

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

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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.

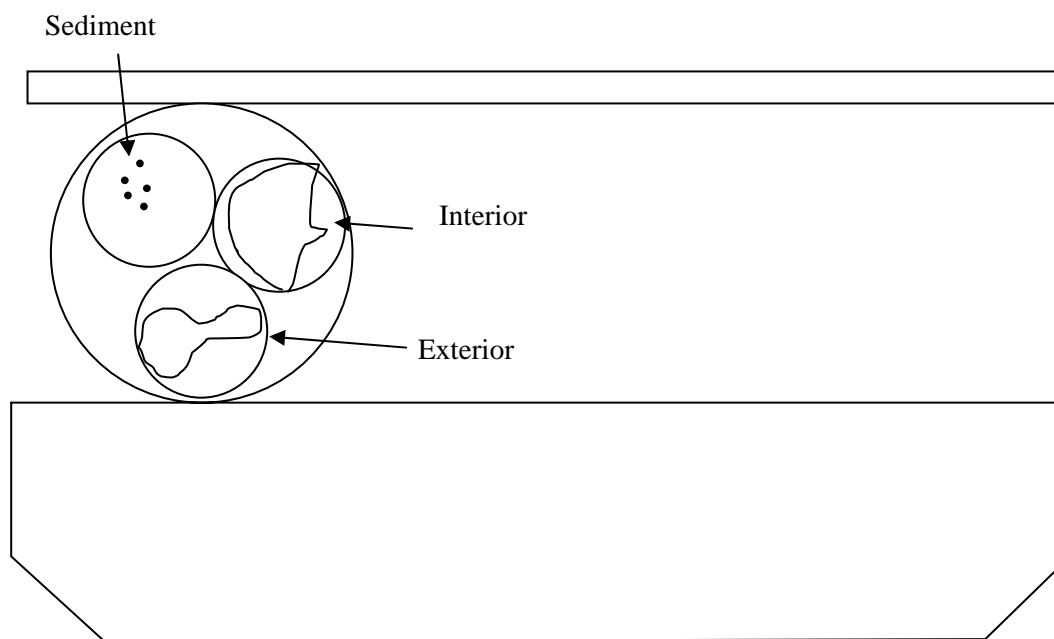
Test #3 Day-4 low-flow fiberglass samples were obtained on April 9, 2005 (4th day for Test #3), and SEM/EDS data presented here were obtained shortly thereafter. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

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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

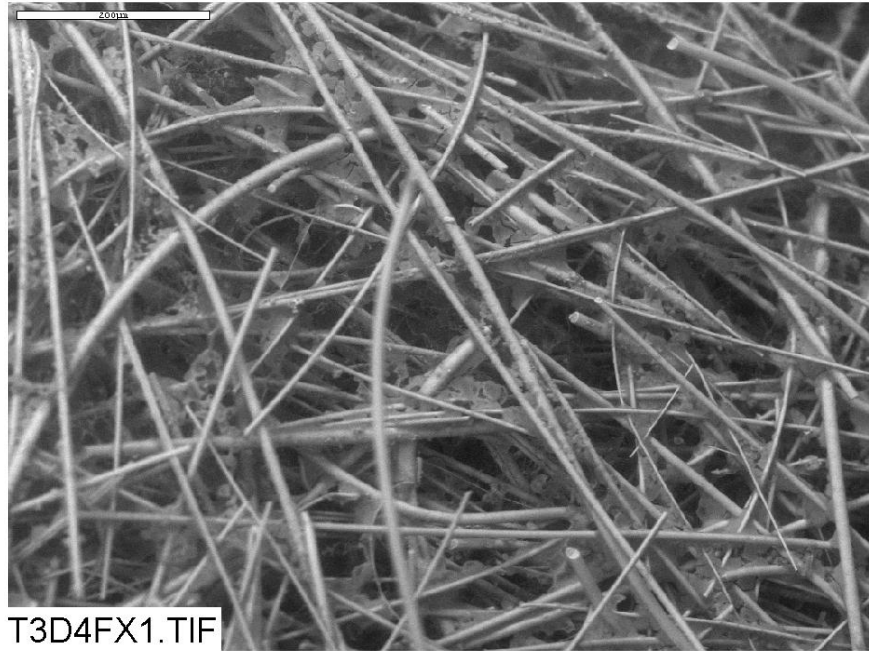


Figure A-1. Environmental SEM image magnified 150 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FX1)

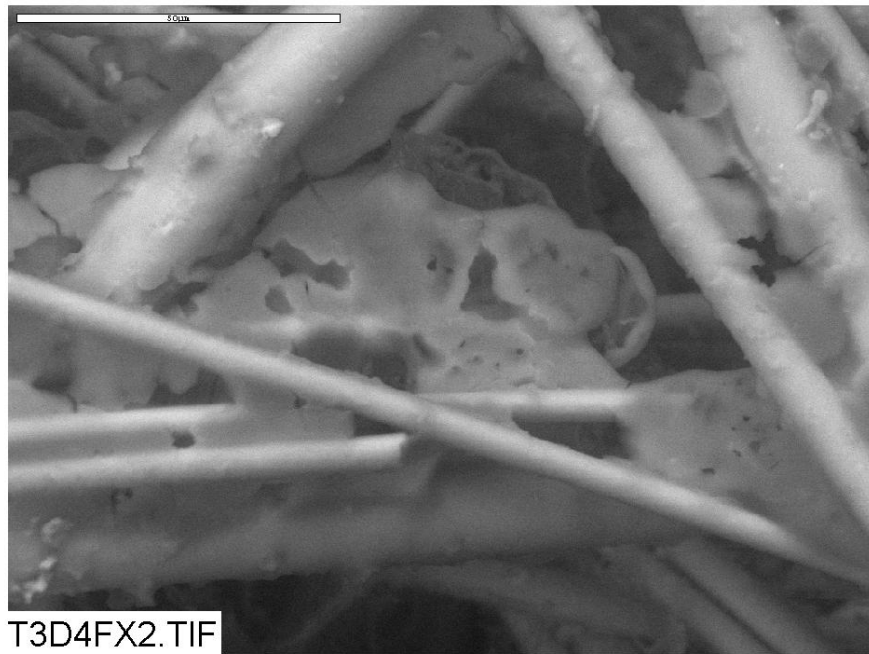


Figure A-2. Environmental SEM image magnified 1000 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FX2)

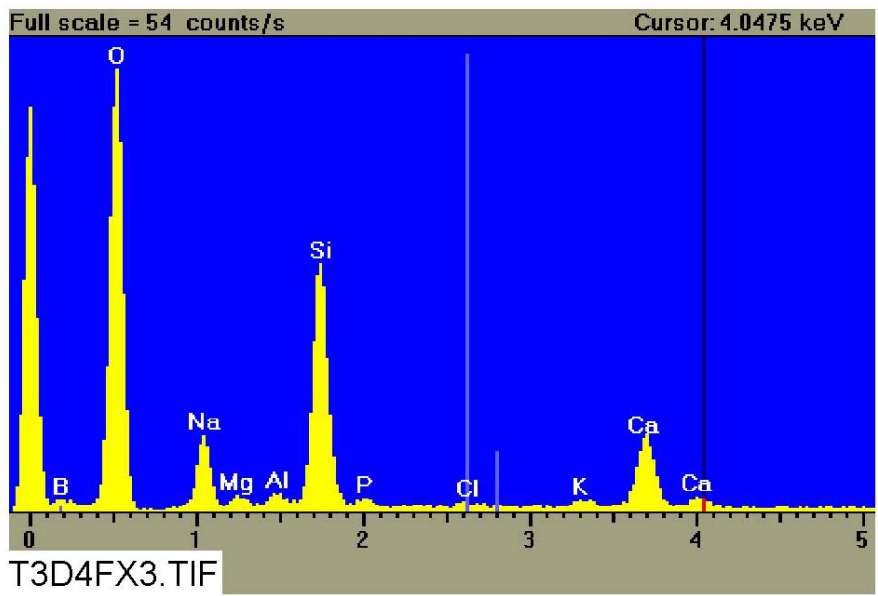


Figure A-3. EDS counting spectrum for the deposits between the fibers on ESEM image shown in Figure A-2. (T3D4FX3)

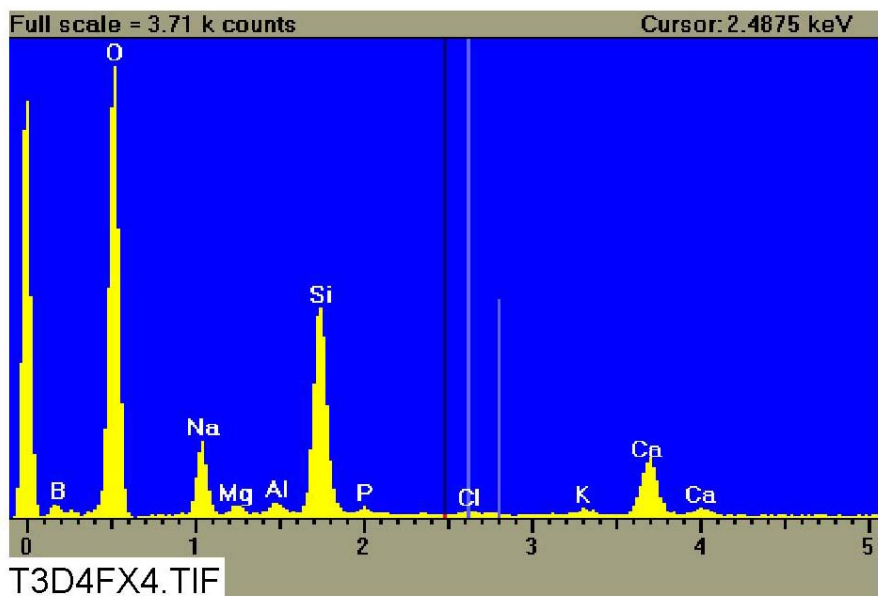


Figure A-4. EDS counting spectrum (after calibration) for the deposits between the fibers on ESEM image shown in Figure A-2. (T3D4FX4)

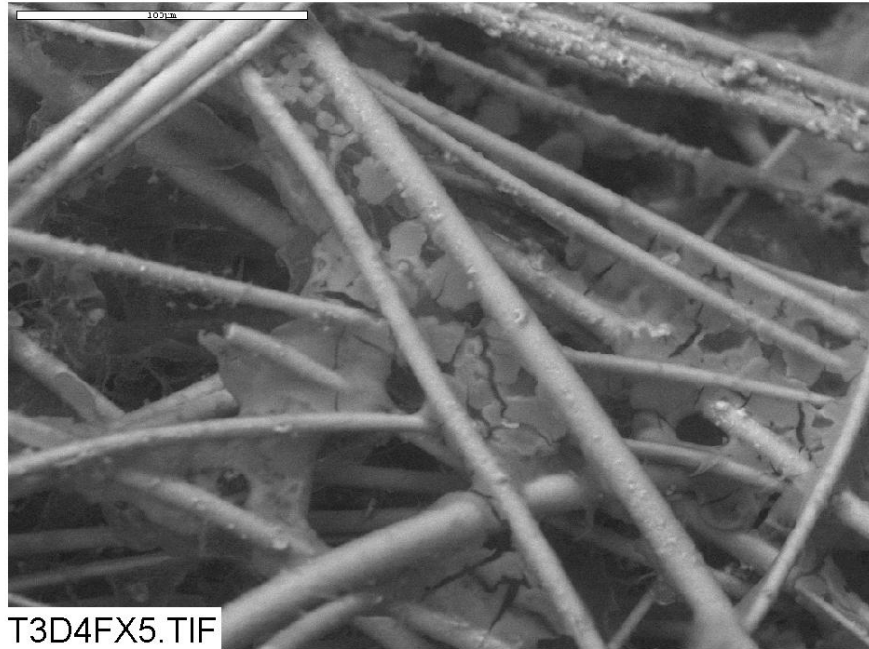


Figure A-5. Environmental SEM image magnified 500 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FX5)

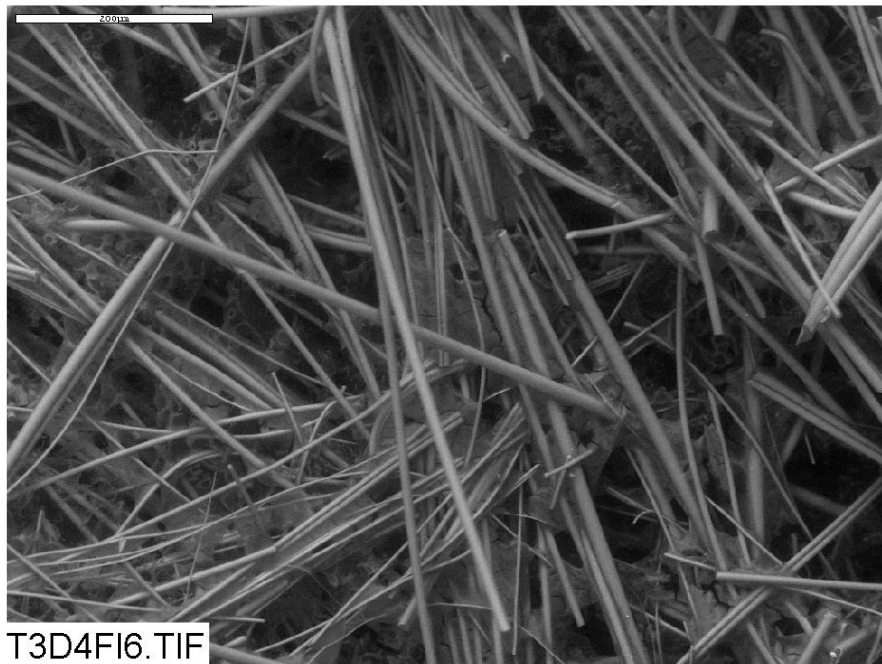


Figure A-6. Environmental SEM image magnified 150 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FI6)

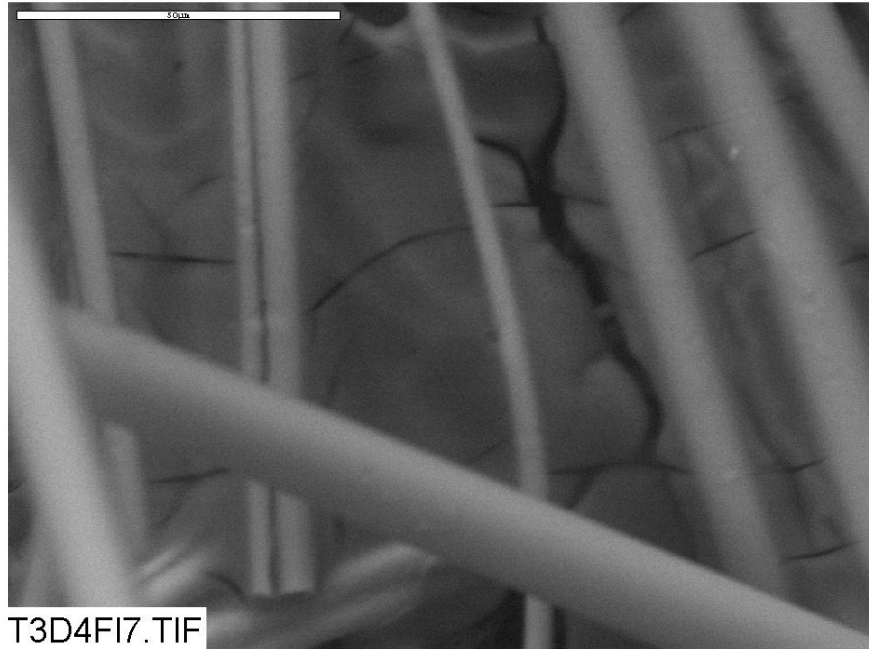


Figure A-7. Environmental SEM image magnified 1000 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FI7)

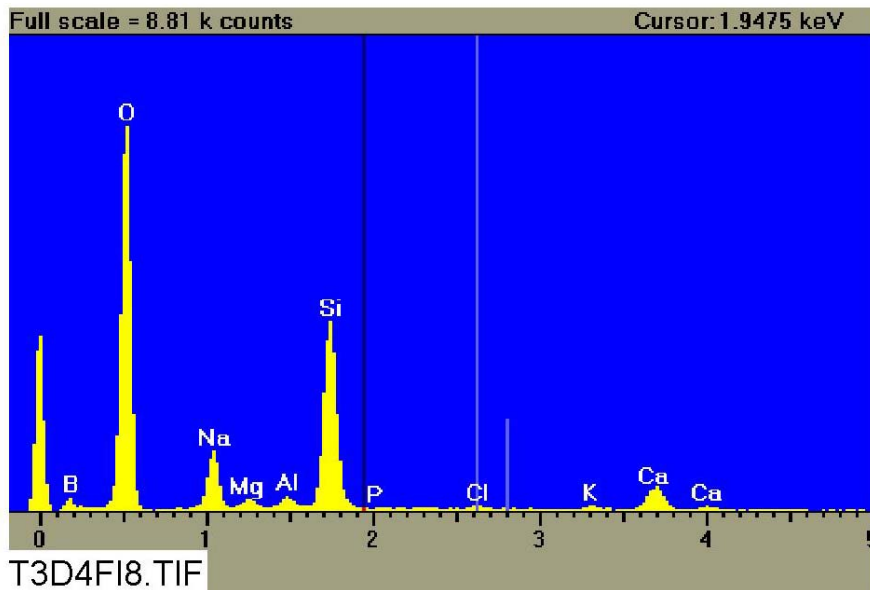


Figure A-8. EDS counting spectrum (after calibration) for the deposits between the fibers on ESEM image shown in Figure A-7. (T3D4FI8)

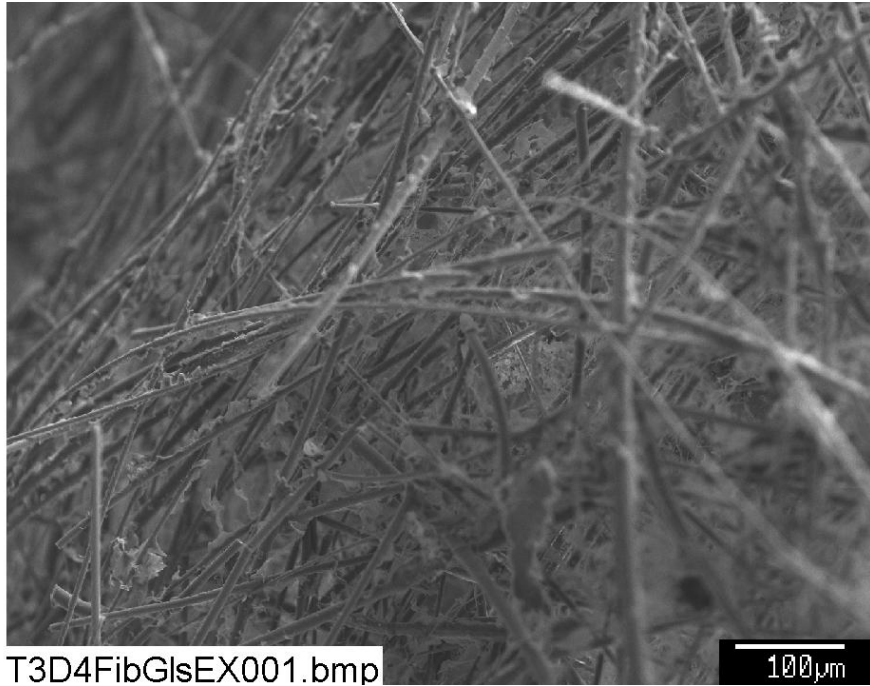


Figure A-9. SEM image magnified 150 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FibGlsEX001)

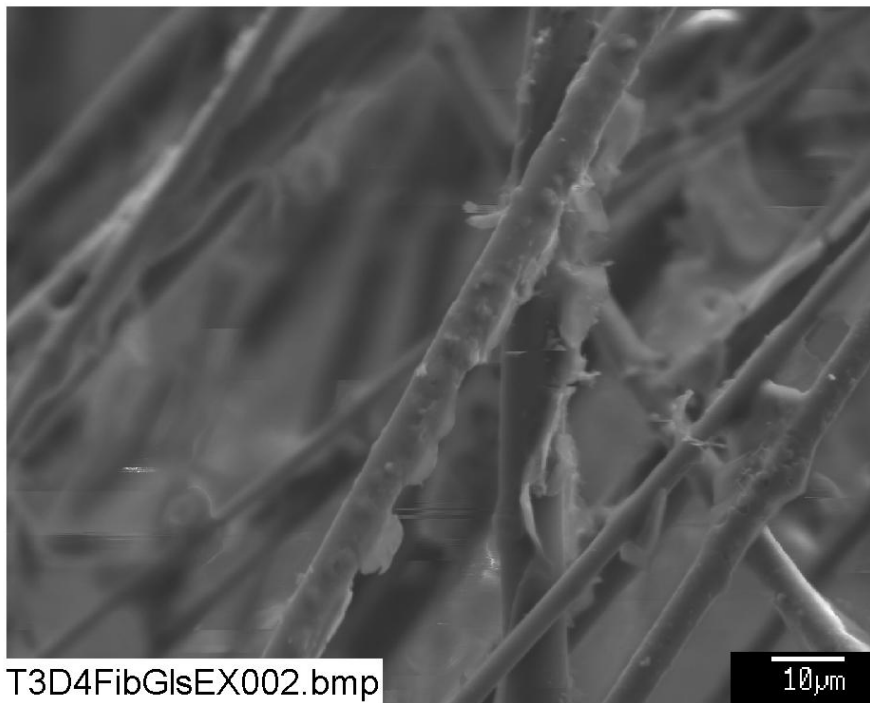
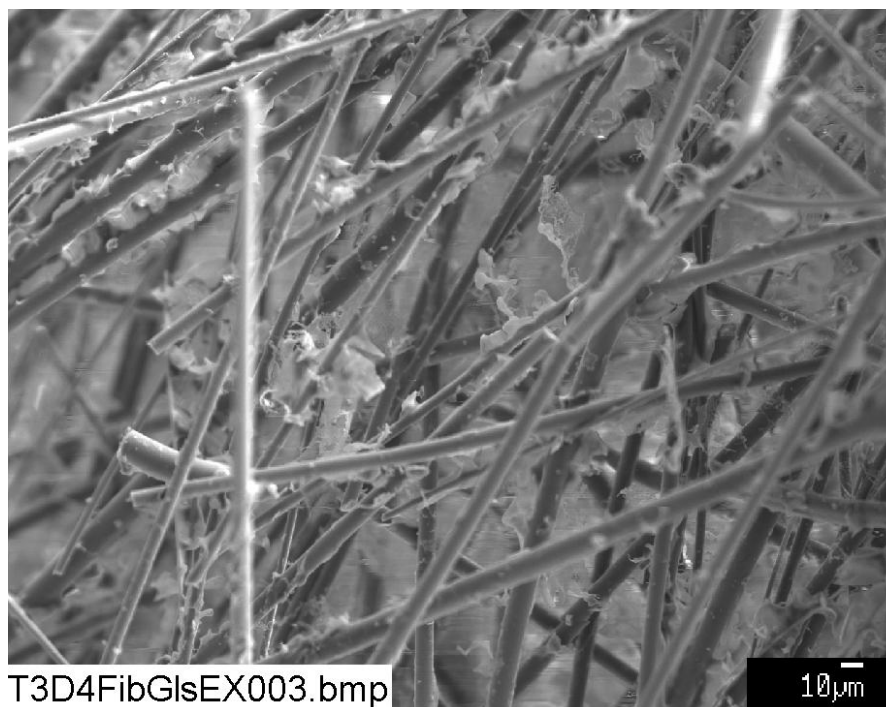


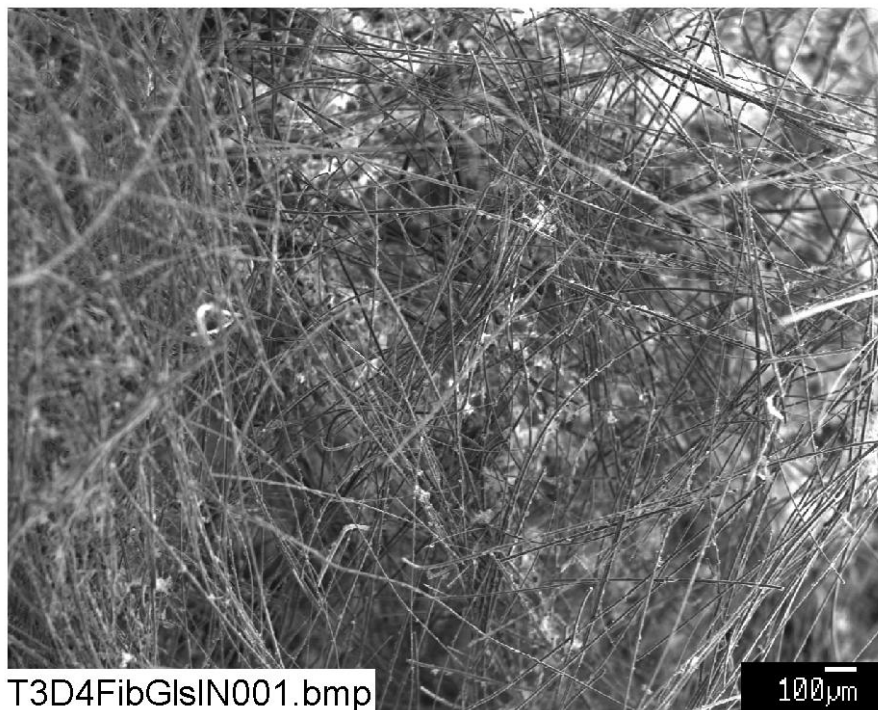
Figure A-10. SEM image magnified 1000 times for a Test #3, Day-4 low-flow exterior fiberglass sample. (T3D4FibGlsEX002)



T3D4FibGlsEX003.bmp

10 μ m

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



T3D4FibGlsIN001.bmp

100 μ m

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

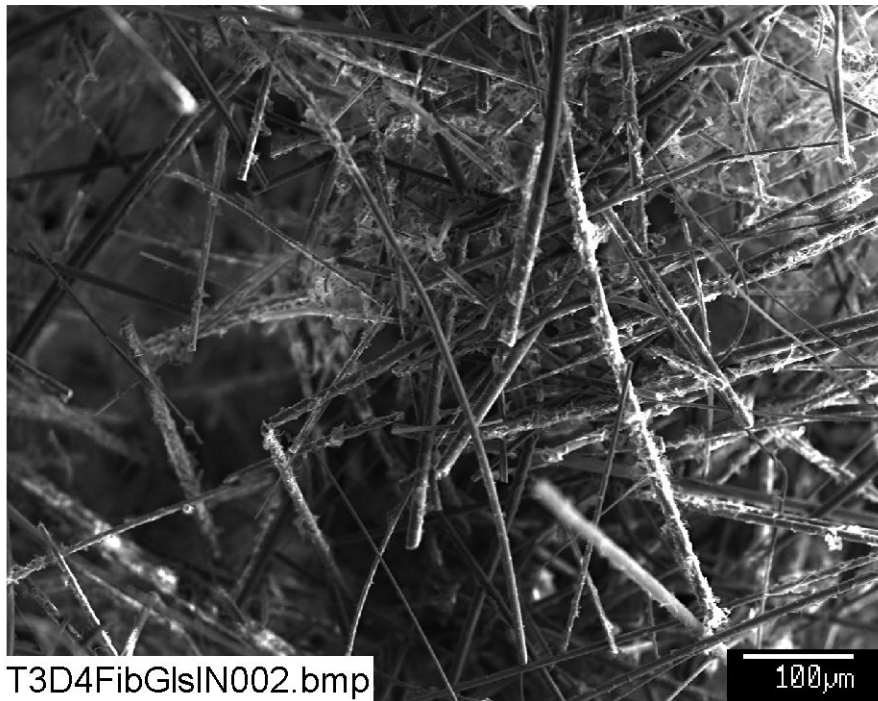


Figure A-13. SEM image magnified 150 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN002)

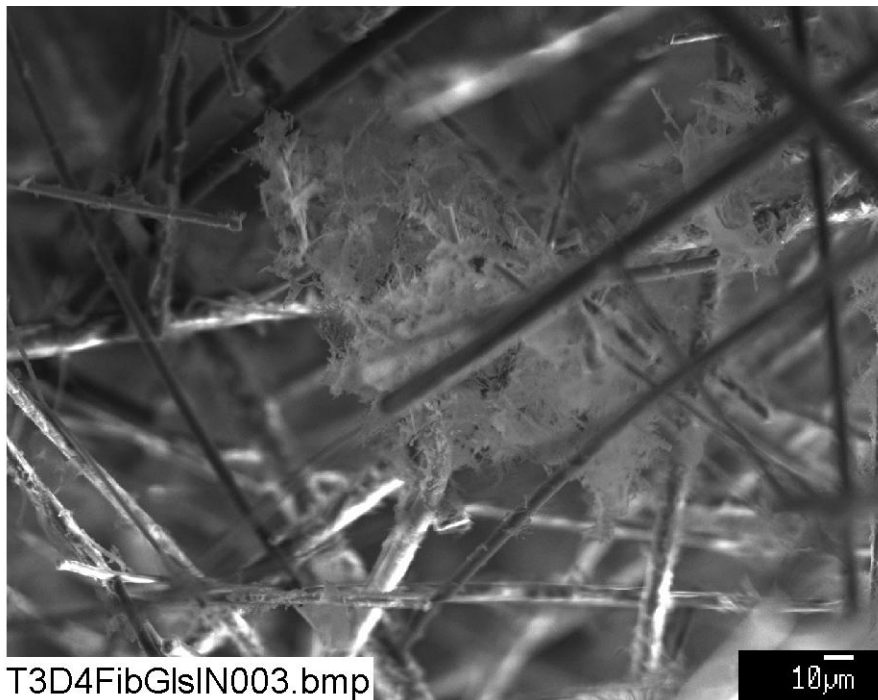


Figure A-14. SEM image magnified 400 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN003)

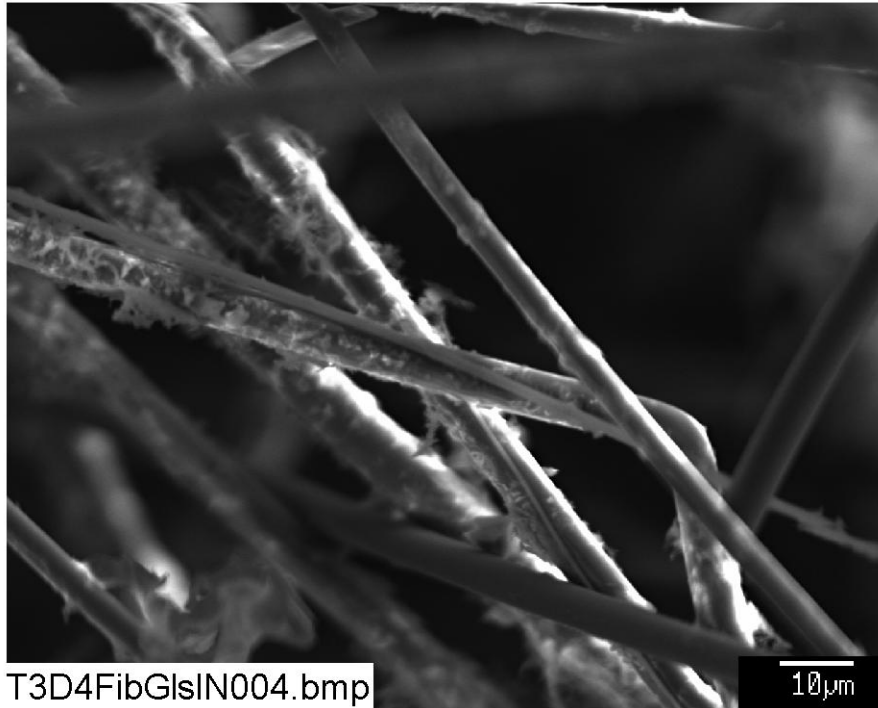


Figure A-15. SEM image magnified 1000 times for a Test #3, Day-4 low-flow interior fiberglass sample. (T3D4FibGlsIN004)