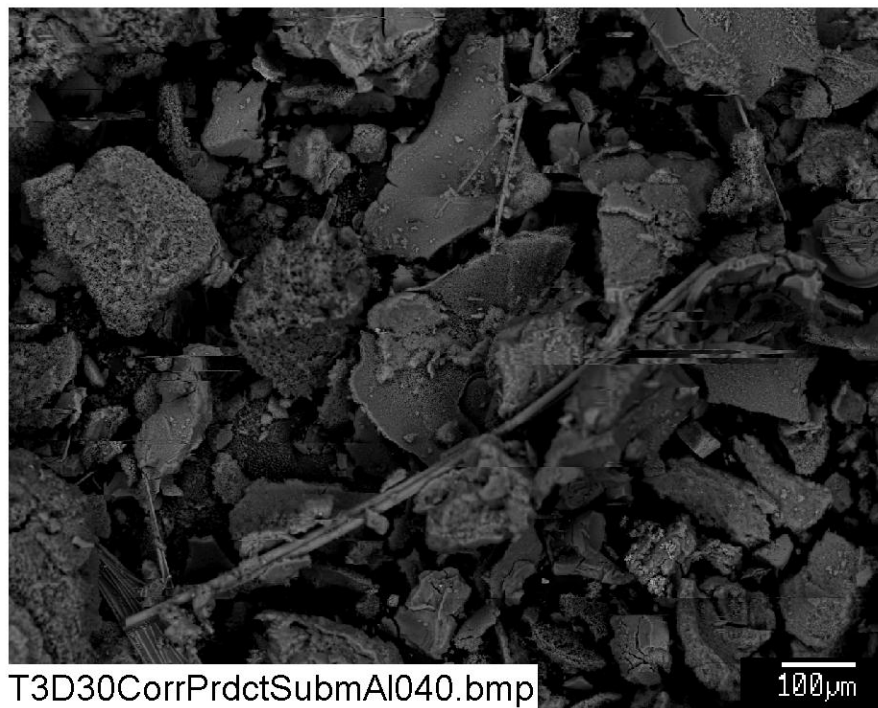

T3D30CorrPrdctSubmAl040.bmp 100μm

Figure D-9: Backscattered SEM image magnified 100 times for Test #3 Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdctSubmAl040)
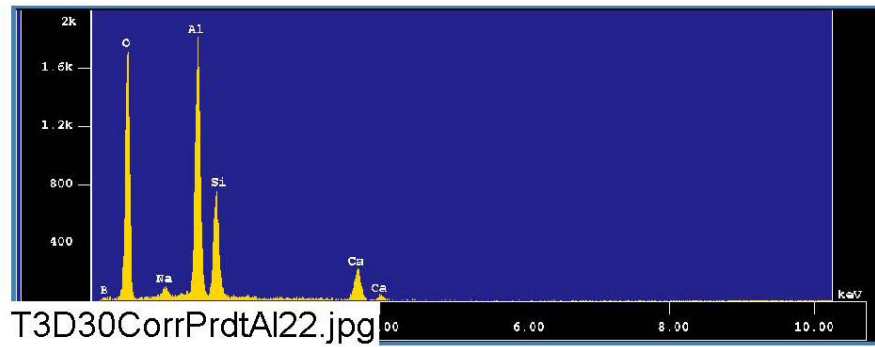
Figure D-10: EDS counting spectrum for the corrosion products (particles) shown in Figure D-9. (T3D30CorrPrdtAl22)

The results from the chemical composition analysis for T3D30CorrPrdtAl22 are given in Table D-4.

Table D-4. Chemical Compositions for T3D30CorrPrdtAl22, Figure D-10.

```
May 17          2005



  Group      : NRC
  Sample     : T3D30  ID# : 25
  Comment    : Corr Prodct on Submerged Al
  Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
               Live Time   :  60.000 sec    Aperture #    : 2
               Acc. Volt   : 15.0 KV        Probe Current : 7.686E-09 A
               Stage Point : X=17.100 Y=58.819 Z=11.000
               Acq. Date   : Tue May 17 16:07:30 2005


  Element     Mode      ROI(KeV)    K-ratio(%)  +/-     Net/Background
    O K      Normal    0.25- 0.77    43.5604   0.0044    10399 /      29
   Na K      Normal    0.81- 1.27     0.7246   0.0010      497 /      45
   Al K      Normal    1.26- 1.78    15.1592   0.0021    15016 /     184
   Si K      Normal    1.50- 2.05     6.6963   0.0008     6185 /     674
   Ca K      Normal    3.40- 4.30     4.2145   0.0082     1892 /      14
    B K      Normal    0.00- 0.36     0.6943   0.0002       69 /      10
  ----------------------------------------------------------------
                         Chi_square = 107.5732

  Element Mass%    Atomic%   ZAF      Z      A      F
      O   52.728  58.8383  1.0170  0.9767  1.0413 1.0000
     Na    1.120   0.8699  1.2989  1.0311  1.2612 0.9988
     Al   19.542  12.9304  1.0831  0.9932  1.0934 0.9974
     Si   10.480   6.6618  1.3149  0.9816  1.3397 0.9999
     Ca    4.923   2.1928  0.9814  0.9916  0.9896 1.0001
      B   11.207  18.5068 13.5610  1.1342 11.9568 1.0000
  ----------------------------------------------------------------
  Total   100.000 100.0000
  Normalization factor =  1.1902
       Fe    2.399   1.5375 0.9896 1.0503  1.0060 0.9366
  ----------------------------------------------------------------
  Total   100.000 100.0000
  Normalization factor =  3.2486
```
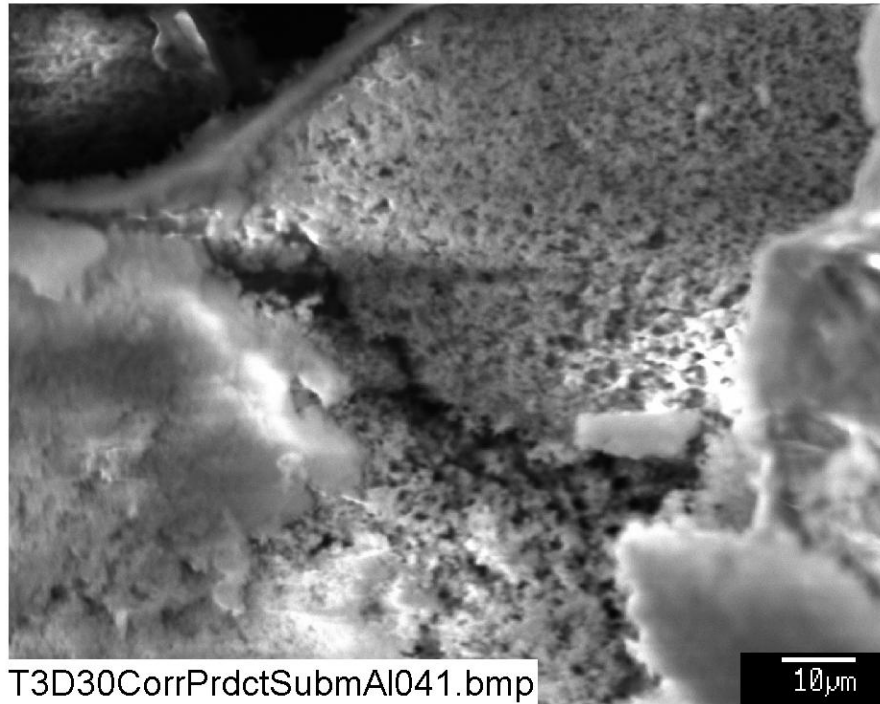
Figure D-11: SEM image magnified 1000 times for Test #3 Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdctSubmAl041)
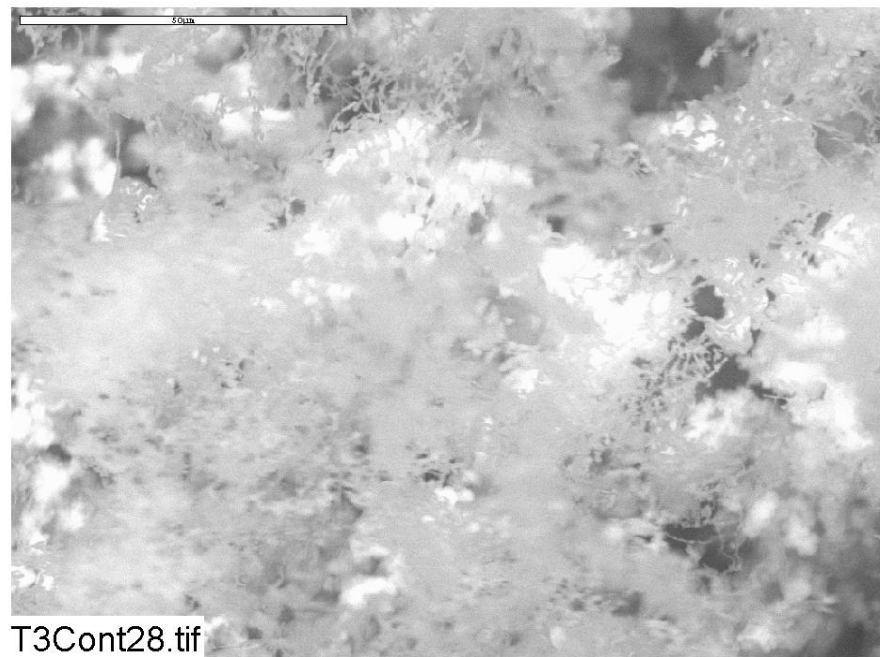

Figure D-12: ESEM image magnified 1000 times for Test #3 Day-30 corrosion products on a submerged concrete coupon. (T3cont28)
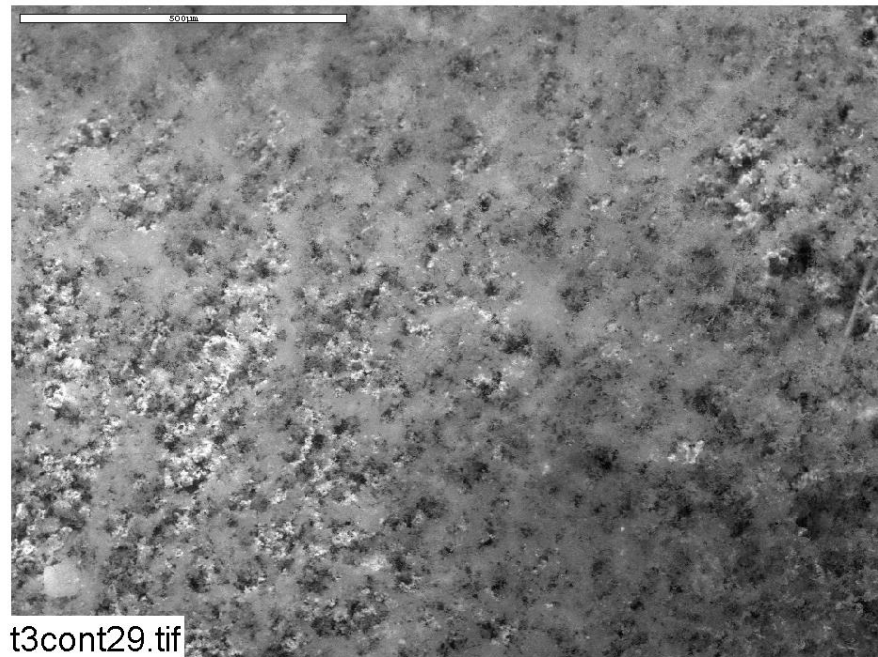
Figure D-13: ESEM image magnified 100 times for Test #3 Day-30 corrosion products on a submerged concrete coupon. (t3cont29)
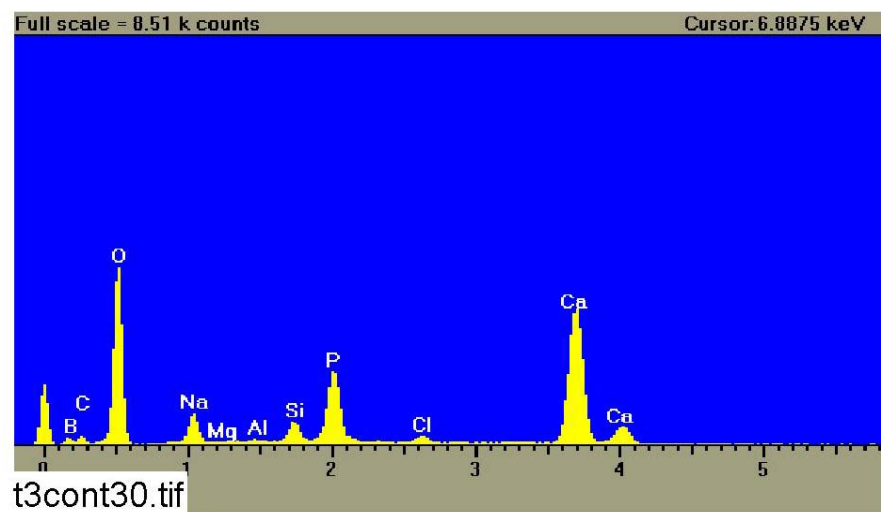

Figure D-14: EDS counting spectrum for the corrosion products (particles) shown in Figure D-13. (t3cont30)