

PREFACE TO APPENDICES

Ten separate appendices were developed to capture more of the images and information obtained for Test #4. Several appendices are further divided into subappendices to better segregate the information according to the time point in the test when the samples were extracted from the test apparatus, the location of the samples in the tank, the type of samples being evaluated, and the type of examinations performed. With the exception of Appendix J, each appendix represents a separate session of laboratory work that can be traced to a batch of samples; these samples were typically processed in chronological order. Appendix J provides the detailed project instructions that were used to initiate Test #4, conduct routine operations during the test, and 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 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.

Appendix titles are listed below for reference.

Appendix A. ESEM/EDS Data for Test #4, Day-5 Fiberglass in Low-Flow Zone

Appendix B. ESEM Day-15 Fiberglass

- B1. ESEM/EDS Data for Test #4, Day-15 Fiberglass in Low-Flow Zones
- B2. ESEM/EDS Data for Test #4, Day-15 Fiberglass in High-Flow Zones

Appendix C. ESEM Day-30 Fiberglass

- C1. ESEM/EDS Data for Test #4, Day-30 Fiberglass in Low-Flow Zones
- C2. ESEM/EDS Data for Test #4, Day-30 Fiberglass Inserted in Nylon Mesh in a Low-Flow Zone
- C3. ESEM Data for Test #4, Day-30 Low-Flow Fiberglass Samples in a Big Envelope
- C4. ESEM/EDS Data for Test #4, Day-30 Fiberglass in High-Flow Zones
- C5. ESEM/EDS Data for Test #4, Day-30 Fiberglass Inserted in Front of Header in a High-Flow Zone
- C6. ESEM/EDS Data for Test #4, Day-30 Drain Collar Fiberglass
- C7. ESEM/EDS Data for Test #4, Day-30 Birdcage Fiberglass

Appendix D. ESEM/EDS Data for Test #4, Day-30 Low-Flow Cal-Sil Samples

Appendix E. ESEM and SEM/EDS Data for Test #4, Day-30 Deposition Products

Appendix F. SEM Day-30 Coupons

- F1. SEM/EDS Data for Test #4, Day-30 Aluminum Coupons
- F2. SEM/EDS Data for Test #4, Day-30 Copper Coupons
- F3. SEM/EDS Data for Test #4, Day-30 Galvanized Steel Coupons
- F4. SEM/EDS Data for Test #4, Day-30 Steel Coupons

Appendix G. SEM/EDS Data for Test #4, Day-30 Sediment

Appendix H. TEM Data for Test #4 Solution Samples

Appendix I. UV Absorbance Spectrum—Day-30 Solution Samples

Appendix J. ICET Test #4: Pre-Test, Test, and Post-Test Project Instructions

Appendix A

ESEM/EDS Data for Test #4, Day-5 Fiberglass in Low-Flow Zone

Figures

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| Figure A-7. | Environmental SEM image magnified 1000 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lfx12.jpg)..... | A-7 |
| Figure A-8. | EDS counting spectrum for the film between the fibers shown in Figure A-7. (t4d5lfx13.jpg)..... | A-7 |
| Figure A-9. | Environmental SEM image magnified 80 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lfi5.jpg)..... | A-8 |
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| Figure A-11. | Environmental SEM image magnified 500 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lfi7.jpg)..... | A-9 |
| Figure A-12. | Environmental SEM image magnified 2000 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lfi8.jpg)..... | A-9 |
| Figure A-13. | Environmental SEM image magnified 1000 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lfi9.jpg)..... | A-10 |

Figure A-14. Environmental SEM image magnified 1000 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5li10.jpg)..... A-10

Figure A-15. Environmental SEM image magnified 500 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5li15)..... A-11

During ICET Test #4, 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 is to identify the composition of debris on fiberglass and the particulate substances in the test solution. To address this question partially, low-flow fiberglass samples on Test #4, Day 5 were examined by ESEM/EDS, including both of the exterior and the interior location of the fiberglass samples.

ESEM was employed to analyze the hydrated fiberglass samples without any coating under a low-vacuum condition (i.e., 80 Pa). This process minimizes the modification of the fiberglass samples as compared with normal SEM that would involve a drying process. EDS results provide a semi-quantitative elemental analysis of the debris attached on fiberglass.

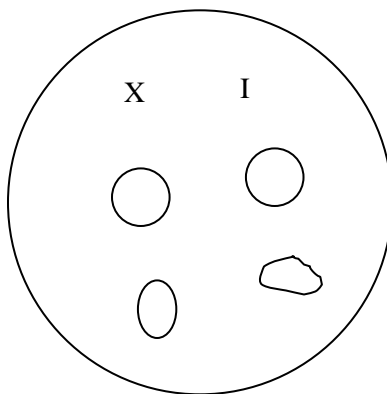
Test #4, Day-5 low-flow fiberglass samples were obtained on May 29, 2005 (fifth date for Test #4). ESEM/EDS data presented here were obtained on May 31, 2005.

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Transcribed Laboratory Log

Laboratory session from May 31, 2005.

ESEM/EDS Test #4, Day-5 Fiberglass in Low-Flow Zone



ESEM Exterior Low-Flow Fiberglass Samples

| | | | | |
|--------|----------|--------|------------------------------|------------|
| Image: | T4D5LFX1 | 80 × | ESEM image | Figure A-1 |
| | t4d5lfx4 | 80 × | ESEM at higher magnification | Figure A-2 |
| | t4d5lfx2 | 500 × | | Figure A-3 |
| | t4d5lfx3 | 1000 × | | Figure A-4 |
| | t4d5lx11 | 1000 × | | Figure A-5 |
| | t4d5lx14 | 500 × | | Figure A-6 |
| | t4d5lx12 | 1000 × | | Figure A-7 |
| EDS: | t4d5lx13 | | | Figure A-8 |

ESEM Interior Low-Flow Fiberglass Samples

| | | | | |
|--------|----------|--------|------------------------------|-------------|
| Image: | t4d5lfi5 | 80 × | ESEM image | Figure A-9 |
| | t4d5lfi6 | 500 × | ESEM at higher magnification | Figure A-10 |
| | t4d5lfi7 | 500 × | | Figure A-11 |
| | t4d5lfi8 | 2000 × | | Figure A-12 |
| | t4d5lfi9 | 1000 × | | Figure A-13 |
| | t4d5li10 | 1000 × | | Figure A-14 |
| | t4d5li15 | 500 × | | Figure A-15 |

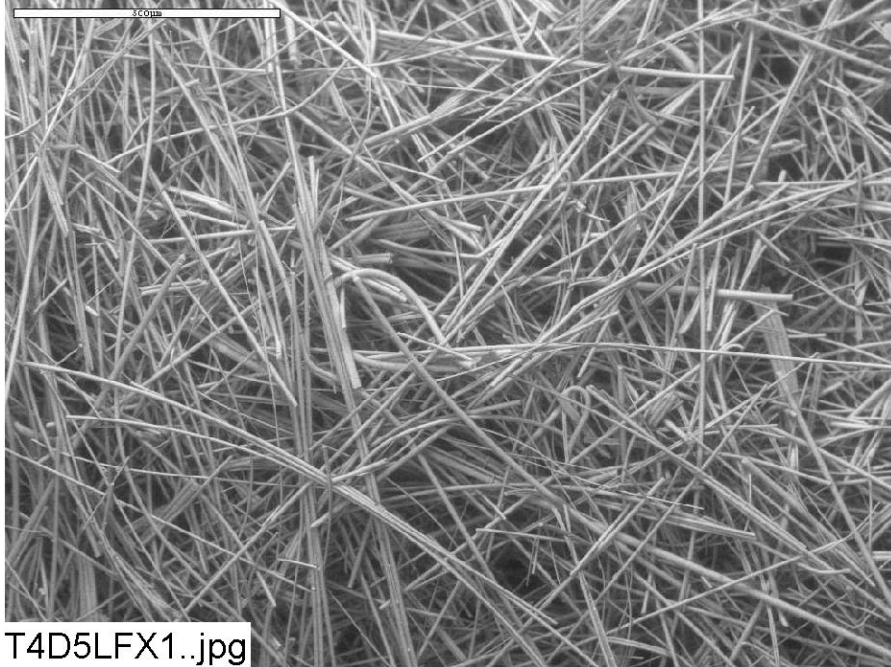


Figure A-1. Environmental SEM image magnified 80 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (T4D5LFX1.jpg)

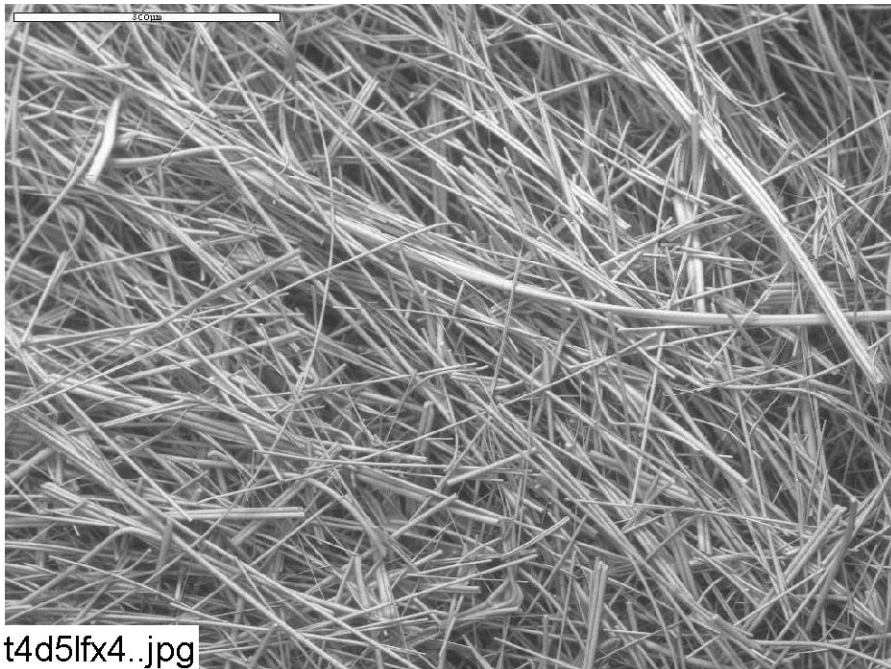


Figure A-2. Environmental SEM image magnified 80 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lfx4.jpg)

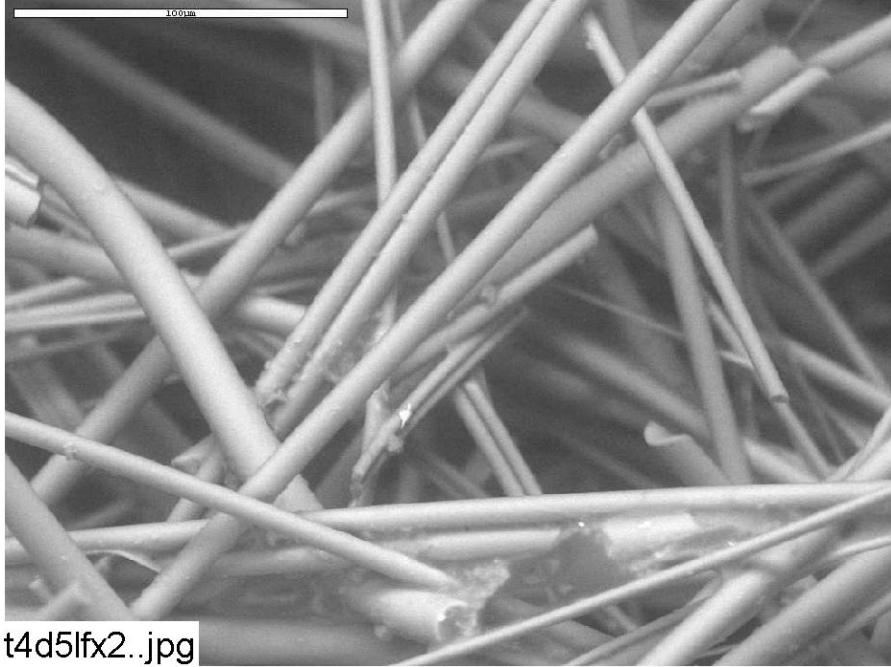


Figure A-3. Environmental SEM image magnified 500 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lfx2.jpg)

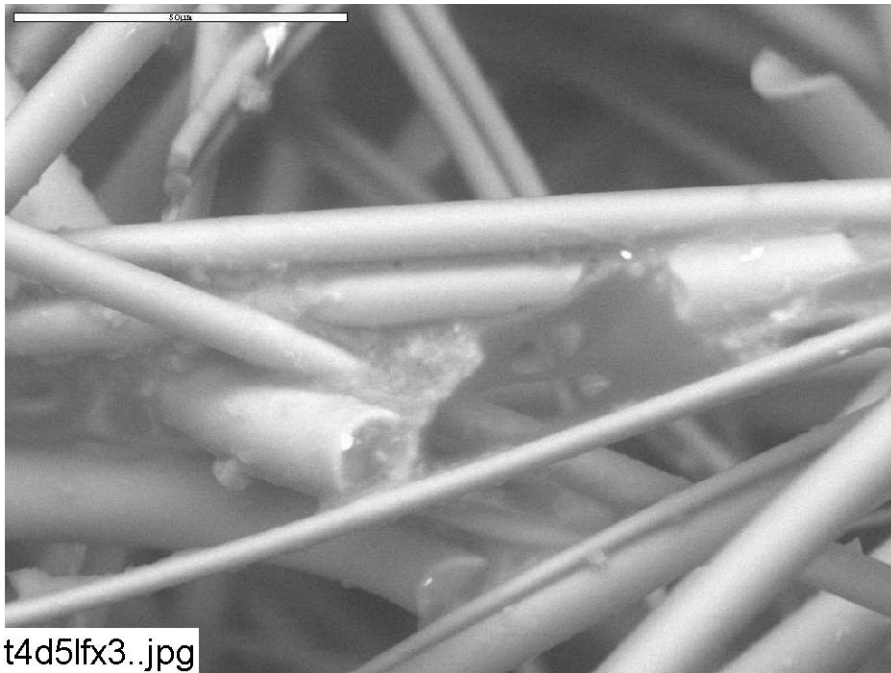


Figure A-4. Environmental SEM image magnified 1000 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lfx3.jpg)

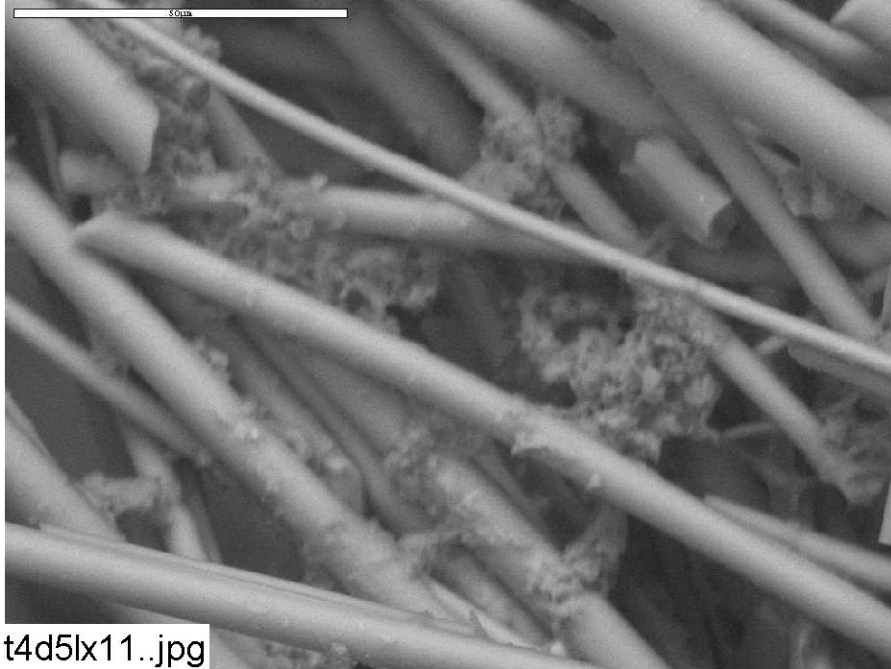


Figure A-5. Environmental SEM image magnified 1000 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lx11.jpg)

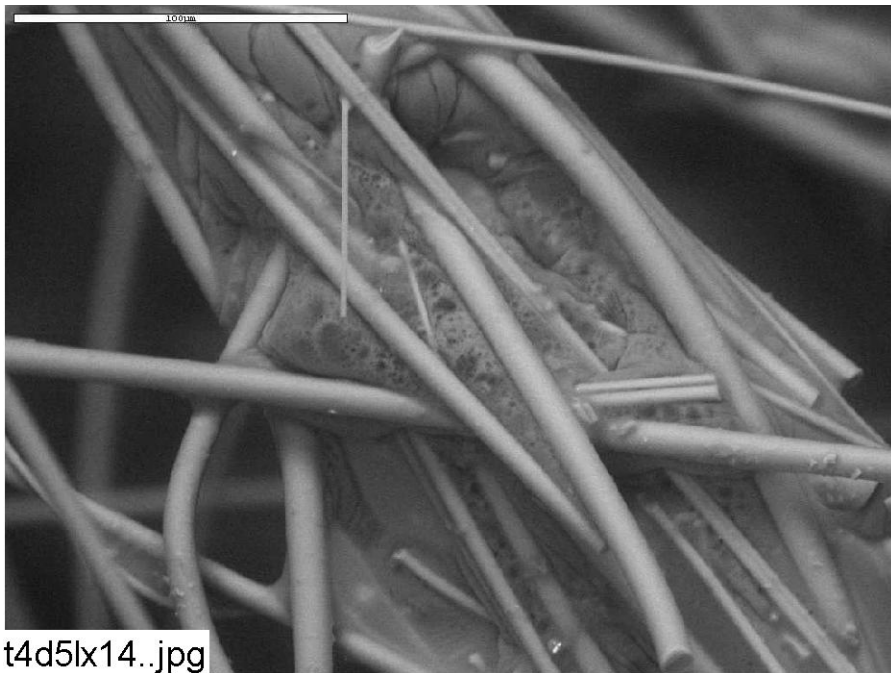


Figure A-6. Environmental SEM image magnified 500 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lx14.jpg)

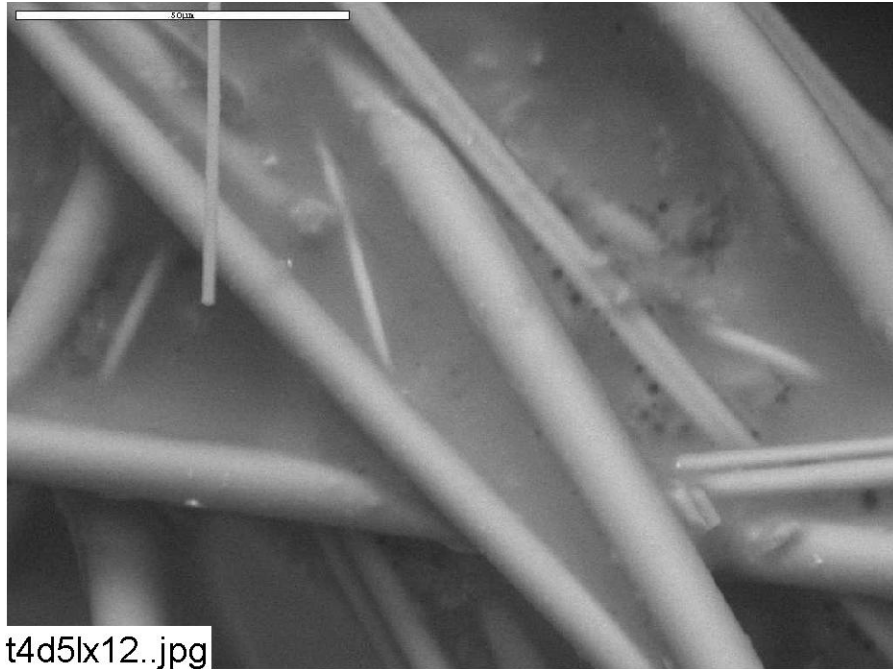


Figure A-7. Environmental SEM image magnified 1000 times for a Test #4, Day-5 low-flow exterior fiberglass sample. (t4d5lx12.jpg)

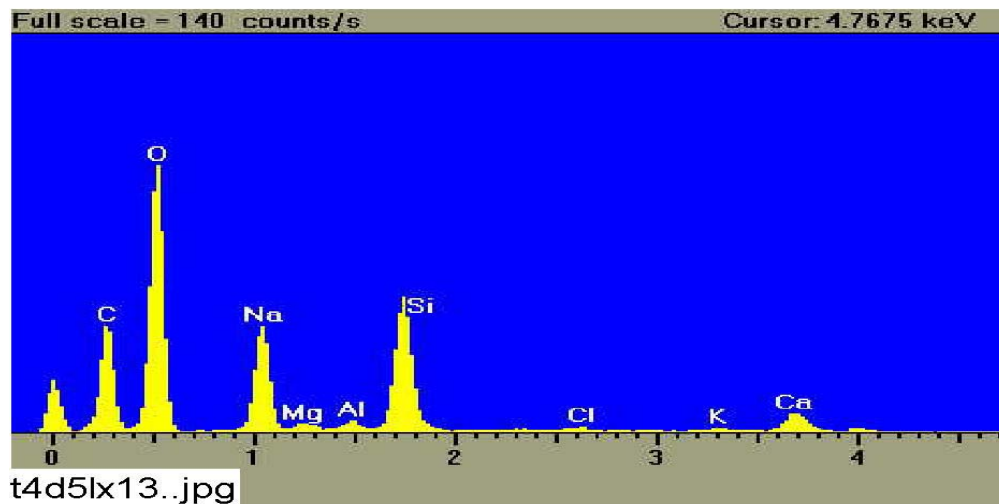


Figure A-8. EDS counting spectrum for the film between the fibers shown in Figure A-7. (t4d5lx13.jpg)

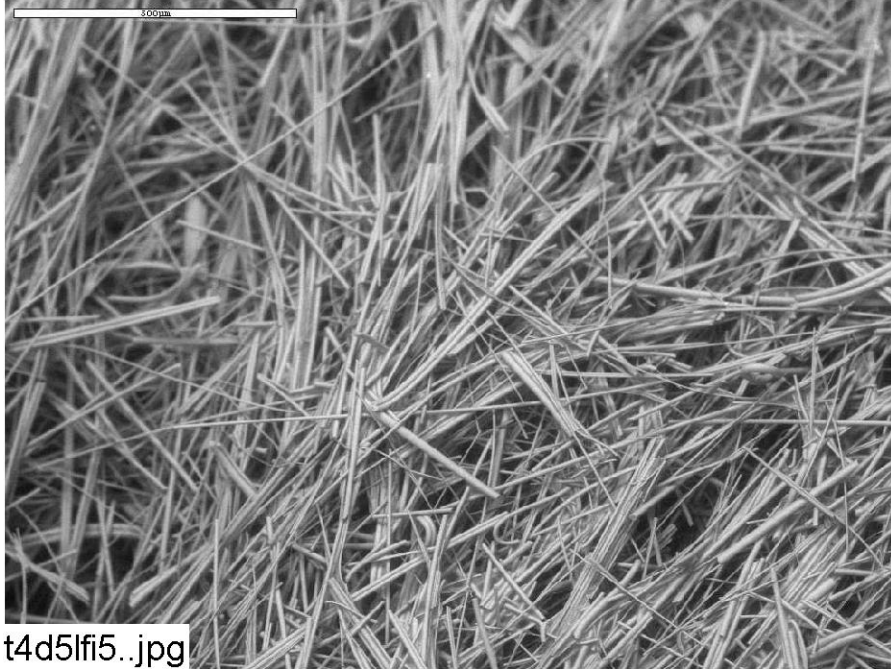


Figure A-9. Environmental SEM image magnified 80 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lf15.jpg)

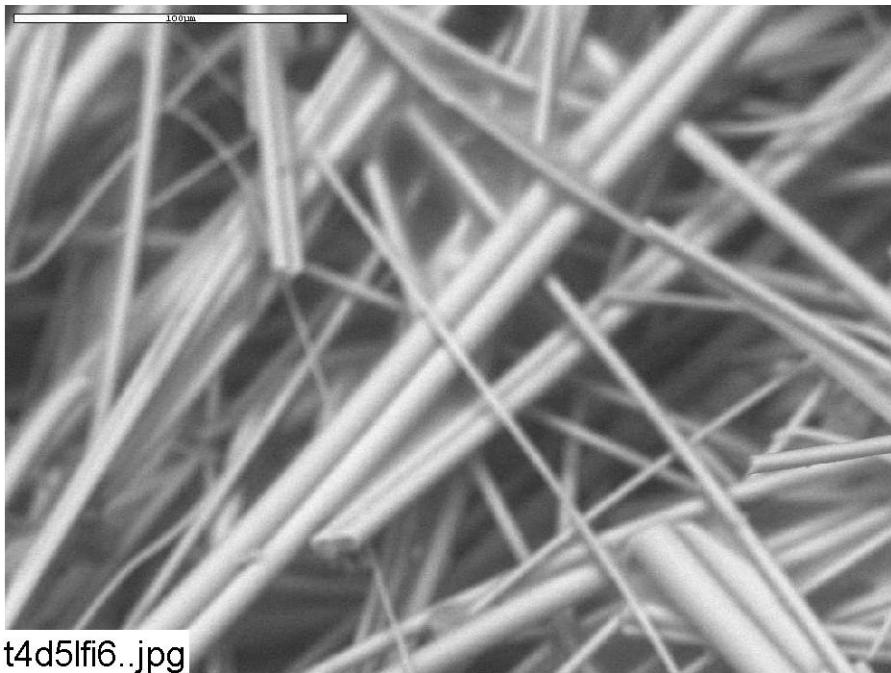


Figure A-10. Environmental SEM image magnified 500 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lf16.jpg)

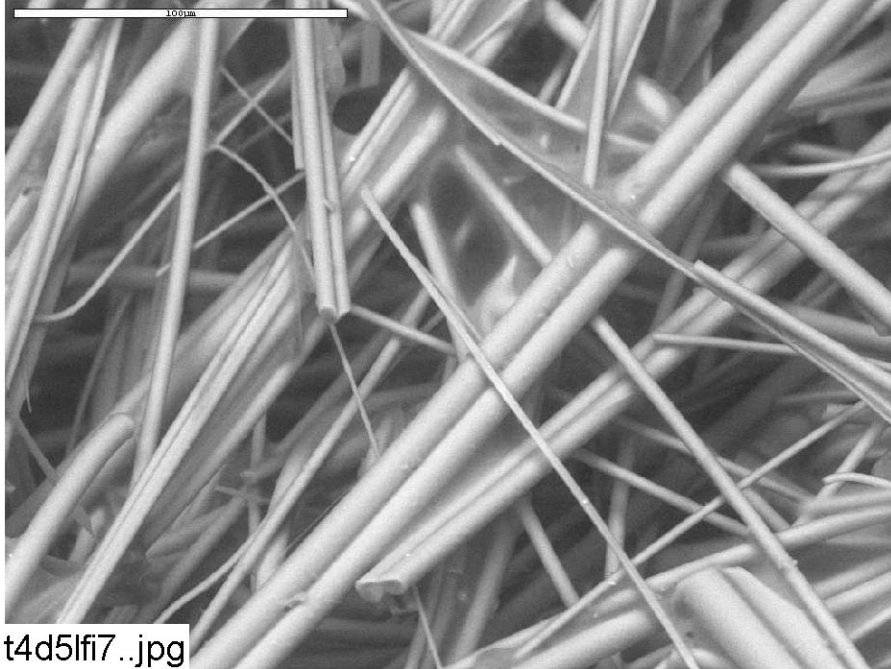


Figure A-11. Environmental SEM image magnified 500 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lf17.jpg)

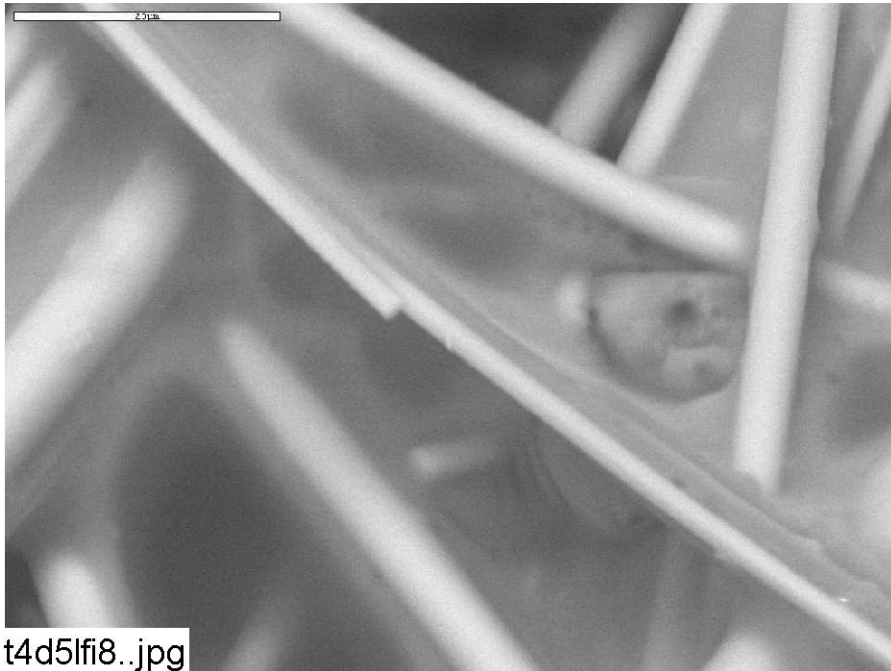


Figure A-12. Environmental SEM image magnified 2000 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lf18.jpg)

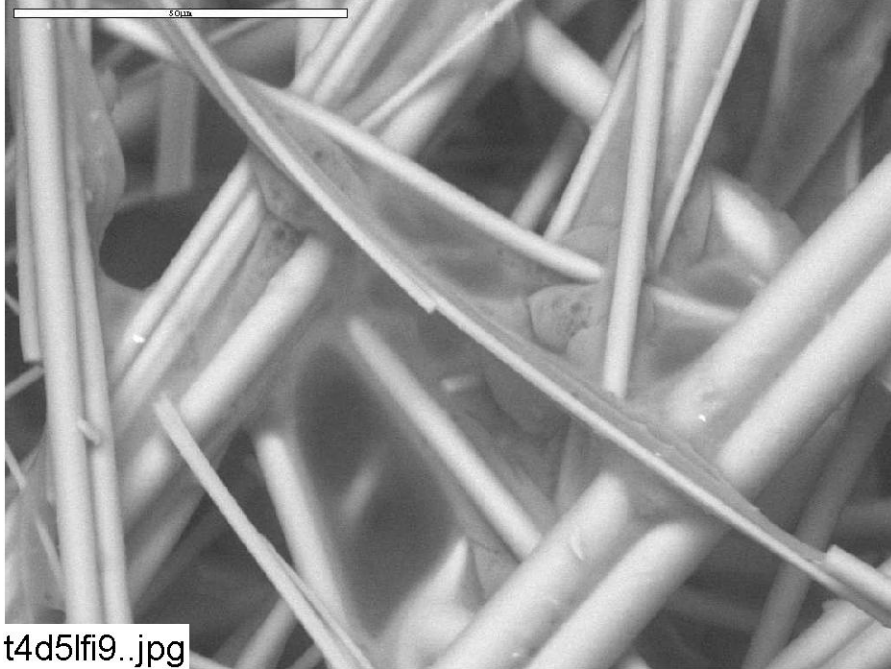


Figure A-13. Environmental SEM image magnified 1000 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5lf9.jpg)

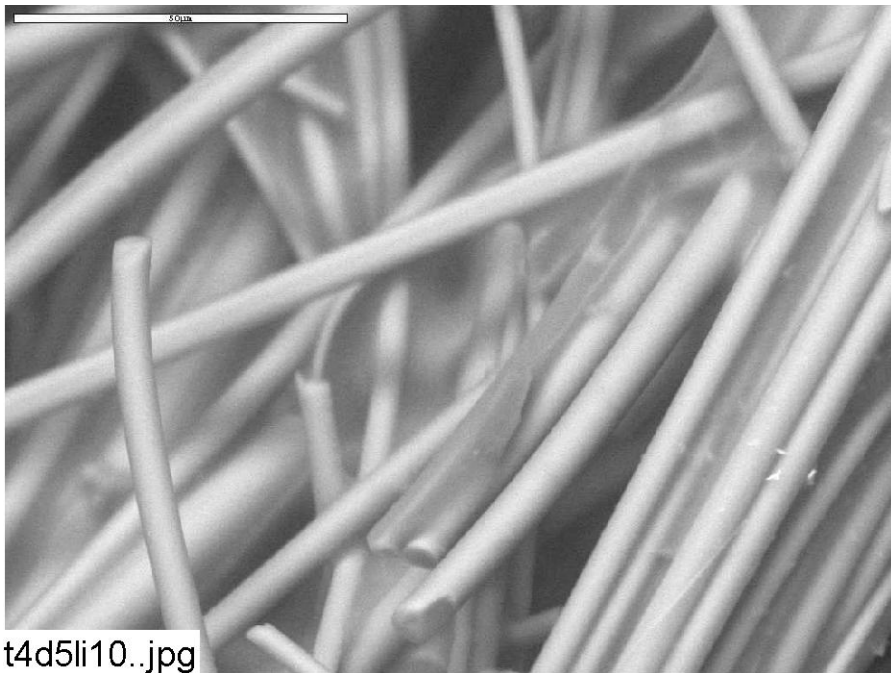


Figure A-14. Environmental SEM image magnified 1000 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5li10.jpg)

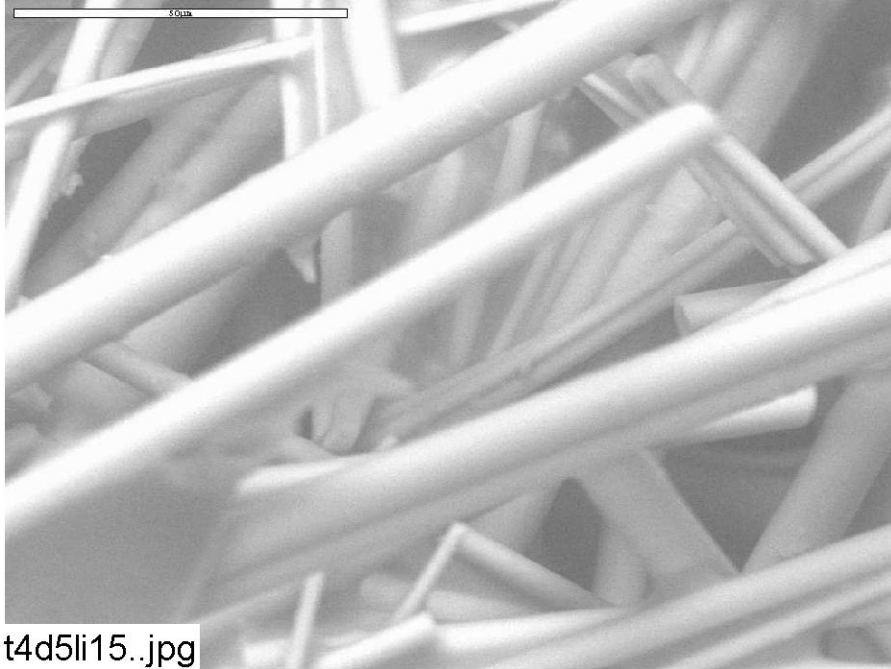


Figure A-15. Environmental SEM image magnified 500 times for a Test #4, Day-5 low-flow interior fiberglass sample. (t4d5li15)

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Appendix B1

ESEM/EDS Data for Test #4, Day-15 Fiberglass in Low-Flow Zones

Figures

- Figure B1-1. Annotated environmental SEM image magnified 100 times for a Test #4, Day-15 exterior low-flow fiberglass sample. (t4d15lx3.jpg)..... B1-4
- Figure B1-2. EDS counting spectrum for the particulate deposit on fiberglass shown in Figure B1-1. (t4d15lx2.jpg) B1-4
- Figure B1-3. Environmental SEM image magnified 500 times for a Test #4, Day-15 exterior low-flow fiberglass sample. (t4d15lx4.jpg)..... B1-5
- Figure B1-4. Environmental SEM image magnified 1000 times for a Test #4, Day-15 exterior low-flow fiberglass sample. (t4d15lx5.jpg)..... B1-5
- Figure B1-5. Environmental SEM image magnified 100 times for a Test #4, Day-15 interior low-flow fiberglass sample. (t4d15li6.jpg) B1-6
- Figure B1-6. Environmental SEM image magnified 500 times for a Test #4, Day-15 interior low-flow fiberglass sample. (t4d15li7.jpg) B1-6
- Figure B1-7. Environmental SEM image magnified 1000 times for a Test #4, Day-15 interior low-flow fiberglass sample. (t4d15li8.jpg) B1-7

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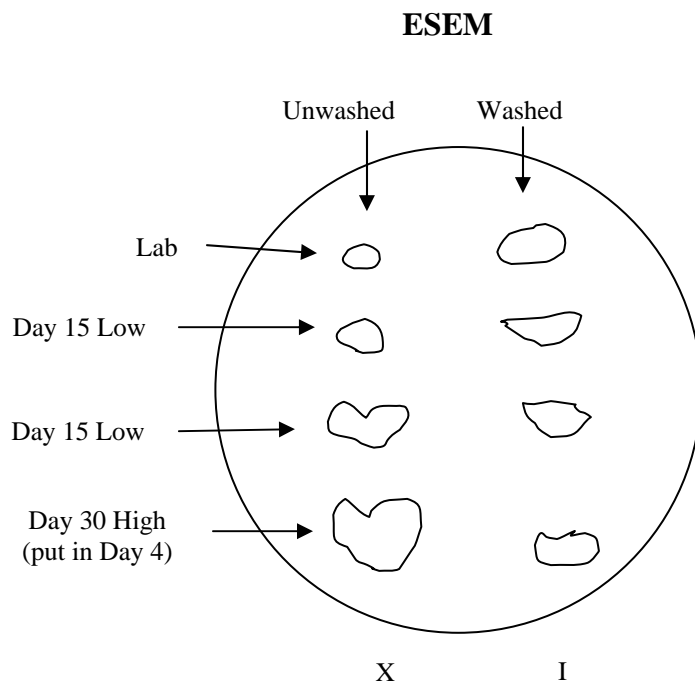
This appendix shows the ESEM/EDS results on ICET Test #4, Day-15 low-flow zone fiberglass samples. The samples were obtained on June 8, 2005 (fifteenth date for Test #4). Both exterior and interior locations of the fiberglass samples were examined. ESEM/EDS data presented here were obtained on June 23, 2005. The hydrated fiberglass samples without any coating were examined by ESEM under a low-vacuum condition (i.e., 80 Pa). EDS results provide a semi-quantitative elemental analysis of the debris attached on fiberglass.

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Transcribed Laboratory Log

Laboratory session from June 23, 2005.

Test #4, Day-15 Fiberglass In Low-Flow Zones



ESEM Fiberglass Low-Flow Exterior

| | | | | |
|--------|----------|--------|------------------------------------|-------------|
| Image: | t4d15lx3 | 100 × | ESEM annotated image | Figure B1-1 |
| EDS: | t4d15lx2 | | EDS image on particles in t4d15lx3 | Figure B1-2 |
| Image: | t4d15lx4 | 500 × | ESEM image of fiberglass | Figure B1-3 |
| | t4d15lx5 | 1000 × | ESEM image at higher magnification | Figure B1-4 |

ESEM Fiberglass Low-Flow Interior

| | | | | |
|--------|----------|--------|------------------------------------|-------------|
| Image: | t4d15li6 | 100 × | ESEM image of fiberglass | Figure B1-5 |
| | t4d15li7 | 500 × | ESEM image of fiberglass | Figure B1-6 |
| | t4d15li8 | 1000 × | ESEM image at higher magnification | Figure B1-7 |

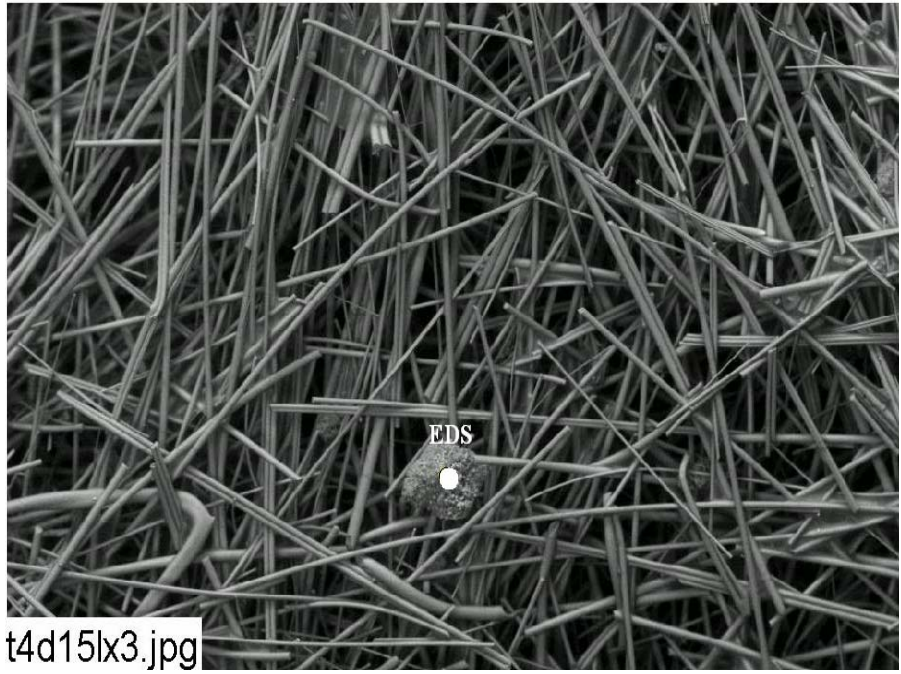


Figure B1-1. Annotated environmental SEM image magnified 100 times for a Test #4, Day-15 exterior low-flow fiberglass sample. (t4d15lx3.jpg)

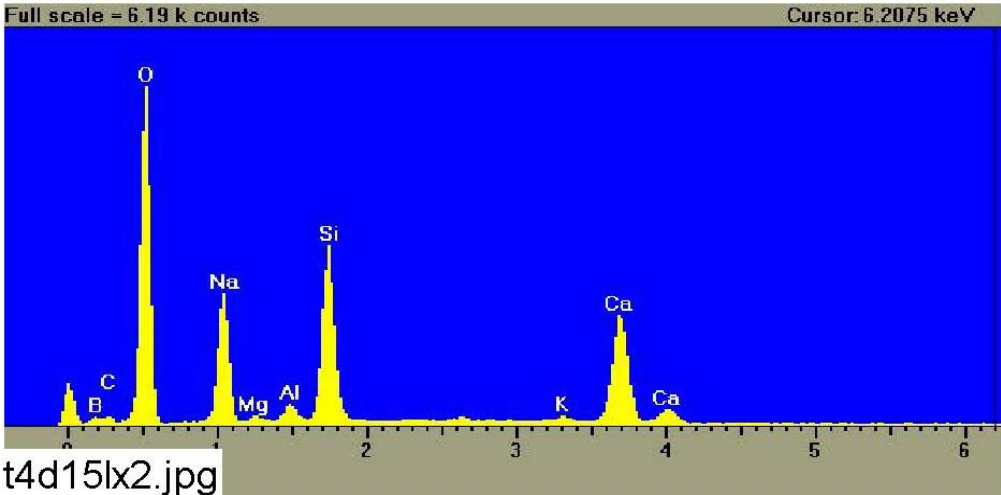


Figure B1-2. EDS counting spectrum for the particulate deposit on fiberglass shown in Figure B1-1. (t4d15lx2.jpg)

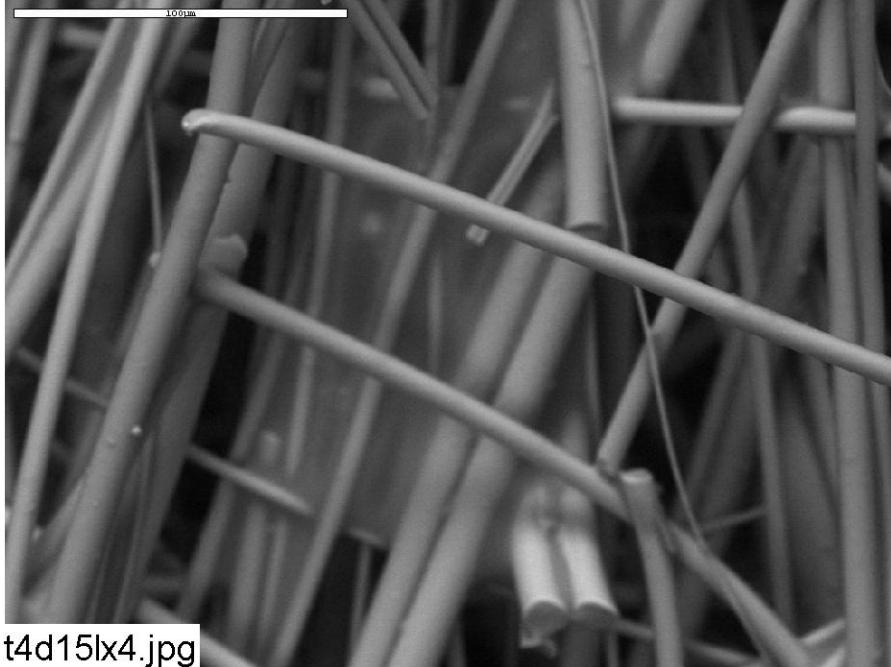


Figure B1-3. Environmental SEM image magnified 500 times for a Test #4, Day-15 exterior low-flow fiberglass sample. (t4d15lx4.jpg)

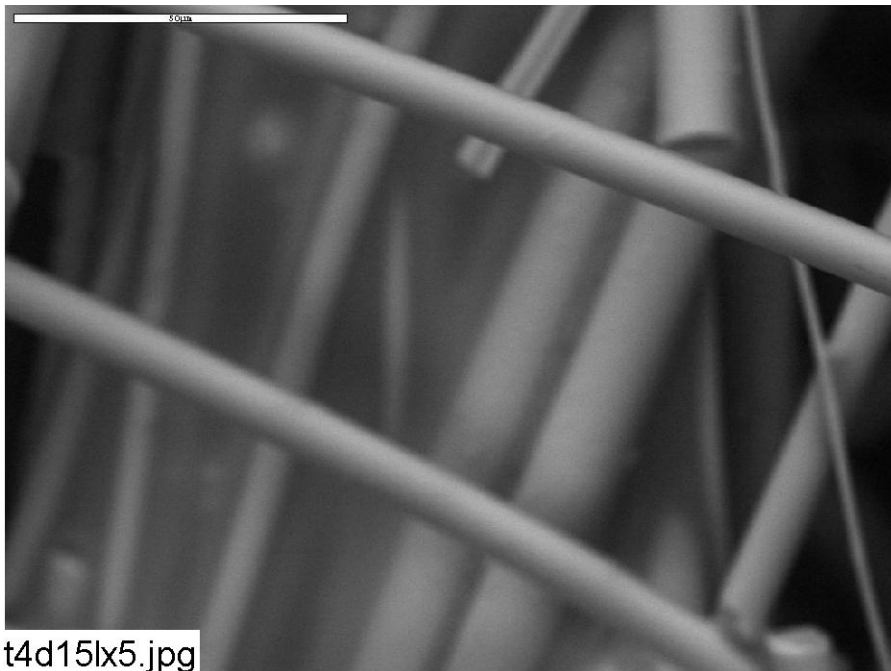


Figure B1-4. Environmental SEM image magnified 1000 times for a Test #4, Day-15 exterior low-flow fiberglass sample. (t4d15lx5.jpg)

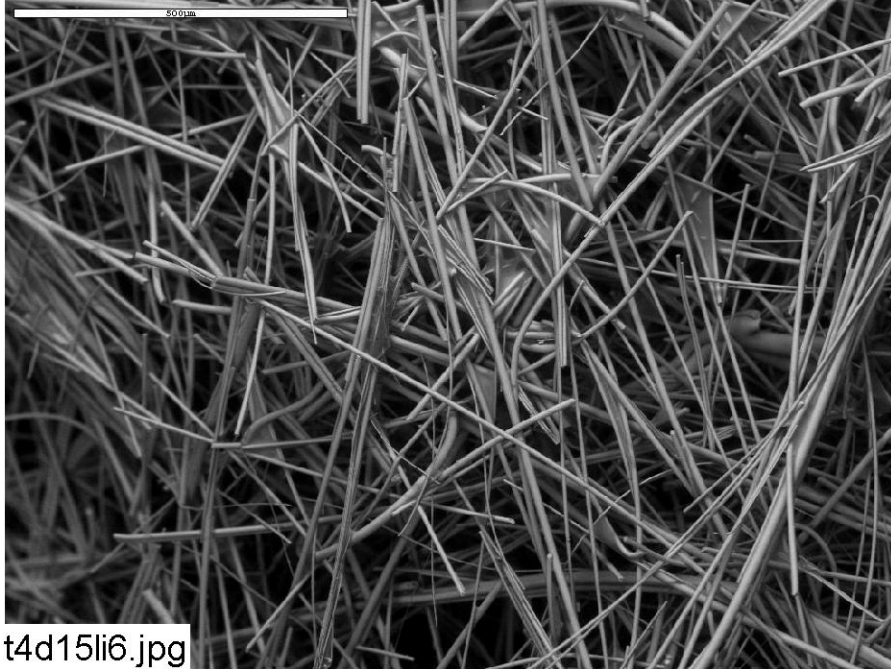


Figure B1-5. Environmental SEM image magnified 100 times for a Test #4, Day-15 interior low-flow fiberglass sample. (t4d15li6.jpg)

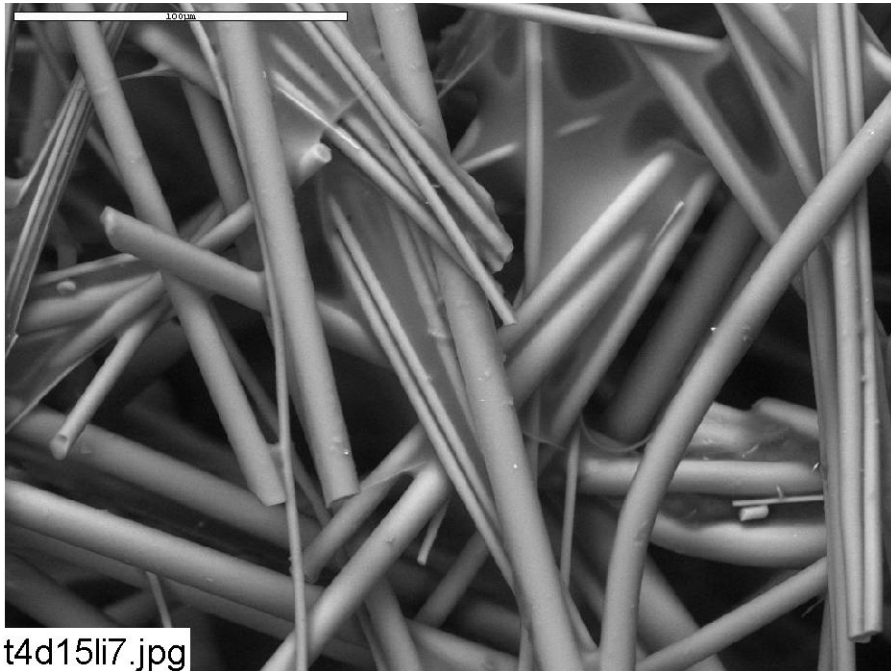


Figure B1-6. Environmental SEM image magnified 500 times for a Test #4, Day-15 interior low-flow fiberglass sample. (t4d15li7.jpg)

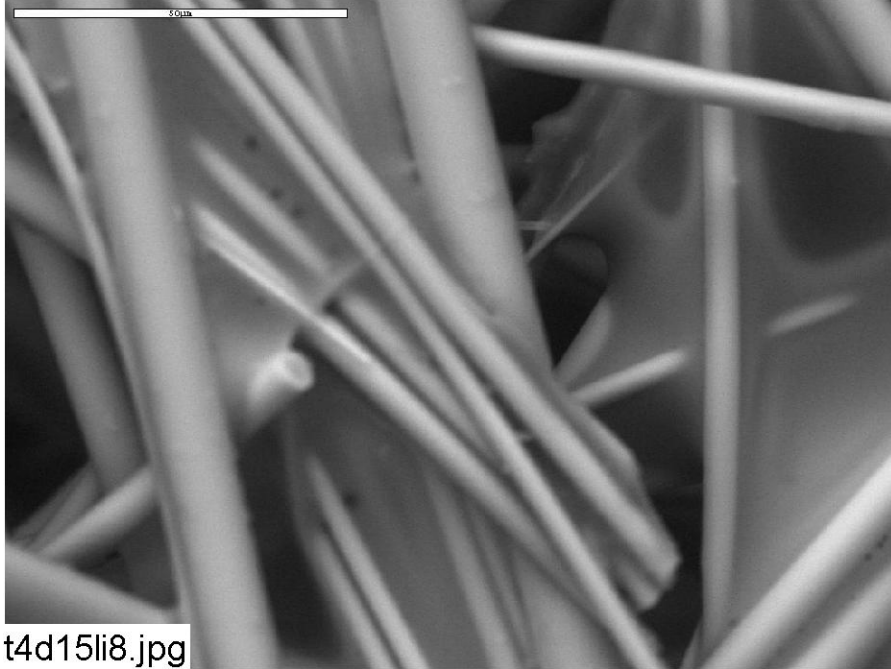


Figure B1-7. Environmental SEM image magnified 1000 times for a Test #4, Day-15 interior low-flow fiberglass sample. (t4d15li8.jpg)

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