# 4. SUMMARY OF KEY OBSERVATIONS

ICET Test #3 was conducted successfully, maintaining the critical physical and chemical parameters called out in the Test Plan. The test ran uninterrupted for 30 days. The solution chemistry behaved as expected, with the turbidity declining from its early values to steady numbers from Day 1 until the end of the test. TSS was relatively steady near its baseline value, with some increases during the test. The kinematic viscosity was steady for the entire test; and the pH was steady, averaging a value of 8.0.

Samples of the solution were taken daily. The chemical elements present were calcium, magnesium, silica, and sodium. Aluminum, copper, iron, zinc, and nickel were present in trace amounts. Strain-rate viscosity measurements indicated that the solution remained Newtonian throughout the test. No precipitates were observed in the solution, even after it had cooled to room temperature.

The submerged aluminum, IOZ-coated steel, copper, and uncoated steel coupons developed significant amounts of white particulate deposits. The aluminum coupons gained an average of 0.6 g, the IOZ-coated steel coupons 1.8 g, the copper 0.3 g, and the uncoated steel coupon lost 1.1 g. The submerged galvanized steel coupons were covered with a dense, gray particulate deposition, and they gained an average of 15.0 g.

The unsubmerged coupons exhibited light patterns of deposition, and they all experienced uniform weight gains. The aluminum coupons gained an average of 0.4 g, the galvanized steel 0.2 g, the copper 0.2 g, the IOZ-coated steel 2.0 g, and the uncoated steel 1.0 g.

Deposits on the fiberglass samples increased over time, and the deposits appeared to be chemically originated for the samples not lying on the tank bottom. These deposits covered individual fiberglass strands and in some cases formed webs between strands. Based on the SEM and ESEM results, the deposits likely originated from chemical precipitation during the sample-drying process. Comparing Days 4, 15, and 30 fiberglass samples showed deposits that were similar in property and amount. There was no significant difference in the amount of deposits found in the exterior and interior samples. Deposits found on the drain collar fiberglass were likely physically attached, and the exterior samples had significantly more deposits than the interior samples. Two different deposits were identified with EDS, one with a higher percentage of P and one with a higher percentage of Si. The former is likely calcium phosphate particles and the latter cal-sil particles. Deposits on and in the birdcage fiberglass similarly had the two different particulate deposits present. In addition, a pinkish-white gel-like precipitate covered the birdcage and much of the sediment. This gel had a consistency much like face cream, and it was composed primarily of Ca, O, and P, making it likely calcium phosphate.

Sediment on the tank bottom was prevalent, accumulating to depths of over 8 in. The sediment contained crystalline substances and calcite, making it primarily cal-sil, although some fugitive fiberglass was also present.

## REFERENCES


1. "Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring inside a PWR Containment Following a LOCA, Rev. 12c," March 30, 2005.

2. J. Dallman, J. Garcia, B. Letellier, and K. Howe, "Integrated Chemical Effects Tests: Data Compilation for Test 1," LA-UR-05-0124, July 2005.

3. "Pre-Test Operations, Test #3," ICET-PI-012, Rev. 1, April 1, 2005.

4. "Test Operations, Test #3 (cal-sil and fiberglass, with TSP)," ICET-PI-014, Rev. 0, April 5, 2005.

5. "Post-Test Operations, Test #3," ICET-PI-008, Rev. 3, May 2, 2005.

# Preface to Appendices

Eleven separate appendices were developed to capture more of the images and information obtained for Test #3. Several appendices are further divided into sub-appendices to better segregate the information according to the time point in the test when the samples were extracted from the test apparatus, the type of samples being evaluated, and the type of examinations performed. With the exception of Appendix L, each appendix represents a separate session of laboratory work that can be traced to a batch of samples that were typically processed in chronological order. Appendix L provides some of the detailed project instructions that were used to initiate Test #3, to conduct routine operations during the test, and to terminate the test with sample recovery and cleaning procedures.

Section 2.4.1.1 of this report reviewed the nomenclature adopted for reporting ICET results. This nomenclature is used in the caption labels for most of the figures presented in the appendices.

As noted in Section 2.4.1.1, the data presented in the appendices are largely qualitative in nature, consisting primarily of SEM and TEM micrographs and EDS spectra. The SEM data are further subdivided into environmental (or low-vacuum) SEM of hydrated samples and microprobe SEM of fully desiccated samples. Microprobe images can be generated using secondary electrons, which are sensitive to attenuation, to reveal fine structural details in a sample or backscatter electrons from the primary beam. Backscatter images indicate in shades of grey with high contrast the relative atomic number of materials across a sample. White or "bright" regions contain high-Z elements; dark regions contain lower-Z elements by comparison.

Transcriptions of the laboratory logbooks are provided for each appendix to document better commonalities that existed among the samples at the time of analysis. Logbook information was developed for most, but not all, of the images presented in the appendices. Interpretation and understanding of the images and their accompanying EDS spectra can be improved by referring frequently to the logbook sample descriptions and sequences.

Typically, a relatively large quantity of a test sample was delivered for SEM or TEM analysis, and then several small subsamples of each item were examined. Note that each subsample was assigned a sequential reference number during the laboratory session. These reference numbers have been cited in the figure captions whenever possible to preserve the connection between the micrographs and the notebook descriptions. Electronic filenames have also been stamped on the images to permit retrieval of the original data files, which are archived elsewhere. Individual data sets for a given sample item have been collated into a typical sequence of (1) visual image, (2) EDS spectra, and (3) semiquantitative mass composition.

Semiquantitative mass compositions are also presented for many of the EDS spectra. These results are obtained from a commercial algorithm that decomposes the spectra into

the separate contributions of each element. Composition estimates should be interpreted with the caveats stated in Section 2.4.1.1 fully in mind.

The appendix titles are listed below.

**Appendix A. SEM and ESEM/EDS Data for Test #3 Day-4 Fiberglass in Low-Flow Zone**

**Appendix B. ESEM and SEM Day-15 Fiberglass**

- B1. ESEM/EDS and SEM/EDS Data for Test #3, Day-15 Fiberglass in High-Flow Zones
- B2. ESEM/EDS and SEM/EDS Data for Test #3, Day-15 Fiberglass in Low-Flow Zones

**Appendix C. ESEM and SEM Day-30 Fiberglass**

- C1. ESEM/EDS Data for Test #3, Day-30 Fiberglass in High-Flow Zones
- C2. ESEM Data for Test #3, Day-30 Fiberglass in Low-Flow Zones
- C3. ESEM/EDS Data for Test #3, Day-30 Drain Collar Fiberglass
- C4. SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Birdcage Fiberglass

**Appendix D. ESEM and SEM/EDS Data for Test #3 Day-30 Corrosion Products**

**Appendix E. SEM Day-30 Coupons**

- E1. SEM/EDS Data for Test #3, Day-30 Aluminum Coupons
- E2. SEM/EDS Data for Test #3, Day-30 Copper Coupons
- E3. SEM/EDS Data for Test #3, Day-30 Galvanized Steel Coupons
- E4. SEM/EDS Data for Test #3, Day-30 Steel Coupons

**Appendix F. SEM/EDS Data for Test #3, Day-30 Flow Meter**

**Appendix G. SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Gel**

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

**Appendix I. ESEM/EDS Data for Test #3, Day-30 Sediment**

**Appendix J. TEM Data for Test #3 Solution Samples**

**Appendix K. UV Absorbance Spectrum—Day-30 Solution Samples**

**Appendix L. ICET Test #3: Pre-Test, Test, and Post-Test Project Instructions**

# Appendix A

# SEM and ESEM/EDS Data for Test #3, Day-4 Fiberglass in Low-Flow Zone

## Figures


Figure A-1. Environmental SEM image magnified 150 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FX1) ..... A-5

Figure A-2. Environmental SEM image magnified 1000 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FX2) ..... A-5

Figure A-3. EDS counting spectrum for the deposits between the fibers on ESEM image shown in Figure A-2. (T3D4FX3) ..... A-6

Figure A-4. EDS counting spectrum (after calibration) for the deposits between the fibers on ESEM image shown in Figure A-2. (T3D4FX4) ..... A-6

Figure A-5. Environmental SEM image magnified 500 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FX5) ..... A-7

Figure A-6. Environmental SEM image magnified 150 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FI6) ..... A-7

Figure A-7. Environmental SEM image magnified 1000 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FI7) ..... A-8

Figure A-8. EDS counting spectrum (after calibration) for the deposits between the fibers on ESEM image shown in Figure A-7. (T3D4FI8) ..... A-8

Figure A-9. SEM image magnified 150 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FibGlsEX001) ..... A-9

Figure A-10. SEM image magnified 1000 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FibGlsEX002) ..... A-9

Figure A-11. SEM image magnified 300 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FibGlsEX003) ..... A-10

Figure A-12. SEM image magnified 50 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN001) ..... A-10

Figure A-13. SEM image magnified 150 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN002) ..... A-11
Figure A-14. SEM image magnified 400 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN003) ..... A-11
Figure A-15. SEM image magnified 1000 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN004) ..... A-12

During ICET Test #3, work was continued for the purpose of identifying the origin and chemical composition of the products that were formed during the test. One objective of ICET tests was to identify the composition of the debris on the fiberglass and of the particulate substances in the test solution. This identification was partially done by examining fiberglass samples located in low-flow-velocity sections of the ICET tank. These samples were removed from the tank on day 4 of the test and examined via Environmental SEM (ESEM)/probe SEM/EDS. The samples were taken both from the exterior and interior locations of the fiberglass package.

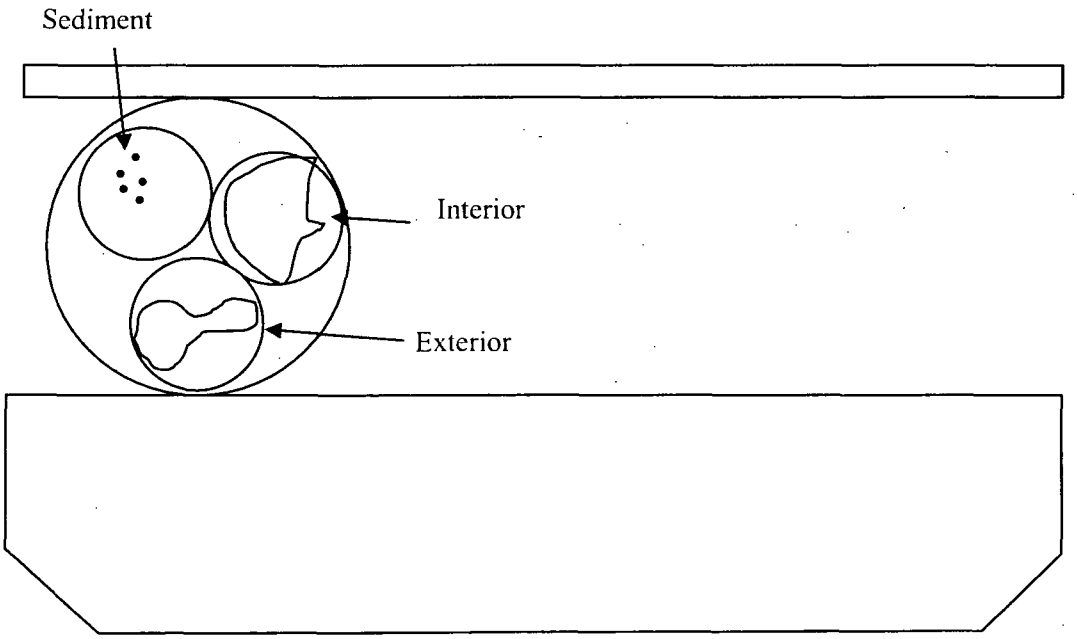
Probe SEM was used to examine the fiberglass samples that had been air dried at room temperature and coated with carbon. In addition to probe SEM, ESEM was employed to analyze uncoated, wet fiberglass samples. The ESEM was performed under low vacuum conditions (i.e., 80 Pa) in order to minimize the modification of the fiberglass through the drying process. The EDS results provide a semi-quantitative elemental analysis of the debris attached to the fiberglass.

Test #3 Day-4 low-flow fiberglass samples were obtained on April 9, 2005 (4th day for Test #3), and SEM/EDS data presented here were obtained shortly thereafter. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

## Transcribed Laboratory Log

Laboratory session from April 12, 2005.

ESEM & SEM Test #3, Day-4 Fiberglass.




### ESEM Exterior Low-Flow Submerged Fiberglass Samples

| | | | | |
|---|---|---|---|---|
| Image: | T3D4FX1 | 150 × | ESEM image | Figure A-1 |
| | T3D4FX2 | 1000 × | ESEM at higher magnification | Figure A-2 |
| EDS: | T3D4FX3 | | Particles on Image FX2 | Figure A-3 |
| | T3D4FX4 | | FX2 particles after calibration with primed elemental analysis | Figure A-4 |
| Image: | T3D4FX5 | 500 × | ESEM image | Figure A-5 |

### ESEM Interior Low-flow Submerged Fiberglass Samples

| | | | | |
|---|---|---|---|---|
| Image: | T3D4FI6 | 150 × | ESEM image | Figure A-6 |
| | T3D4FI7 | 1000 × | ESEM at higher magnification | Figure A-7 |
| EDS: | T3D4FI8 | | EDS of particles on FI7 | Figure A-8 |

**SEM Exterior Low-flow Submerged Fiberglass Samples**

| | | | | |
|---|---|---|---|---|
| Image: | T3D4FibGlsEX001 | 150 × | SEM image | Figure A-9 |
| | T3D4FibGlsEX002 | 1000 × | SEM at higher magnification | Figure A-10 |
| | T3D4FibGlsEX003 | 300 × | | Figure A-11 |

**SEM Interior Low-flow Submerged Fiberglass Samples**

| | | | | |
|---|---|---|---|---|
| Image: | T3D4FibGlsIn001 | 50 × | SEM image | Figure A-12 |
| | T3D4FibGlsIn002 | 150 × | SEM image | Figure A-13 |
| | T3D4FibGlsIn003 | 400 × | SEM image | Figure A-14 |
| | T3D4FibGlsIn004 | 1000 × | SEM at higher magnification | Figure A-15 |

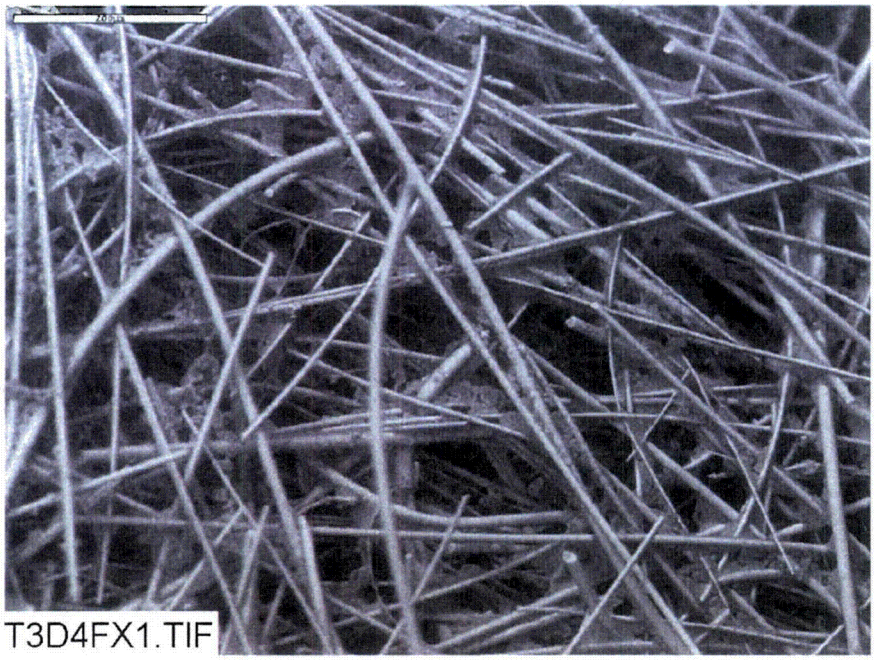



**Figure A-1.** **Environmental SEM image magnified 150 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FX1)**

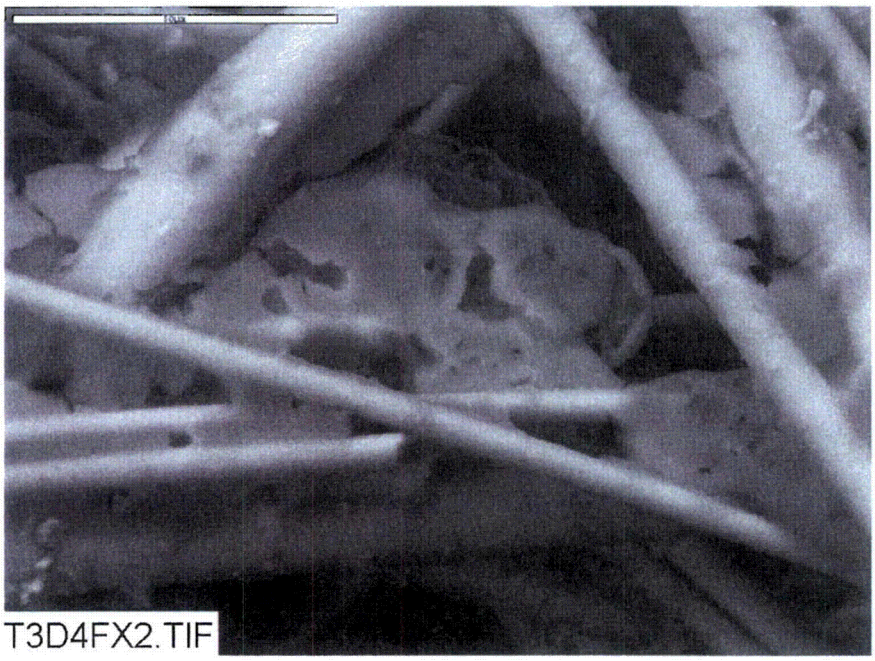



**Figure A-2.** **Environmental SEM image magnified 1000 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FX2)**

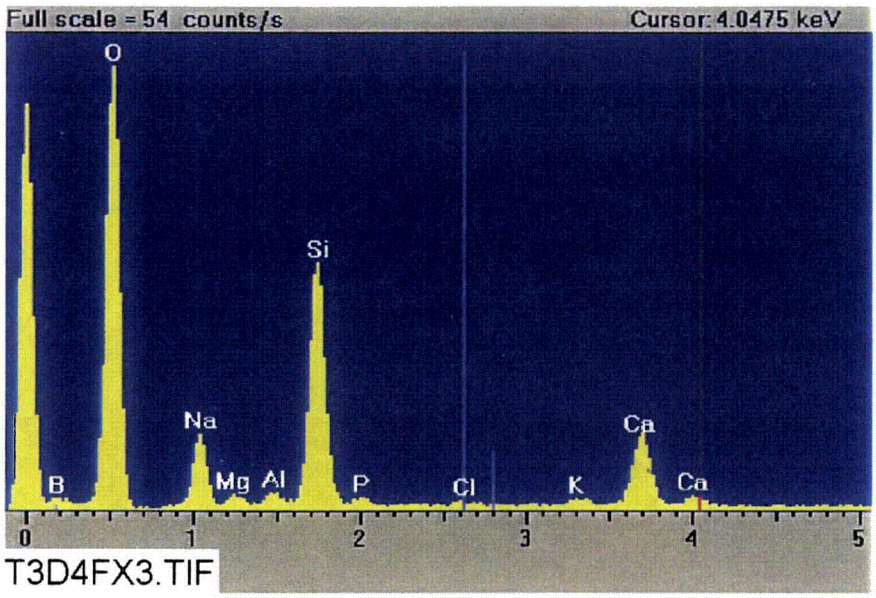



**Figure A-3.** **EDS counting spectrum for the deposits between the fibers on ESEM image shown in Figure A-2. (T3D4FX3)**

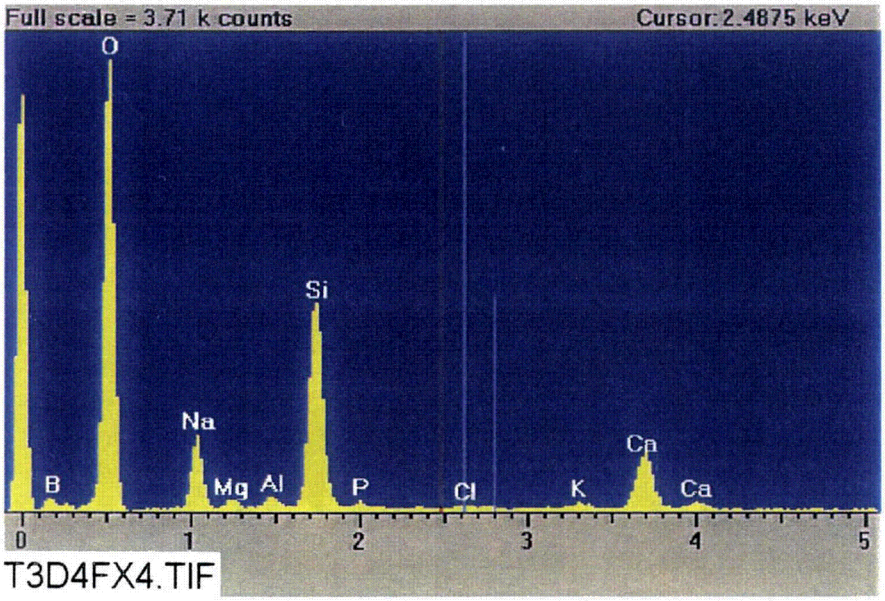



**Figure A-4.** **EDS counting spectrum (after calibration) for the deposits between the fibers on ESEM image shown in Figure A-2. (T3D4FX4)**

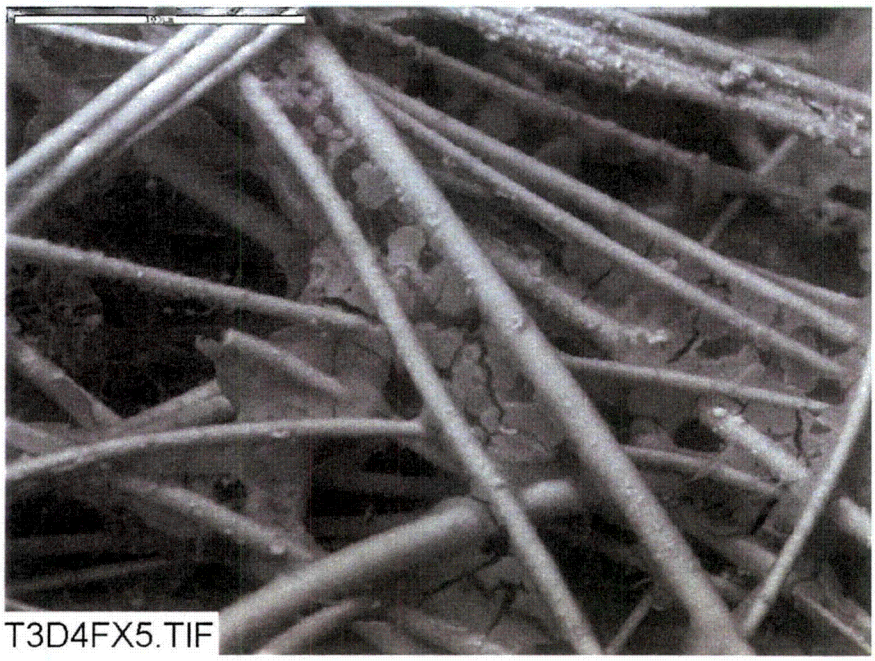



**Figure A-5.** **Environmental SEM image magnified 500 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FX5)**


**Figure A-6.** **Environmental SEM image magnified 150 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FI6)**


**Figure A-7.** **Environmental SEM image magnified 1000 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FI7)**

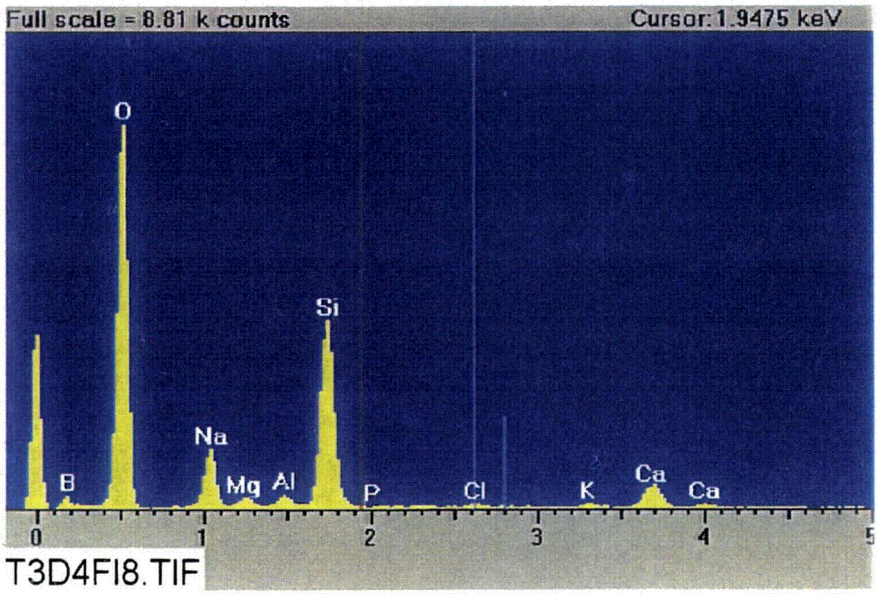



**Figure A-8.** **EDS counting spectrum (after calibration) for the deposits between the fibers on ESEM image shown in Figure A-7. (T3D4FI8)**

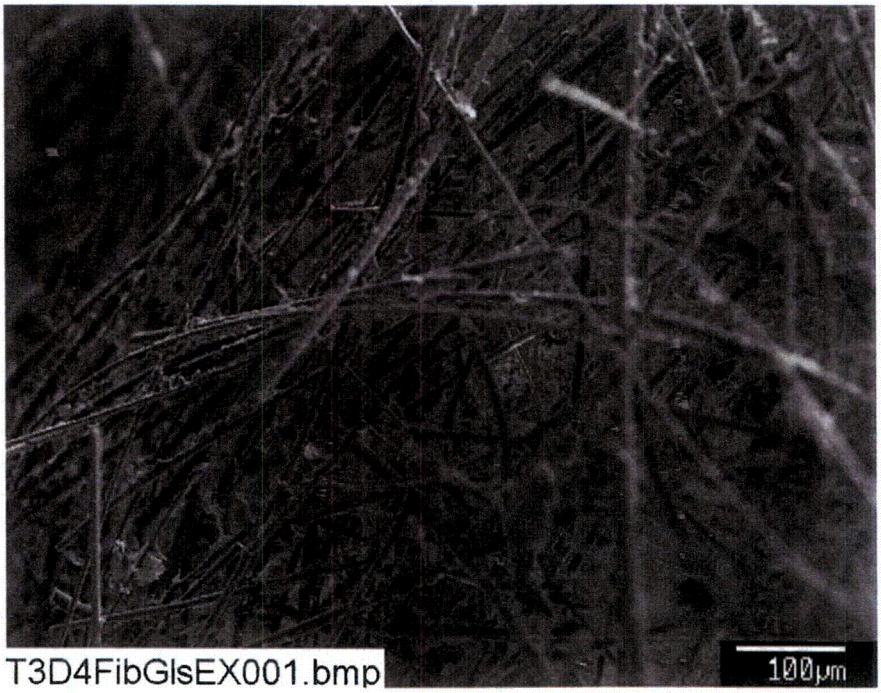



**Figure A-9.** **SEM image magnified 150 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FibGlsEX001)**

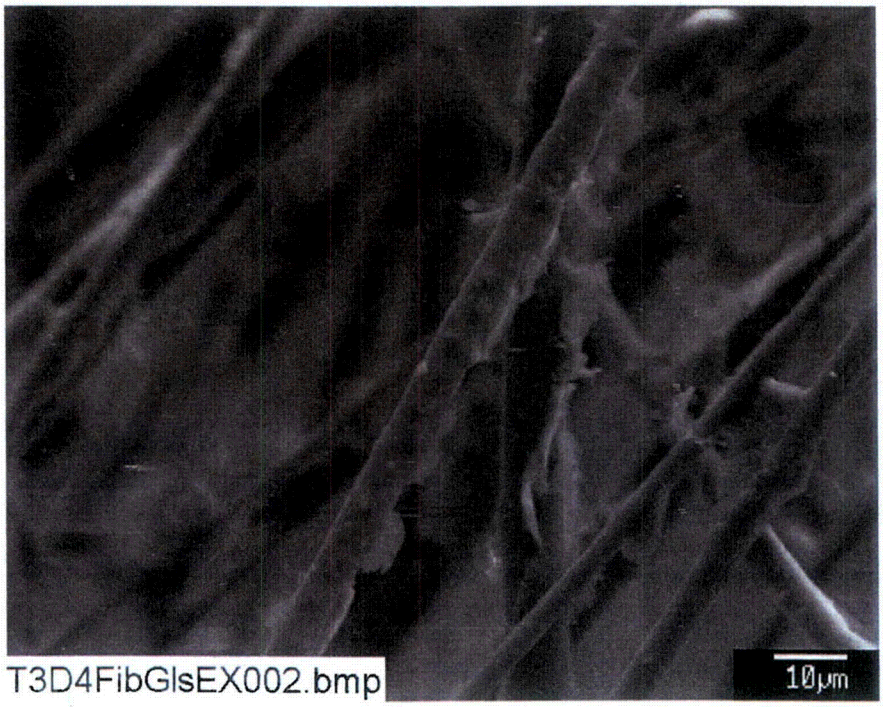



**Figure A-10.** **SEM image magnified 1000 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FibGlsEX002)**

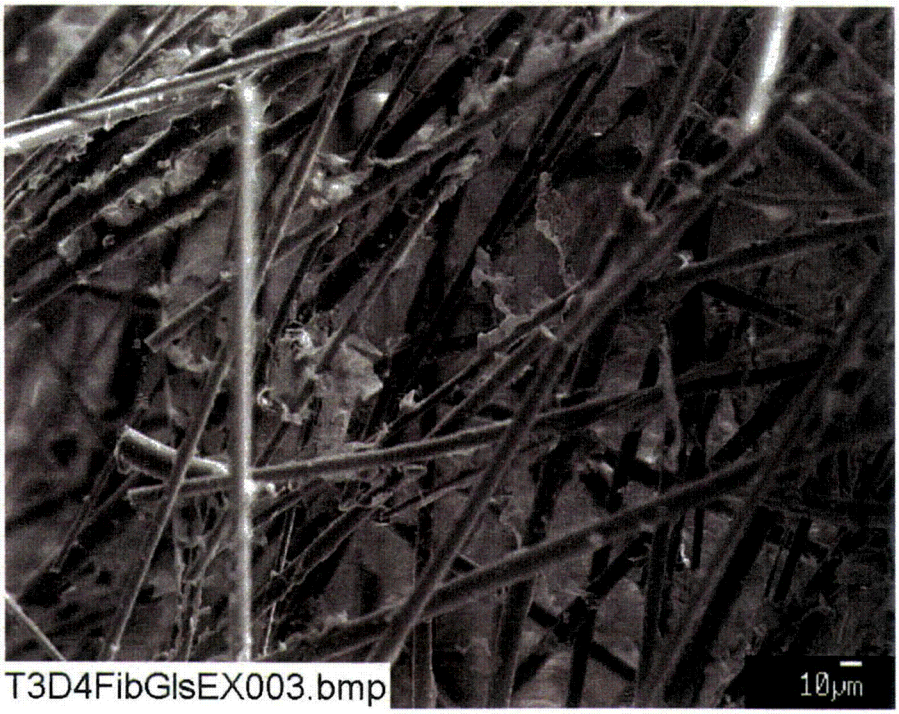



**Figure A-11.** **SEM image magnified 300 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FibGlsEX003)**

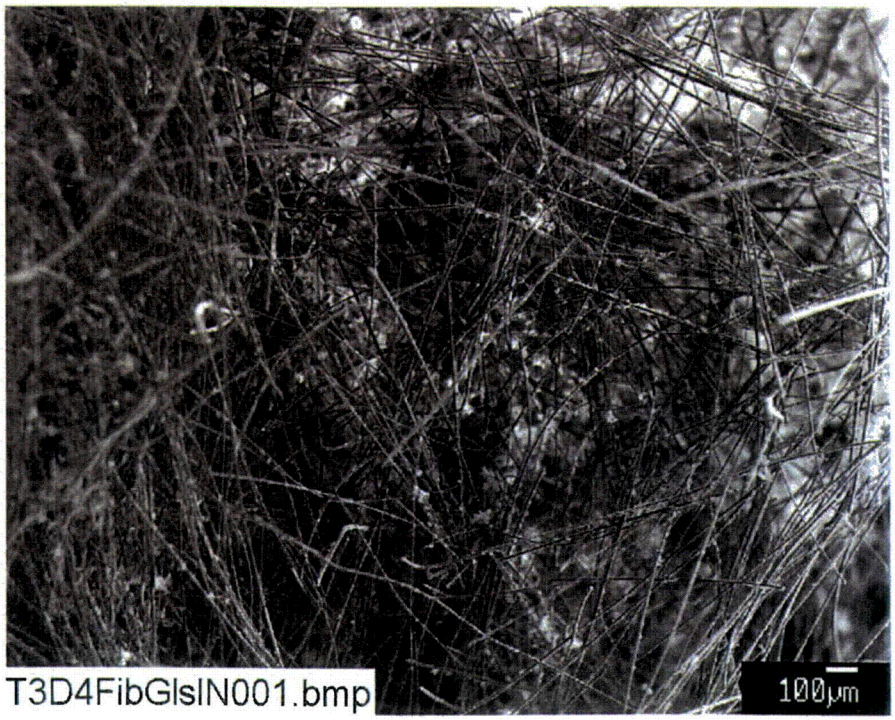



**Figure A-12.** **SEM image magnified 50 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN001)**

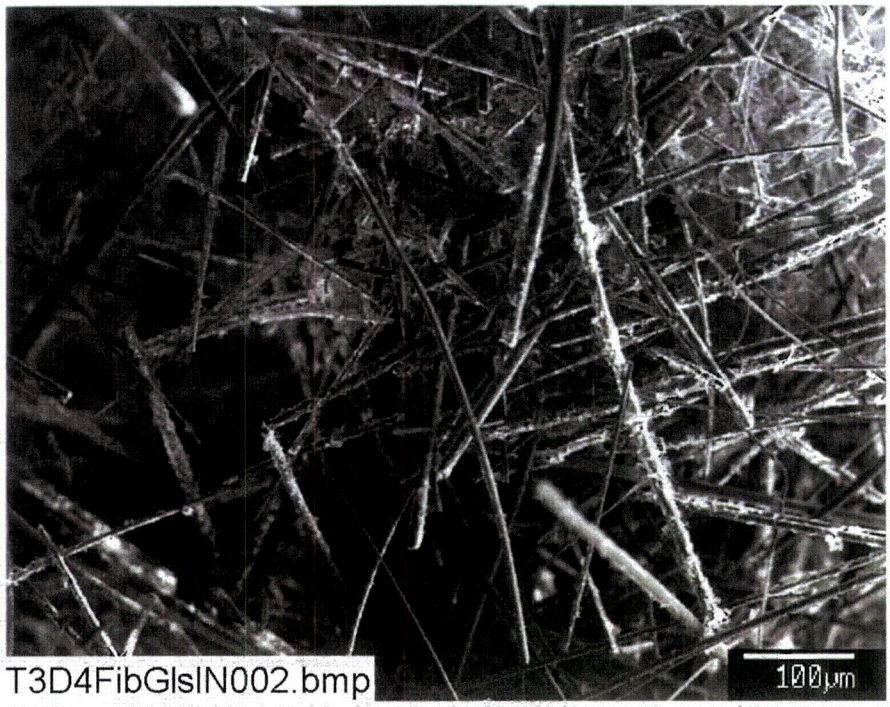



**Figure A-13.** **SEM image magnified 150 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN002)**

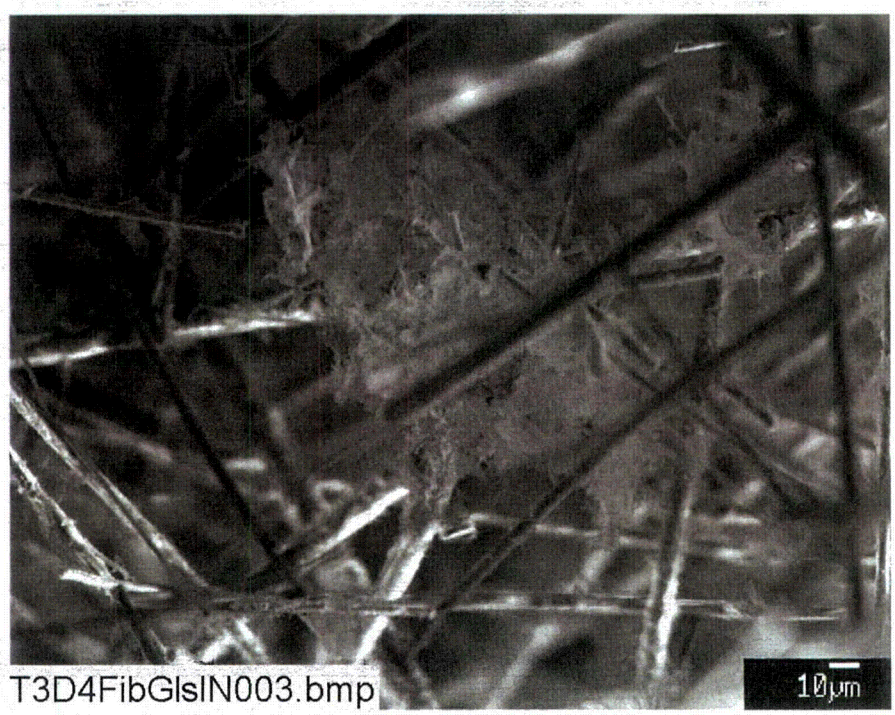



**Figure A-14.** **SEM image magnified 400 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN003)**

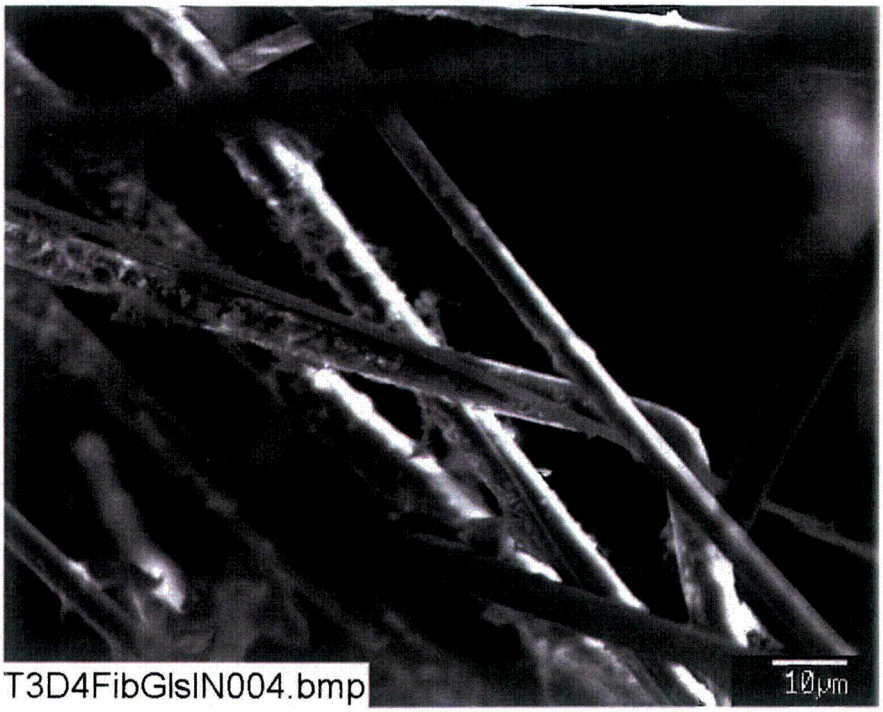



**Figure A-15.** **SEM image magnified 1000 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlslN004)**

# Appendix B1

# ESEM/EDS and SEM/EDS Data for Test #3, Day-15 Fiberglass in High-Flow Zones

## Figures


Figure B1-1. Environmental SEM image magnified 100 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HI1) ..... B1-5

Figure B1-2. Environmental SEM image magnified 1000 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HI2) ..... B1-5

Figure B1-3. Environmental SEM image magnified 2000 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HI3) ..... B1-6

Figure B1-4. EDS counting spectrum for the flocculence deposits between the fibers on ESEM image shown in Figure B1-3. (T3D15HIA) ..... B1-6

Figure B1-5. Environmental SEM image magnified 110 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HX4) ..... B1-7

Figure B1-6. Environmental SEM image magnified 1000 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HX5) ..... B1-7

Figure B1-7. EDS counting spectrum for the deposits between the fibers on ESEM image shown in Figure B1-6. (T3D15HXB) ..... B1-8

Figure B1-8. SEM image magnified 100 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HIFlowInt002) ..... B1-8

Figure B1-9. SEM image magnified 1000 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HIFlowInt003) ..... B1-9

Figure B1-10. SEM image magnified 1000 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HIFlowInt004) ..... B1-9

Figure B1-11. EDS counting spectrum for the flocculence deposits between the fibers on SEM image shown in Figure B1-10. (T3D15HiFlowIntEDS1) ..... B1-10

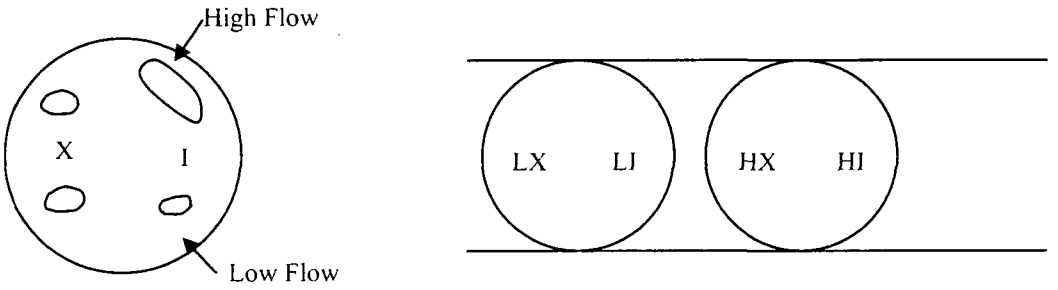
Figure B1-12. SEM image magnified 100 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HIFlowExt005) ..... B1-10
Figure B1-13. SEM image magnified 1000 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HIFlowExt006) ..... B1-11
Figure B1-14. SEM image magnified 500 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HIFlowExt007) ..... B1-11
Figure B1-15. EDS counting spectrum for the flocculence deposits between the fibers on SEM image shown in Figure B1-14. (T3D15HiFlowExtEDS2) ..... B1-12

This appendix shows the ESEM/probe SEM/EDS results for ICET Test #3, Day-15 high-flow zone fiberglass samples. Both exterior and interior locations of the fiberglass samples were examined. These fiberglass samples were obtained on April 20, 2005 (15th day of Test #3). For probe SEM examination, the fiberglass samples were air dried at room temperature and then were coated with Au/Pd. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

## Transcribed Laboratory Log

Laboratory session from April 22, 2005.
Test #3, Day-15 fiberglass in high-flow zones

**ESEM** **SEM**




Instrument Conditions: ESEM, Pressure = 80 Pa
SEM, Electron beam of $1 \times 10^{-9}$ (A)

### ESEM Fiberglass High-Flow Interior

| | | | | |
|---|---|---|---|---|
| Image: | T3D15HI1 | 100 × | | Figure B1-1 |
| | T3D15HI2 | 1000 × | | Figure B1-2 |
| | T3D15HI3 | 2000 × | | Figure B1-3 |
| EDS: | T3D15HIA | | EDS on particles shown in T3D15HI3 | Figure B1-4 |

### ESEM Fiberglass High-Flow Exterior

| | | | | |
|---|---|---|---|---|
| Image: | T3D15HX4 | 110 × | | Figure B1-5 |
| | T3D15HX5 | 1000 × | | Figure B1-6 |
| EDS: | T3D15HXB | | EDS on particles shown in T3D15HX5 | Figure B1-7 |

**SEM Fiberglass High-Flow Interior**

| | | | | |
|---|---|---|---|---|
| Image: | T3D15HIFlowInt002 | 100 × | | Figure B1-8 |
| | T3D15HIFlowInt003 | 1000 × | | Figure B1-9 |
| | T3D15HIFlowInt004 | 1000 × | | Figure B1-10 |
| EDS: | T3D15HIFlowIntEDS1 | | EDS on particles shown in Int004 | Figure B1-11 |

**SEM Fiberglass High-Flow Exterior**

| | | | | |
|---|---|---|---|---|
| Image: | T3D15HIFlowExt005 | 100 × | | Figure B1-12 |
| | T3D15HIFlowExt006 | 1000 × | | Figure B1-13 |
| | T3D15HIFlowExt007 | 500 × | | Figure B1-14 |
| EDS: | T3D15HIFlowExtEDS 2 | | EDS on particles shown in Ext007 | Figure B1-15 |


**Figure B1-1.** **Environmental SEM image magnified 100 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HI1)**

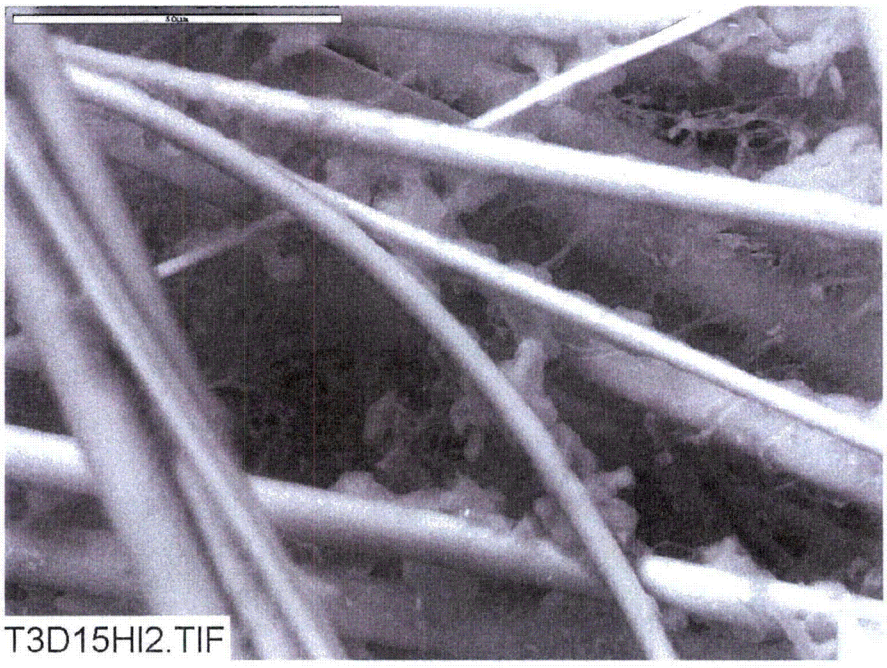



**Figure B1-2.** **Environmental SEM image magnified 1000 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HI2)**

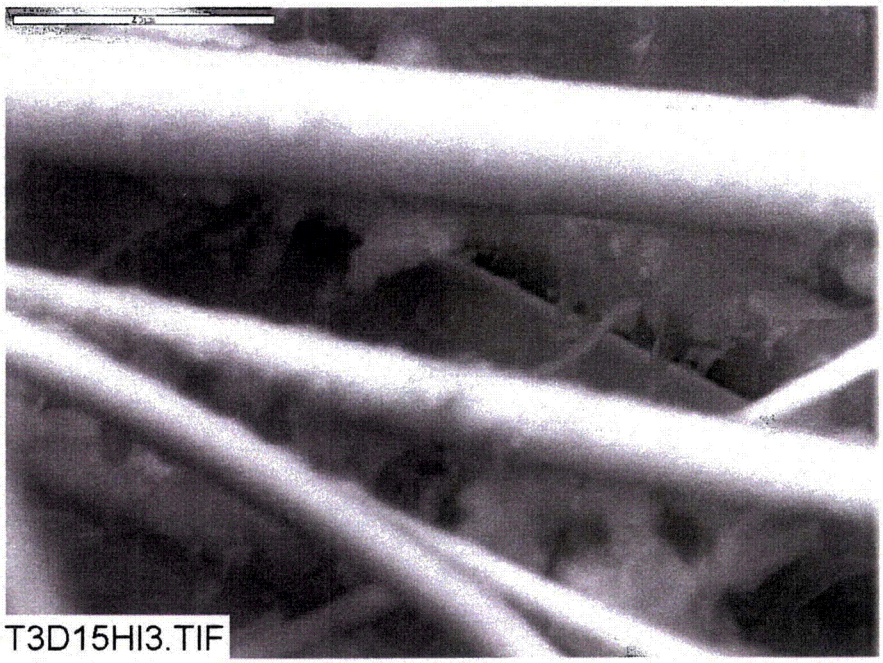



**Figure B1-3.** **Environmental SEM image magnified 2000 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HI3)**

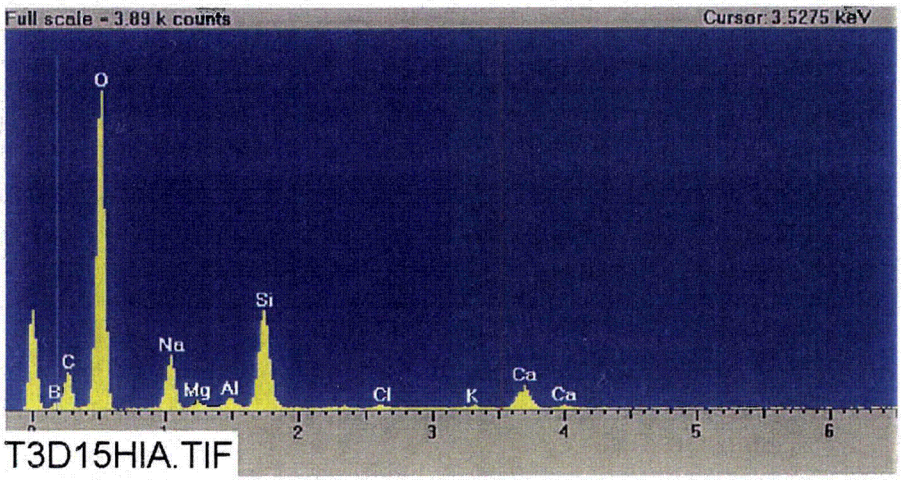



**Figure B1-4.** **EDS counting spectrum for the flocculence deposits between the fibers on ESEM image shown in Figure B1-3. (T3D15HIA)**


**Figure B1-5.** **Environmental SEM image magnified 110 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HX4)**

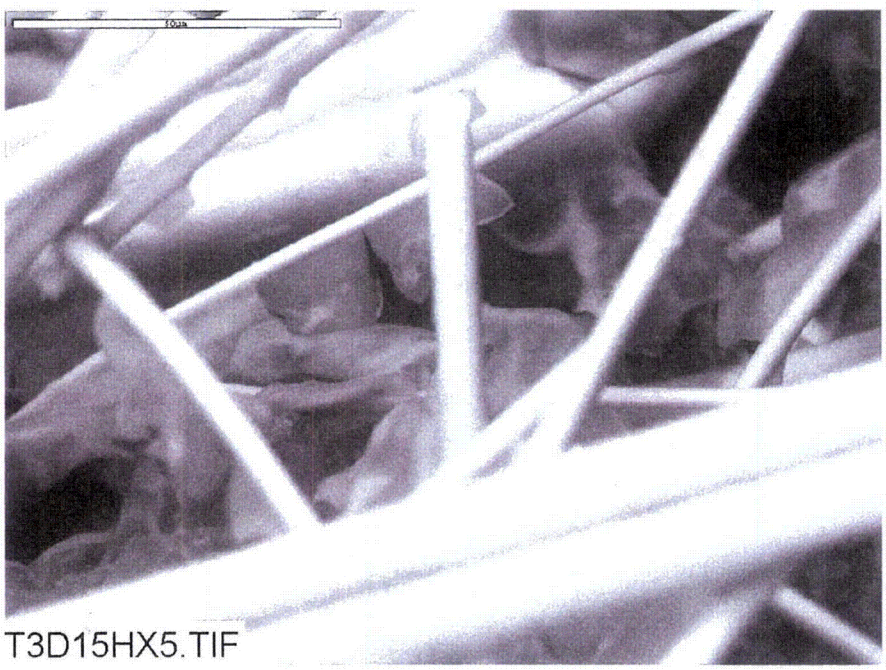



**Figure B1-6.** **Environmental SEM image magnified 1000 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HX5)**

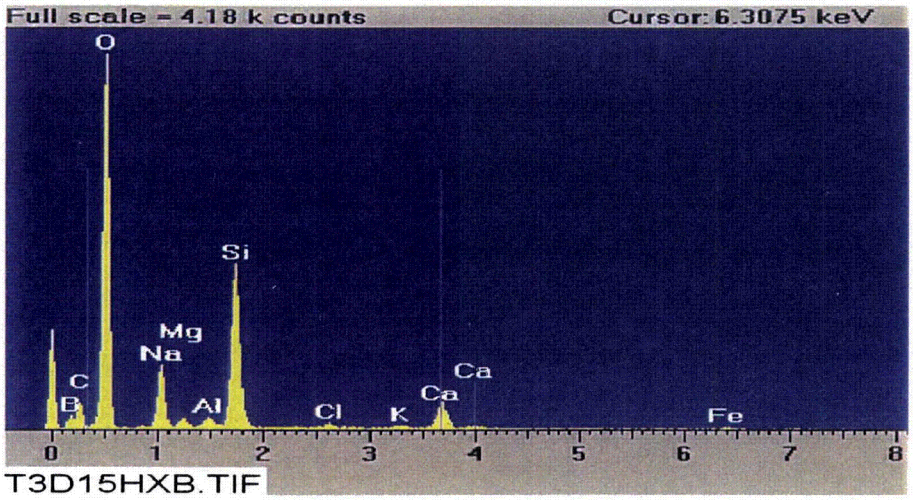



**Figure B1-7.** **EDS counting spectrum for the deposits between the fibers on ESEM image shown in Figure B1-6. (T3D15HXB)**

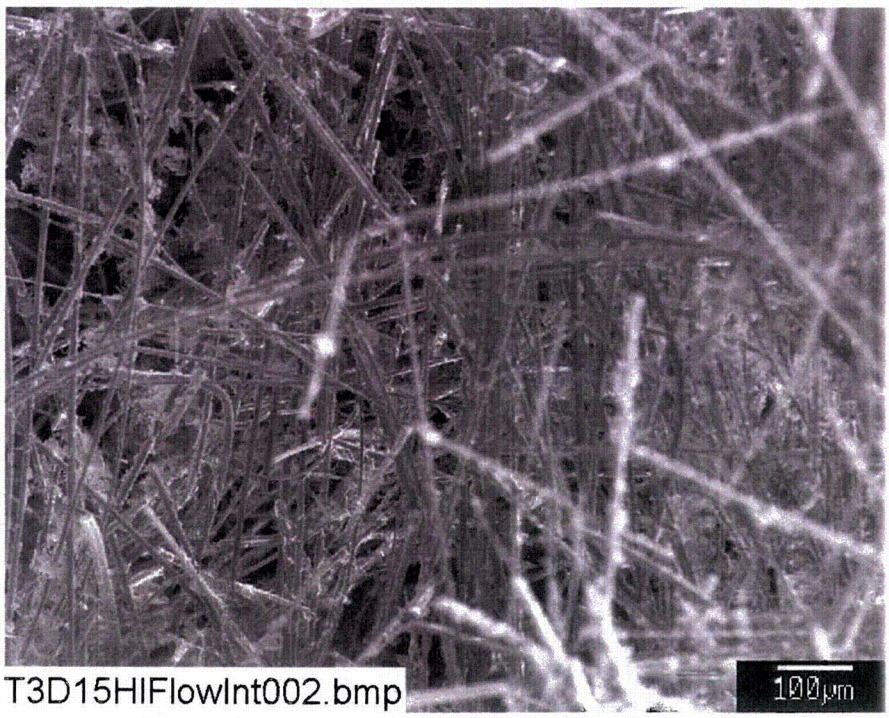



**Figure B1-8.** **SEM image magnified 100 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HIFlowInt002)**

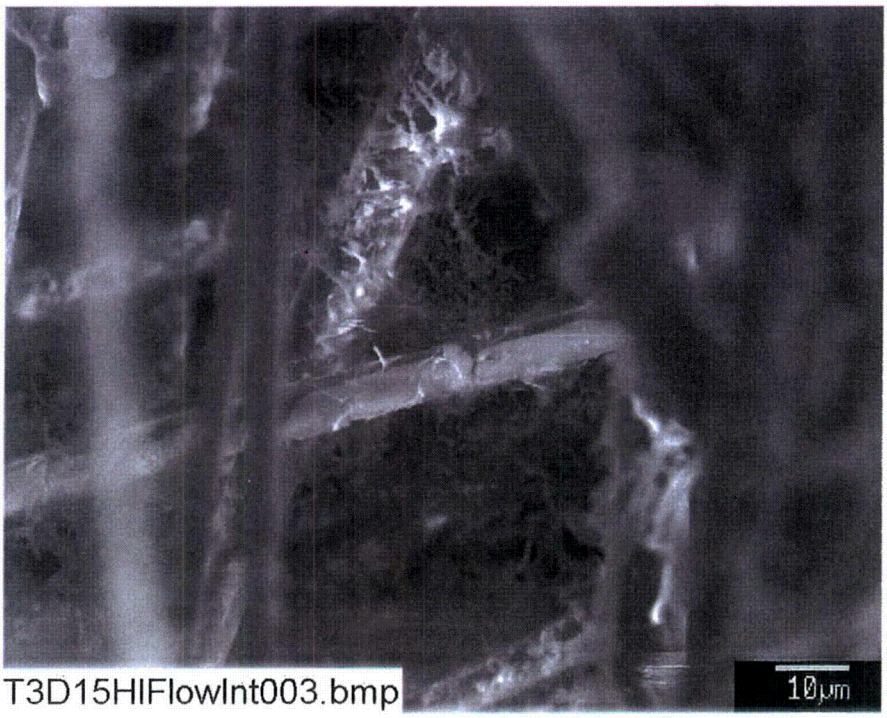



**Figure B1-9.** **SEM image magnified 1000 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HIFlowInt003)**

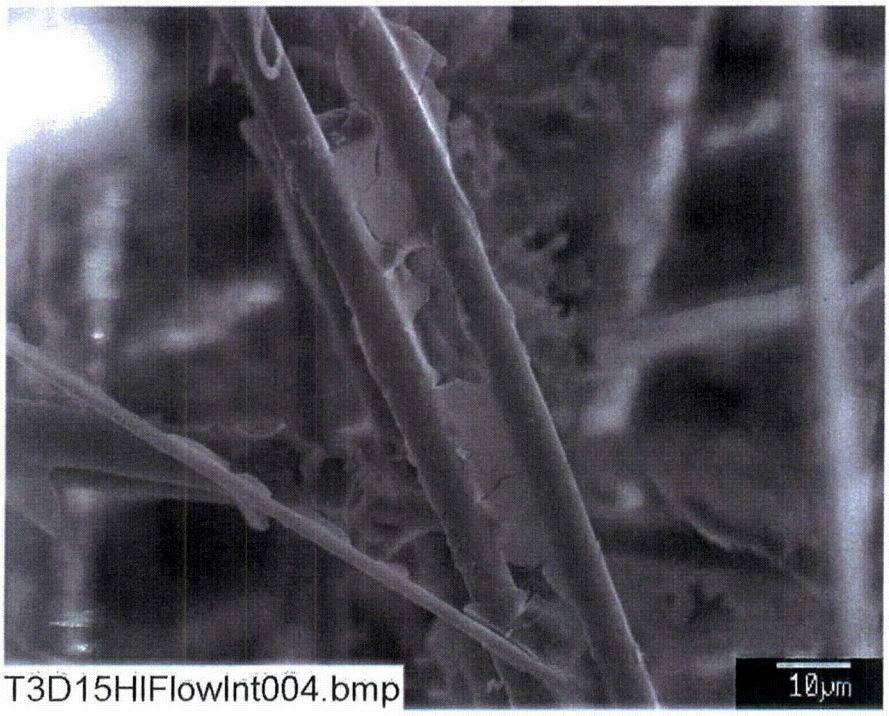



**Figure B1-10.** **SEM image magnified 1000 times for a Test #3, Day-15 high-flow interior fiberglass sample. (T3D15HIFlowInt004)**

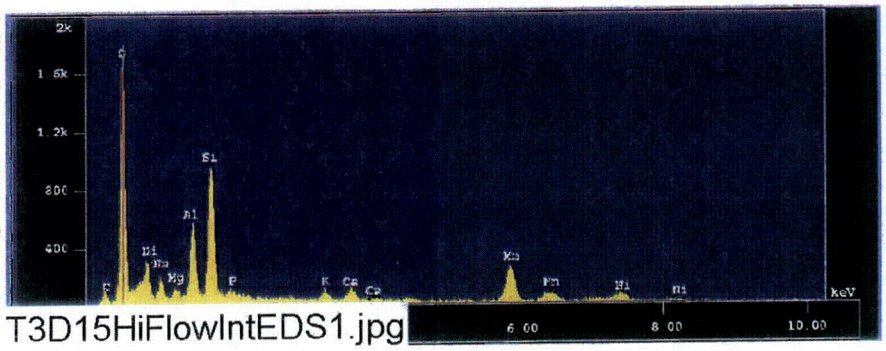



**Figure B1-11.** **EDS counting spectrum for the flocculence deposits between the fibers on SEM image shown in Figure B1-10. (T3D15HiFlowIntEDS1)**

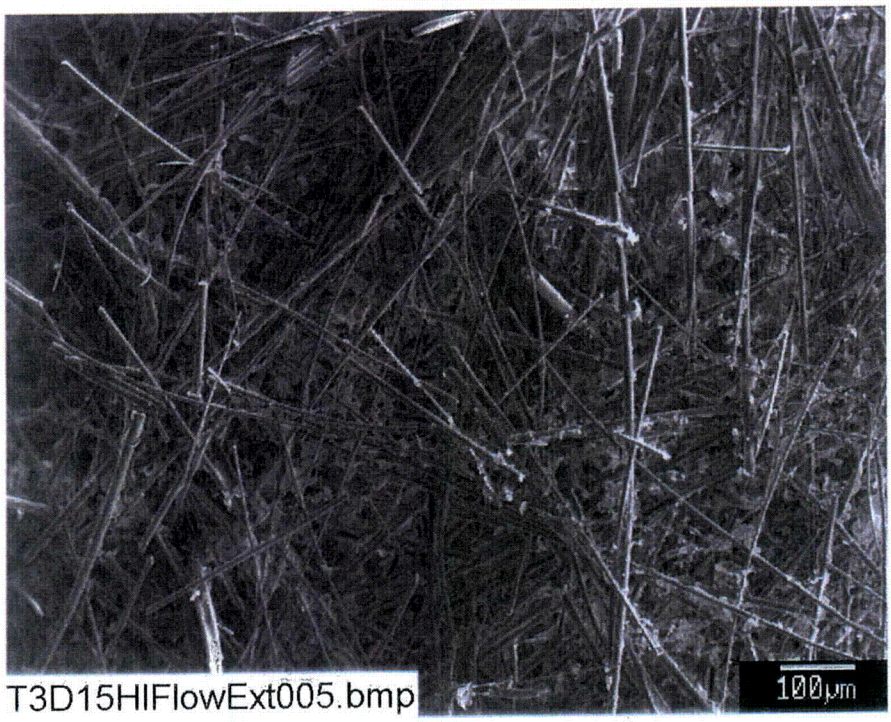



**Figure B1-12.** **SEM image magnified 100 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HIFlowExt005)**

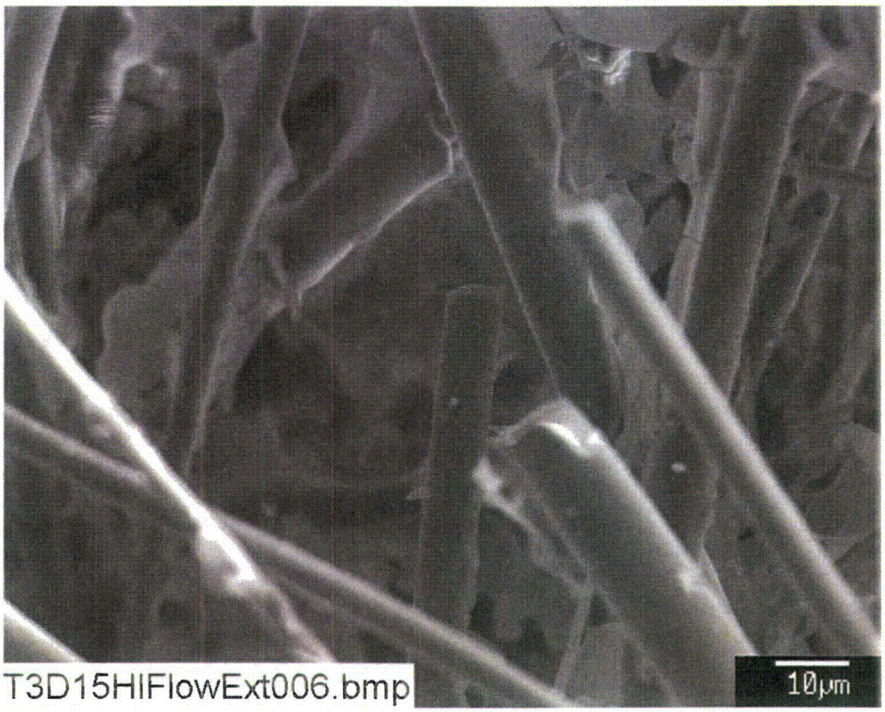



**Figure B1-13. SEM image magnified 1000 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HIFlowExt006)**

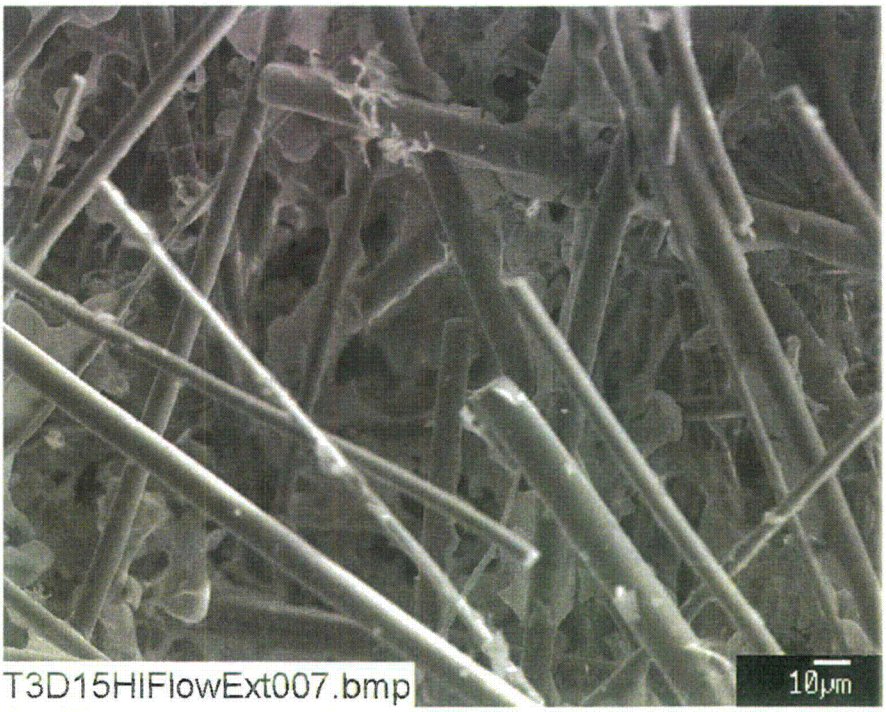



**Figure B1-14. SEM image magnified 500 times for a Test #3, Day-15 high-flow exterior fiberglass sample. (T3D15HIFlowExt007)**

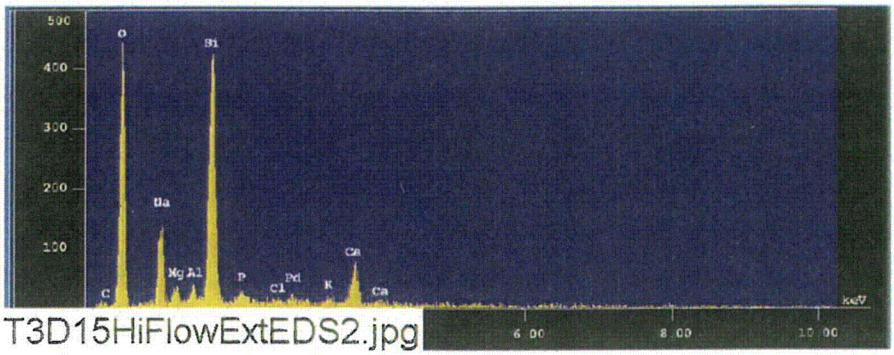



**Figure B1-15.** **EDS counting spectrum for the flocculence deposits between the fibers on SEM image shown in Figure B1-14. (T3D15HiFlowExtEDS2)**

# Appendix B2

# ESEM/EDS and SEM/EDS Data for Test #3, Day-15 Fiberglass in Low-Flow Zones

## Figures


Figure B2-1. Environmental SEM image magnified 100 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LI6) ..... B2-5

Figure B2-2. Environmental SEM image magnified 1000 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LI7) ..... B2-5

Figure B2-3. Environmental SEM image magnified 1000 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LI8) ..... B2-6

Figure B2-4. EDS counting spectrum for the flocculence deposits between the fibers on ESEM image shown in Figure B2-3. (T3D15LIC) ..... B2-6

Figure B2-5. Environmental SEM image magnified 110 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LX9) ..... B2-7

Figure B2-6. Environmental SEM image magnified 1000 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LX0) ..... B2-7

Figure B2-7. EDS counting spectrum for the deposits between the fibers on ESEM image shown in Figure B2-6. (T3D15LXD) ..... B2-8

Figure B2-8. SEM image magnified 100 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LowFlowInt008) ..... B2-8

Figure B2-9. SEM image magnified 1000 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LowFlowInt009) ..... B2-9

Figure B2-10. SEM image magnified 1000 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LowFlowInt010) ..... B2-9

Figure B2-11. SEM image magnified 100 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LowFlowExt011) ..... B2-10

Figure B2-12. SEM image magnified 1000 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LowFlowExt012) ..... B2-10

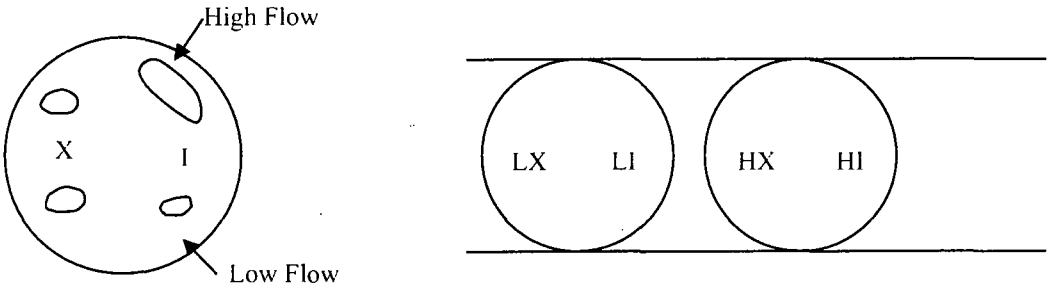
Figure B2-13. SEM image magnified 1000 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LowFlowExt013) ..... B2-11
Figure B2-14. EDS counting spectrum for the flocculence deposits between the fibers on SEM image shown in Figure B2-13. (T3D15LowFlowExtEDS3) ..... B2-11

This appendix shows the ESEM/probe SEM/EDS results on ICET Test #3, Day-15 low-flow zone fiberglass samples. The samples were obtained on April 20, 2005 (15th day of Test #3). Both exterior and interior locations of the fiberglass samples were examined. For probe SEM examination, the fiberglass samples were dried in air at room temperature followed by being coated with Au/Pd. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

## Transcribed Laboratory Log

Laboratory session from April 22, 2005.
Test #3, Day-15 fiberglass in low-flow zones

**ESEM** **SEM**




Instrument Conditions: ESEM, Pressure = 80 Pa
SEM, Electron beam of $1 \times 10^{-9}$ (A)

### ESEM Fiberglass Low-Flow Interior

| | | | | |
|---|---|---|---|---|
| Image: | T3D15LI6 | 100 × | | Figure B2-1 |
| | T3D15LI7 | 1000 × | | Figure B2-2 |
| | T3D15LI8 | 1000 × | | Figure B2-3 |
| EDS: | T3D15LIC | | EDS on particles shown in T3D15LI8 | Figure B2-4 |

### ESEM Fiberglass Low-Flow Exterior

| | | | | |
|---|---|---|---|---|
| Image: | T3D15LX9 | 110 × | | Figure B2-5 |
| | T3D15LX0 | 1000 × | | Figure B2-6 |
| EDS: | T3D15LXD | | EDS on particles shown in T3D15LX0 | Figure B2-7 |

**SEM Fiberglass Low-Flow Interior**

| | | | | |
|---|---|---|---|---|
| Image: | T3D15LowFlowInt008 | 100 × | | Figure B2-8 |
| | T3D15LowFlowInt009 | 1000 × | | Figure B2-9 |
| | T3D15LowFlowInt010 | 1000 × | | Figure B2-10 |

**SEM Fiberglass Low-Flow Exterior**

| | | | | |
|---|---|---|---|---|
| Image: | T3D15LowFlowExt011 | 100 × | | Figure B2-11 |
| | T3D15LowFlowExt012 | 1000 × | | Figure B2-12 |
| | T3D15LowFlowExt013 | 1000 × | | Figure B2-13 |
| EDS: | T3D15LowFlowExtEDS3 | | EDS on particles shown in Ext007 | Figure B2-14 |

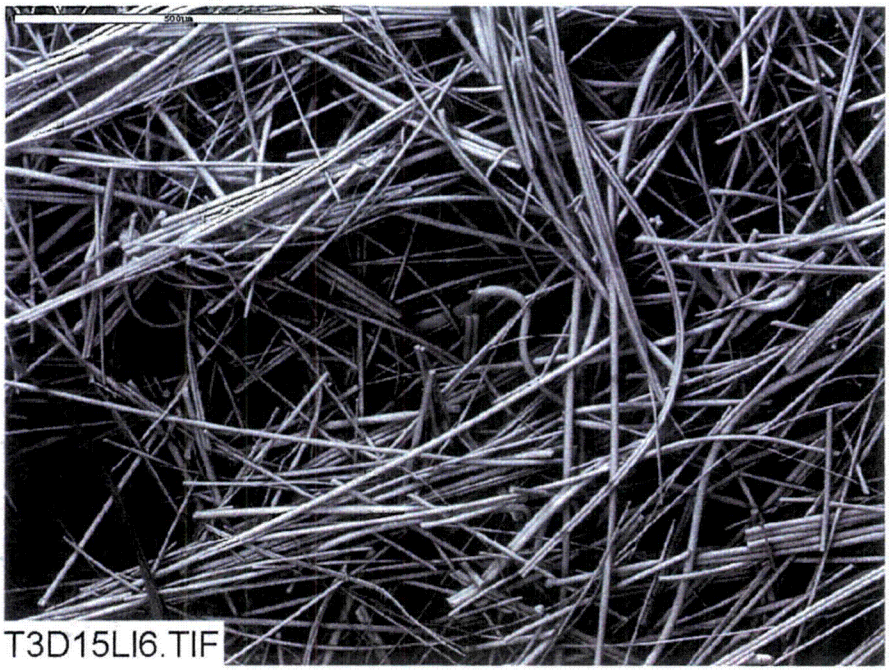



**Figure B2-1.** **Environmental SEM image magnified 100 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LI6)**

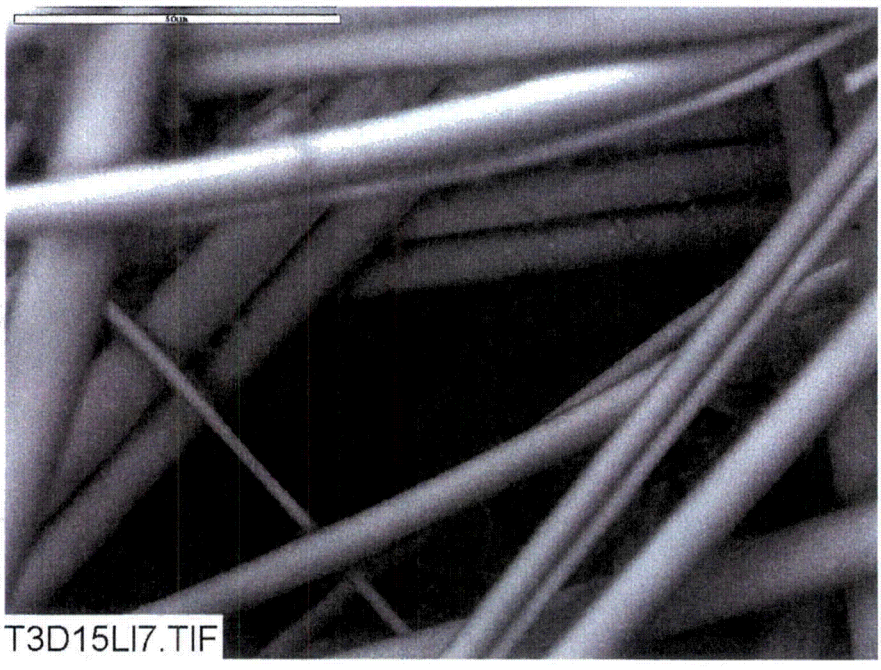



**Figure B2-2.** **Environmental SEM image magnified 1000 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LI7)**

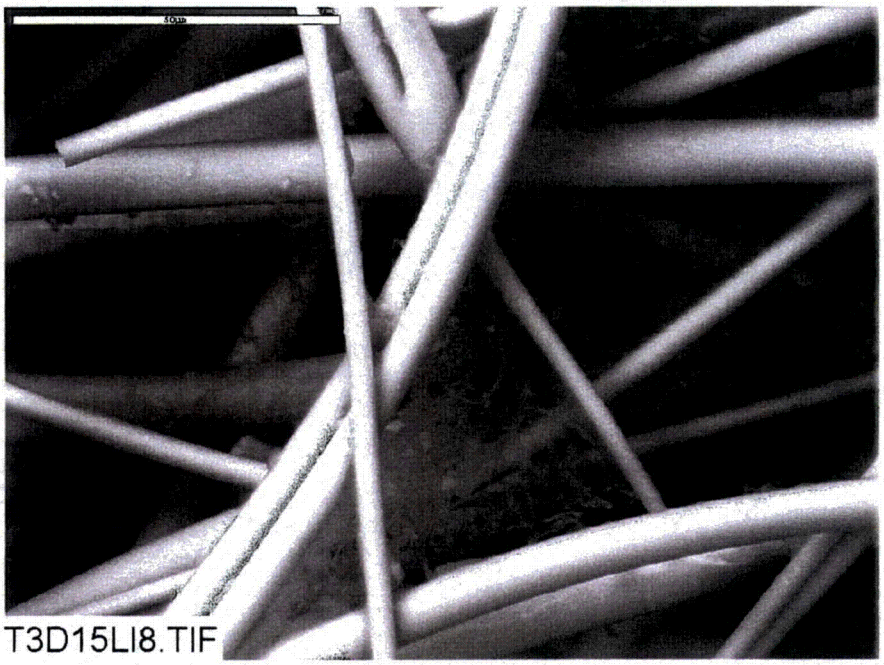



**Figure B2-3.** **Environmental SEM image magnified 1000 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LI8)**

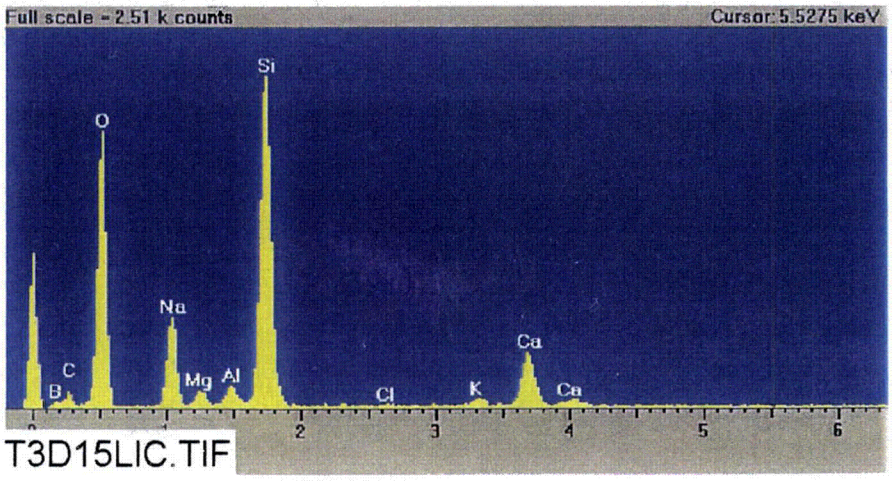



**Figure B2-4.** **EDS counting spectrum for the flocculence deposits between the fibers on ESEM image shown in Figure B2-3. (T3D15LIC)**

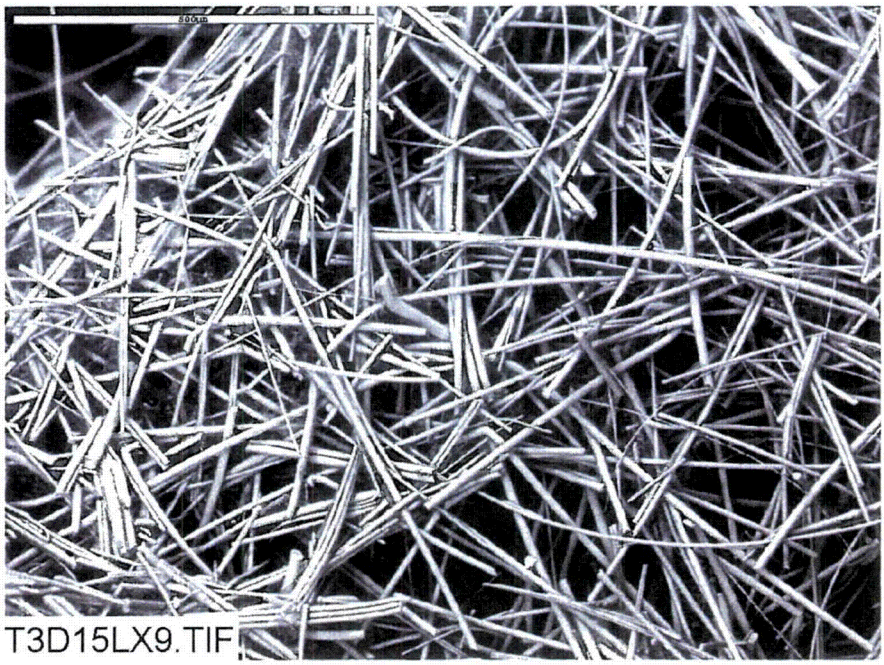



**Figure B2-5.** **Environmental SEM image magnified 110 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LX9)**

c

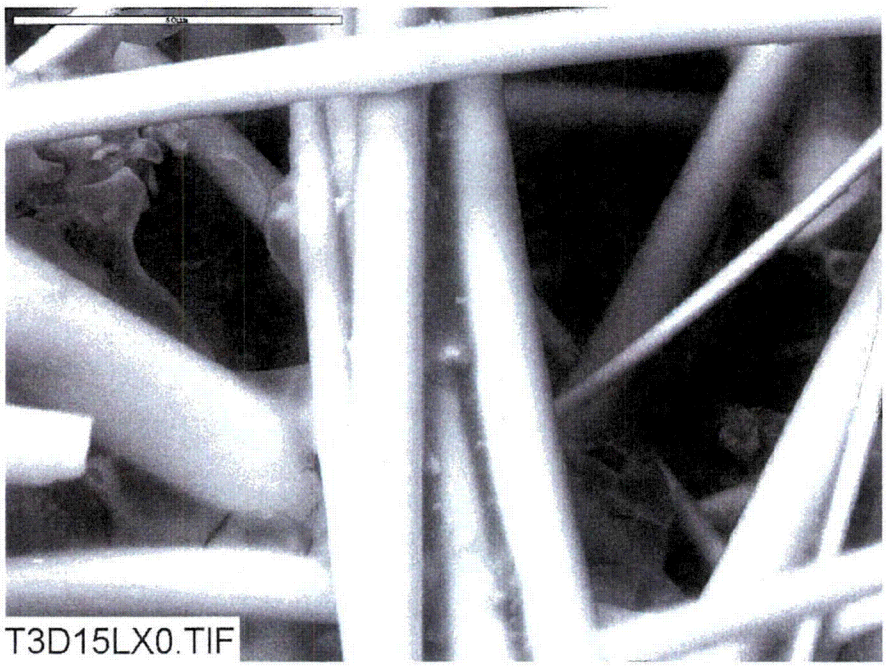



**Figure B2-6.** **Environmental SEM image magnified 1000 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LX0)**

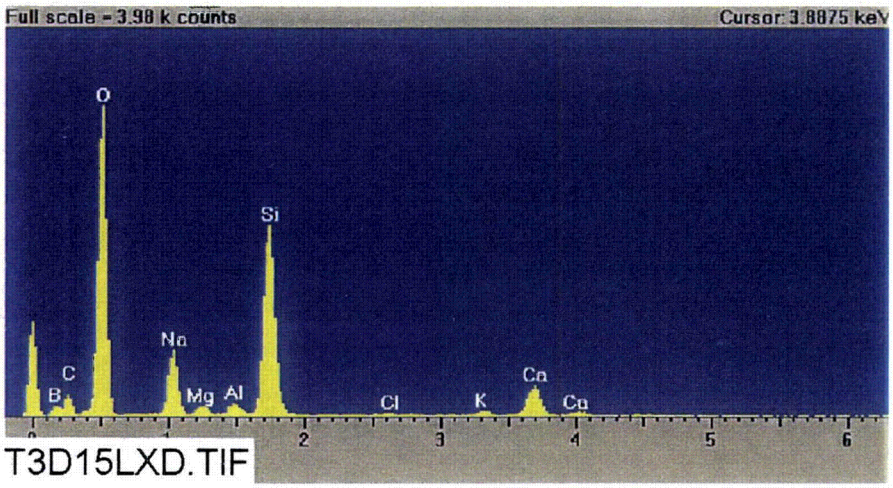



**Figure B2-7.** **EDS counting spectrum for the deposits between the fibers on ESEM image shown in Figure B2-6. (T3D15LXD)**


**Figure B2-8.** **SEM image magnified 100 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LowFlowInt008)**

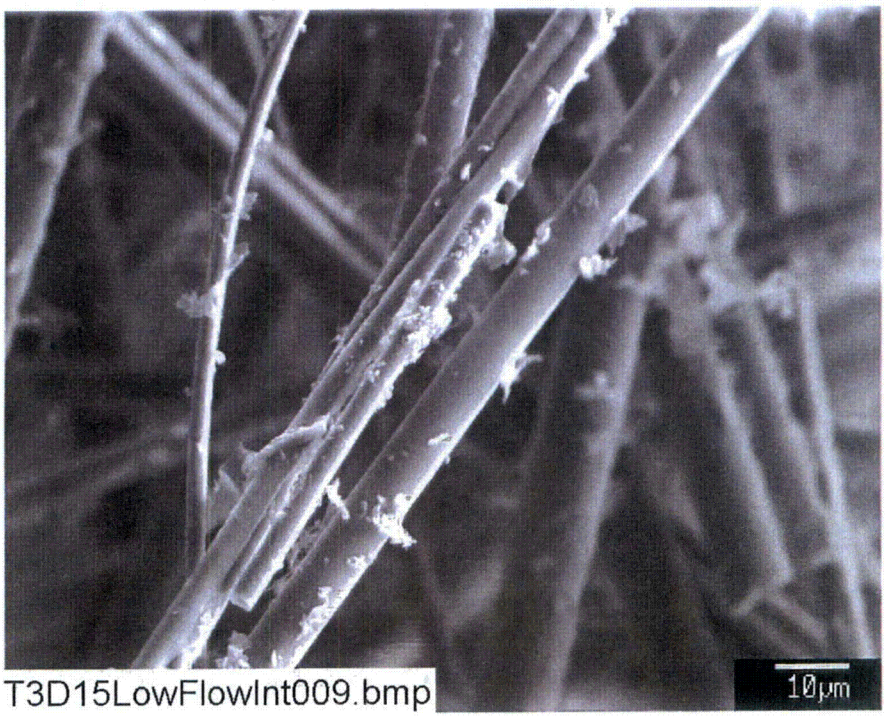



**Figure B2-9.** **SEM image magnified 1000 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LowFlowInt009)**

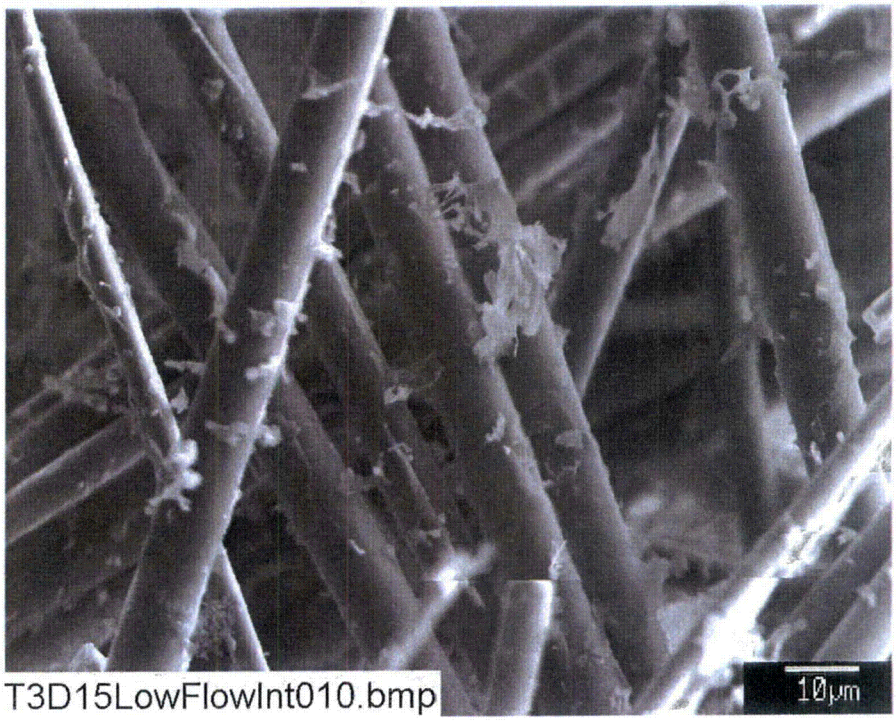



**Figure B2-10.** **SEM image magnified 1000 times for a Test #3, Day-15 low-flow interior fiberglass sample. (T3D15LowFlowInt010)**

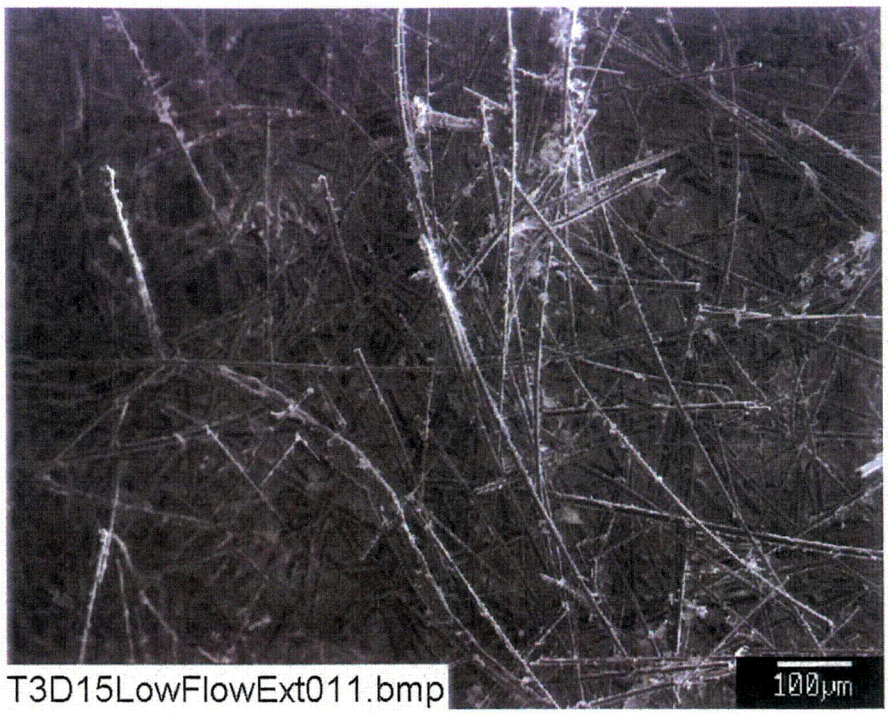



**Figure B2-11.** **SEM image magnified 100 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LowFlowExt011)**

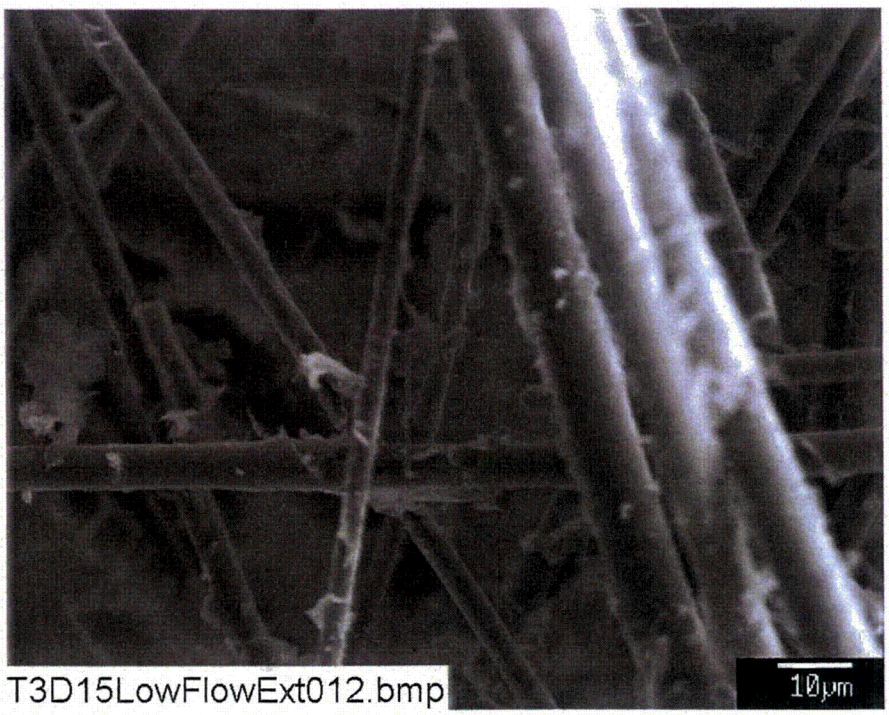



**Figure B2-12.** **SEM image magnified 1000 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LowFlowExt012)**


**Figure B2-13.** **SEM image magnified 1000 times for a Test #3, Day-15 low-flow exterior fiberglass sample. (T3D15LowFlowExt013)**

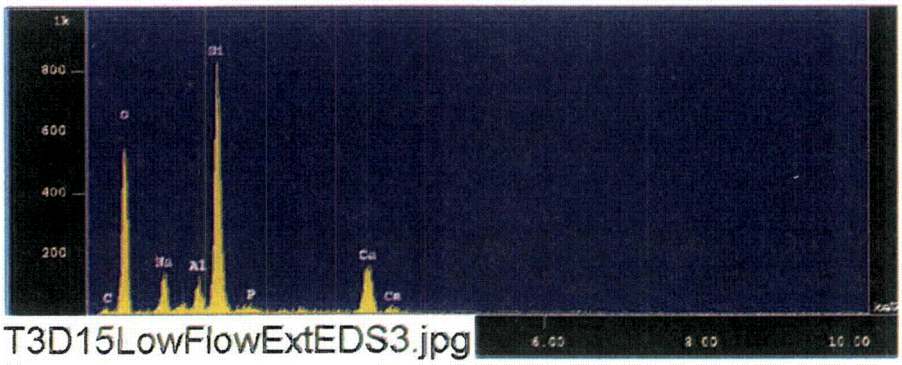



**Figure B2-14.** **EDS counting spectrum for the flocculence deposits between the fibers on SEM image shown in Figure B2-13. (T3D15LowFlowExtEDS3)**

# Appendix C1

# ESEM/EDS Data for Test #3, Day-30 Fiberglass in High-Flow Zones

## Figures


Figure C1-1. ESEM image magnified 100 times for a Test #3, Day-30 exterior high-flow fiberglass sample. (T3HiFX31) ..... C1-4

Figure C1-2. ESEM image magnified 1000 times for a Test #3, Day-30 exterior high-flow fiberglass sample. (t3hifx32) ..... C1-4

Figure C1-3. ESEM image magnified 100 times for a Test #3, Day-30 exterior high-flow fiberglass sample. (t3hifx33) ..... C1-5

Figure C1-4. EDS counting spectrum for the large masses of particulate deposits shown in Figure C1-3. (t3hifx34) ..... C1-5

Figure C1-5. ESEM image magnified 600 times for a Test #3, Day-30 exterior high-flow fiberglass sample. (t3hifx35) ..... C1-6

Figure C1-6. ESEM image magnified 100 times for a Test #3, Day-30 interior high-flow fiberglass sample. (T3HiFI36) ..... C1-6

Figure C1-7. ESEM image magnified 1000 times for a Test #3, Day-30 interior high-flow fiberglass sample. (t3hifi37) ..... C1-7

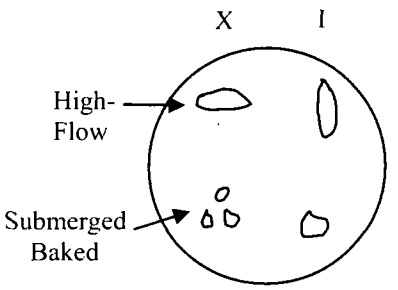
Figure C1-8. ESEM image magnified 100 times for a Test #3, Day-30 interior high-flow fiberglass sample. (t3hifi38) ..... C1-7

For ICET tests, the debris accumulated on the fiberglass is of great concern because it may cause significant head loss for the recirculation of the coolant during LOCAs. To evaluate these potential debris accumulations, fiberglass samples submerged in high-flow zones in the test tank were examined by ESEM/EDS.

The fiberglass samples examined in this appendix were extracted on the shutdown date of Test #3 (May 5, 2005). Both exterior and interior locations of the fiberglass samples were examined. Environmental SEM (ESEM) was employed to analyze the wet fiberglass samples under low-vacuum conditions (i.e., 80 Pa) and without any carbon coating, to minimize the possible modification of the fiberglass through the drying process. EDS results provide a semi-quantitative elemental analysis of the debris attached on fiberglass. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

## Transcribed Laboratory Log

Laboratory session from May 11, 2005.
Test #3, Day-30 High-Flow Fiberglass.




### High-Flow Exterior

| | | | | |
|---|---|---|---|---|
| Image: | T3HiFX31 | 100 × | ESEM image | Figure C1-1 |
| | t3hifX32 | 1000 × | ESEM image higher magnification | Figure C1-2 |
| | t3hifX33 | 100 × | ESEM image | Figure C1-3 |
| EDS: | t3hifX34 | | EDS on particles in T3HiFX33 | Figure C1-4 |
| Image: | t3hifX35 | 600 × | ESEM image on fiberglass | Figure C1-5 |

### High-Flow Interior

| | | | | |
|---|---|---|---|---|
| Image: | T3HiFI36 | 100 × | ESEM image of fiberglass | Figure C1-6 |
| | t3hifI37 | 1000 × | ESEM image higher magnification | Figure C1-7 |
| | t3hifI38 | 100 × | ESEM image | Figure C1-8 |


**Figure C1-1.** **ESEM image magnified 100 times for a Test #3, Day-30 exterior high-flow fiberglass sample. (T3HiFX31)**

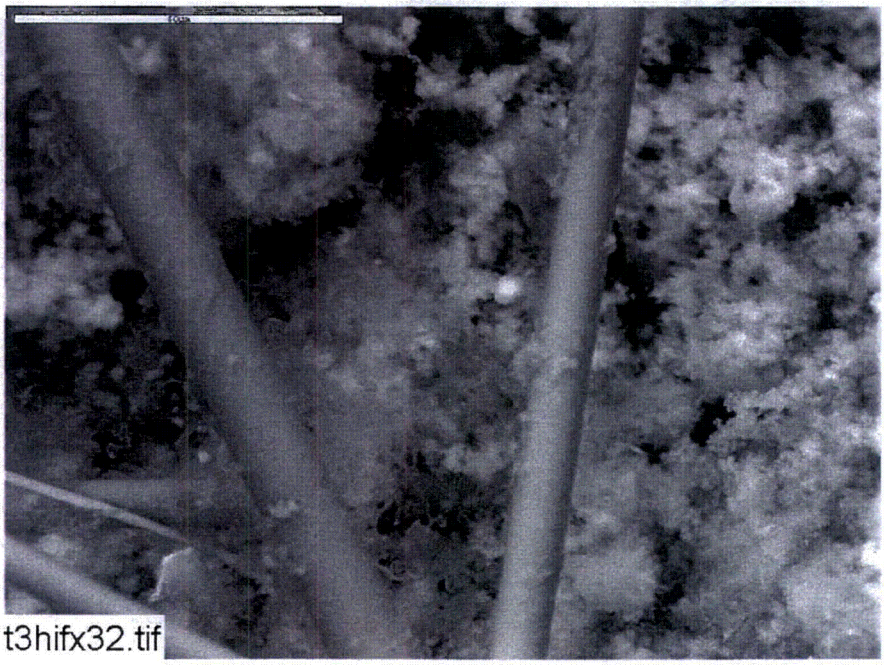



**Figure C1-2.** **ESEM image magnified 1000 times for a Test #3, Day-30 exterior high-flow fiberglass sample. (t3hifx32)**

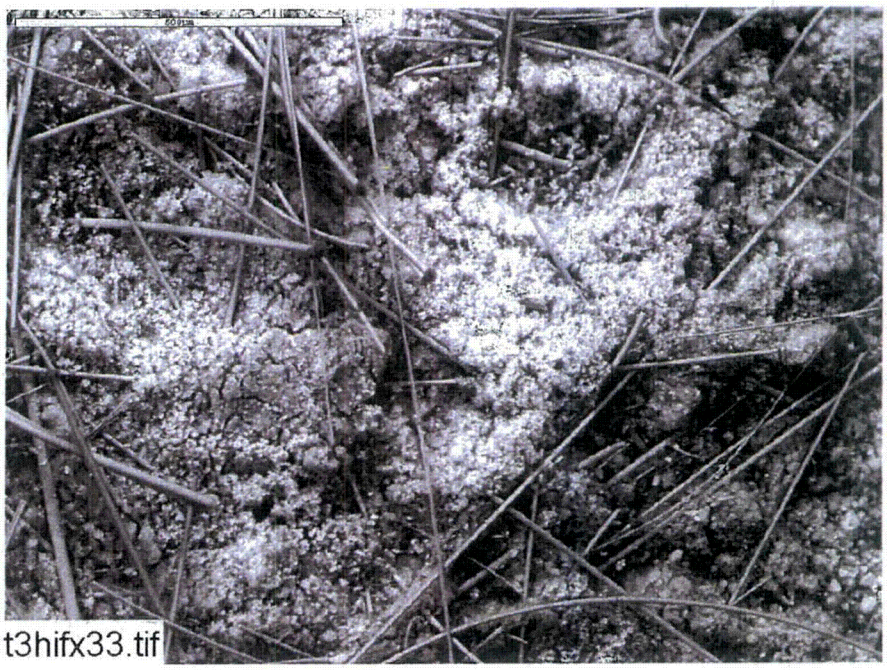



**Figure C1-3.** **ESEM image magnified 100 times for a Test #3, Day-30 exterior high-flow fiberglass sample. (t3hifx33)**

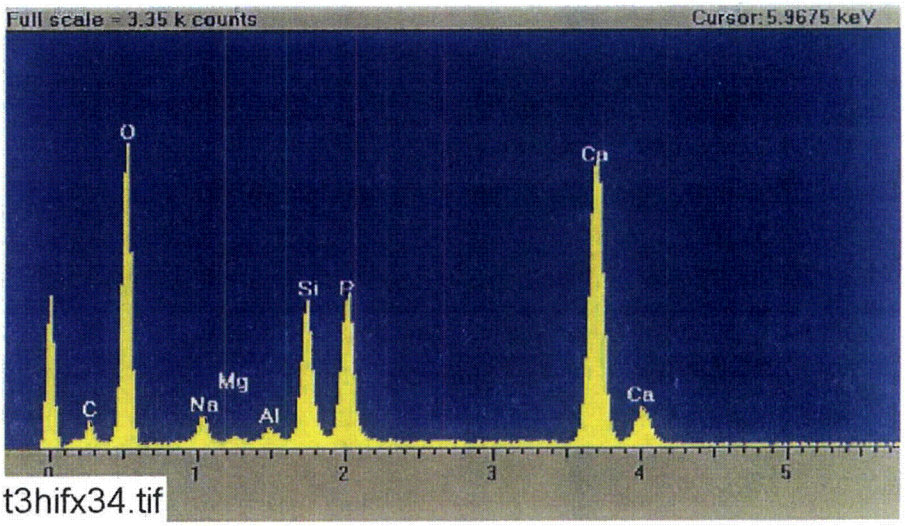



**Figure C1-4.** **EDS counting spectrum for the large masses of particulate deposits shown in Figure C1-3. (t3hifx34)**

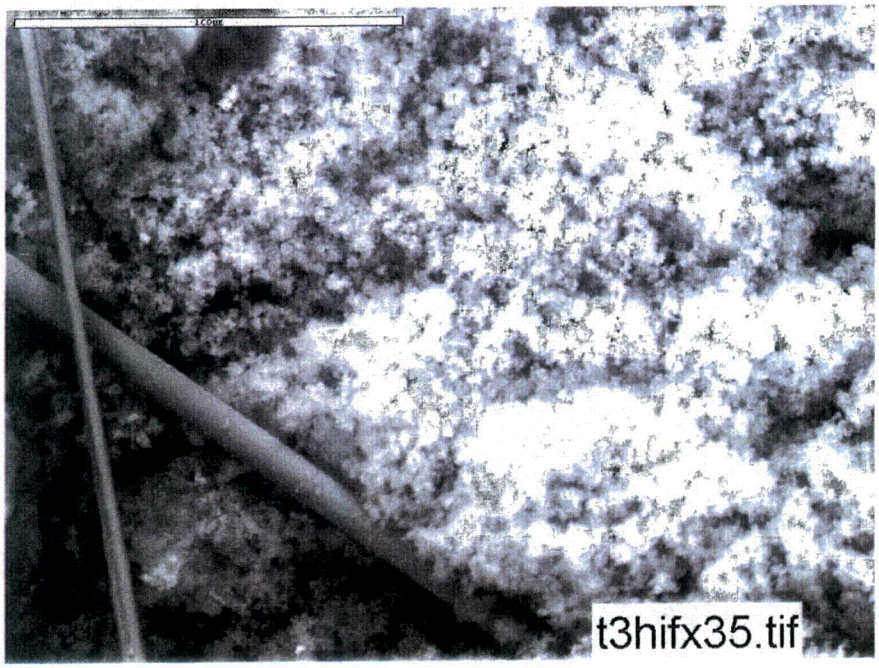



**Figure C1-5.** **ESEM image magnified 600 times for a Test #3, Day-30 exterior high-flow fiberglass sample. (t3hifx35)**

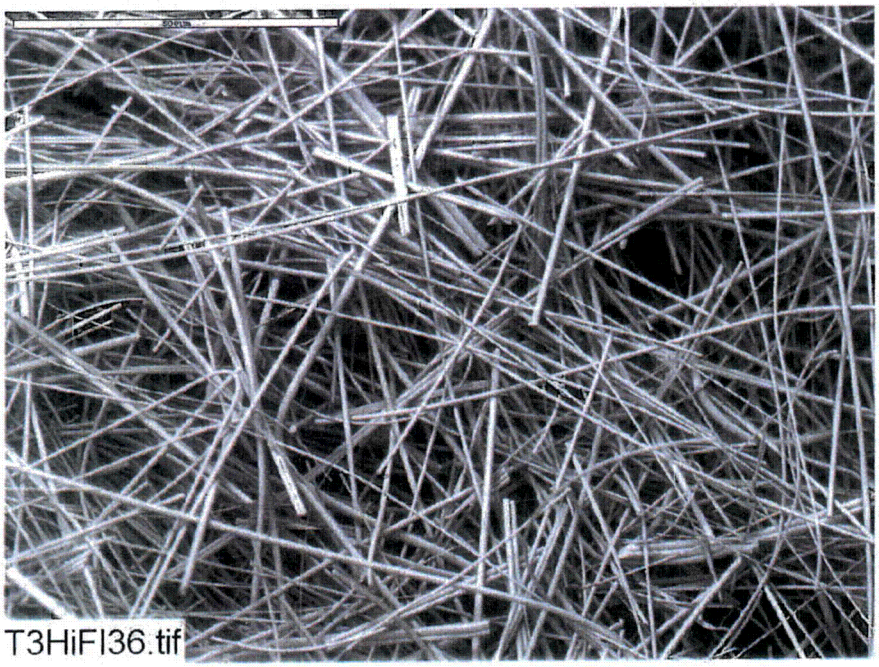



**Figure C1-6.** **ESEM image magnified 100 times for a Test #3, Day-30 interior high-flow fiberglass sample. (T3HiFI36)**

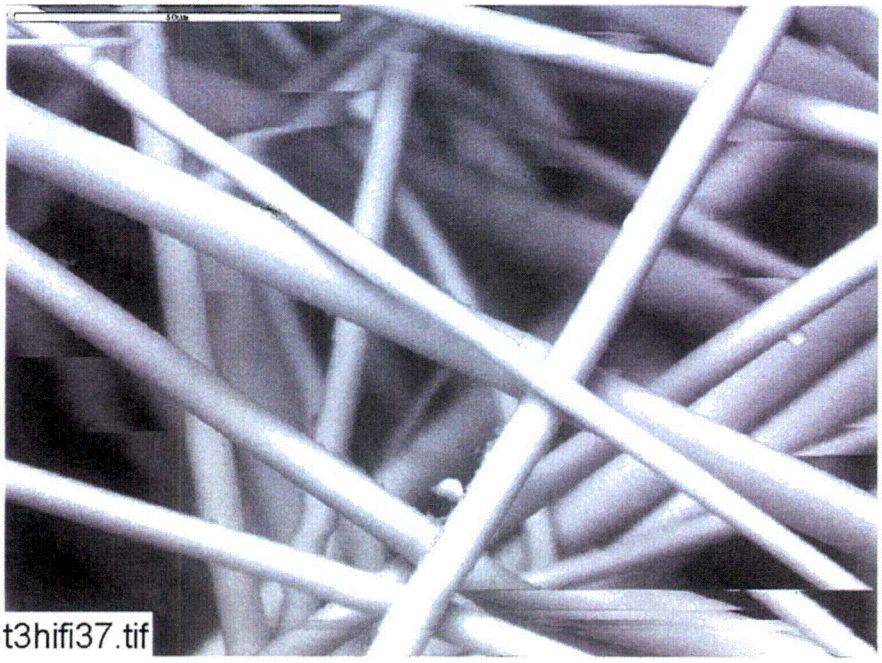



**Figure C1-7.** **ESEM image magnified 1000 times for a Test #3, Day-30 interior high-flow fiberglass sample. (t3hifi37)**


**Figure C1-8.** **ESEM image magnified 100 times for a Test #3, Day-30 interior high-flow fiberglass sample. (t3hifi38)**

# Appendix C2

# ESEM Data for Test #3, Day-30 Fiberglass in Low-Flow Zones

## Figures


Figure C2-1. ESEM image magnified 70 times for a Test #3, Day-30 exterior low-flow fiberglass sample. (t3lfex09) ..... C2-4

Figure C2-2. ESEM image magnified 1000 times for a Test #3, Day-30 exterior low-flow fiberglass sample. (t3lfex10) ..... C2-4

Figure C2-3. ESEM image magnified 1000 times for a Test #3, Day-30 exterior low-flow fiberglass sample. (t3lfex11) ..... C2-5

Figure C2-4. ESEM image magnified 70 times for a Test #3, Day-30 interior low-flow fiberglass sample. (t3lfin12) ..... C2-5

Figure C2-5. ESEM image magnified 1000 times for a Test #3, Day-30 interior low-flow fiberglass sample. (t3lfin13) ..... C2-6

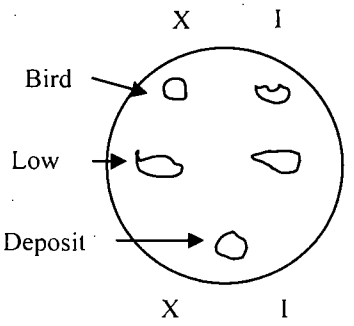
Figure C2-6. ESEM image magnified 1000 times for a Test #3, Day-30 interior low-flow fiberglass sample. (t3lfin14) ..... C2-6

Figure C2-7. ESEM image magnified 1000 times for a Test #3, Day-30 interior low-flow fiberglass sample. (t3lfin15) ..... C2-7

This appendix presents the ESEM results on fiberglass samples submerged in the low-flow zone in the tank. The fiberglass samples were extracted on the date Test #3 was shut down (May 5, 2005). Both exterior and interior locations of the fiberglass samples were examined. ESEM was employed to analyze the uncoated, wet fiberglass samples. Additionally, the ESEM was performed under a low vacuum condition (i.e., 80 Pa) to minimize the modification of the fiberglass that could occur through the drying process. 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 Birdcage Fiberglass.




### Low-Flow Exterior

| | | | | |
|---|---|---|---|---|
| Image: | t3lfex09 | 70 × | ESEM image of fiberglass sample | Figure C2-1 |
| | t3lfex10 | 1000 × | ESEM at higher magnification | Figure C2-2 |
| | t3lfex11 | 1000 × | ESEM image | Figure C2-3 |

### Low-Flow Interior

| | | | | |
|---|---|---|---|---|
| Image: | t3lfin12 | 70 × | ESEM image of fiberglass sample | Figure C2-4 |
| | t3lfin13 | 1000 × | ESEM image at higher mag. | Figure C2-5 |
| | t3lfin14 | 1000 × | ESEM image | Figure C2-6 |
| | t3lfin15 | 1000 × | ESEM image | Figure C2-7 |

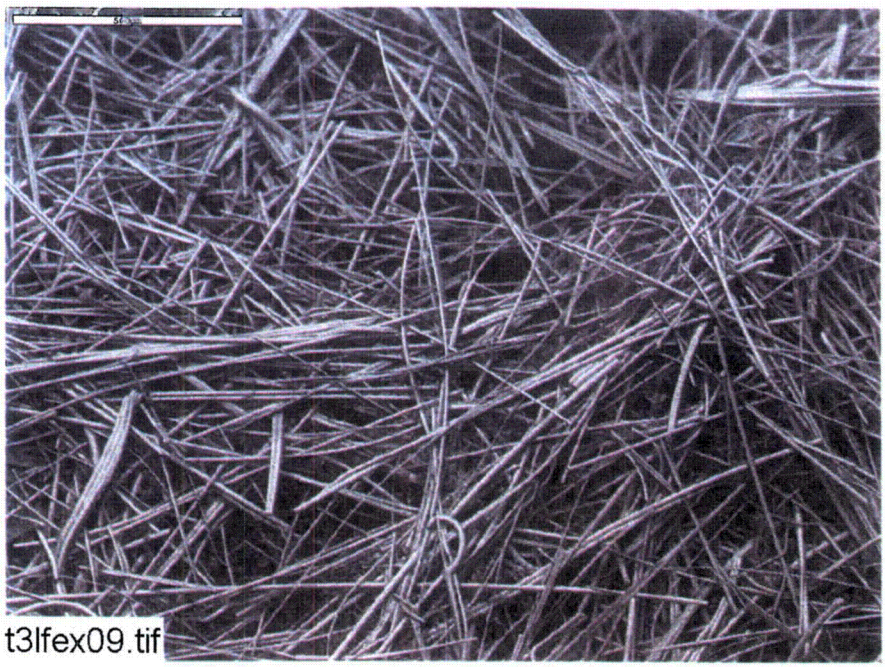



**Figure C2-1.** **ESEM image magnified 70 times for a Test #3, Day-30 exterior low-flow fiberglass sample. (t3lfex09)**

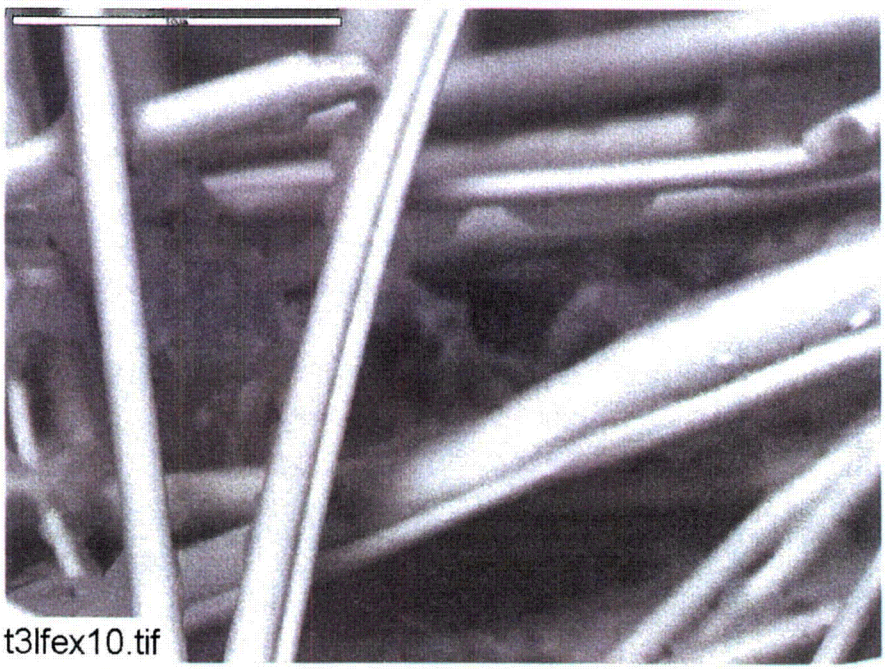



**Figure C2-2.** **ESEM image magnified 1000 times for a Test #3, Day-30 exterior low-flow fiberglass sample. (t3lfex10)**

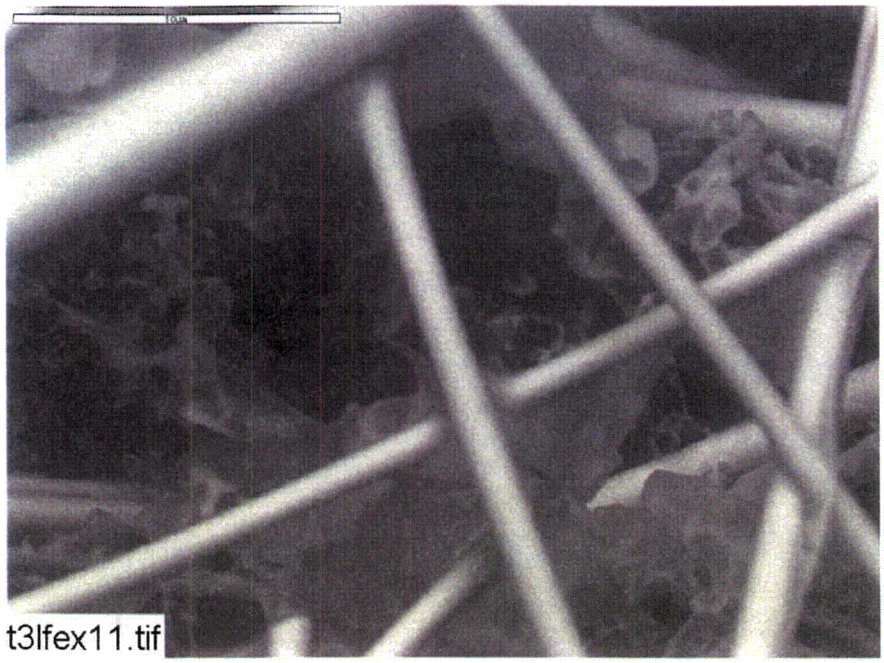



**Figure C2-3.** **ESEM image magnified 1000 times for a Test #3, Day-30 exterior low-flow fiberglass sample. (t3lfex11)**

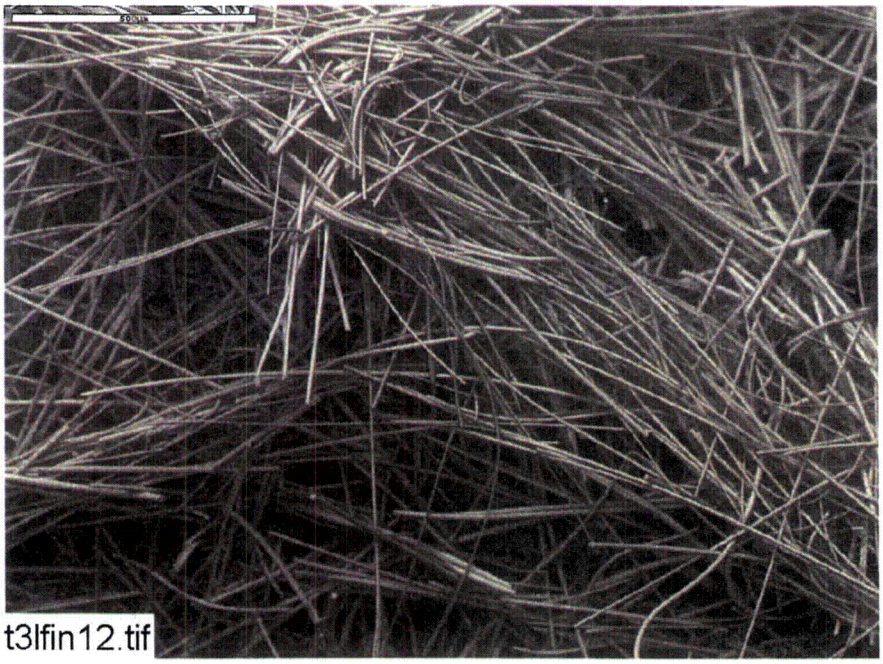



**Figure C2-4.** **ESEM image magnified 70 times for a Test #3, Day-30 interior low-flow fiberglass sample. (t3lfin12)**

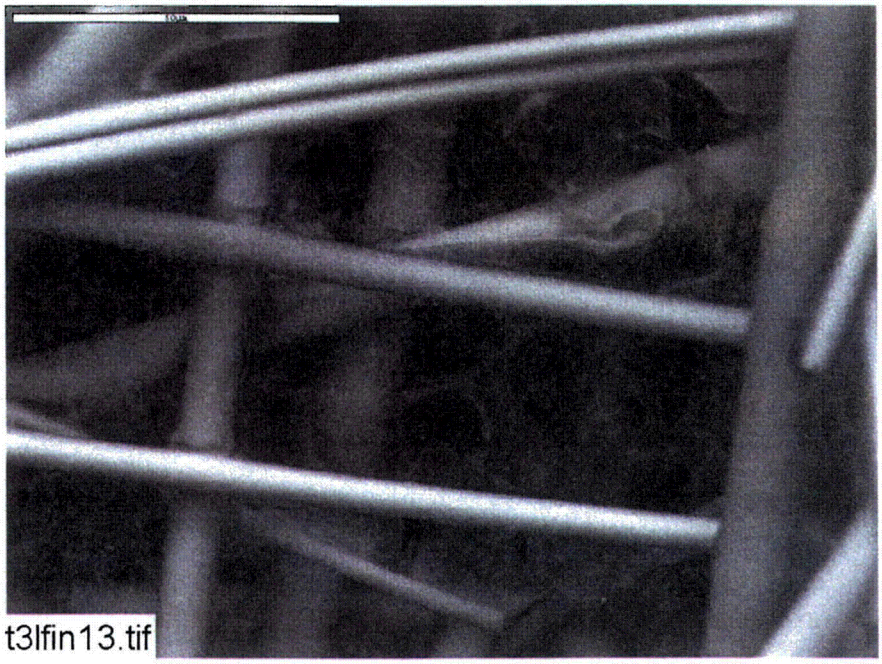



**Figure C2-5.** **ESEM image magnified 1000 times for a Test #3, Day-30 interior low-flow fiberglass sample. (t3lfin13)**

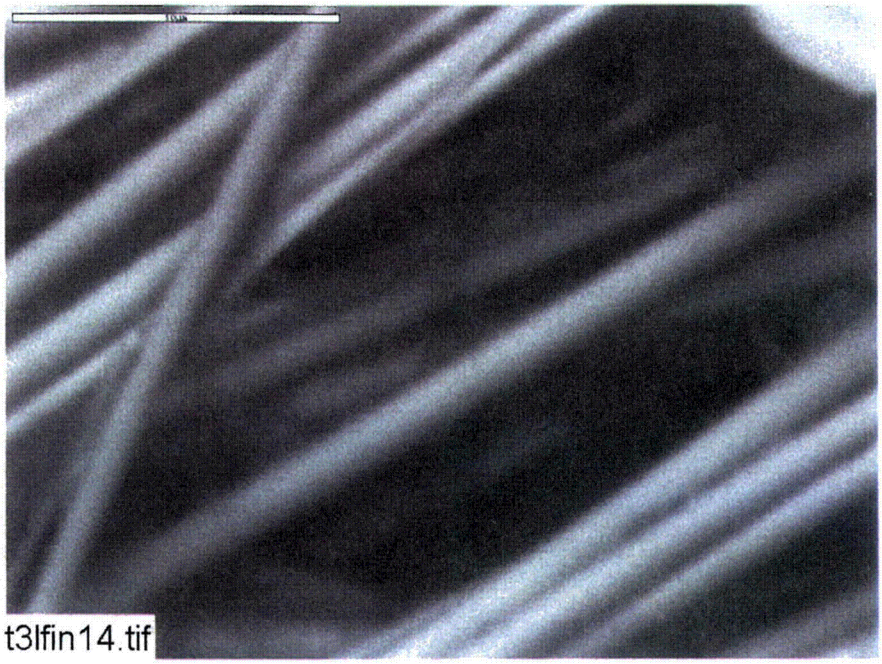



**Figure C2-6.** **ESEM image magnified 1000 times for a Test #3, Day-30 interior low-flow fiberglass sample. (t3lfin14)**

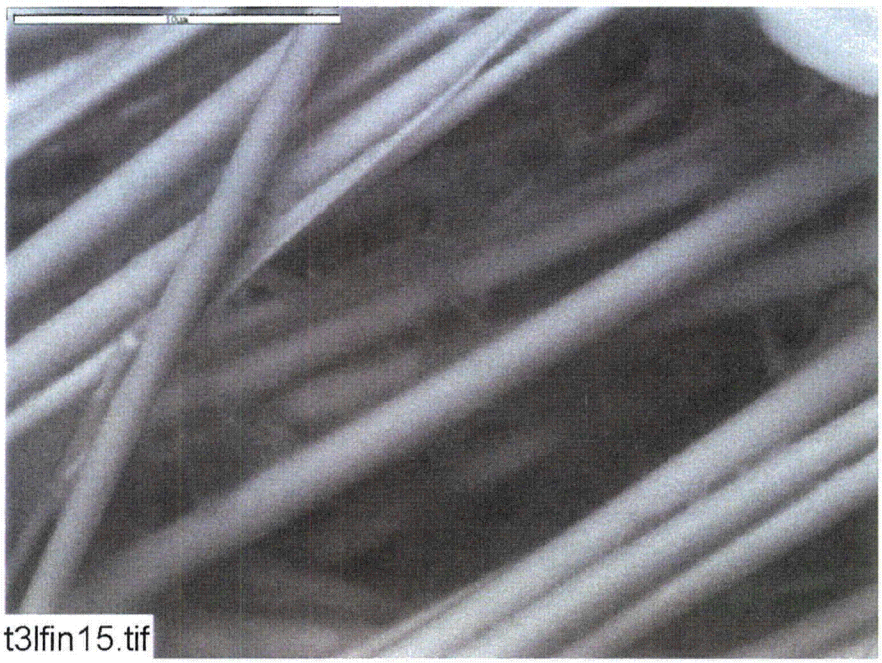



**Figure C2-7.** **ESEM image magnified 1000 times for a Test #3, Day-30 interior low-flow fiberglass sample. (t3lfin15)**

# Appendix C3

# ESEM/EDS Data for Test #3, Day-30 Drain Collar Fiberglass

## Figures


Figure C3-1. ESEM image magnified 100 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen). (t3DCSC16) ..... C3-4

Figure C3-2. ESEM image magnified 1000 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen). (t3dcsc17) ..... C3-4

Figure C3-3. EDS counting spectrum for the particulate deposits shown in Figure C3-2. (t3dcsc18) ..... C3-5

Figure C3-4. ESEM image magnified 70 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen). (t3dcsc19) ..... C3-5

Figure C3-5. ESEM image magnified 70 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen). (t3dcEx20) ..... C3-6

Figure C3-6. ESEM image magnified 1000 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen). (t3dcex21) ..... C3-6

Figure C3-7. EDS counting spectrum for the light particulate deposits (EDS1) shown in Figure C3-6. (t3dcex22) ..... C3-7

Figure C3-8. EDS counting spectrum for the dark deposits (EDS2) shown in Figure C3-6. (t3dcex23) ..... C3-7

Figure C3-9. Comparison of EDS counting spectra between Figure C3-7 (yellow) and Figure C3-8 (red). (t3dcex24) ..... C3-8

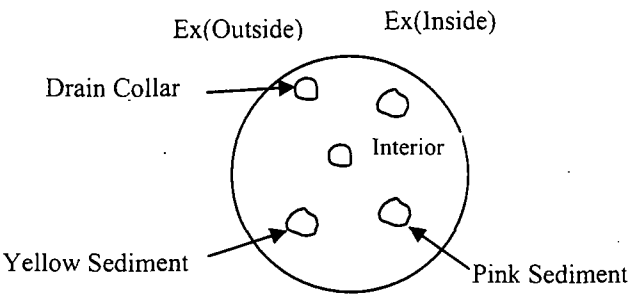
Figure C3-10. ESEM image magnified 100 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen). (t3dcex25) ..... C3-8
Figure C3-11. ESEM image magnified 80 times for a Test #3, Day-30 interior fiberglass sample on the drain collar. (t3dcin26) ..... C3-9
Figure C3-12. ESEM image magnified 1000 times for a Test #3, Day-30 interior fiberglass sample on the drain collar. (t3dcin27) ..... C3-9
Figure C3-13. ESEM image magnified 100 times for a Test #3, Day-30 interior fiberglass sample on the drain collar. (t3dcin28) ..... C3-10

As mentioned before, the debris accumulated on fiberglass during ICET tests is of great concern because such debris may cause significant head loss for the recirculation of coolant during a LOCA. The ICET tank drain collar was used to simulate the sump screen used in nuclear reactor containment systems. Therefore, it is very important to perform SEM/EDS examinations of the fiberglass samples within the drain collar submerged in the tank.

In this appendix, the fiberglass samples within the drain collar were extracted on the date that Test #3 was shut down (May 5, 2005). The fiberglass samples located at the outside exterior (away from the drain screen), the inside exterior (next to the drain screen), and the interior were examined. ESEM was employed to analyze the wet fiberglass samples; it was performed without any coating on the samples and under low-vacuum conditions (i.e., 80 Pa). This evaluation technique minimizes the modification of the fiberglass that could occur through a drying process. EDS results provide an elemental composition analysis of the debris attached on the fiberglass. 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 Drain Collar Fiberglass.




### Drain Collar Inside

| | | | | |
|---|---|---|---|---|
| Image: | t3DCSC16 | 100 × | | Figure C3-1 |
| | t3dcsc17 | 1000 × | Particles | Figure C3-2 |
| EDS: | t3dcsc18 | | EDS on particles | Figure C3-3 |
| Image: | t3dcsc19 | 70 × | | Figure C3-4 |

### Drain Collar Outside

| | | | | |
|---|---|---|---|---|
| Image: | t3dcEx20 | 70 × | | Figure C3-5 |
| | t3dcex21 | 1000 × | | Figure C3-6 |
| EDS: | t3dcex22 | | EDS on white bright particles shown in 21 | Figure C3-7 |
| | t3dcex23 | | EDS on dark particles shown in 21 | Figure C3-8 |
| | t3dcex24 | | Comparing t3dcex22 & t3dcex23 | Figure C3-9 |
| Image: | t3ccex25 | 100 × | | Figure C3-10 |

### Drain Collar Interior

| | | | |
|---|---|---|---|
| Image: | t3dcin26 | 80 × | Figure C3-11 |
| | t3dcin27 | 1000 × | Figure C3-12 |
| | t3dcin28 | 100 × | Figure C3-13 |


**Figure C3-1.** **ESEM image magnified 100 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen). (t3DCSC16)**

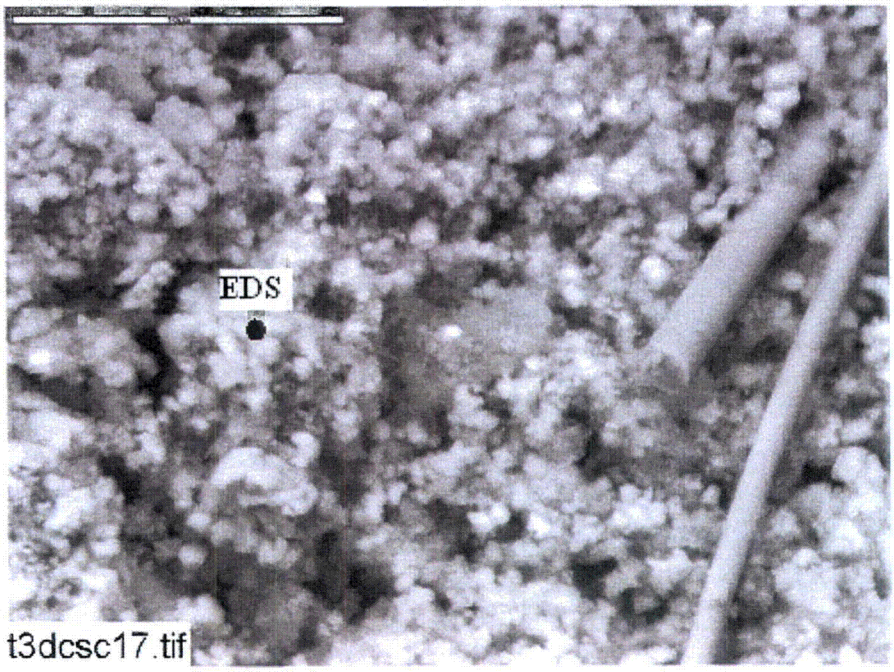



**Figure C3-2.** **ESEM image magnified 1000 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen). (t3dcsc17)**

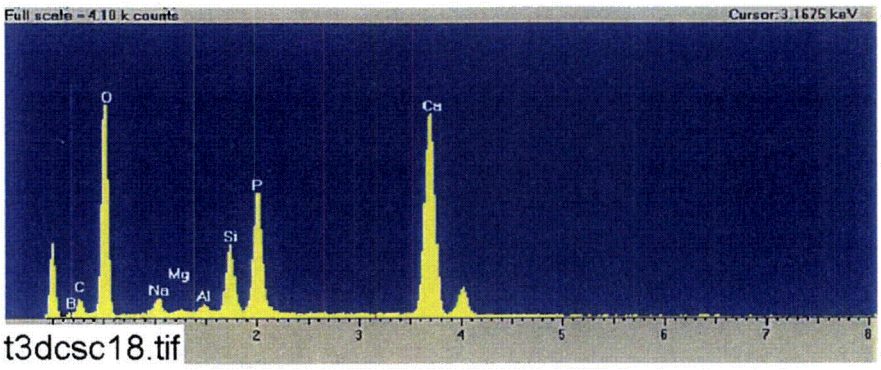



**Figure C3-3.** **EDS counting spectrum for the particulate deposits shown in Figure C3-2. (t3dcsc18)**

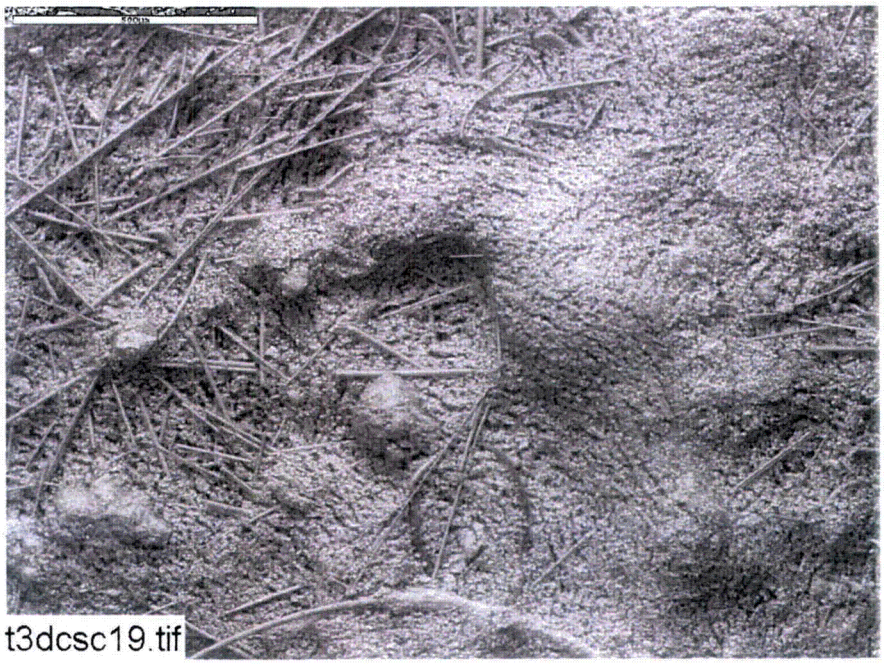



**Figure C3-4.** **ESEM image magnified 70 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen). (t3dcsc19)**

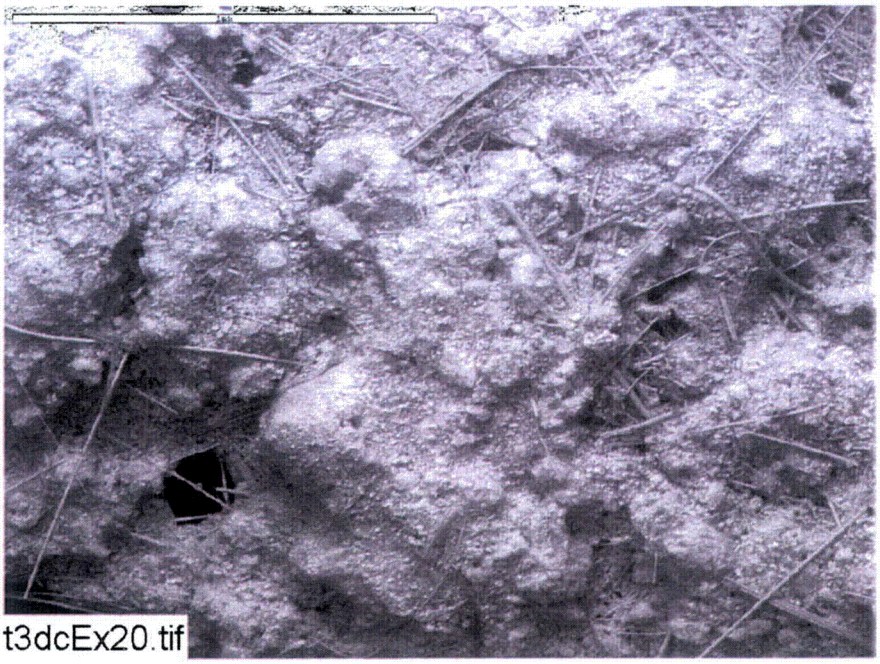



**Figure C3-5.** **ESEM image magnified 70 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen). (t3dcEx20)**

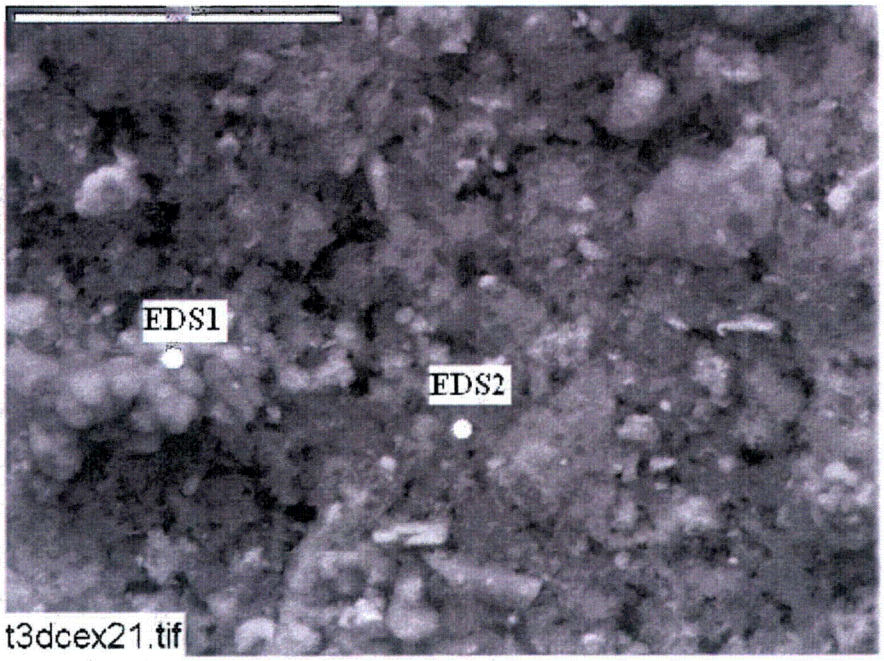



**Figure C3-6.** **ESEM image magnified 1000 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen). (t3dcex21)**

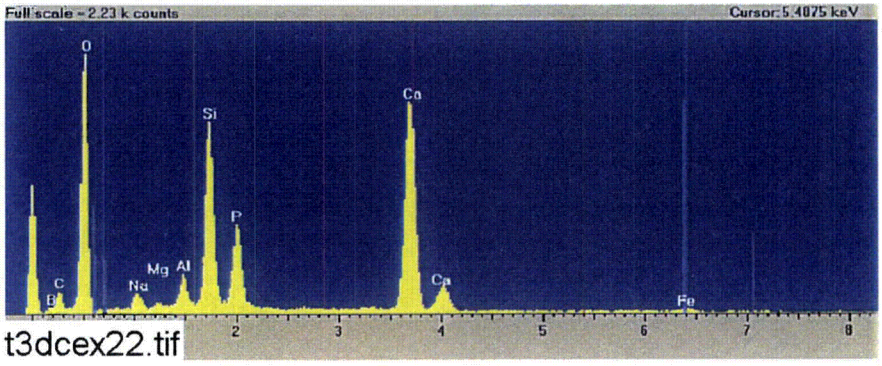



**Figure C3-7.** **EDS counting spectrum for the light particulate deposits (EDS1) shown in Figure C3-6. (t3dcex22)**

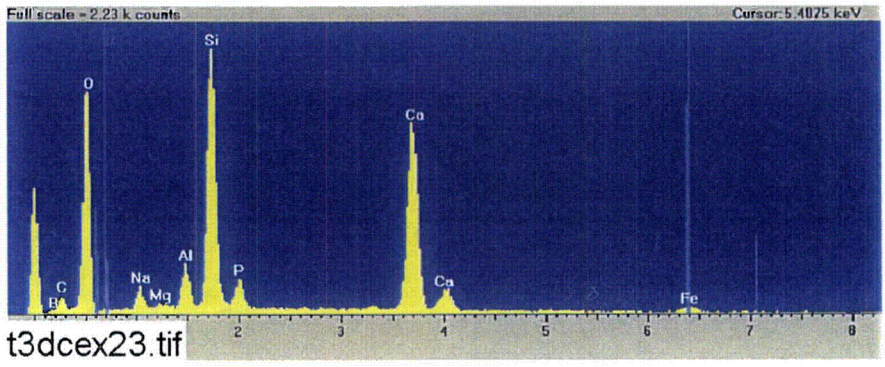



**Figure C3-8.** **EDS counting spectrum for the dark deposits (EDS2) shown in Figure C3-6. (t3dcex23)**

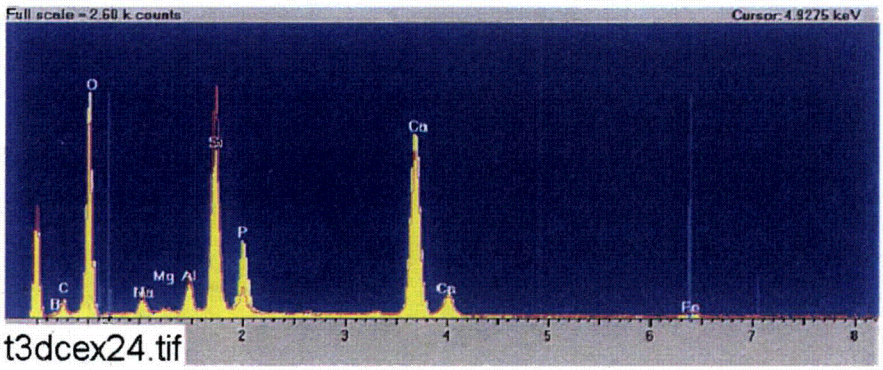



**Figure C3-9.** **Comparison of EDS counting spectra between Figure C3-7 (yellow) and Figure C3-8 (red). (t3dcex24)**

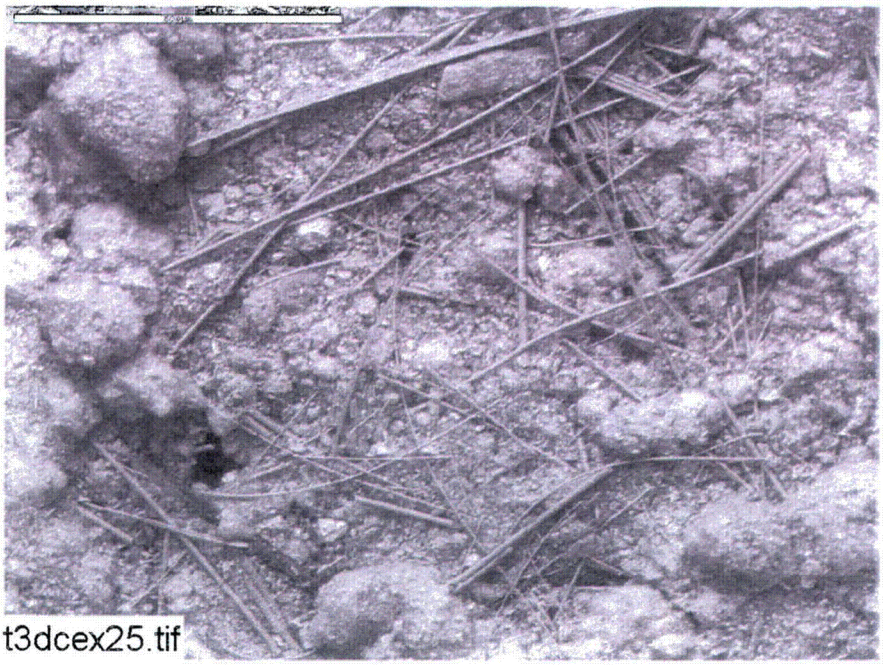



**Figure C3-10.** **ESEM image magnified 100 times for a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen). (t3dcex25)**

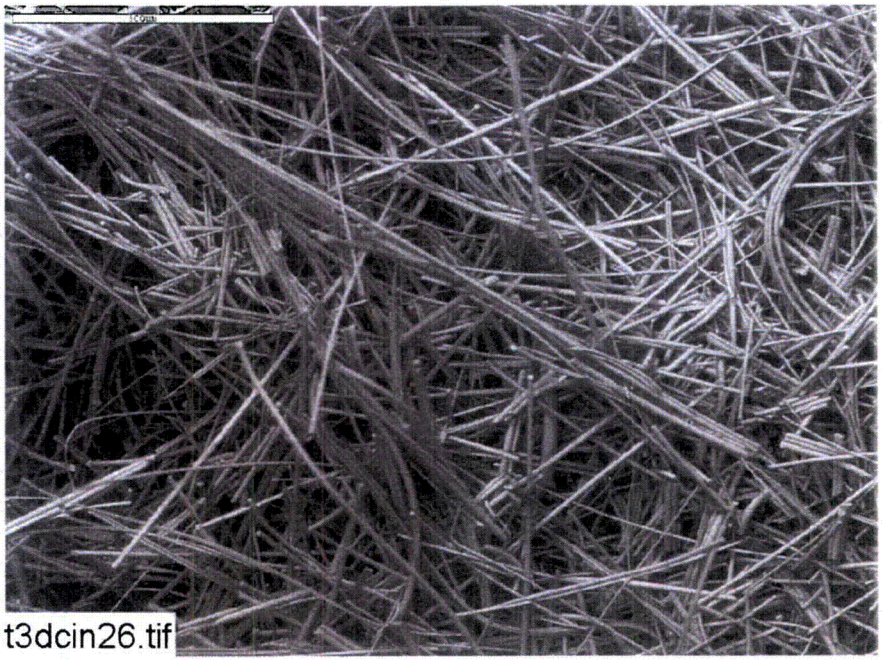



**Figure C3-11. ESEM image magnified 80 times for a Test #3, Day-30 interior fiberglass sample on the drain collar. (t3dcin26)**

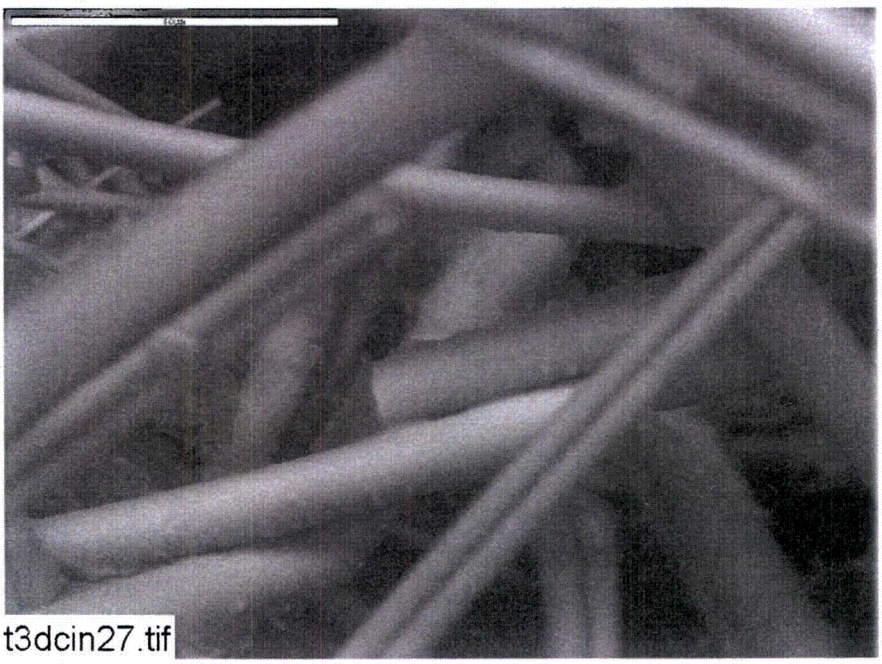



**Figure C3-12. ESEM image magnified 1000 times for a Test #3, Day-30 interior fiberglass sample on the drain collar. (t3dcin27)**

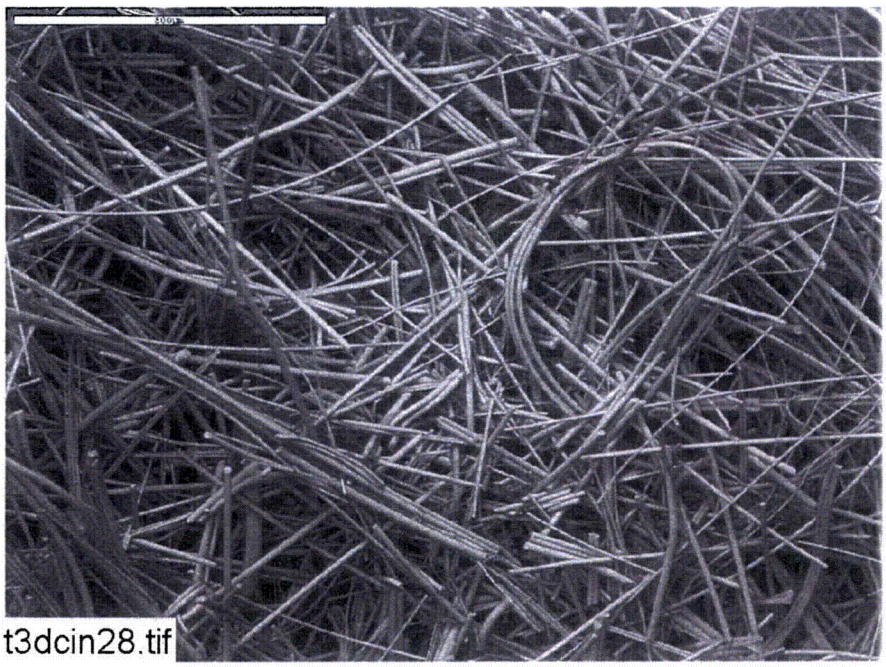



**Figure C3-13. ESEM image magnified 100 times for a Test #3, Day-30 interior fiberglass sample on the drain collar. (t3dcin28)**

# Appendix C4

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

## Figures


Figure C4-1. SEM image magnified 100 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (T3D30BirdExt001) ..... C4-6

Figure C4-2. SEM image magnified 550 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (T3D30BirdExt002) ..... C4-6

Figure C4-3. EDS counting spectrum for the large masses of particulate deposits shown in Figure C4-2. (T3D30BirdExtPt01) ..... C4-7

Figure C4-4. ESEM image magnified 80 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (T3BCEX01) ..... C4-7

Figure C4-5. EDS counting spectrum for the the large deposits shown in Figure C4-4. (t3bcexe2) ..... C4-8

Figure C4-6. ESEM image magnified 80 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (t3bcex02) ..... C4-8

Figure C4-7. ESEM image magnified 500 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (t3bcex03) ..... C4-9

Figure C4-8. EDS counting spectrum for the deposits shown in Figure C4-7. (T3BCExE1) ..... C4-9

Figure C4-9. Comparison of EDS counting spectra of Figure C4-5 (yellow) and Figure C4-8 (red). (t3bcexe3) ..... C4-10

Figure C4-10. ESEM image magnified 1000 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (t3bcex04) ..... C4-10

Figure C4-11. ESEM image magnified 80 times for a Test #3, Day-30 interior fiberglass sample within a birdcage. (t3bcin05) ..... C4-11

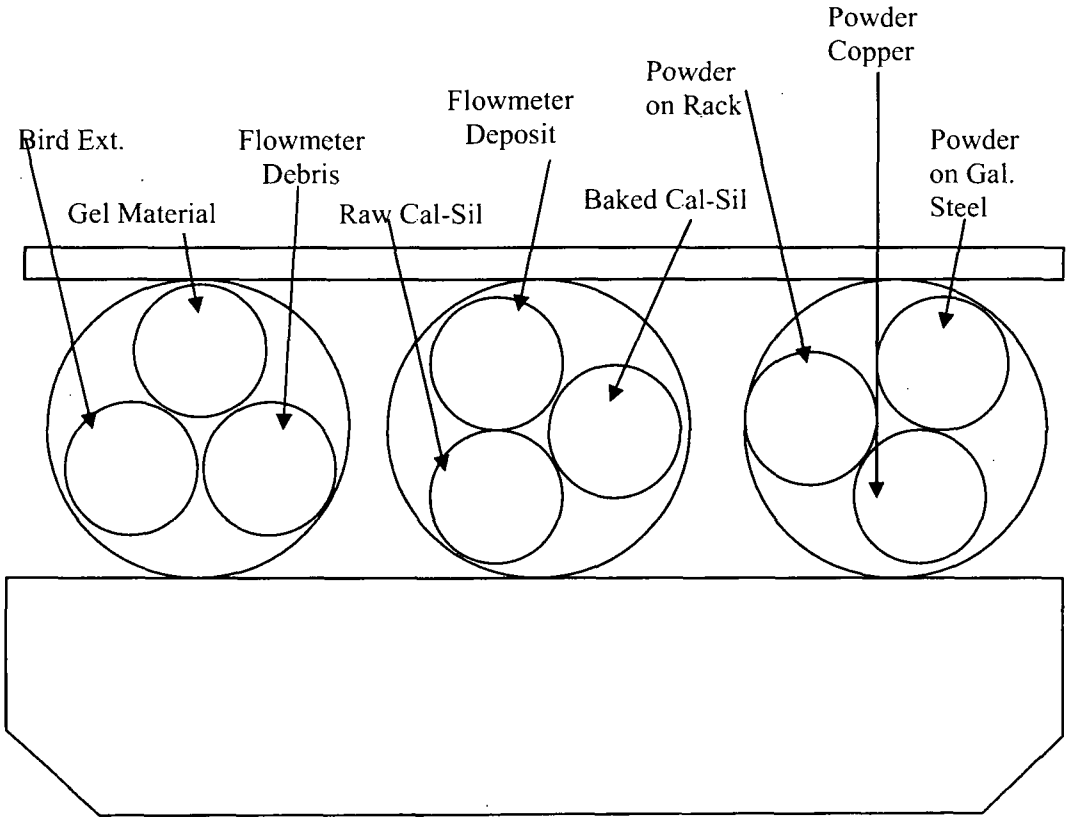
Figure C4-12. ESEM image magnified 950 times for a Test #3, Day-30 interior fiberglass sample within a birdcage. (t3bcin06) ..... C4-11

Figure C4-13. ESEM image magnified 1000 times for a Test #3, Day-30 interior fiberglass sample within a birdcage. (t3bcin07) ..... C4-12

This appendix lists the ESEM/SEM/EDS results for the fiberglass samples within a birdcage submerged in the testing solution. The purpose of this analysis was to determine the degree and the extent of particulate debris that migrated and attached on fiberglass. In this appendix, the fiberglass samples within the birdcage were extracted on the shutdown date of Test #3 (May 5, 2005). Both exterior and interior fiberglass samples were examined. Probe SEM was used to examine the fiberglass samples after drying at room temperature followed by being coated with Au/Pd. In addition to probe SEM, ESEM was employed to analyze the uncoated, wet fiberglass samples under a low-vacuum condition (i.e., 80 Pa) to minimize the potential for modification of the fiberglass samples that might occur through the drying process. 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 Birdcage



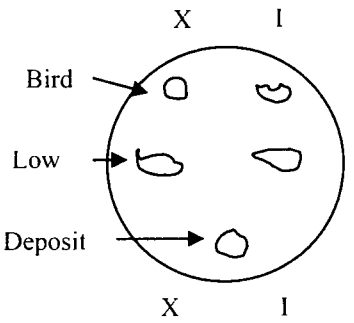

### Birdcage Exterior

| | | | | |
|---|---|---|---|---|
| Image: | T3D30BirdExt001 | 100 × | | Figure C4-1 |
| | T3D30BirdExt002 | 550 × | | Figure C4-2 |
| EDS: | T3D30BirdExtPt01 | | Particles on fiberglass, image 002 | Figure C4-3 |

This page intentionally left blank.

## Transcribed Laboratory Log

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




### Birdcage Exterior

| | | | | |
|---|---|---|---|---|
| Image: | T3BCEX01 | 80 × | | Figure C4-4 |
| EDS: | t3bcexe2 | | EDS on particles-T3BCEXE2 | Figure C4-5 |
| Image: | t3bcex02 | 80 × | | Figure C4-6 |
| | t3bcex03 | 500 × | | Figure C4-7 |
| EDS: | T3BCExE1 | | EDS on particles in t3bcex03 | Figure C4-8 |
| | t3bcexe3 | | Comparing t3bcexe2 & T3BCExE1 | Figure C4-9 |
| Image: | t3bcex04 | 1000 × | | Figure C4-10 |

### Birdcage Interior

| | | | |
|---|---|---|---|
| Image: | t3bcin05 | 80 × | Figure C4-11 |
| | t3bcin06 | 950 × | Figure C4-12 |
| | t3bcin07 | 1000 × | Figure C4-13 |


**Figure C4-1.** **SEM image magnified 100 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (T3D30BirdExt001)**


**Figure C4-2.** **SEM image magnified 550 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (T3D30BirdExt002)**

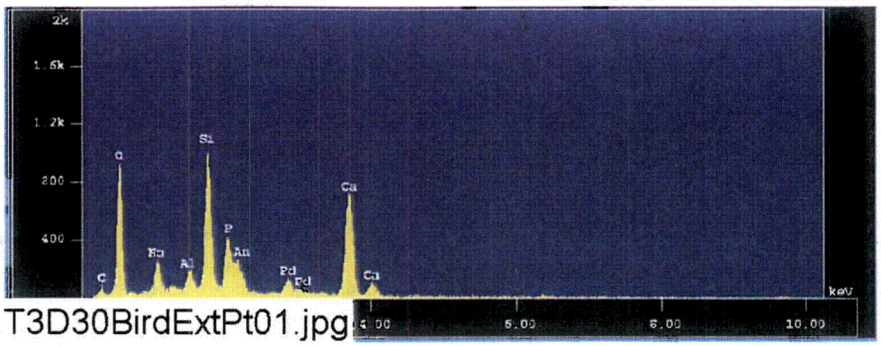



**Figure C4-3.** **EDS counting spectrum for the large masses of particulate deposits shown in Figure C4-2. (T3D30BirdExtPt01)**

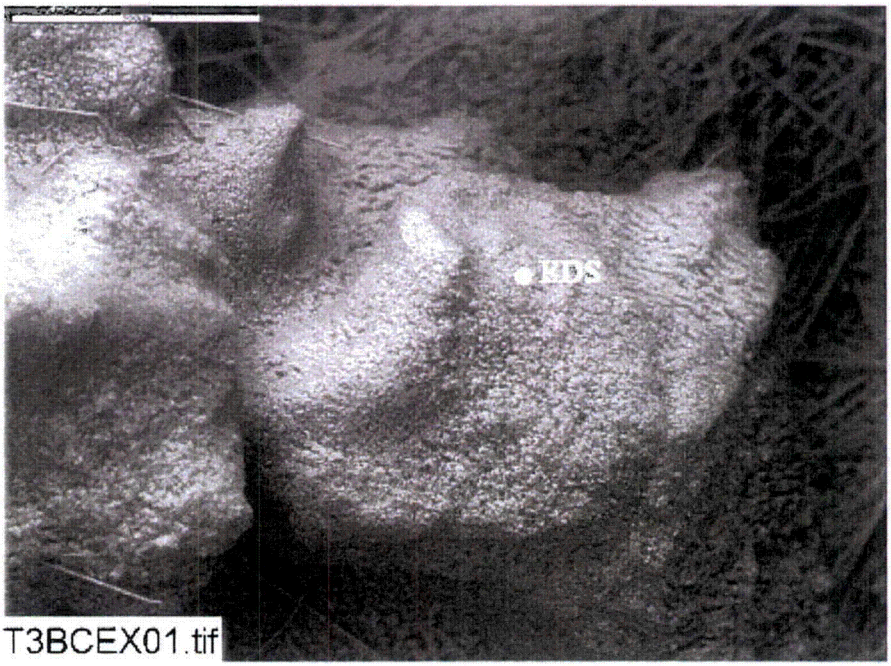



**Figure C4-4.** **ESEM image magnified 80 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (T3BCEX01)**

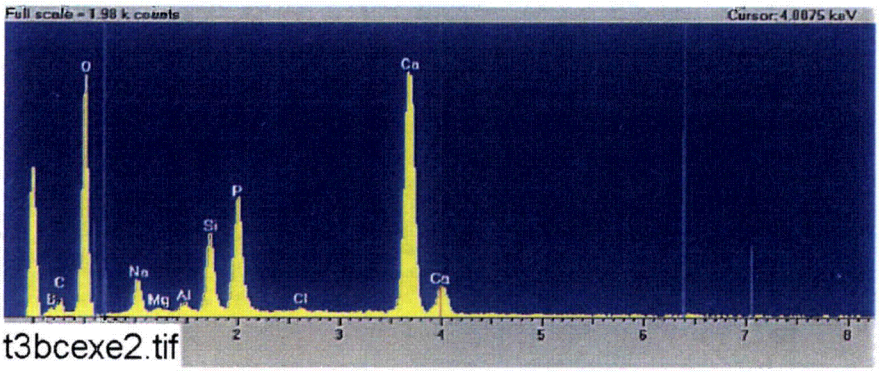



**Figure C4-5.** **EDS counting spectrum for the the large deposits shown in Figure C4-4. (t3bcexe2)**


**Figure C4-6.** **ESEM image magnified 80 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (t3bcex02)**

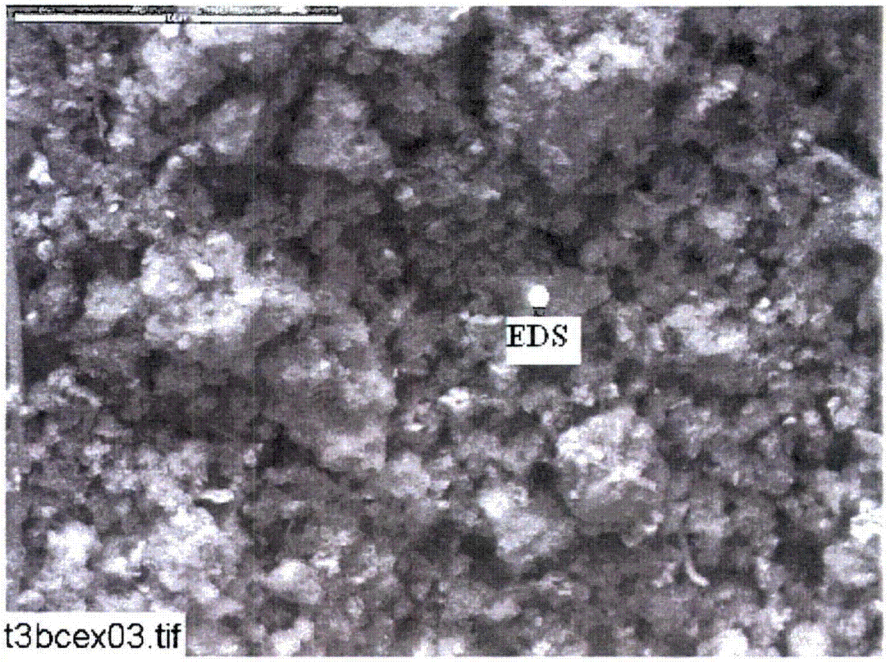



**Figure C4-7.** **ESEM image magnified 500 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (t3bcex03)**

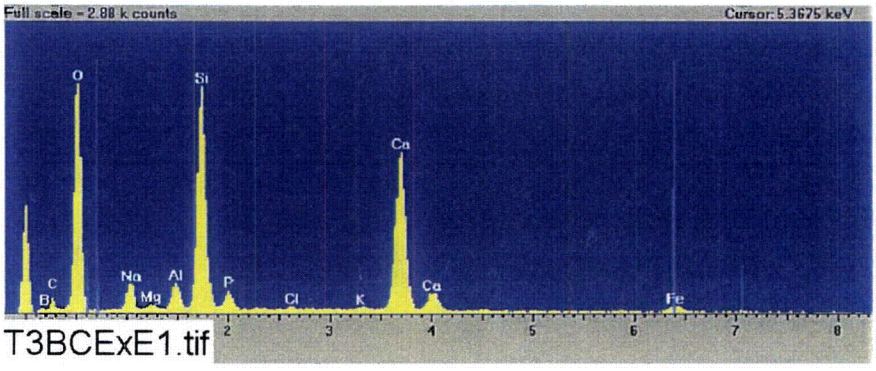



**Figure C4-8.** **EDS counting spectrum for the deposits shown in Figure C4-7. (T3BCExE1)**

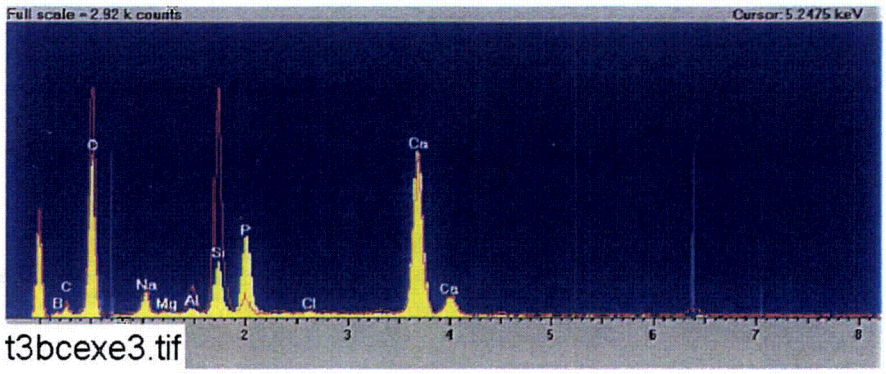



**Figure C4-9.** **Comparison of EDS counting spectra of Figure C4-5 (yellow) and Figure C4-8 (red). (t3bcexe3)**

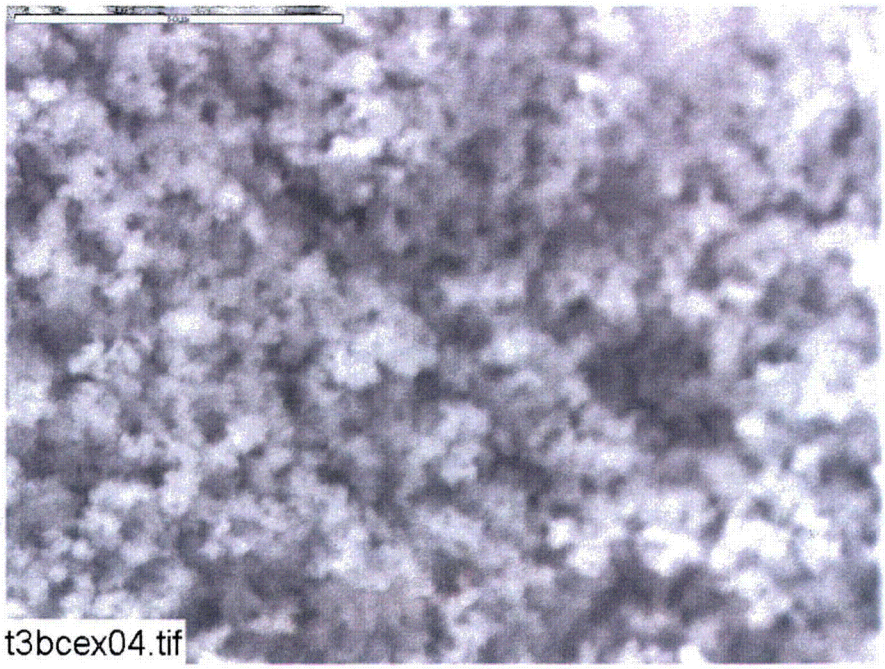



**Figure C4-10.** **ESEM image magnified 1000 times for a Test #3, Day-30 exterior fiberglass sample within a birdcage. (t3bcex04)**

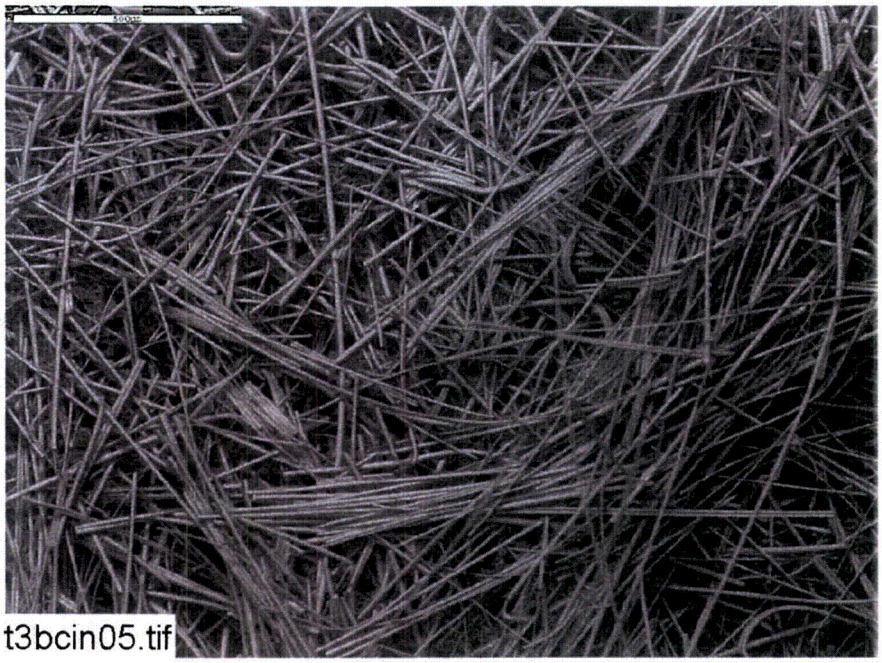



**Figure C4-11. ESEM image magnified 80 times for a Test #3, Day-30 interior fiberglass sample within a birdcage. (t3bcin05)**


**Figure C4-12. ESEM image magnified 950 times for a Test #3, Day-30 interior fiberglass sample within a birdcage. (t3bcin06)**

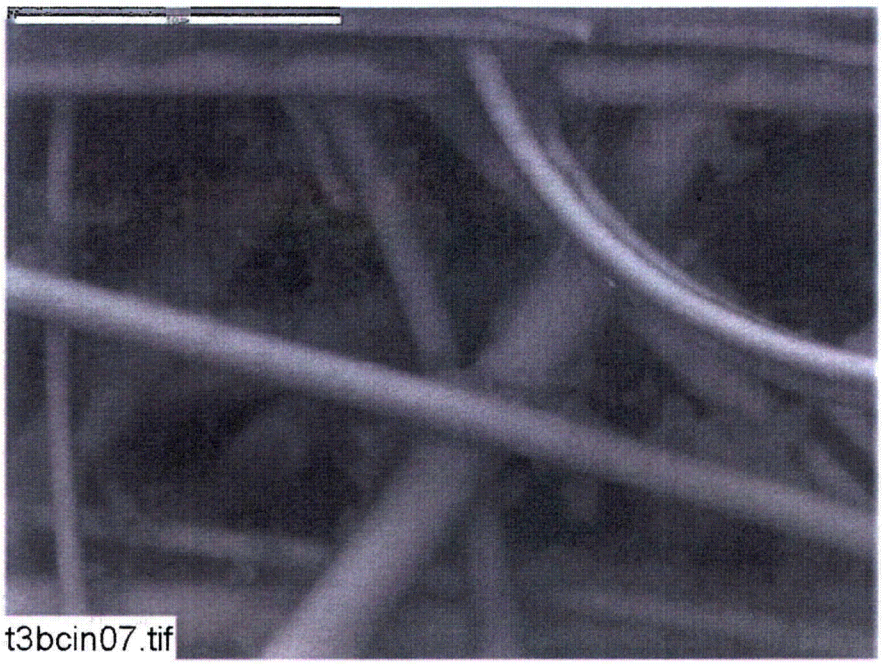



**Figure C4-13. ESEM image magnified 1000 times for a Test #3, Day-30 interior fiberglass sample within a birdcage. (t3bcin07)**

# Appendix D

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

## Figures


Figure D-1. SEM image magnified 200 times for a Test #3, Day-30 powder on the submerged rack. (T3~RackPowder013) ..... D-6

Figure D-2. EDS counting spectrum for the powder on the submerged rack shown in Figure D-1. (T3RackPowder09) ..... D-6

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) ..... D-8

Figure D-4. EDS counting spectrum for the white powder on galvanized steel shown in Figure D-3. (CorPdct~GalStel10) ..... D-8

Figure D-5. EDS counting spectrum for the dark powder on galvanized steel shown in Figure D-3. (CorPrdct~GalSteel11) ..... D-9

Figure D-6. SEM image magnified 500 times for a Test #3, Day-30 powder on a submerged copper coupon. (T3~Copper~Powder015) ..... D-11

Figure D-7. EDS counting spectrum for the powder on copper shown in Figure D-6. (CorPrdct~cu12) ..... D-11

Figure D-8. SEM image magnified 100 times for Test #3, Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdctSubmAl039) ..... D-13

Figure D-9. Backscattered SEM image magnified 100 times for Test #3, Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdctSubmAl040) ..... D-13

Figure D-10. EDS counting spectrum for the corrosion products (particles) shown in Figure D-9. (T3D30CorrPrdtAl22) ..... D-14

Figure D-11. SEM image magnified 1000 times for Test #3, Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdctSubmAl041) ..... D-16
Figure D-12. ESEM image magnified 1000 times for Test #3, Day-30 corrosion products on a submerged concrete coupon. (T3cont28) ..... D-16
Figure D-13. ESEM image magnified 100 times for Test #3, Day-30 corrosion products on a submerged concrete coupon. (t3cont29) ..... D-17
Figure D-14. EDS counting spectrum for the corrosion products (particles) shown in Figure D-13. (t3cont30) ..... D-17

## Tables

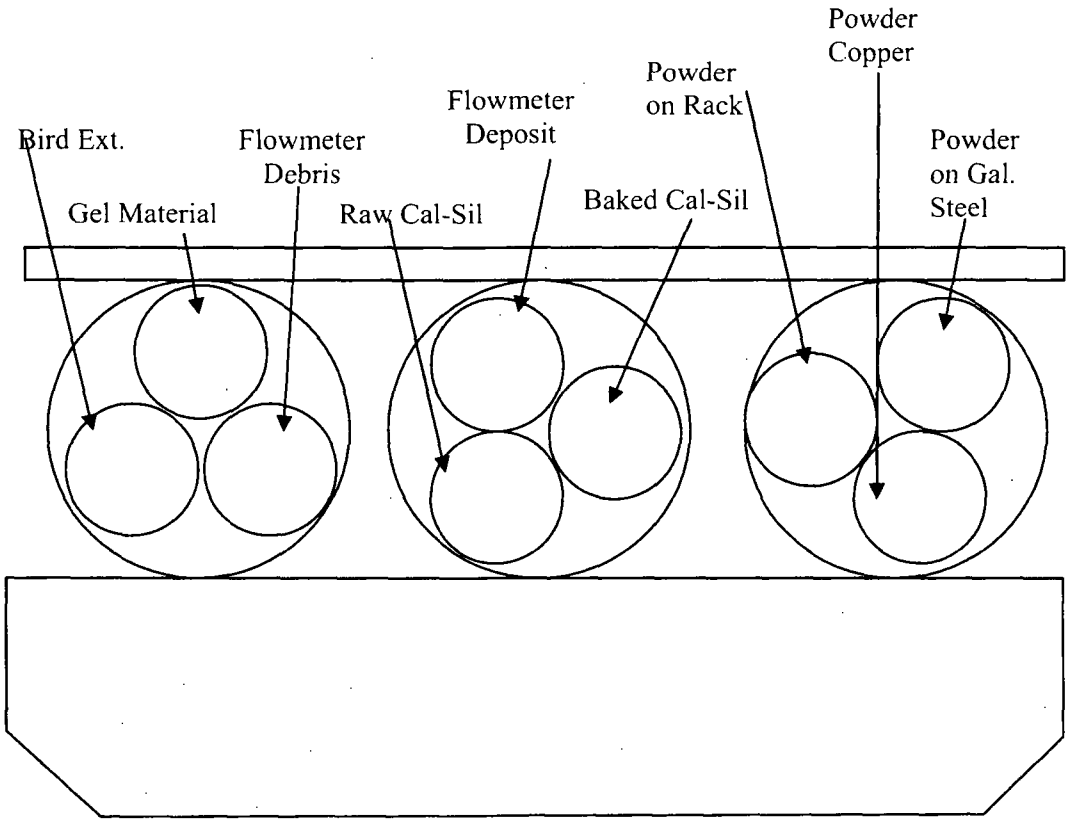
Table D-1. Chemical Compositions for T3RackPowder09, Figure D-2. ..... D-7
Table D-2. Chemical Compositions for CorPrdct~GalSteel11, Figure D-5..... D-10
Table D-3. Chemical Compositions for CorPrdct~cu12, Figure D-7..... D-12
Table D-4. Chemical Compositions for T3D30CorrPrdtAl22, Figure D-10..... D-15

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, an 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

Laboratory session from May 9, 2005.
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**

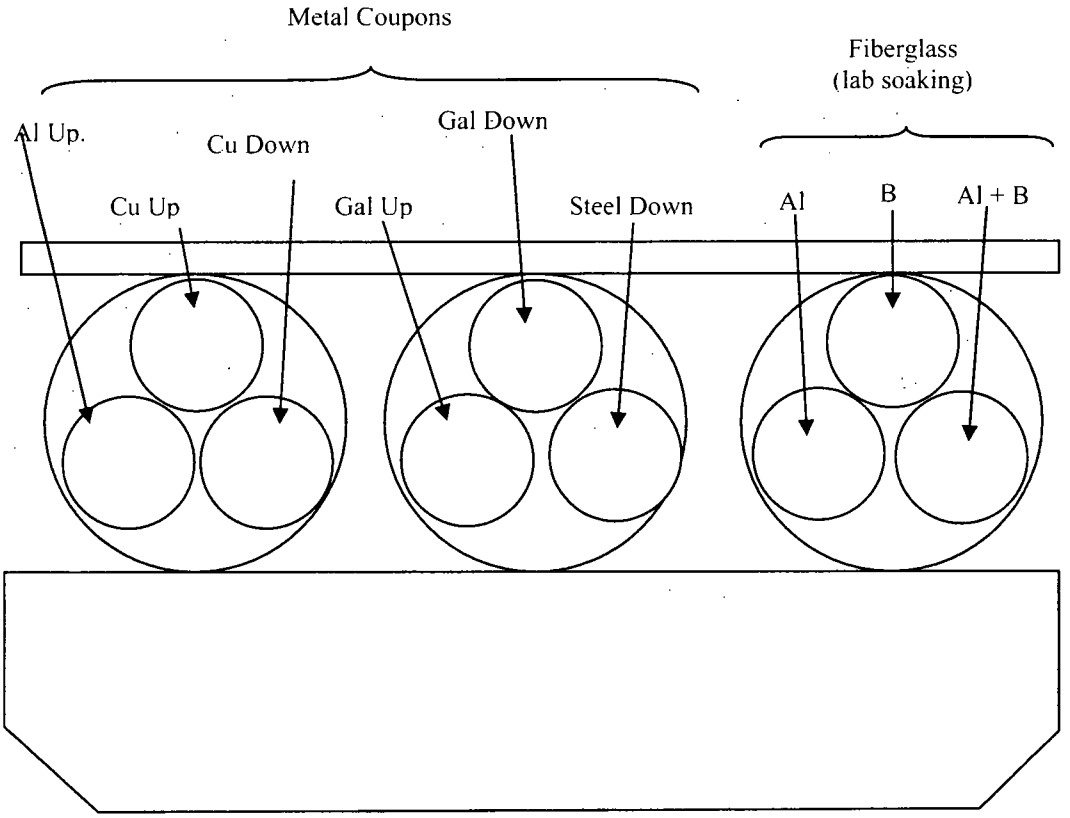
| | | | |
|---|---|---|---|
| Image: | T3~Gal~Steel~Powder014 500 × | | Figure D-3 |
| EDS: | CorPdct~GalStel10 | White powder on image 014 | Figure D-4 |
| | CorPrdct~GalSteel11 | Dark powder on image 014 | Figure D-5 |

**Powder on Copper**

| | | | |
|---|---|---|---|
| Image: | T3~Copper~Powder015 500 × | | Figure D-6 |
| EDS: | CorPrdct_cu12 | Powder on image 015 | Figure D-7 |

## Transcribed Laboratory Log

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



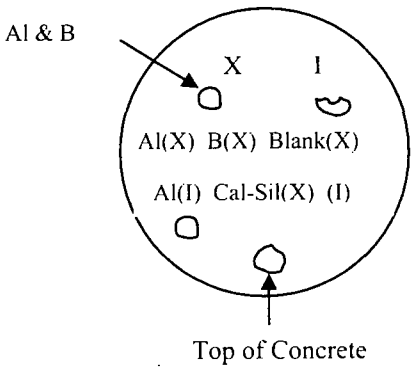

**Coat with Gold

**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.




**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 |

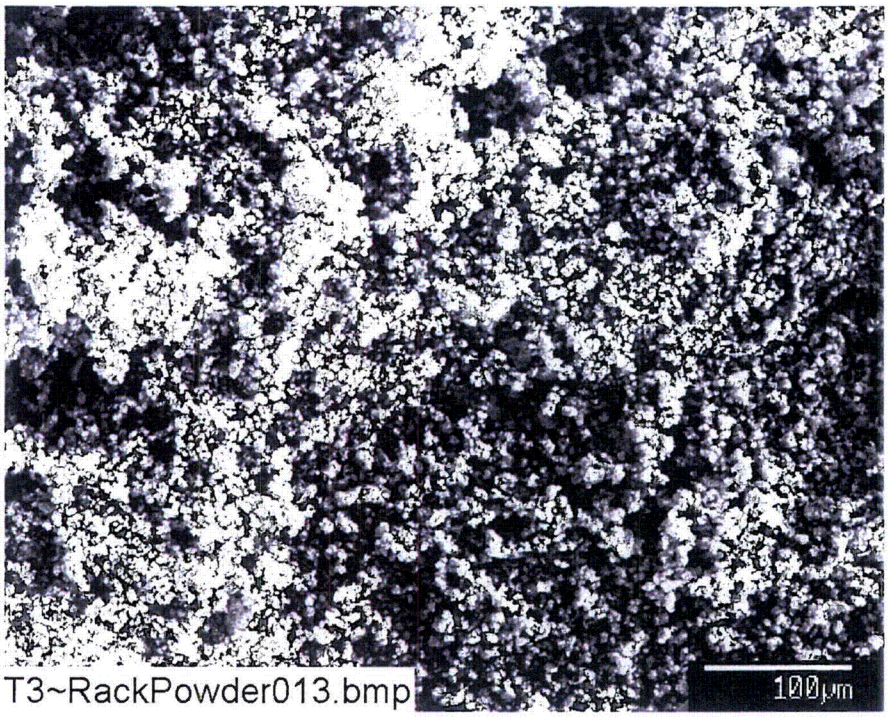



Figure D-1. SEM image magnified 200 times for a Test #3, Day-30 powder on the submerged rack. (T3~RackPowder013)

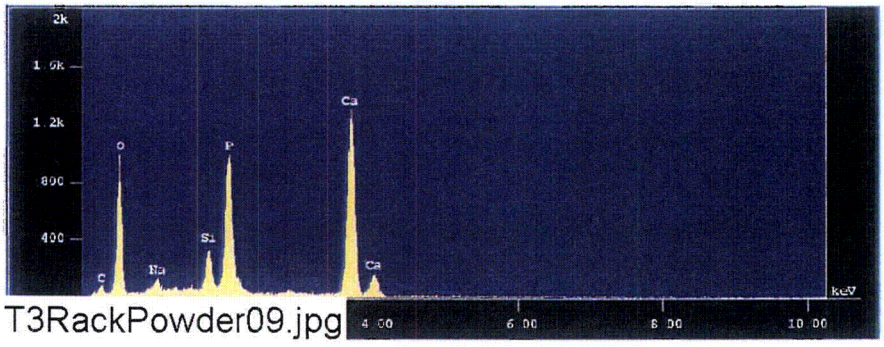



Figure D-2. EDS counting spectrum for the powder on the submerged rack shown in Figure D-1. (T3RackPowder09)

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

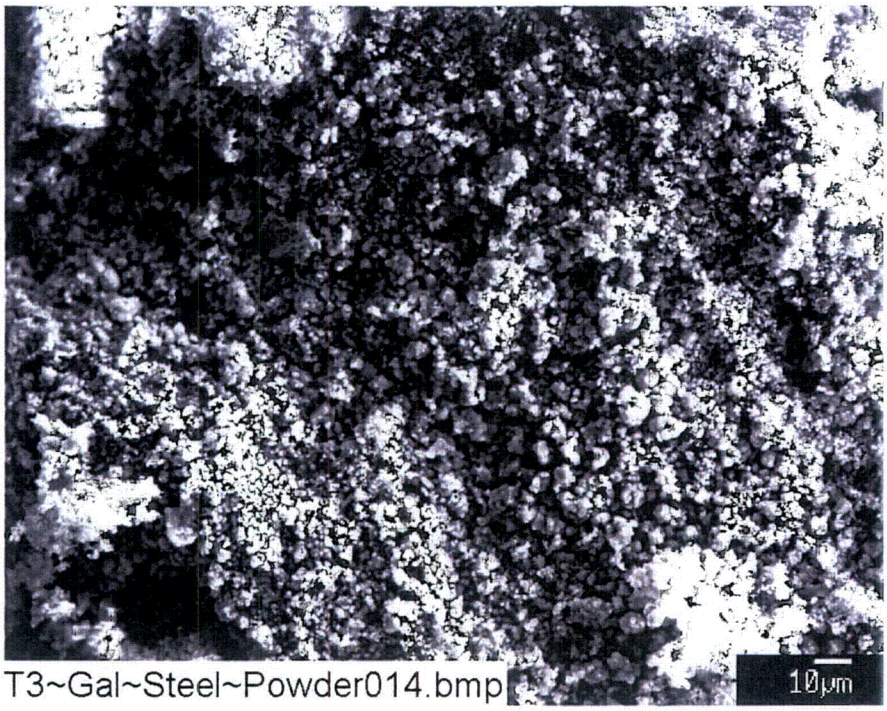



Figure D-3. SEM image magnified 500 times for a Test #3, Day-30 powder on a submerged galvanized steel coupon. (T3~Gal~Steel~Powder014)

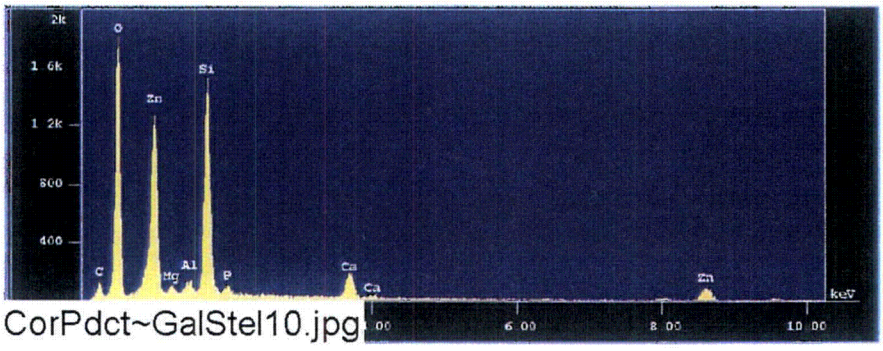



Figure D-4. EDS counting spectrum for the white powder on galvanized steel shown in Figure D-3. (CorPdct~GalStel10)

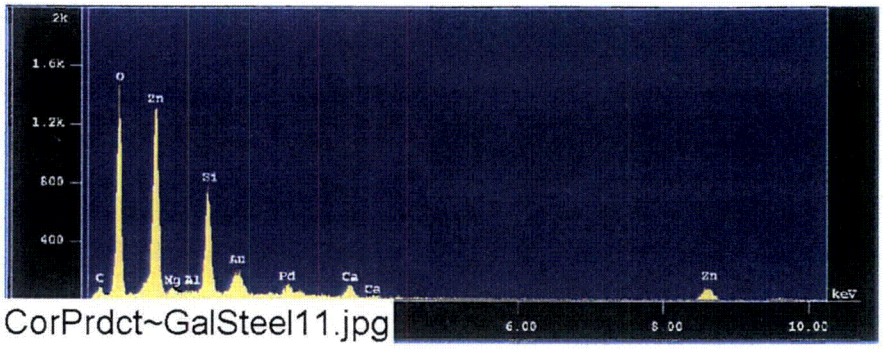



**Figure D-5.** **EDS counting spectrum for the dark powder on galvanized steel shown in Figure D-3. (CorPrdct~GalSteel11)**

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

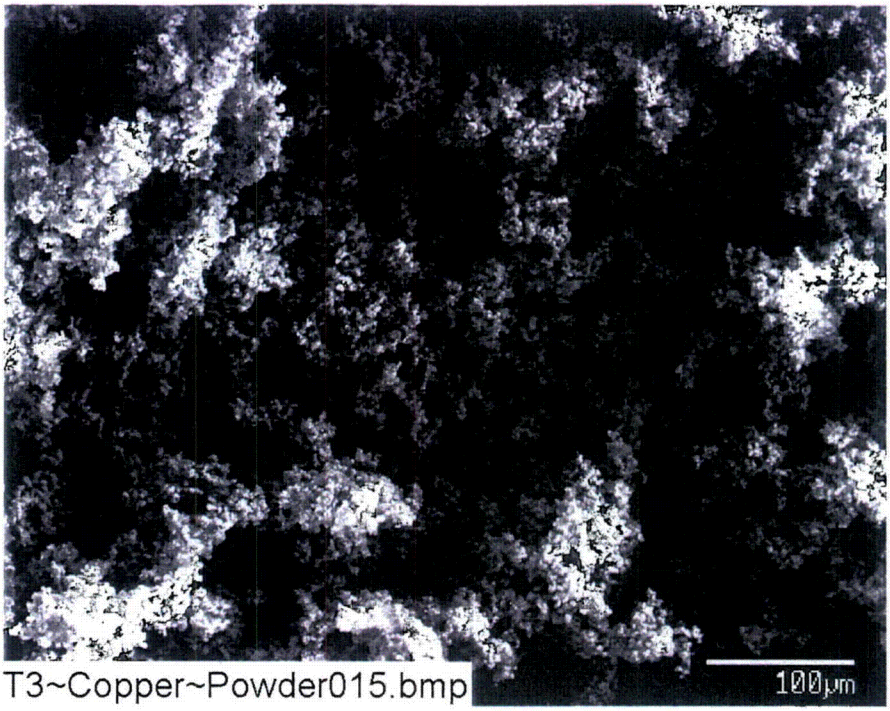



Figure D-6. SEM image magnified 500 times for a Test #3, Day-30 powder on a submerged copper coupon. (T3~Copper~Powder015)

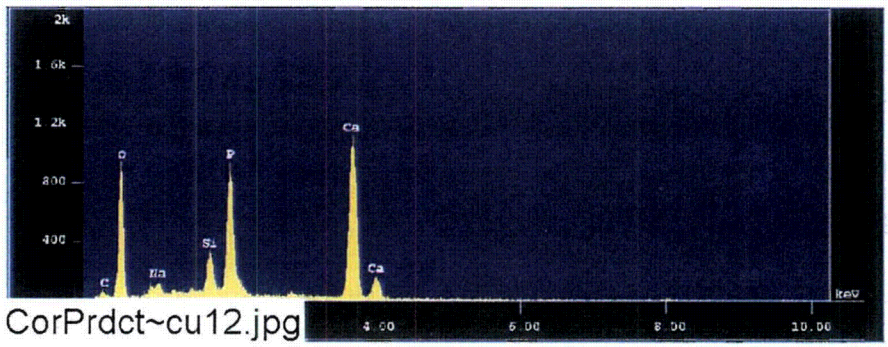



Figure D-7. EDS counting spectrum for the powder on copper shown in Figure D-6. (CorPrdct~cu12)

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


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


**Figure D-9.** **Backscattered SEM image magnified 100 times for Test #3, Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdct SubmAl040)**

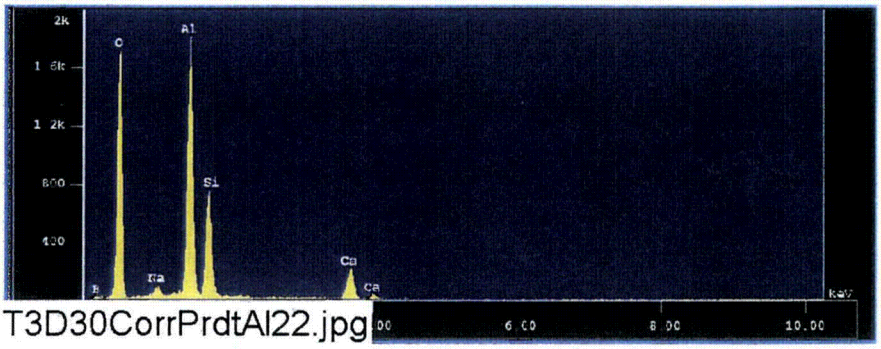



**Figure D-10.** **EDS counting spectrum for the corrosion products (particles) shown in Figure D-9. (T3D30CorrPrdtAl22)**

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

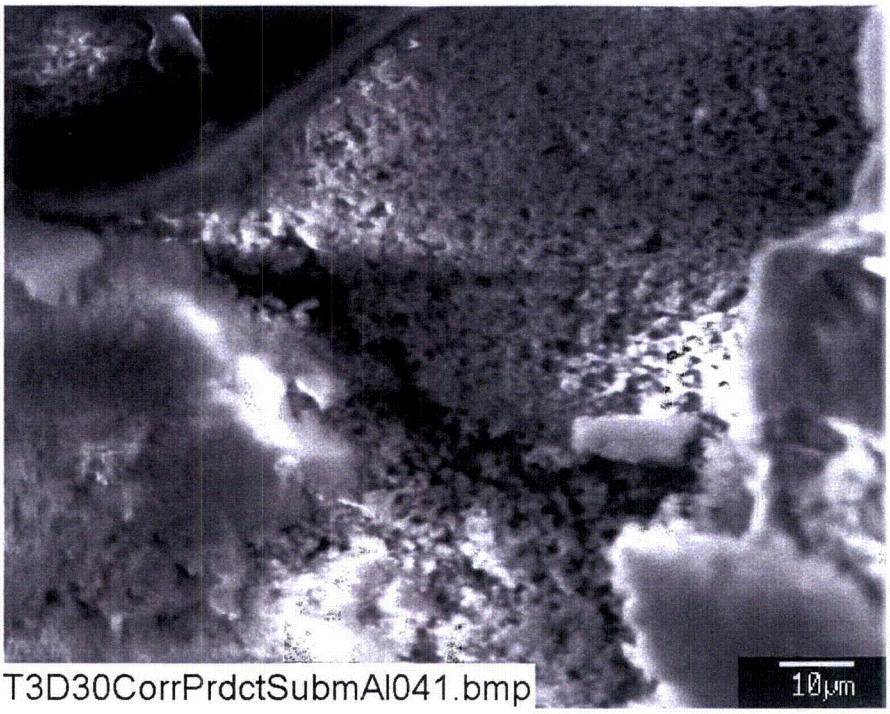



**Figure D-11.** **SEM image magnified 1000 times for Test #3, Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdctSubmAl041)**

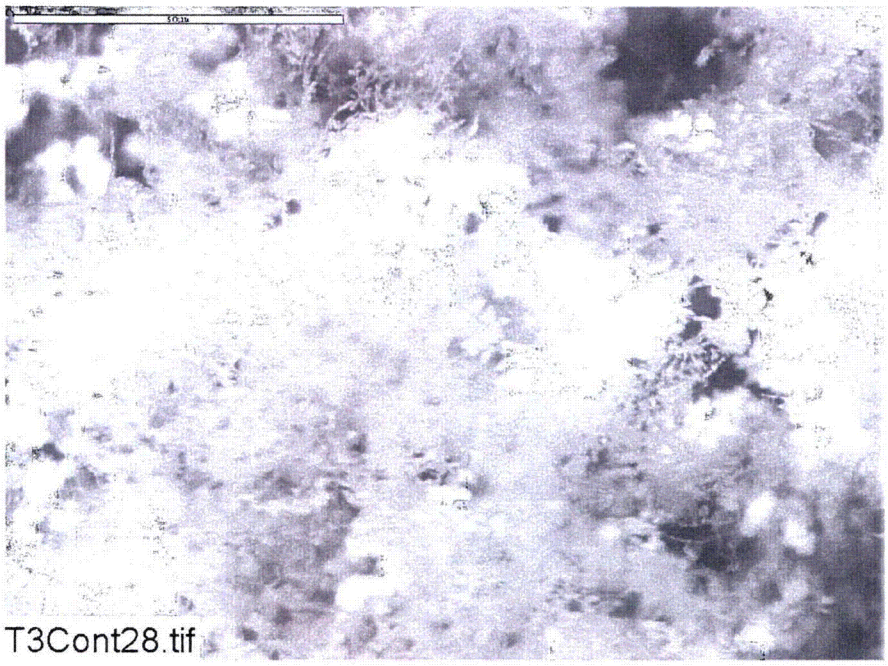



**Figure D-12.** **ESEM image magnified 1000 times for Test #3, Day-30 corrosion products on a submerged concrete coupon. (T3cont28)**

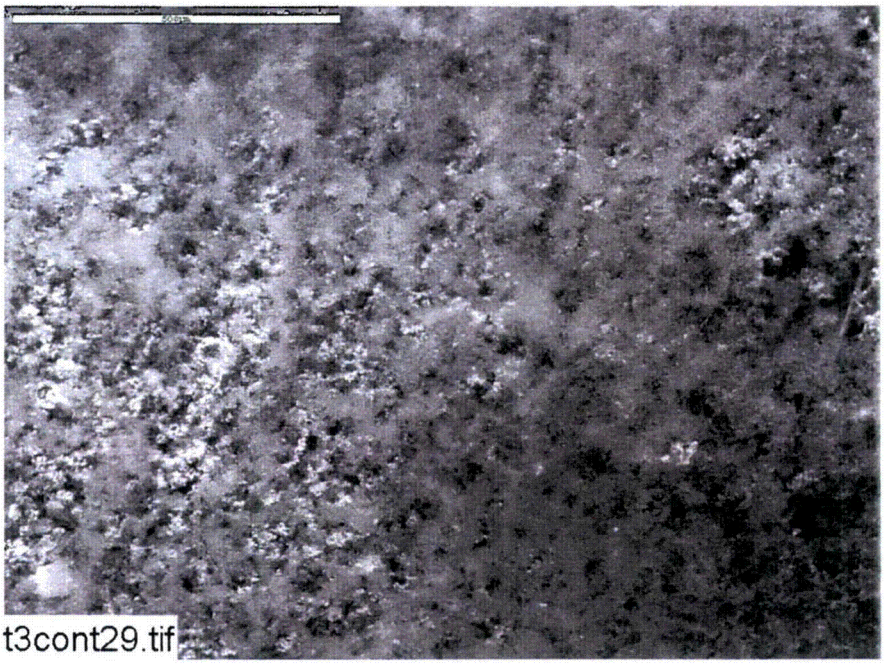



**Figure D-13.** **ESEM image magnified 100 times for Test #3, Day-30 corrosion products on a submerged concrete coupon. (t3cont29)**

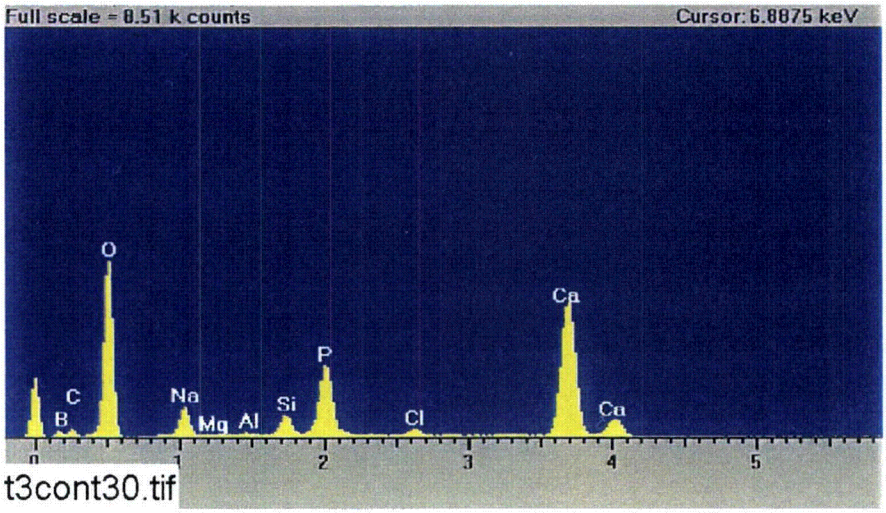



**Figure D-14.** **EDS counting spectrum for the corrosion products (particles) shown in Figure D-13. (t3cont30)**