

Appendix C

SEM/EDS Data for Test #1, Day-30 Fiberglass and Filtrate

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Test #1, Day-30 samples that were examined on December 21, 2004 (the termination date for T1), include filter papers obtained from (1) high-volume filtration extracted at the sample tap, (2) high-volume filtration performed outside of the loop under vacuum, (3) sacrificial fiberglass from the interior of a large blanket, and (4) white precipitate filtered from the T1D21 water sample. In addition to the standard SEM/EDS data, optical microscopy images were taken in both diffuse and plane-polarized light of the high-volume filtrate obtained under vacuum (items 1 and 2 above).

Optical microscopy was used to examine the Day-21 water samples using both plane-polarized and cross-polarized light. Different levels of information can be obtained in plane-polarized light (analyzer out of the optical path) or with crossed polarizers (analyzer inserted into the optical path). Observations in plane-polarized light reveal details of the optical relief of the specimen, which are manifest in the “visibility” of boundaries and which increase with the increase of the refractive index across these boundaries. Differences in the refractive indices of the mounting adhesive and the specimen determine the extent to which light is scattered as it emerges from the uneven specimen surface. Materials with high relief, which appear to stand out from the image, have refractive indices that are appreciably different from that of the sample mount.

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Transcribed Laboratory Log

Optical microscope plane-polarized photographs

#1 Day-21 precipitation from "filtered" water sample

Frame		I.D.	PIC	×	Description
1			PIC00001	10	scattered dry fragments
2			PIC00002	10	desiccated fragments
3			PIC00003	50	single fragment with contraction dome
4			PIC00004	50	similar fragment at adjacent area
5			PIC00005	10	general cracking pattern
6			PIC00006	50	edge of previous area as in PIC00005 larger cracks in the interior and finer cracks near the edge

General Observations: Filters show no crystal diffraction pattern.
Samples may have been disturbed.

#2 High-volume #1 46-ml sample

Frame		I.D.	PIC	×	Description
7			PIC00007	2	general desiccated fragments
8			PIC00008	5	general desiccated fragments
9			PIC00009	5	same field with cross polarized filters showing some evidence of crystal highlights
10			PIC00010	5	same field with different exposure time
11			PIC00011	10	similar contraction bubbles on desiccated fragments
11b			PIC00012	50	zoom in on one of the contraction bubbles

#3 High-volume #2 1-l @ pressure

Frame		I.D.	PIC	×	Description
12			PIC00013	5	general desiccated fragments
13			PIC00014	10	same field as PIC00013 with higher resolution
14			PIC00015	50	same field as PIC00013 and PIC00014, some structure but no clear evidence of crystals
14a			PIC00016	50	cross polarized to see structure around edges of dry flakes
14b			PIC00017	50	cross polarized to see structure around edges of dry flakes with different exposure from PIC00016
15			PIC00018	5	anomalous dendrites with polarizers (not saved)
16			PIC00019	2	anomalous dendrites with polarizers (not saved)

Day-30 SEM Samples

Sample I.D.	#1	T1D30 high-vol. 1, taken in line 46 ml over 2.5 hours
	#2	T1D30 high-vol. 2, taken in lab under pressure, ~1 l (not analyzed because of similarity with Sample #1 and time constraints of the laboratory session)
	#3	T1D30 sacrificial fiberglass from interior of the large blanket Sample from dark crusty perimeter of a lump
	#4	T1D30 sacrificial fiberglass from perimeter of a lump This sample is also taken from perimeter of a lump, and a 1-mm-diam “tiny” white crystal on the surface is included in the sample
	#5	T1D30 sacrificial fiber from interior of larger blanket, internal flock from the clump; very little color change compared with raw fiber
	#6	T1D30 control sample of fiber glass that has not been exposed to solution (data not saved)
	#7	T1D30 filtered precipitate sample from Day 30

Note: Samples all were prepared together and then submitted for sputtering with gold/palladium alloy ~20 nm (nominal thickness). These sample identification (I.D.) numbers serve as the first digit in four-character fields that define the series of images and spectra obtained for each sample type. For example, image #1006 refers to the sixth sequential image taken during this lab session, and it was taken from sample I.D. #1.

SEM Analysis (added coating to avoid charging; thickness of the coating approximately 25 nm)

Operator: Adrian Brearley

File: Day-30 sample #1

Image:	Day-30 #1001	40 ×	on string of fragments
	Day-30 #1002	90 ×	string of blocky fragments
	Day-30 #1003	100 ×	string of blocky fragments
	Day-30 #1004	250 ×	close-up of fragments
	Day-30 #1005	500 ×	close-up of particles
	Day-30 #1006	1500 ×	surface of fragments
EDS:	1-1006		fragment in image 1006 showing Na, Al, O ₂ , Ca
	1-1006a		adjacent particles
	1-1006b		20-s exposure time—lower Na
	1-1006c		60-s scan for the same spot
Image:	Day-30 #1007	40 ×	filtrate on carbon-film substrate
	Day-30 #1008	100 ×	close-up of filtrate on carbon-film substrate
	Day-30 #1009	250 ×	close-up of filtrate on carbon-film substrate
EDS:	1-1009		carbon-film substrate in image 1009
Image:	Day-30 #1010	50 ×	area of filtrate
	Day-30 #1011	100 ×	area of filtrate
	Day-30 #1012	250 ×	area of filtrate
	Day-30 #1013	500 ×	area of filtrate
EDS:	1-1013		analysis of filtrate on carbon-film substrate
	1-1013a		analysis of filtrate on carbon-film substrate
Image:	Day 30 #1014	500 ×	block fragment on carbon-film substrate
EDS:	1-1014		EDS on particulate
Image:	Day-30 #1015	2000 ×	close-up of fracture surface

File: Day-30 sample #3

Image:	Day-30 #3001	50 ×	fiberglass with surface coating
	Day-30 #3002	100 ×	fiberglass with surface coating
	Day-30 #3003	250 ×	close-up of surface coating
EDS:	3003		spectrum of fractured coating (Na, B, O)
Image:	Day-30 #3004	500 ×	close-up of fractured surface
	Day-30 #3005	100 ×	fiber with film
	Day-30 #3006	250 ×	films across fiber
	Day-30 #3007	500 ×	films across fiber
EDS:	3007a		analysis of film
Image:	Day-30 #3008	450 ×	particles on film
EDS:	3008a		analysis of particles
	3008b		analysis of fiber

Image:	Day-30 #3009	100 ×	group of fibers
	Day-30 #3010	250 ×	close-up of film
EDS:	3010a		spectrum of precipitate on film
	3011		film (not saved)

File: Day-30 sample #4

Image:	Day-30 #4001	40 ×	fiber cluster with film
	Day-30 #4002	100 ×	fiber cluster with film
	Day-30 #4003	250 ×	fiber close-up
	Day-30 #4004	500 ×	fiber close-up
EDS:	4004		on fiber showing Na, Al, Si, and B (not saved)
Image:	Day-30 #4005	50 ×	cluster of fibers
	Day-30 #4006	100 ×	close-up
	Day-30 #4007	200 ×	close-up of surface precipitates
	Day-30 #4008	350 ×	surface precipitates
EDS:	4008		spectrum of thin film between fibers (not saved)
	4008a		spectrum of fractured precipitate
	4008b		spectrum of fiber

File: Day-30 sample #5

Image:	Day-30 #5001	40 ×	fiberglass
	Day-30 #5002	100 ×	close-up of fiberglass—charging badly
	Day-30 #5003	100 ×	close-up of fibers
	Day-30 #5004	250 ×	fibers with precipitate on surface
EDS:	5004a		spectrum of fiber with precipitates

File: Day-30 sample #6—fiberglass control

Image:	Day-30 #6001	45 ×	fiberglass cluster (not saved)
	Day-30 #6002	100 ×	fiberglass cluster (not saved)
	Day-30 #6003	270 ×	close-up of fiberglass cluster (not saved)
EDS:	6003		analysis of fiber (not saved)
Image:	Day-30 #6004	1500 ×	close-up of fiberglass cluster (not saved)

File: Day-30 sample #7—precipitate from high-volume filter

Image:	Day-30 #7001	40 ×	precipitate layer
	Day-30 #7002	100 ×	precipitate layer
	Day-30 #7003	250 ×	close-up of surface
	Day-30 #7004	500 ×	close-up of surface
EDS:	7004		just oxygen and B (not saved)

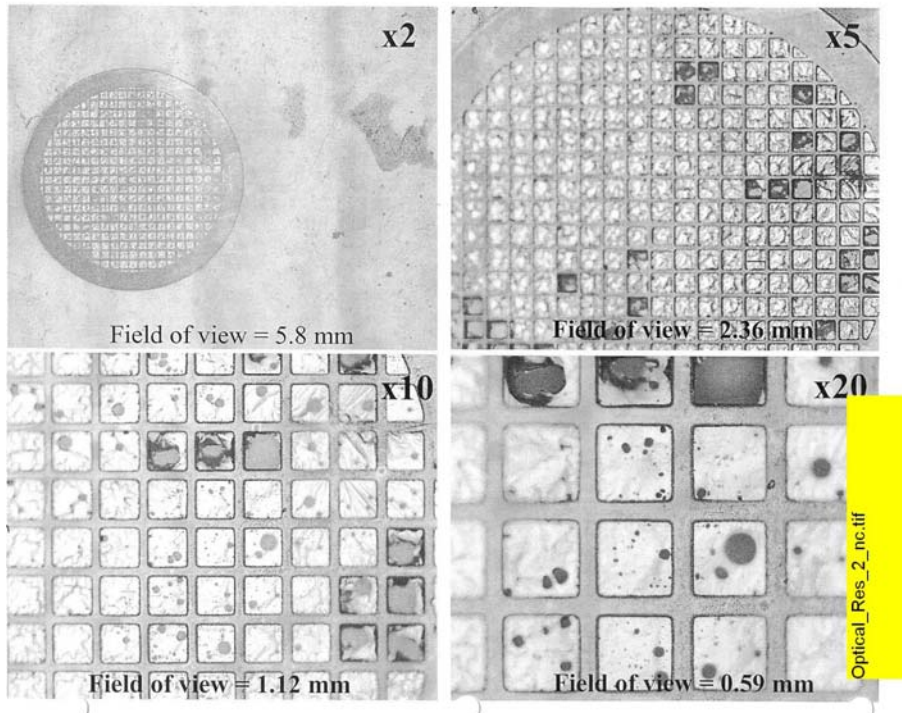


Figure C-1. Spatial resolution reference image for optical microscopy (1 of 2). Field of view refers to the length of the frame in the direction of the text label.

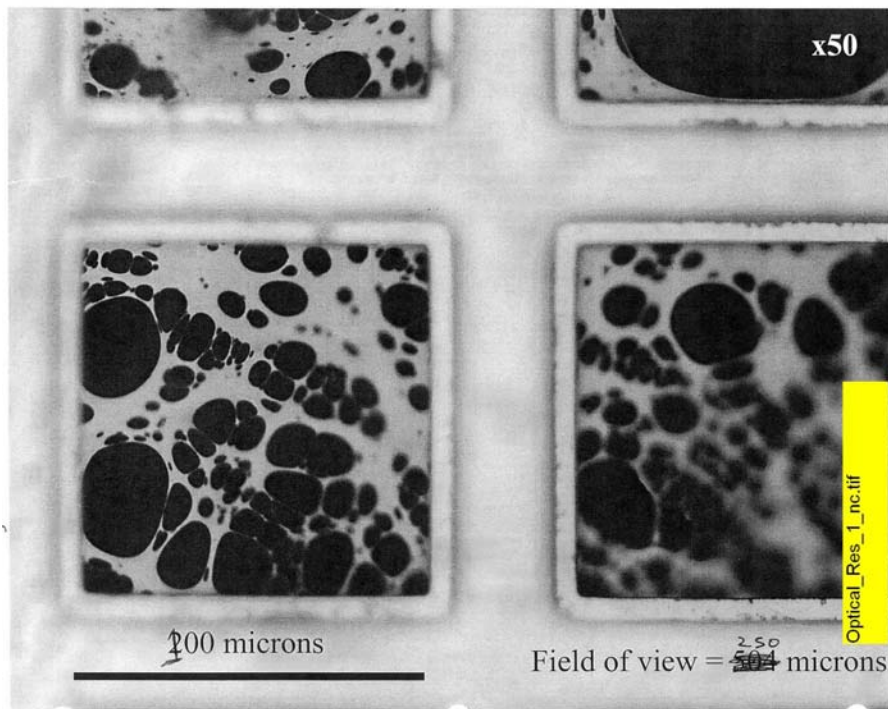


Figure C-2. Spatial resolution reference image (2 of 2). Field of view refers to the length of the respective quadrant in the direction of the text label.



Figure C-3. Plane-polarized optical microscope photograph (PIC00001) for the Day-21 precipitation from the filtered water sample magnified 10 times on the scattered dry fragments.



Figure C-4. Plane-polarized optical microscope photograph (PIC00002) for the Day-21 precipitation from the filtered water sample magnified 10 times on the desiccated fragments.



Figure C-5. Plane-polarized optical microscope photograph (PIC00003) for the Day-21 precipitation from the filtered water sample magnified 50 times on the single fragment with contraction dome, as shown in Figure C-4.

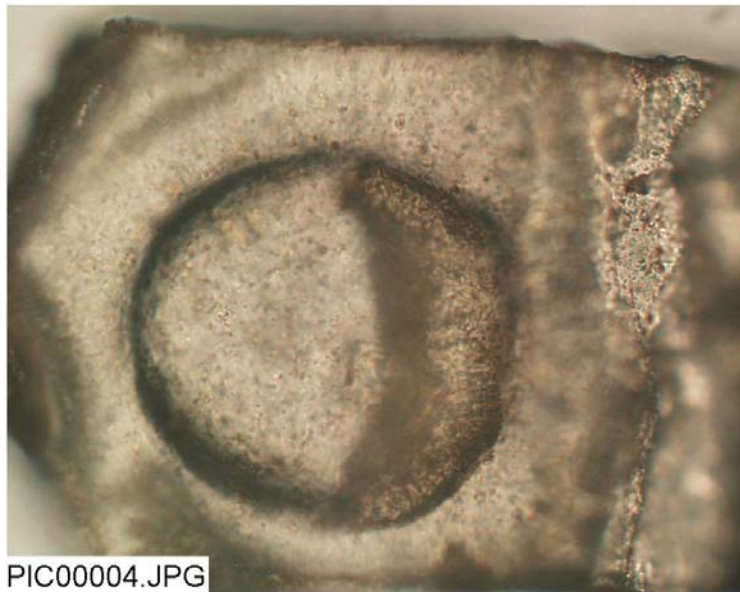


Figure C-6. Plane-polarized optical microscope photograph (PIC00004) for the Day-21 precipitation from the filtered water sample magnified 50 times on the similar adjacent single fragment, as shown in Figure C-4.



Figure C-7. Plane-polarized optical microscope photograph (PIC00005) for the Day-21 precipitation from the filtered water sample magnified 10 times on the dry fragments, showing the general cracking pattern.



Figure C-8. Plane-polarized optical microscope photograph (PIC00006) for the Day-21 precipitation from the filtered water sample magnified 50 times on the desiccated fragments at the edge of the area in Figure C-7, showing larger cracks in the interior and finer cracks near the edge.



Figure C-9. Plane-polarized optical microscope photograph (PIC00007) for the Day-21 precipitation from the 46-ml high-volume filter magnified 2 times on the desiccated fragments.

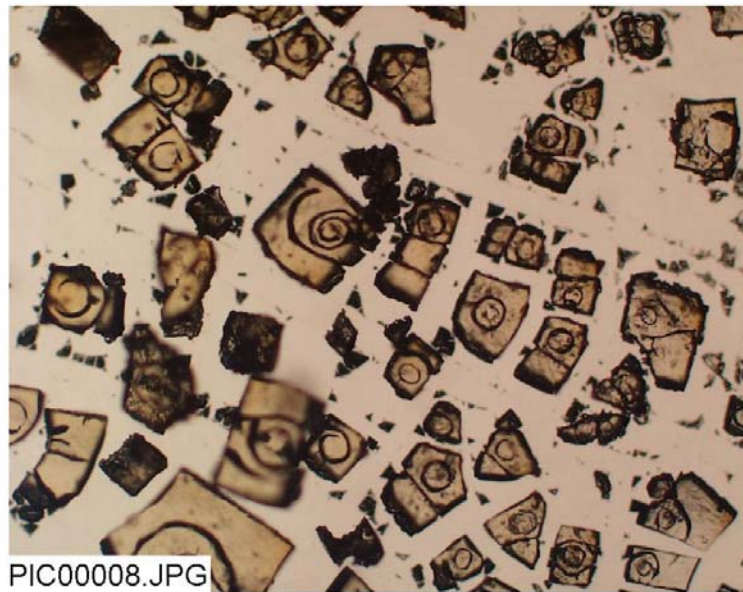


Figure C-10. Plane-polarized optical microscope photograph (PIC00008) for the Day-21 precipitation from the 46-ml high-volume filter magnified 5 times on the desiccated fragments.

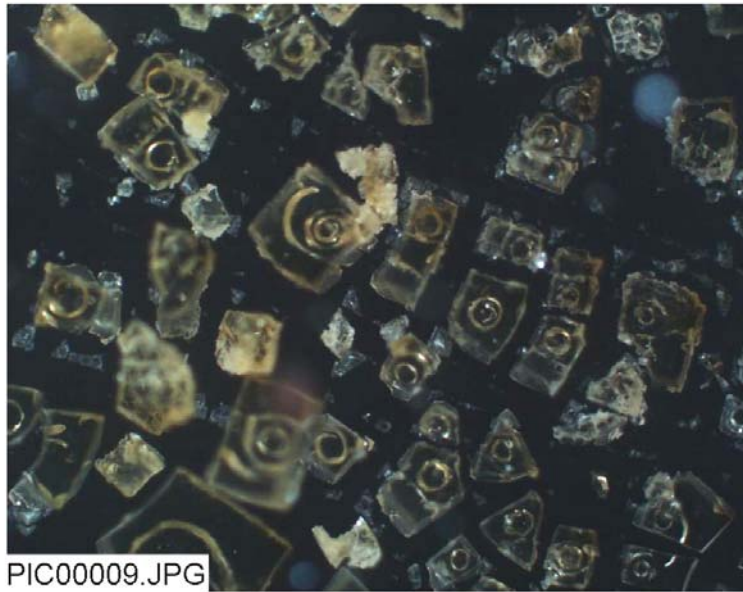


Figure C-11. Cross-polarized optical microscope photograph (PIC00009) of the same field shown in Figure C-10, showing some evidence of crystal highlights in the Day-21 precipitation from the 46-ml high-volume filter; magnified 5 times on the desiccated fragments.

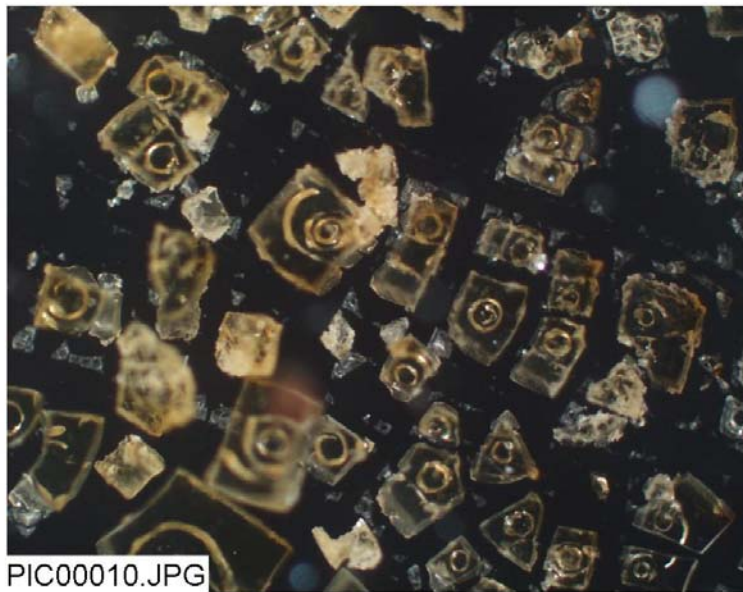


Figure C-12. Optical microscope photograph (PIC00010) with cross polar filters for the Day-21 precipitation from the 46-ml high-volume filter using a different exposure time from Figure C-11.

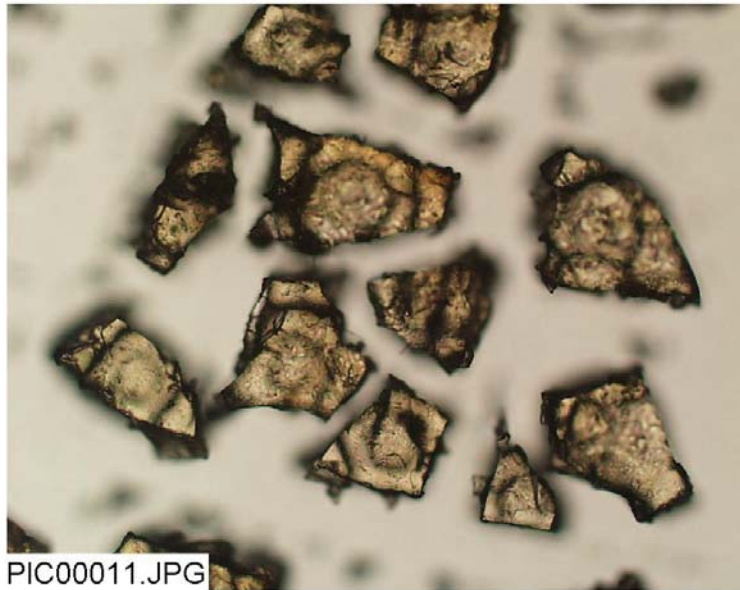


Figure C-13. Plane-polarized optical microscope photograph (PIC00011) for the Day-21 precipitation from the 46-ml high-volume filter magnified 10 times, showing contraction bubbles on the desiccated fragments.

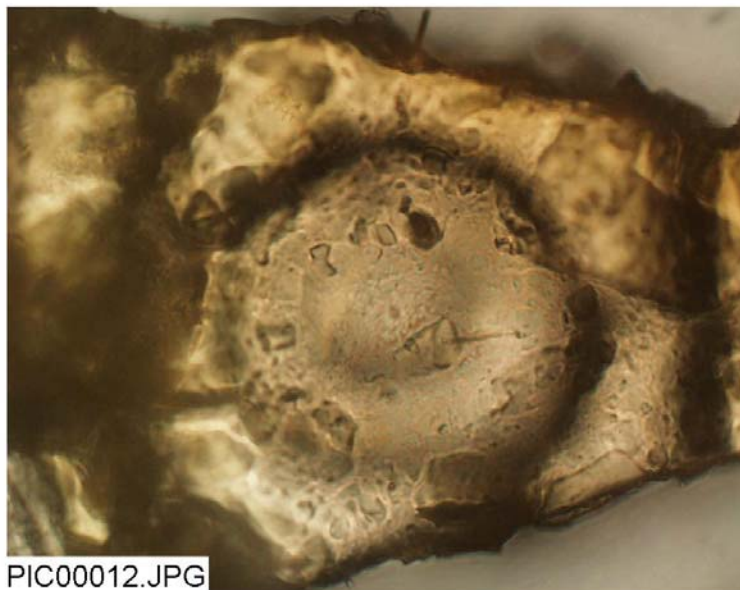


Figure C-14. Plane-polarized optical microscope photograph (PIC00012) for the Day-21 precipitation from the 46-ml high-volume filter magnified 50 times on a single desiccated fragment, showing contraction bubble.

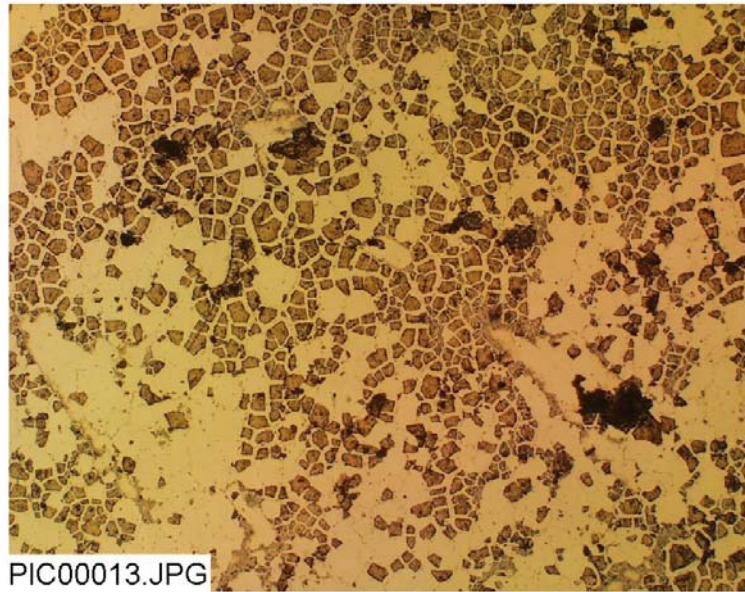


Figure C-15. Plane-polarized optical microscope photograph (PIC00013) for the Day-21 precipitation from the 1-l high-volume filter magnified 5 times on the desiccated fragments.

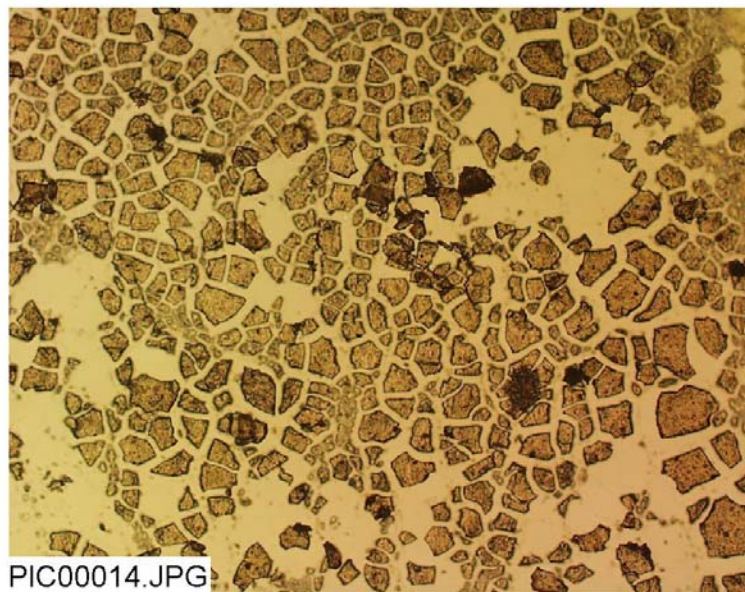


Figure C-16. Plane-polarized optical microscope photograph (PIC00014) for the Day-21 precipitation from the 1-l high-volume filter magnified 10 times on the same desiccated fragments as shown in Figure C-15.

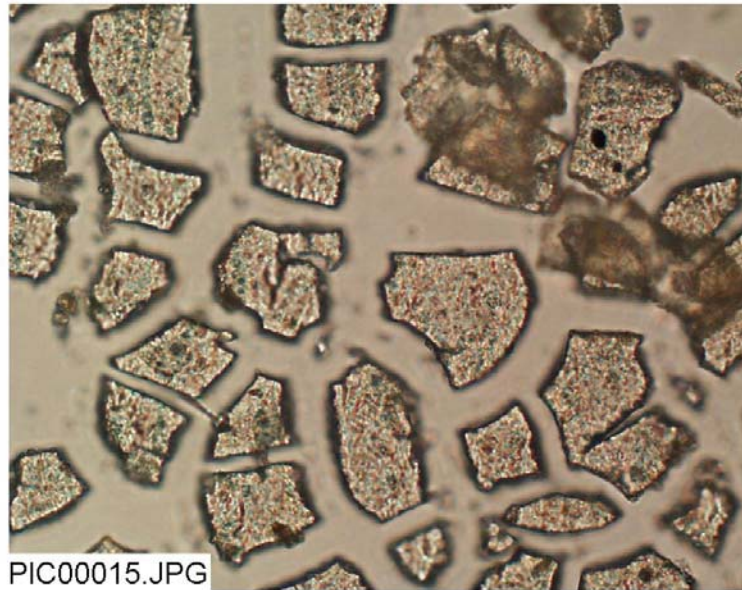


Figure C-17. Plane-polarized optical microscope photograph (PIC00015) for the Day-21 precipitation from the 1-l high-volume filter magnified 50 times on the desiccated fragments, as shown in Figure C-15.



Figure C-18. Cross-polarized optical microscope photograph (PIC00016) for the Day-21 precipitation from the 1-l high-volume filter magnified 50 times to see structure on edges of the dry flakes.



Figure C-19. Filtered optical microscope photograph (PIC00017) with a different exposure time from Figure C-18 for the Day-21 precipitation from the 1-l high-volume filter magnified 50 times on the dry flakes.

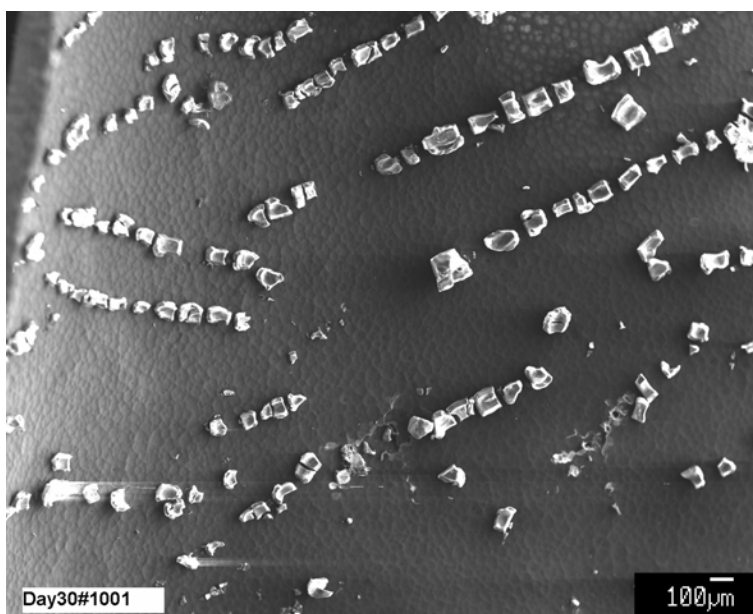


Figure C-20. Day-30, sample #1 SEM image (#1001) magnified 40 times on the string of blocky fragments.

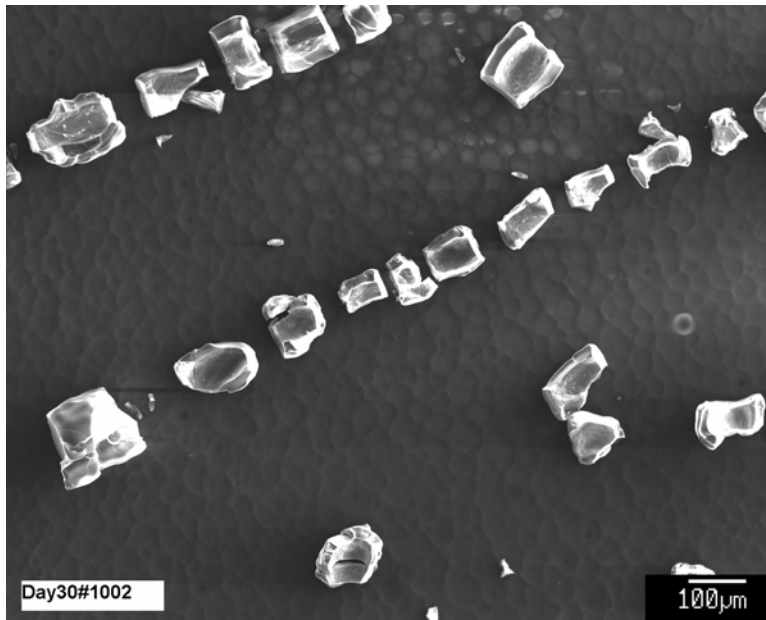


Figure C-21. Day-30, sample #1 SEM image (#1002) magnified 90 times on the string of blocky fragments.

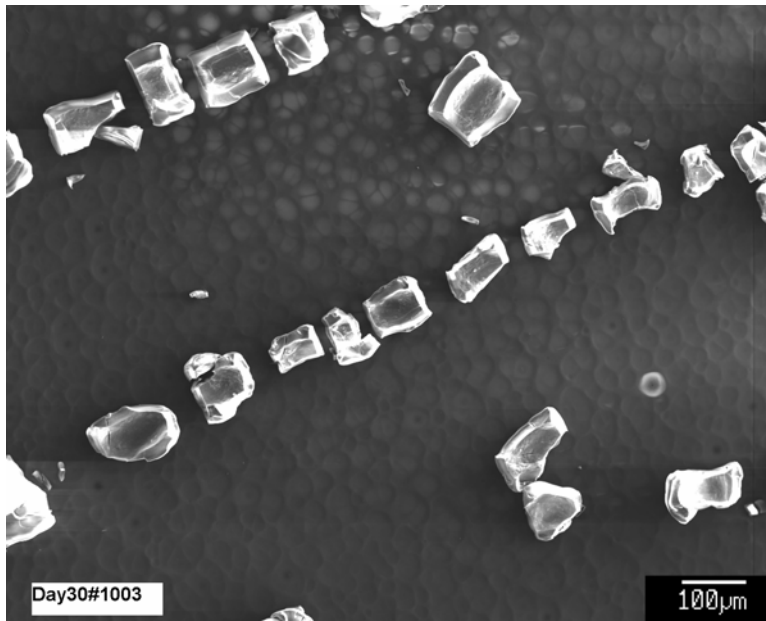


Figure C-22. Day-30, sample #1 SEM image (#1003) magnified 100 times on the string of blocky fragments.

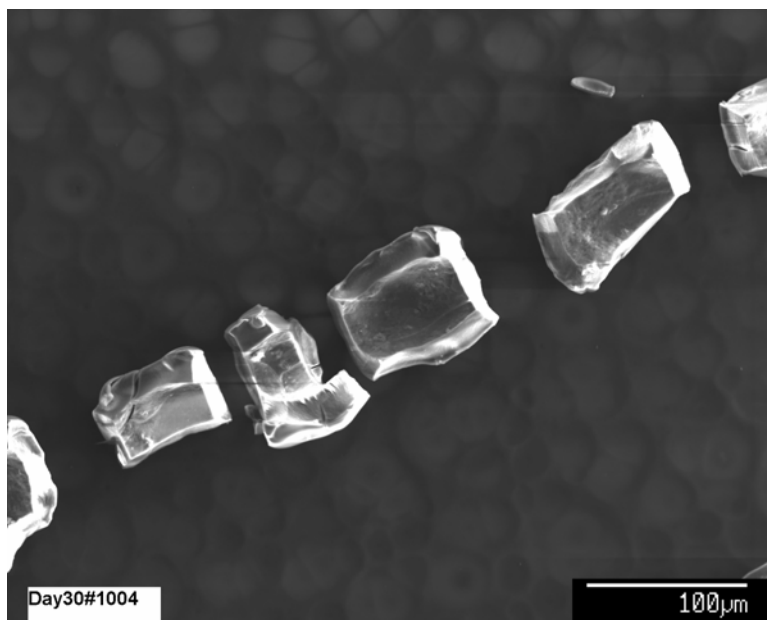


Figure C-23. Day-30, sample #1 SEM image (#1004) magnified 250 times; close-up of the fragments.

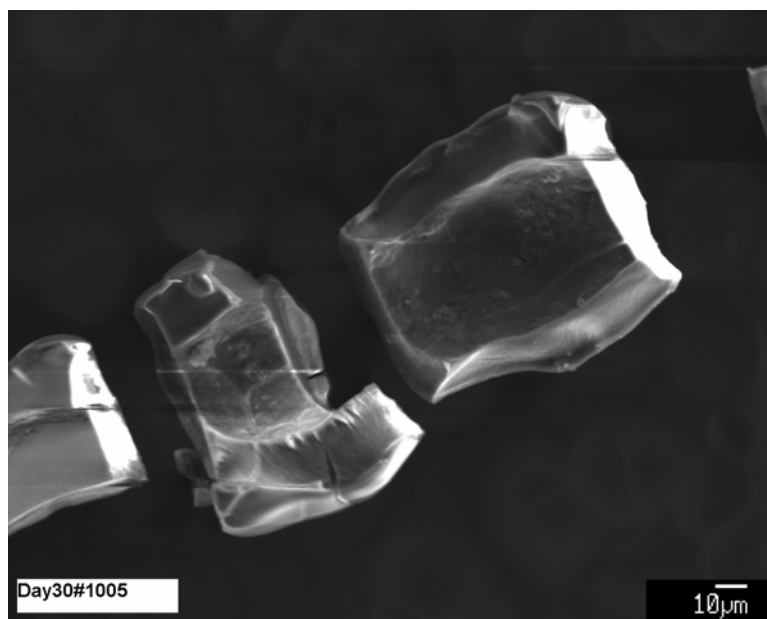


Figure C-24. Day-30, sample #1 SEM image (#1005) magnified 500 times; close-up of the fragments.

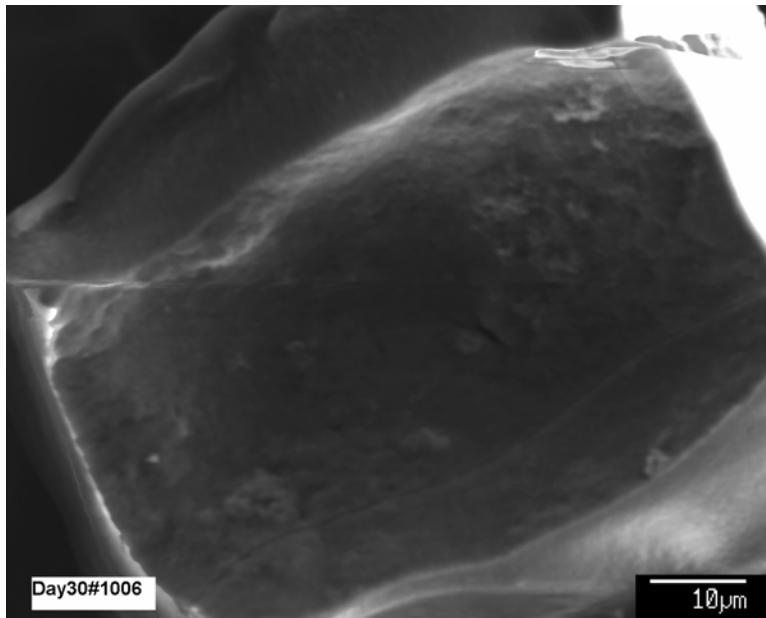


Figure C-25. Day-30, sample #1 SEM image (#1006) magnified 1500 times on the surface of a fragment.

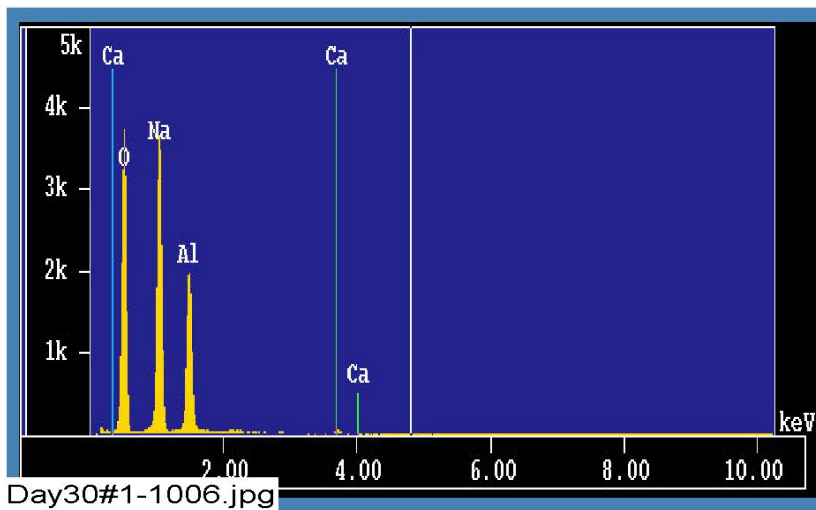


Figure C-26. Day-30, sample #1 counting spectrum (EDS 1-1006) for the fragment in Figure C-25 with a composition of Na, Al, O₂, and Ca.

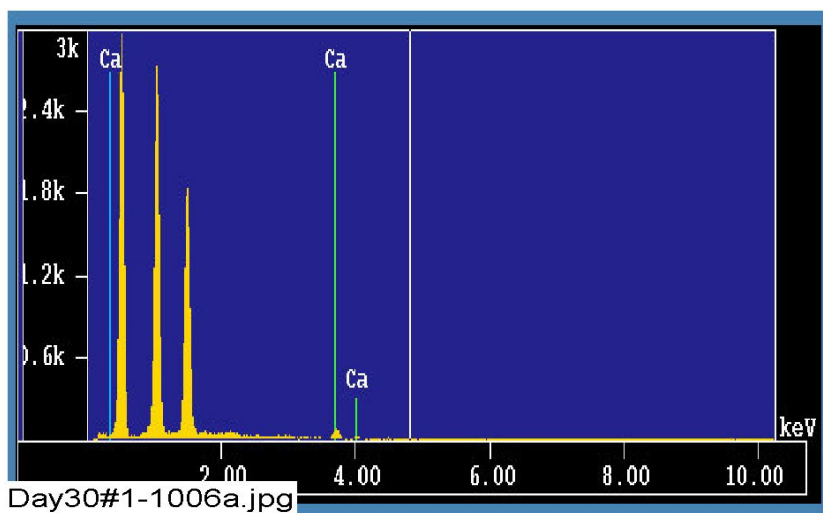


Figure C-27. Day-30, sample #1 counting spectrum (EDS 1-1006a) for the adjacent particles compared with that in Figure C-25 with a composition of Na, Al, O₂, and Ca.

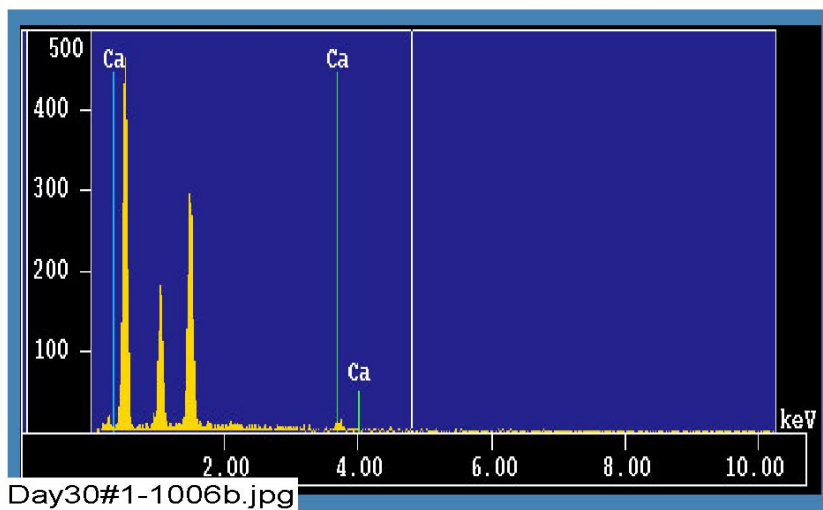


Figure C-28. Day-30, sample #1 counting spectrum (EDS 1-1006b).

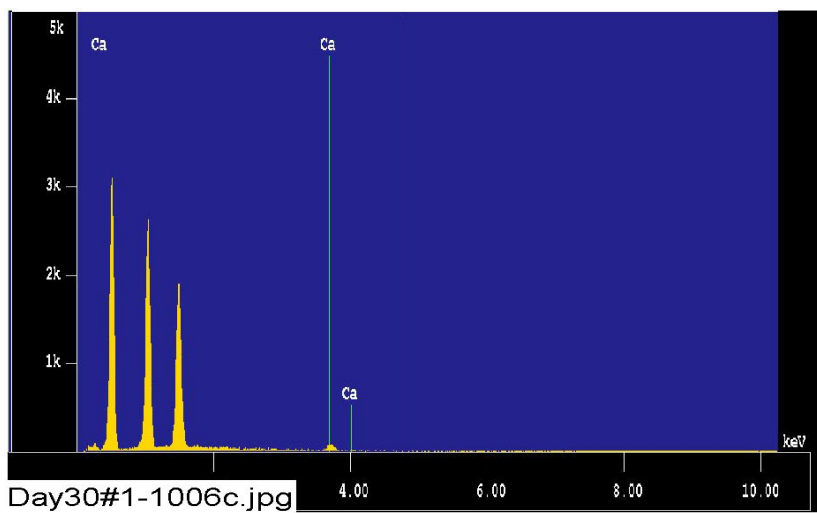


Figure C-29. Day-30, sample #1 counting spectrum (EDS 1-1006c).

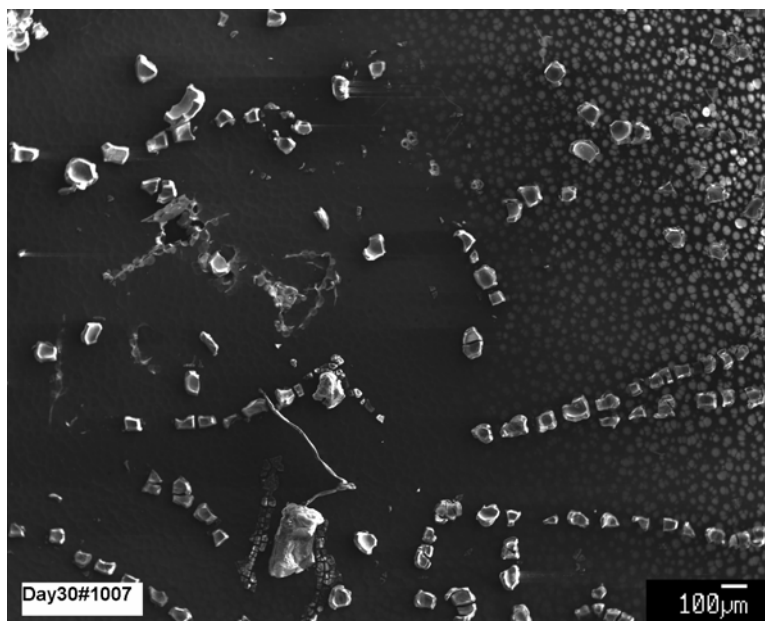


Figure C-30. Day-30, sample #1 SEM image (#1007) magnified 40 times of filtrate on carbon film substrate.

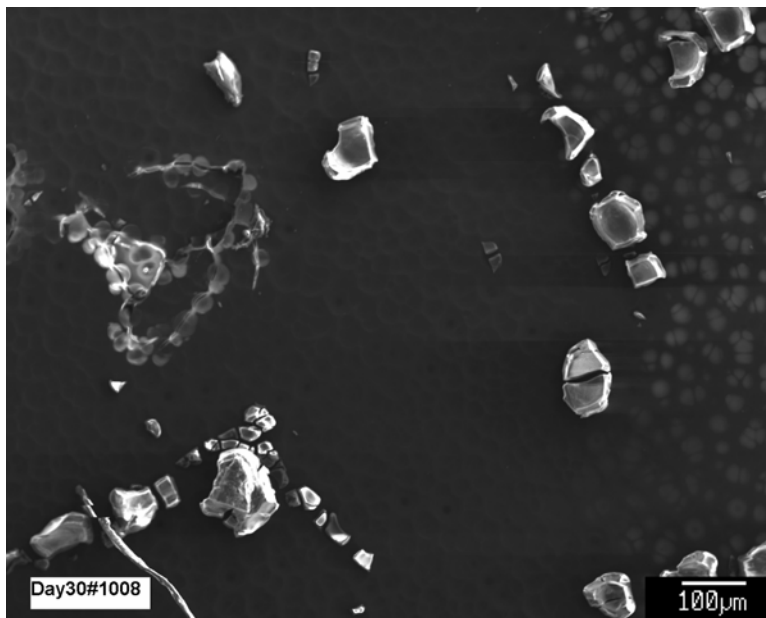


Figure C-31. Day-30, sample #1 SEM image (#1008) magnified 100 times; close-up of filtrate on carbon film substrate, as shown in Figure C-30.

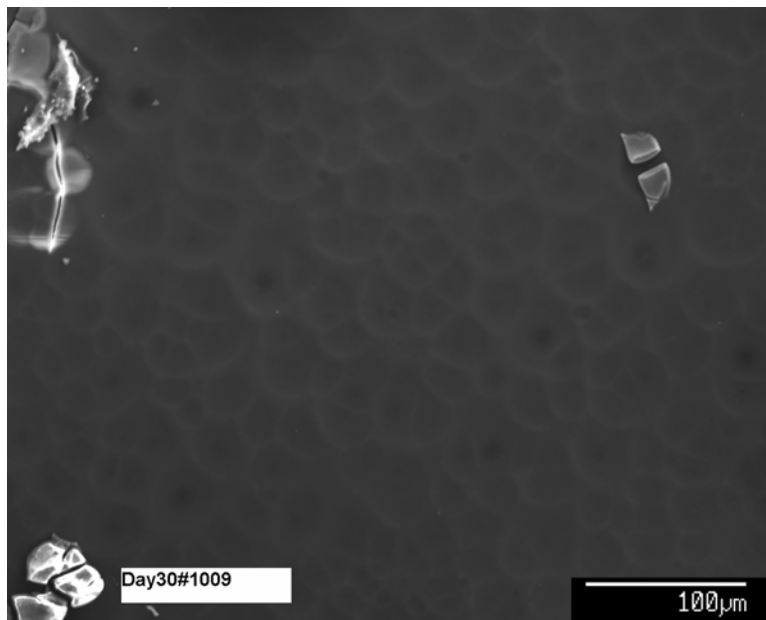


Figure C-32. Day-30, sample #1 SEM image (#1009) magnified 250 times; close-up of the carbon-film substrate, as shown in Figure C-31.

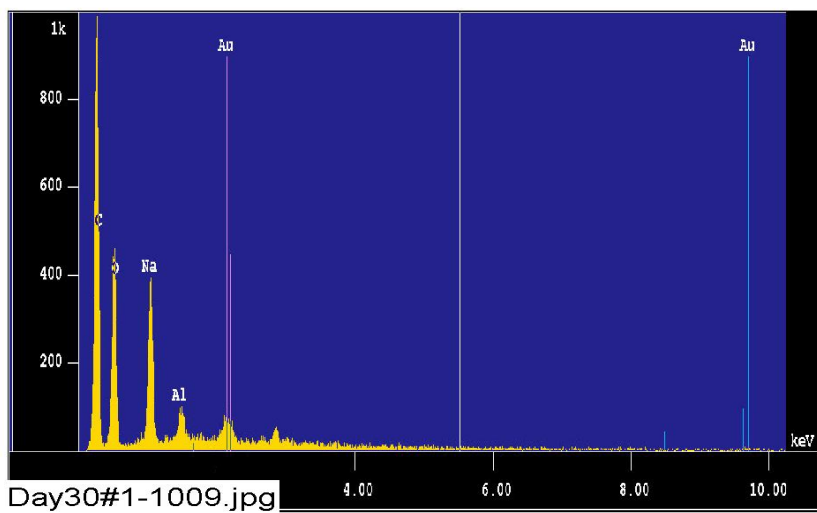


Figure C-33. Day-30, sample #1 counting spectrum (EDS 1-1009) for the carbon-film substrate, as shown in Figure C-32.

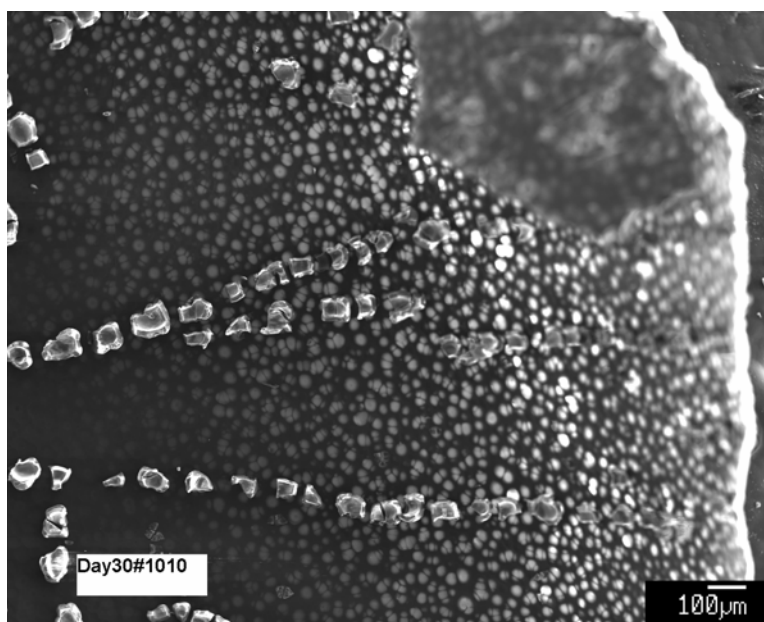


Figure C-34. Day-30, sample #1 SEM image (#1010) magnified 50 times on one area of the filtrate.

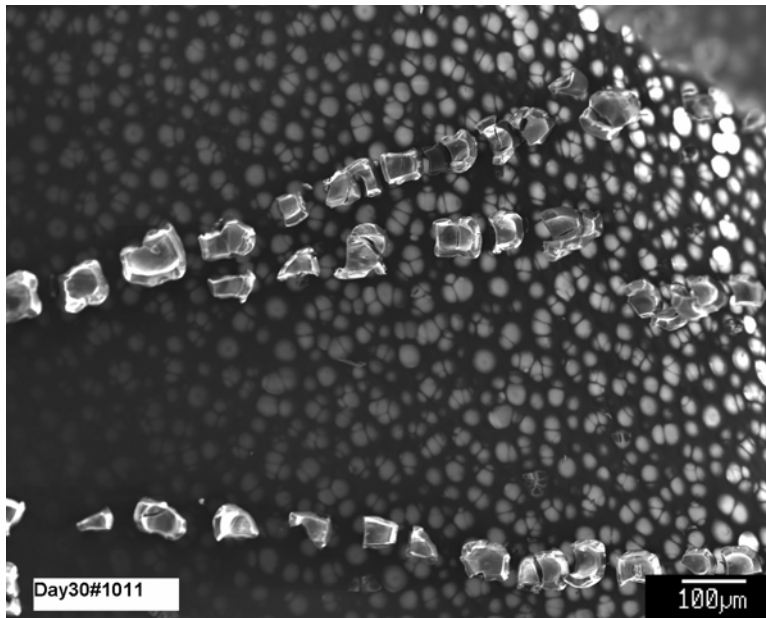


Figure C-35. Day-30, sample #1 SEM image (#1011) magnified 100 times on one area of the filtrate.

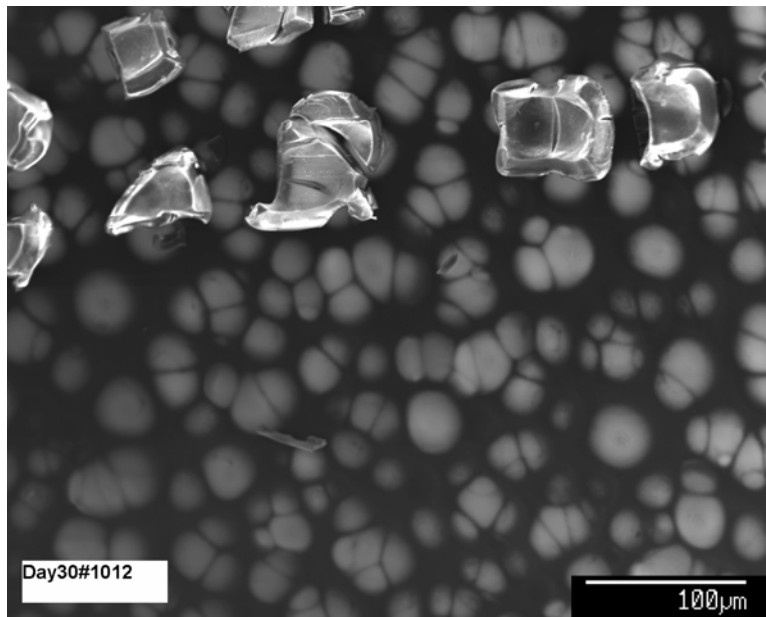


Figure C-36. Day-30, sample #1 SEM image (#1012) magnified 250 times on one area of the filtrate.

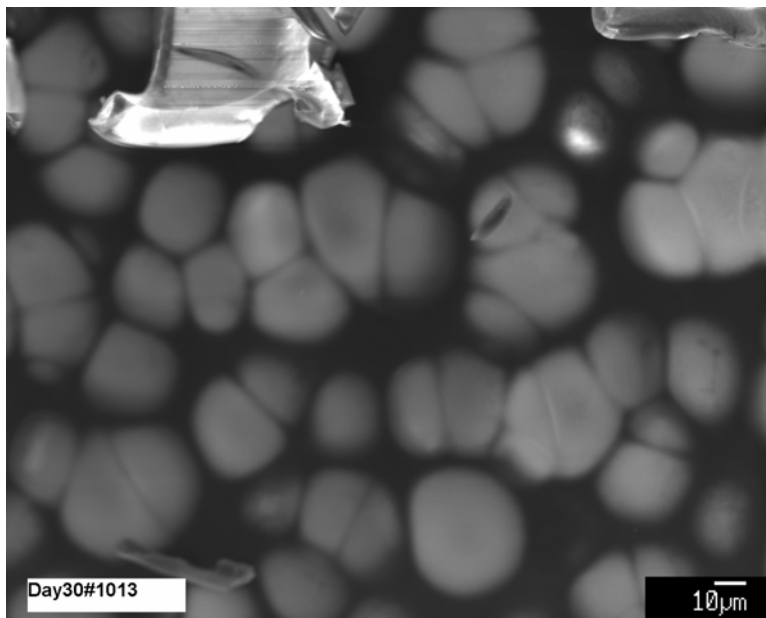


Figure C-37. Day-30, sample #1 SEM image (#1013) magnified 500 times on one area of the filtrate.

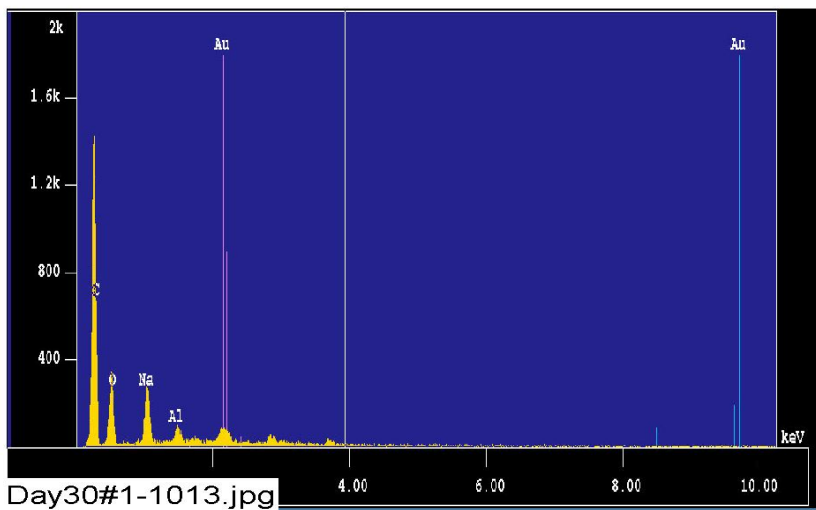


Figure C-38. Day-30, sample #1 counting spectrum (EDS 1-1013) for the filtrate on the carbon-film substrate, as shown in Figure C-37.

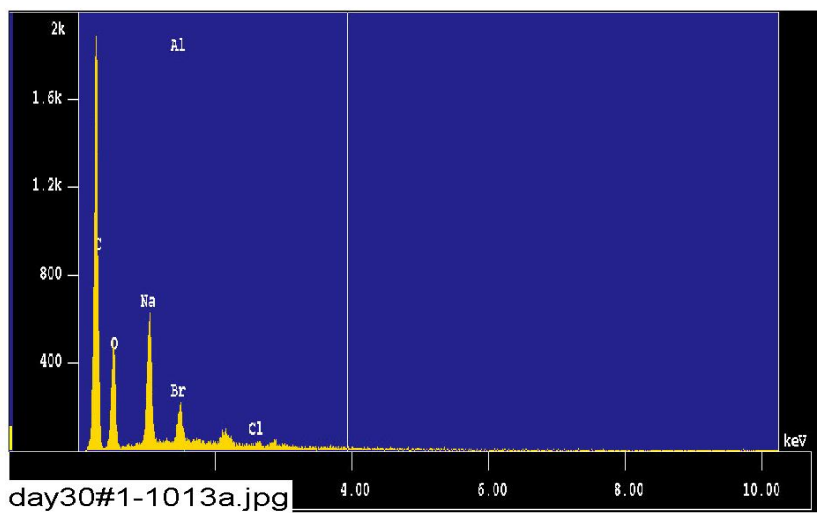


Figure C-39. Day-30, sample #1 counting spectrum (EDS 1-1013a) for the filtrate on the carbon-film substrate, as shown in Figure C-37.

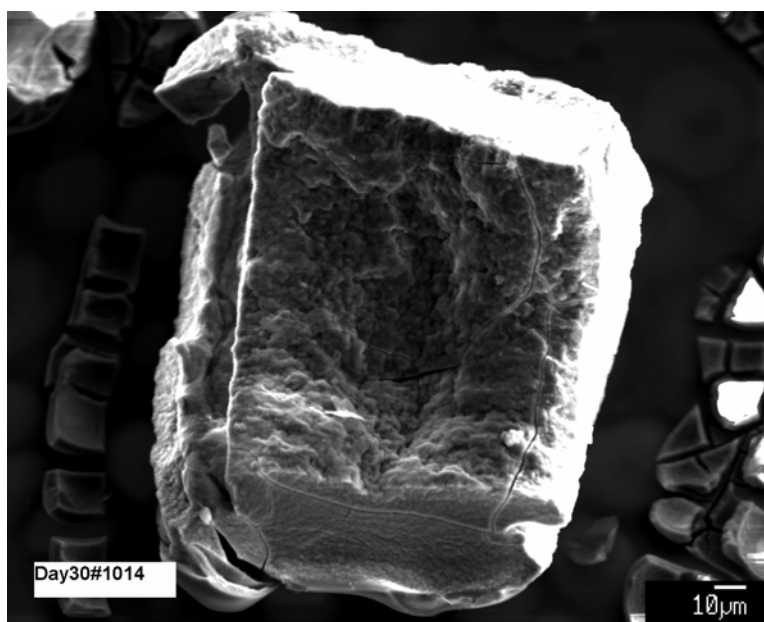


Figure C-40. Day-30, sample #1 SEM image (#1014) magnified 500 times on the block filtrate on the carbon-film substrate.

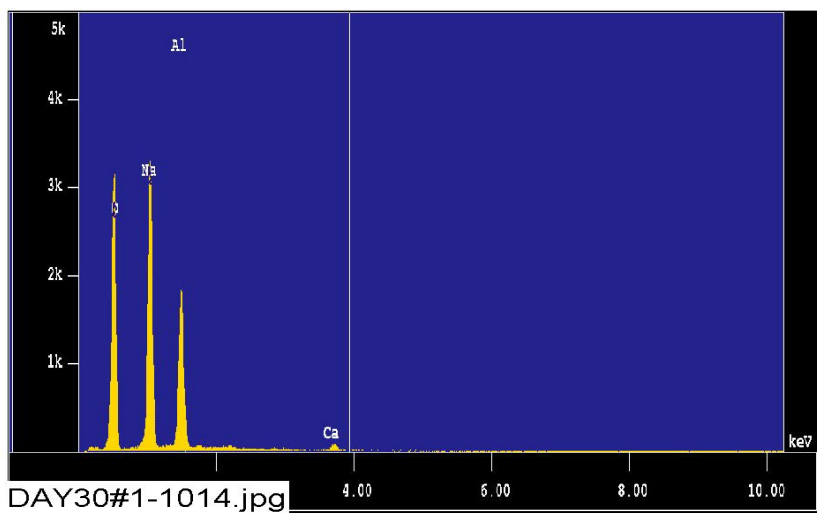


Figure C-41. Day-30, sample #1 counting spectrum (EDS 1-1014) on the particle, as shown in Figure C-40.

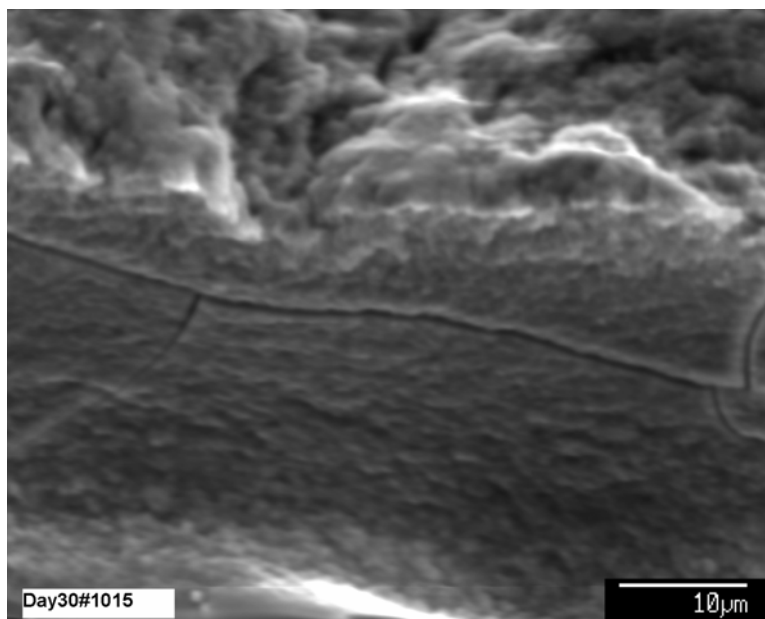


Figure C-42. Day-30, sample #1 SEM image (#1015) magnified 2000 times, close-up of the fracture surface, as shown in Figure C-40.

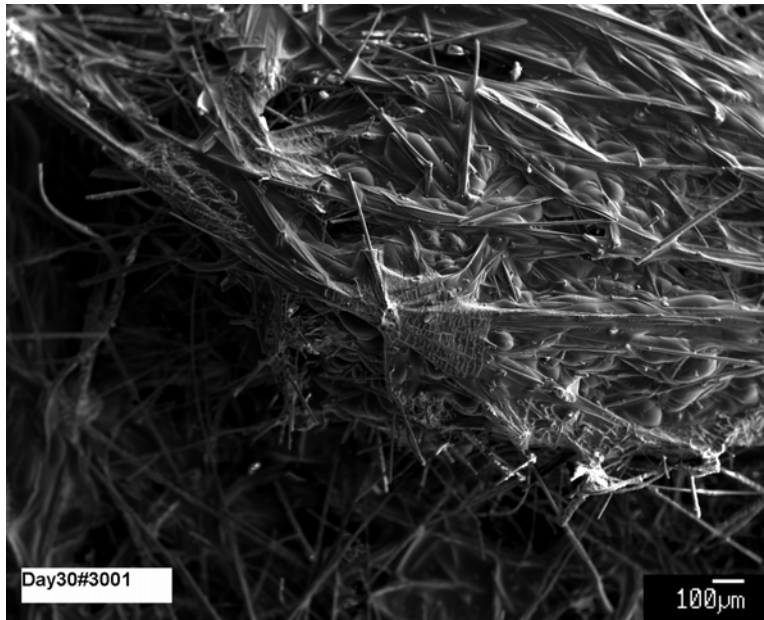


Figure C-43. Day-30, sample #3 SEM image (#3001) magnified 50 times on the fiberglass with surface coating.

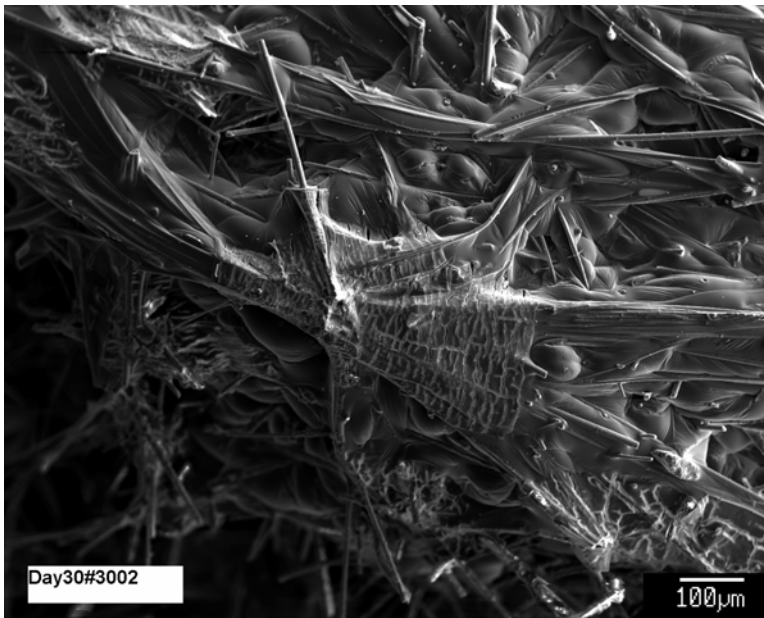


Figure C-44. Day-30, sample #3 SEM image (#3002) magnified 100 times on the fiberglass with surface coating.

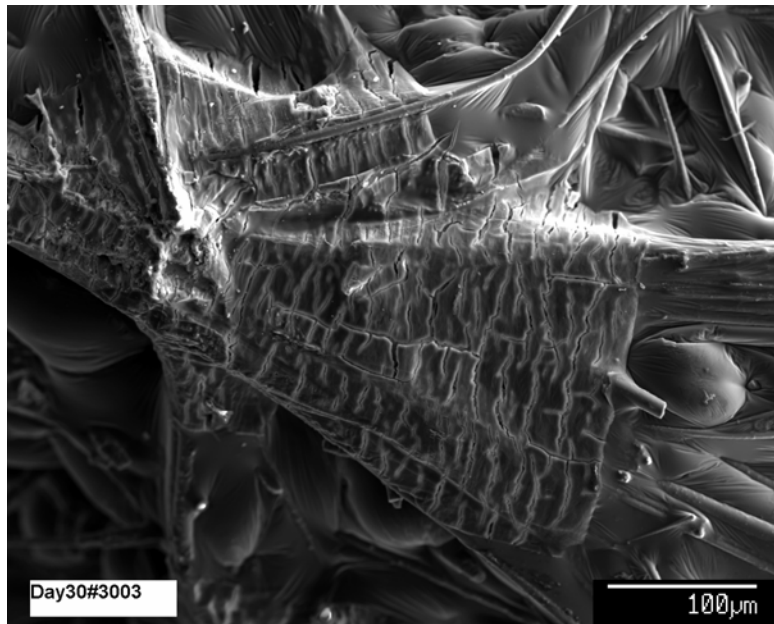


Figure C-45. Day-30, sample #3 SEM image (#3003) magnified 250 times on the fiberglass with surface coating.

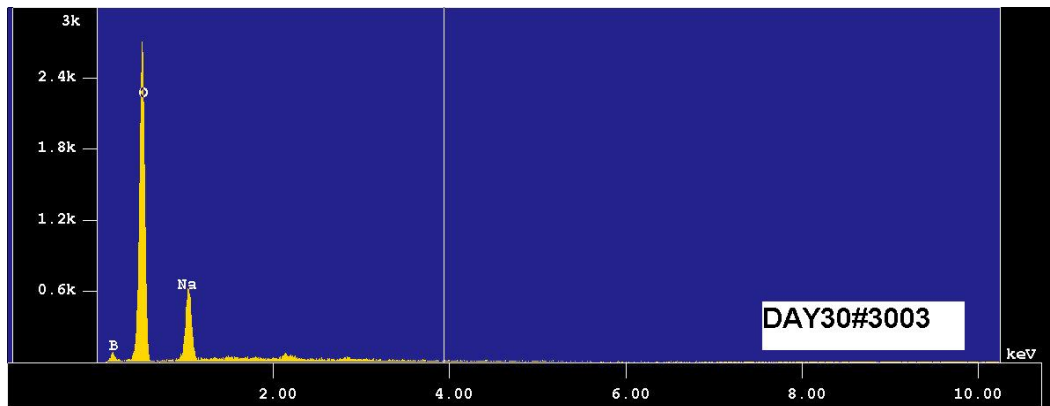


Figure C-46. Day-30, sample #3 counting spectrum (EDS #3003) of the fractured coating, as shown in Figure C-45.

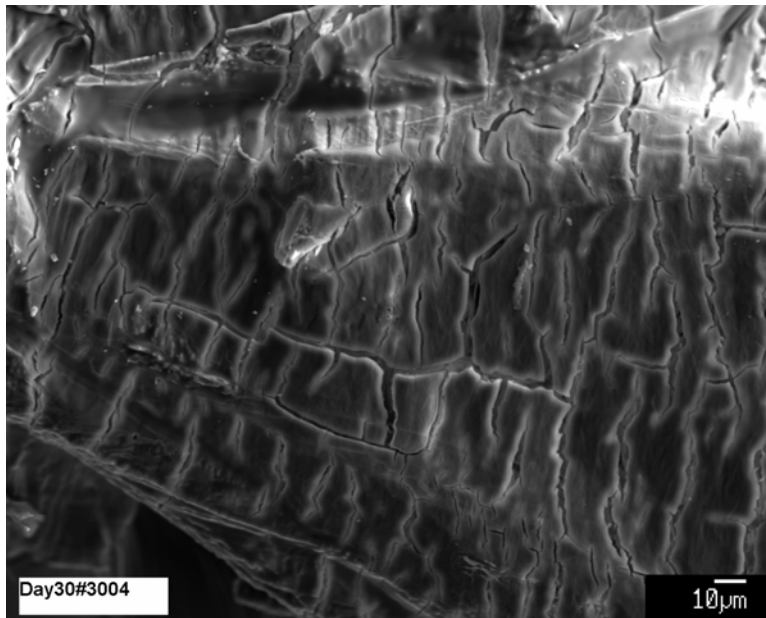


Figure C-47. Day-30, sample #3 SEM image (#3004) magnified 500 times; close-up of the fractured surface.

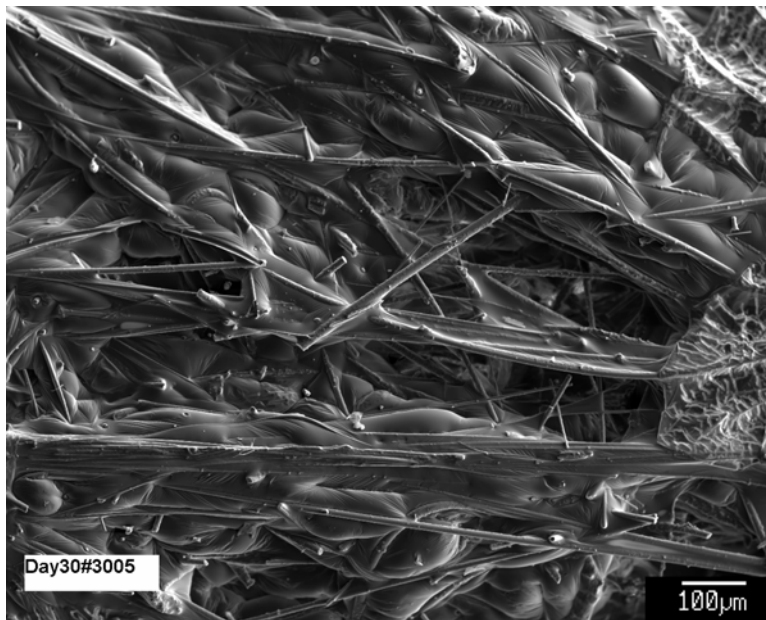


Figure C-48. Day-30, sample #3 SEM image (#3005) magnified 100 times on the fiber with film.

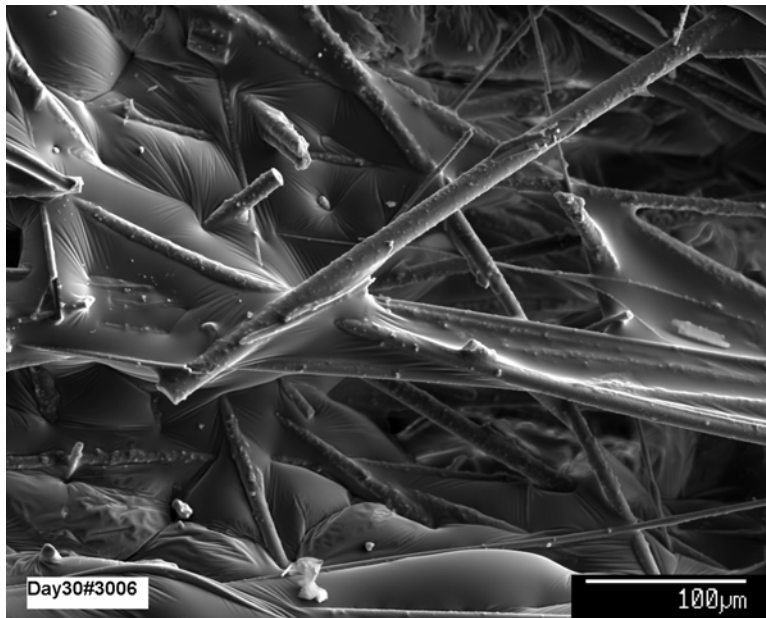


Figure C-49. Day-30, sample #3 SEM image (#3006) magnified 250 times on the film across the fibers.

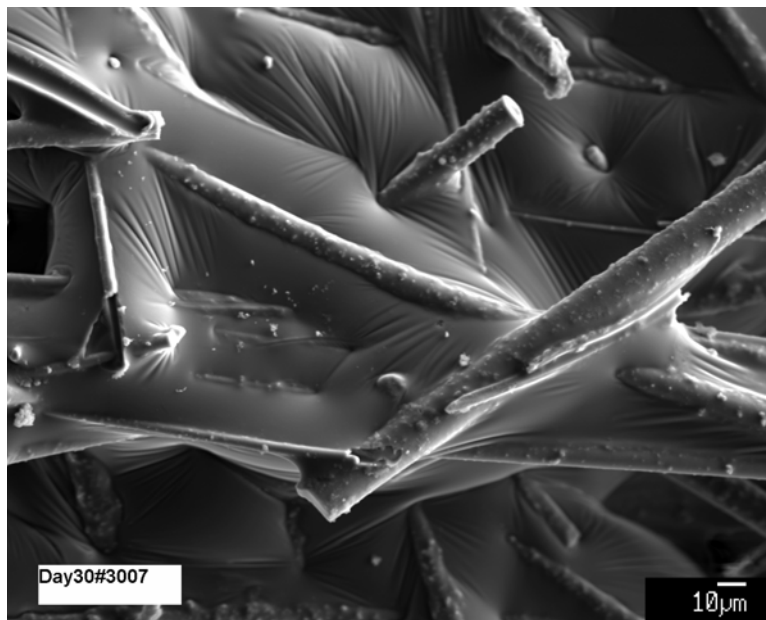


Figure C-50. Day-30, sample #3 SEM image (#3007) magnified 500 times on the film across the fibers.

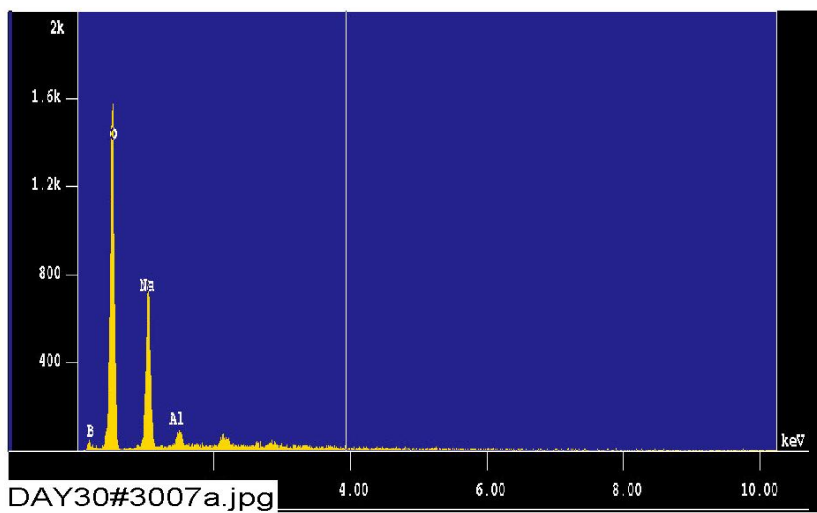


Figure C-51. Day-30, sample #3 counting spectrum (EDS #3007a) for the film, as shown in Figure C-50.

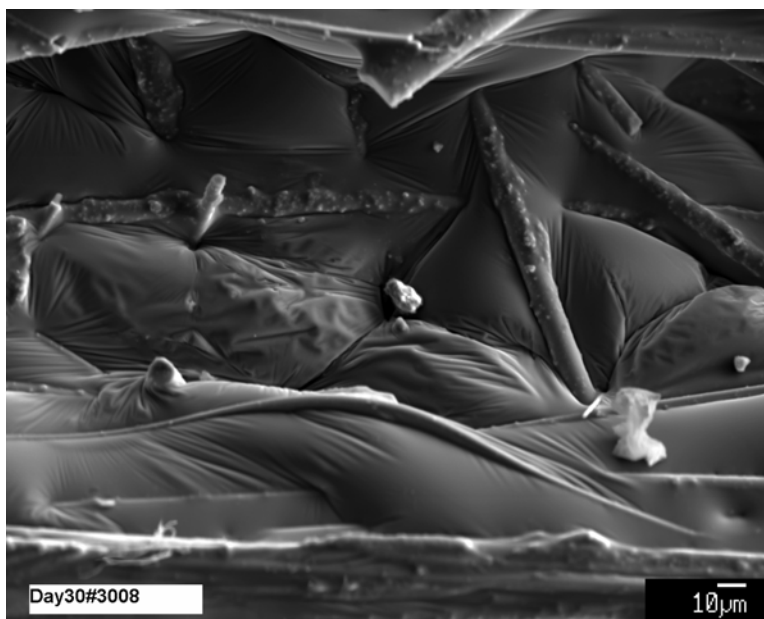


Figure C-52. Day-30, sample #3 SEM image (#3008) of particles on the film, magnified 450 times.

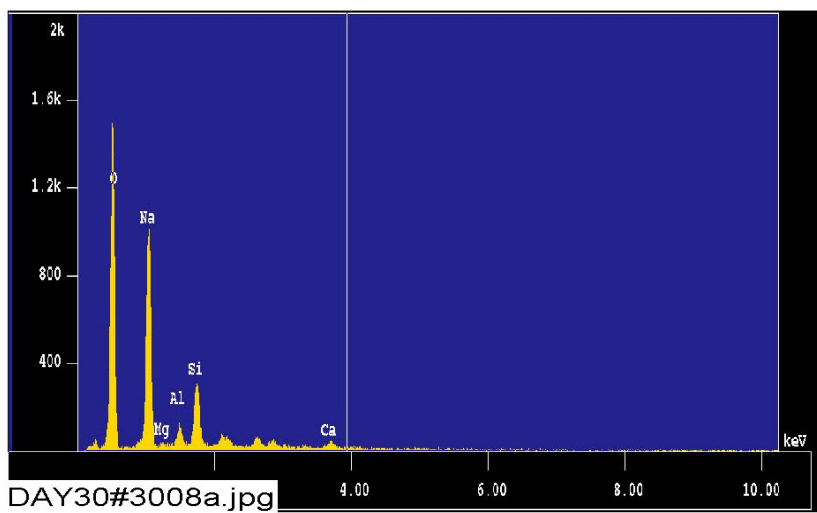


Figure C-53. Day-30, sample #3 counting spectrum (EDS #3008a) for the particles on the film, as shown in Figure C-52.

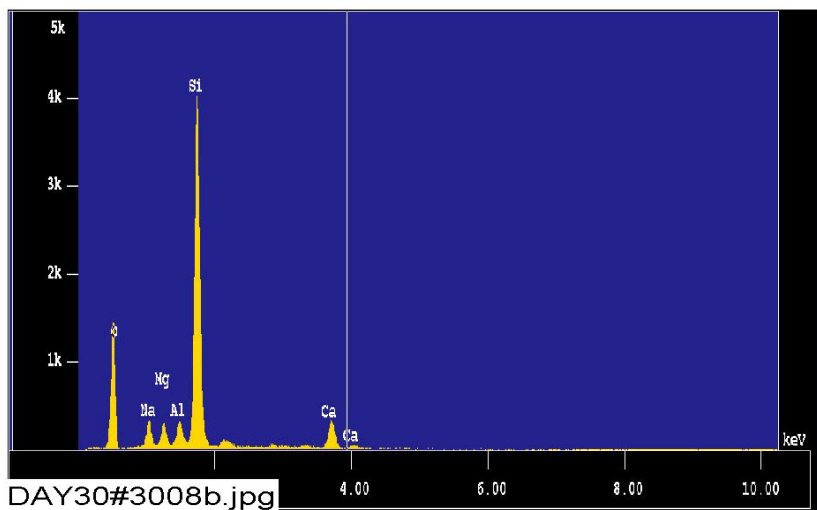


Figure C-54. Day-30, sample #3 counting spectrum (EDS #3008b) for the fiber, as shown in Figure C-52.

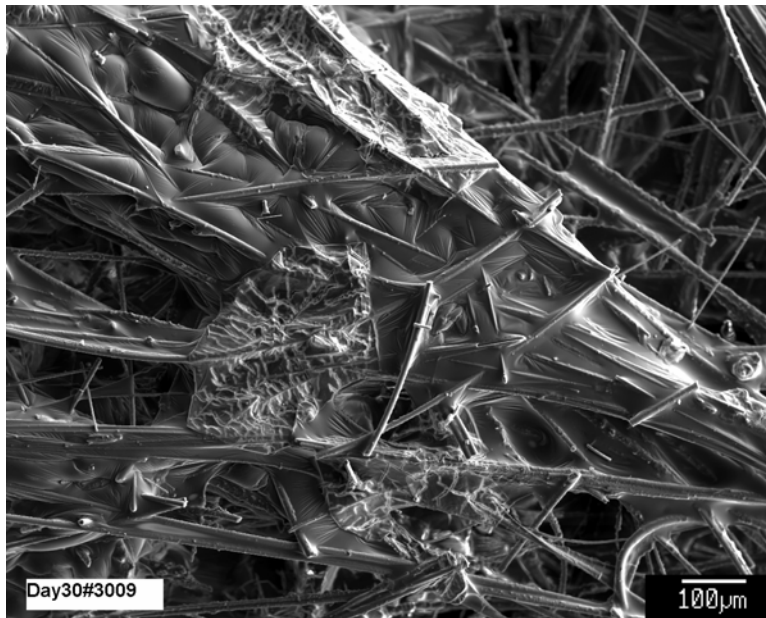


Figure C-55. Day-30, sample #3 SEM image (#3009) magnified 100 times on a group of fibers.

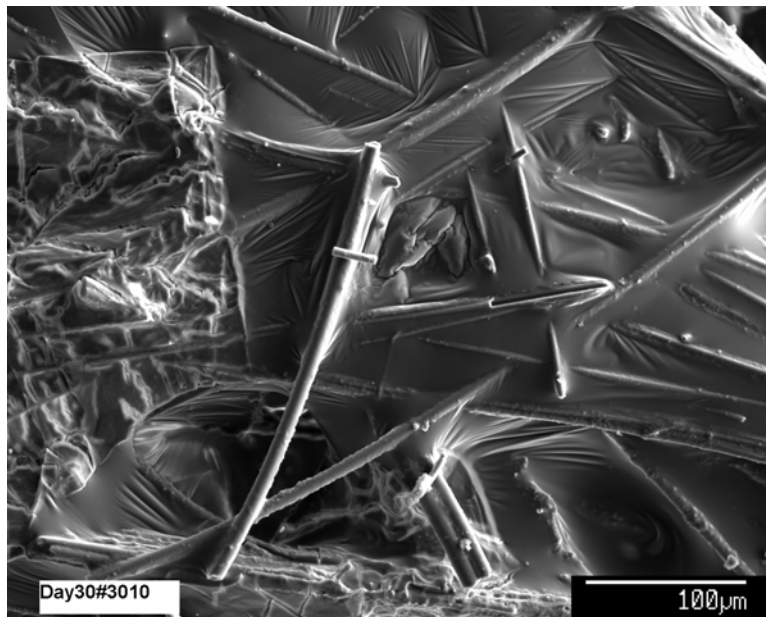


Figure C-56. Day-30, sample #3 SEM image (#3010) magnified 250 times; close-up of the film.

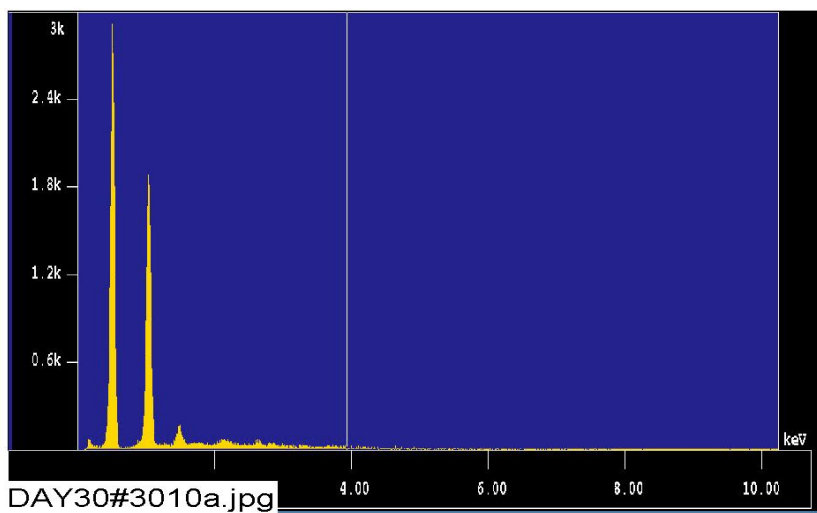


Figure C-57. Day-30, sample #3 counting spectrum (EDS #3010a) of precipitate on the film.

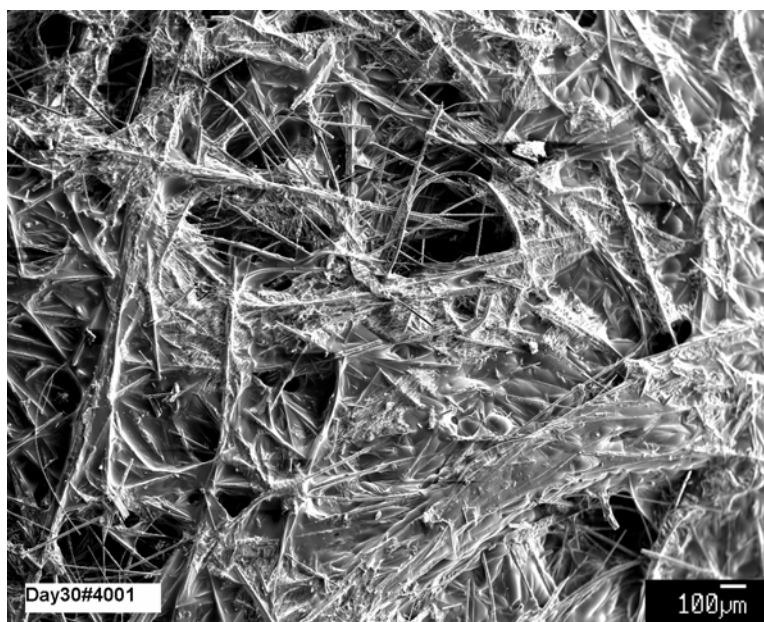


Figure C-58. Day-30, sample #4 SEM image (#4001) of fiber cluster with film, magnified 40 times.

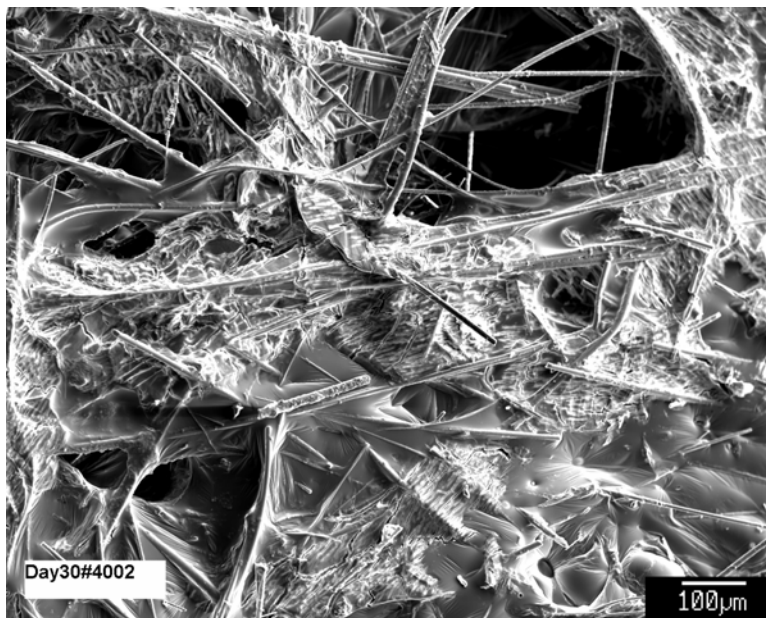


Figure C-59. Day-30, sample #4 SEM image (#4002) of fiber cluster with film, magnified 100 times.

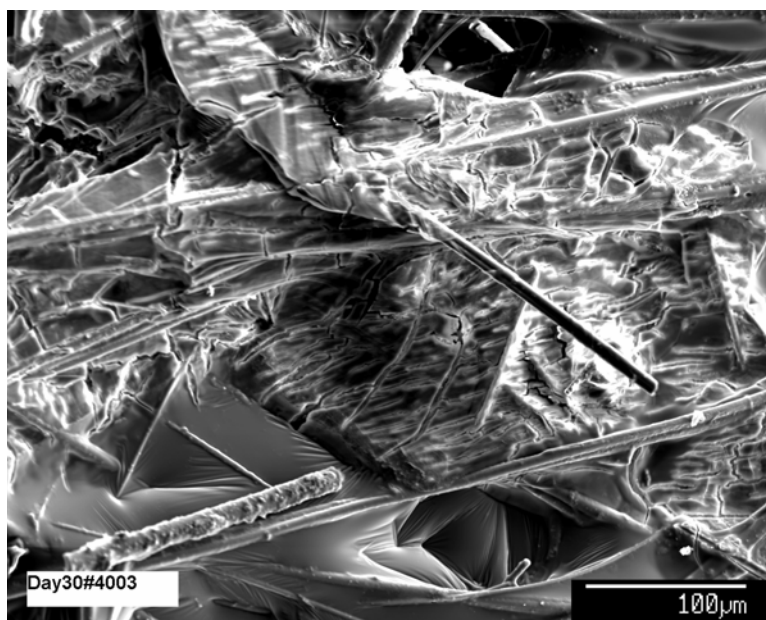


Figure C-60. Day-30, sample #4 SEM image (#4003) of fiber cluster with film, magnified 250 times.

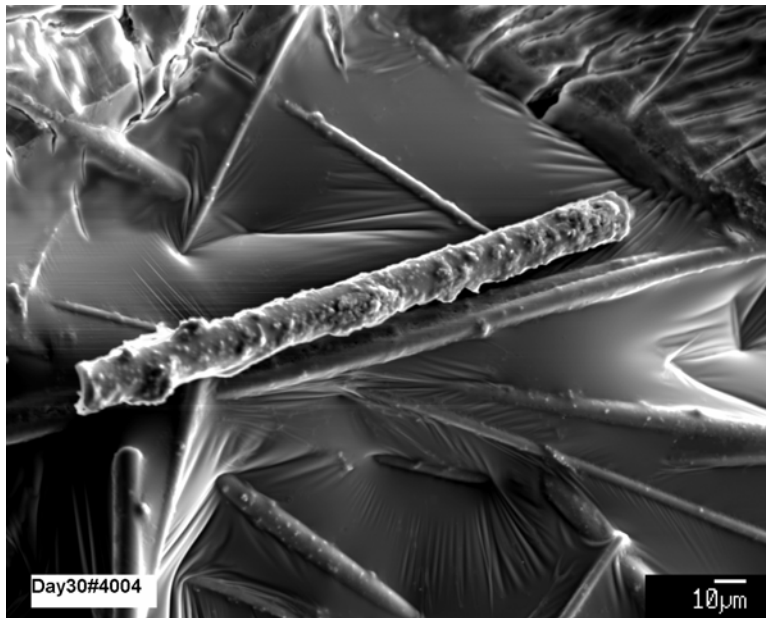


Figure C-61. Day-30, sample #4 SEM image (#4004) close-up of fiber with film, magnified 500 times.

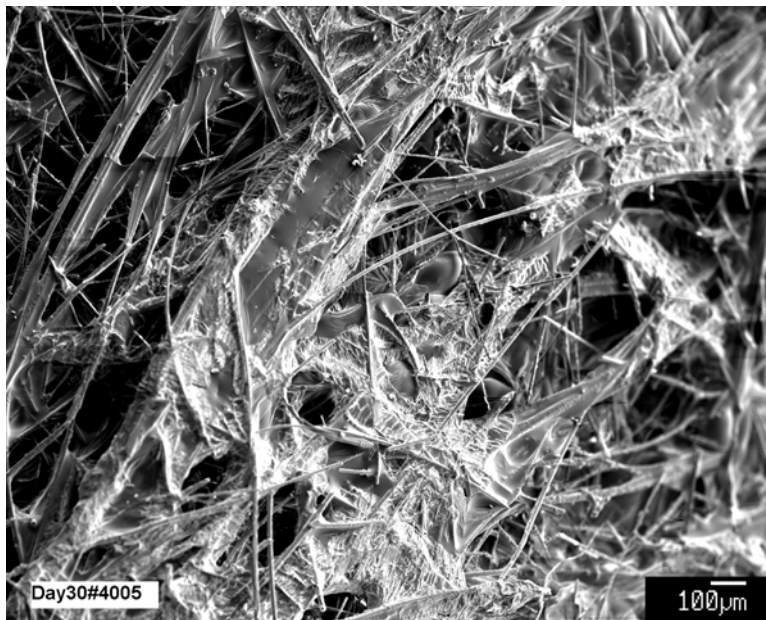


Figure C-62. Day-30, sample #4 SEM image (#4005) of a cluster of fiber, magnified 50 times.

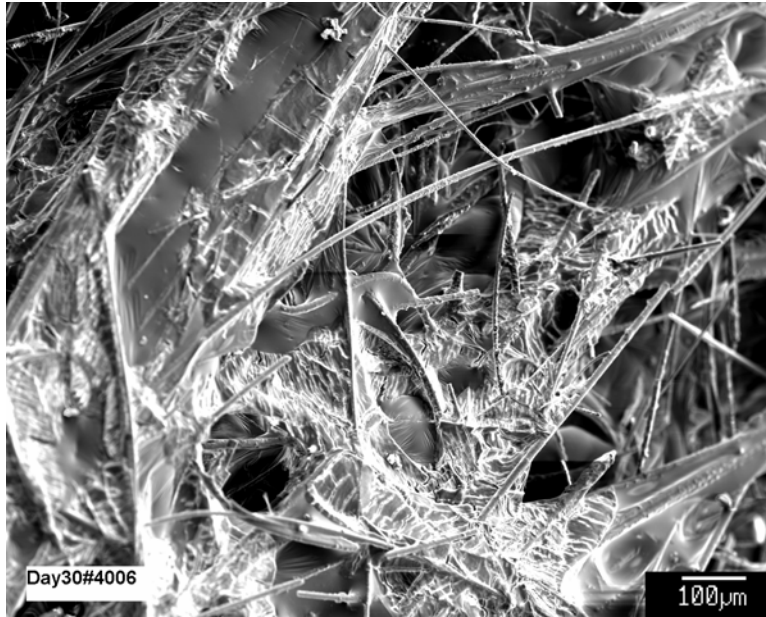


Figure C-63. Day-30, sample #4 SEM image (#4006) magnified 100 times on the cluster of fiber shown in Figure C-62.

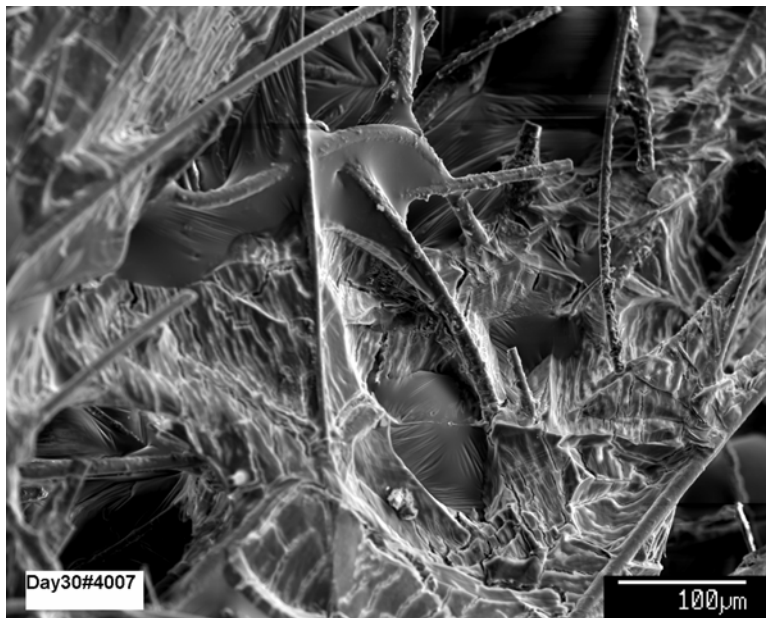


Figure C-64. Day-30, sample #4 SEM image (#4007) magnified 200 times on the surface precipitates shown in Figure C-62.

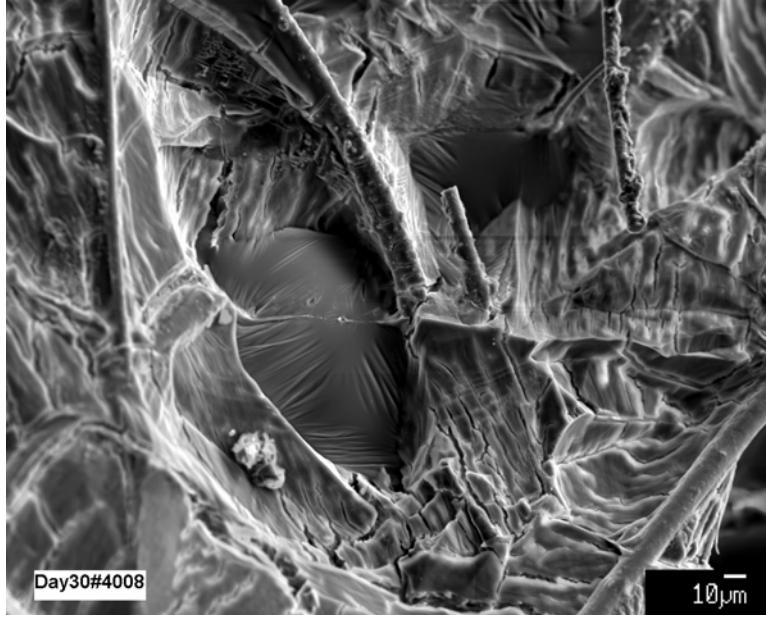


Figure C-65. Day-30, sample #4 SEM image (#4008) magnified 350 times on the surface precipitates shown in Figure C-62.

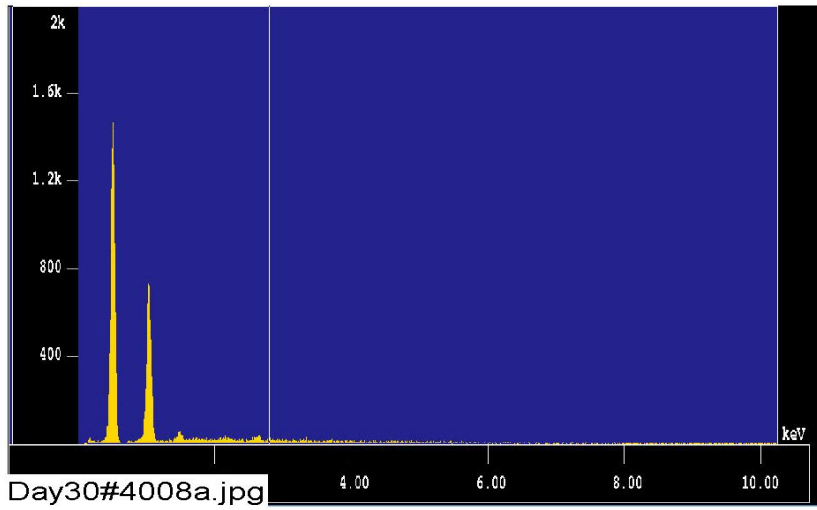


Figure C-66. Day-30, sample #4 counting spectrum (EDS #4008a) for the fractured precipitates.

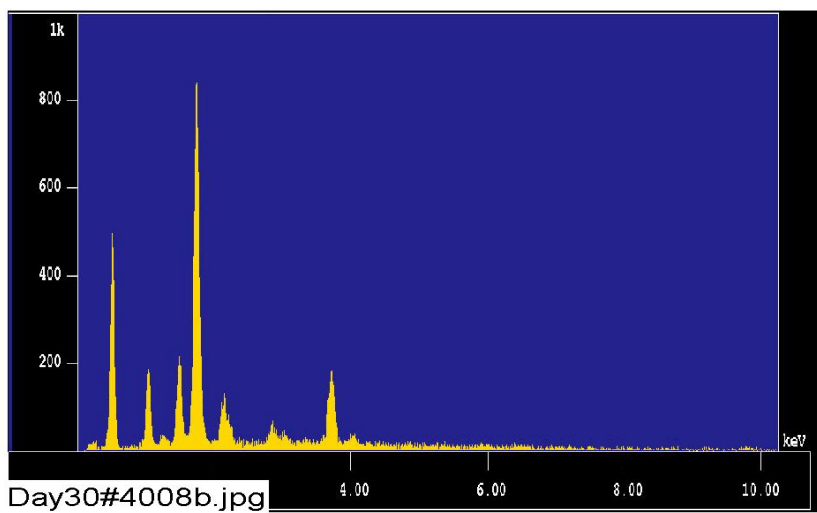


Figure C-67. Day-30, sample #4 counting spectrum (EDS #4008b) for the film between fibers.

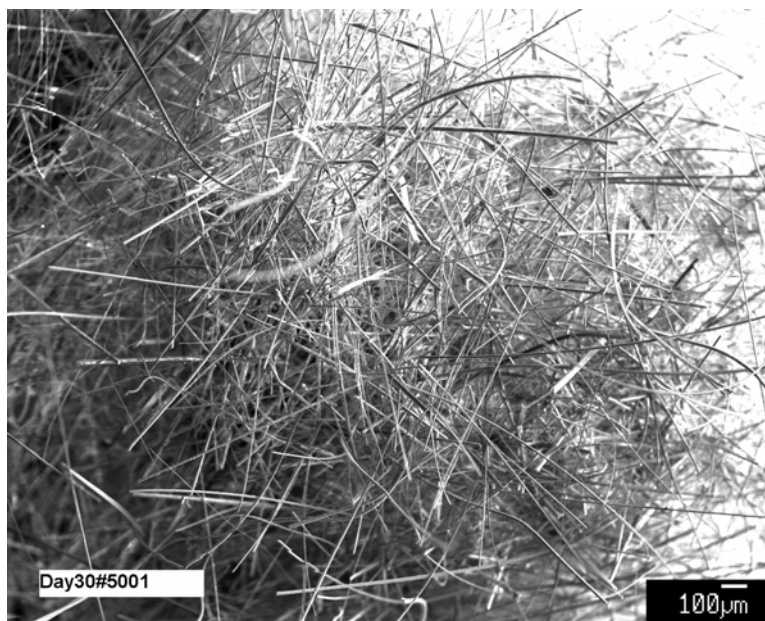


Figure C-68. Day-30, sample #5 SEM image (#5001) magnified 40 times on the fiberglass.

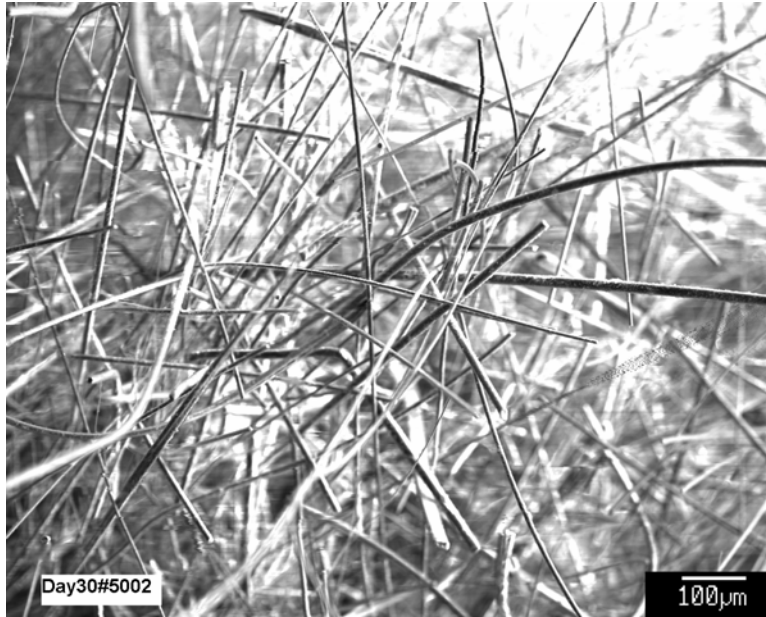


Figure C-69. Day-30, sample #5 SEM image (#5002) magnified 100 times; close-up of the fiberglass.

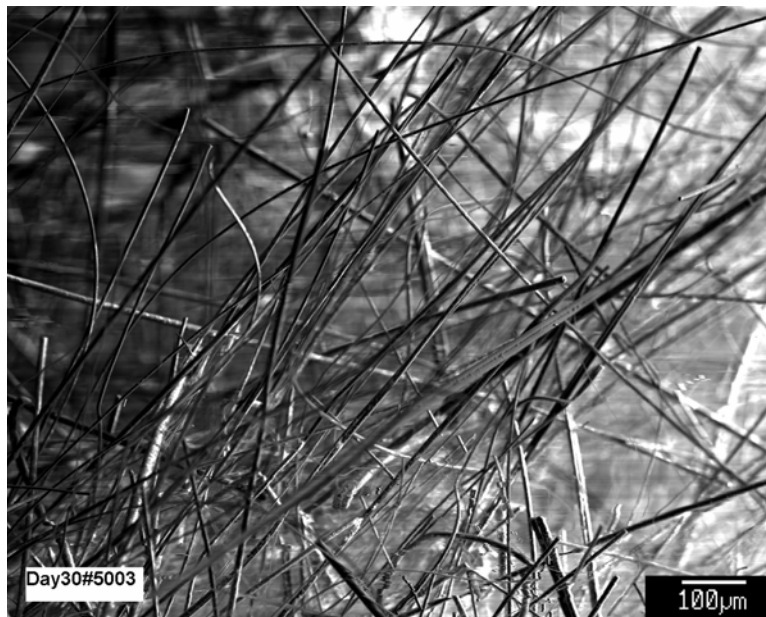


Figure C-70. Day-30, sample #5 SEM image (#5003) magnified 100 times; close-up of fibers.

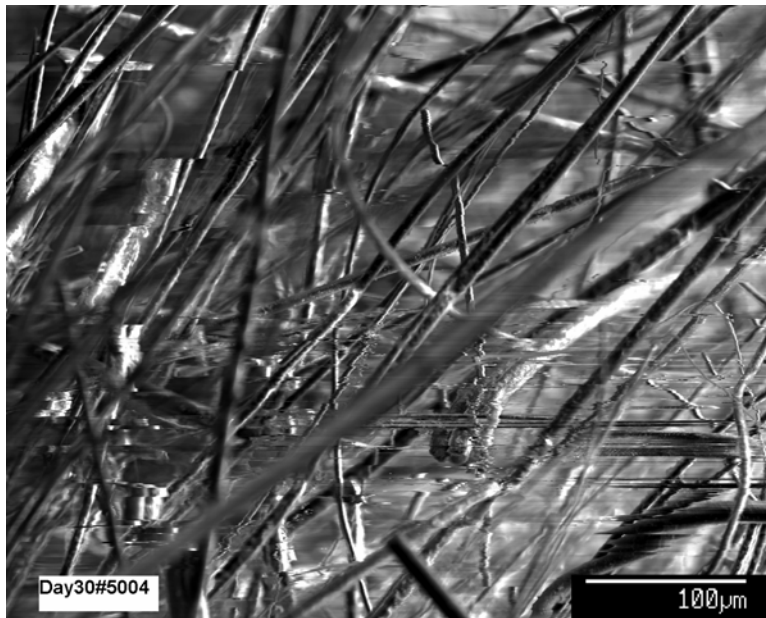


Figure C-71. Day-30, sample #5 SEM image (#5004) magnified 250 times on the fibers with precipitate on surface. Streaks show evidence of charge accumulation on the sample.

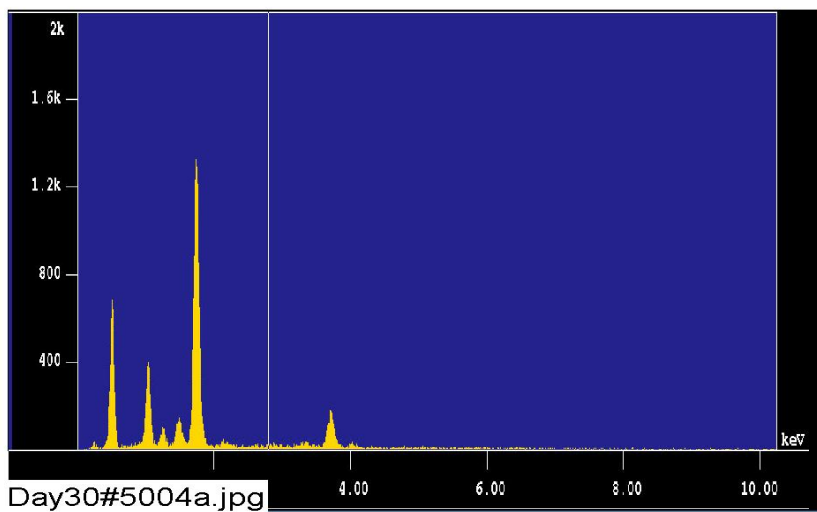


Figure C-72. Day-30, sample #5 counting spectrum (EDS #5004a) for an exposed fiber with precipitate appearing in Figure C-71.

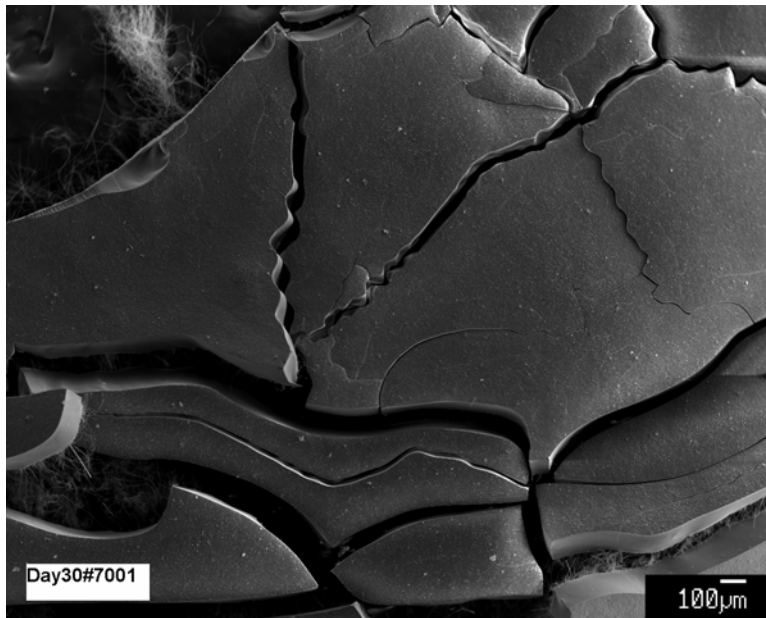


Figure C-73. Day-30, sample #7 SEM image (#7001), magnified 40 times; close-up of precipitate layer.

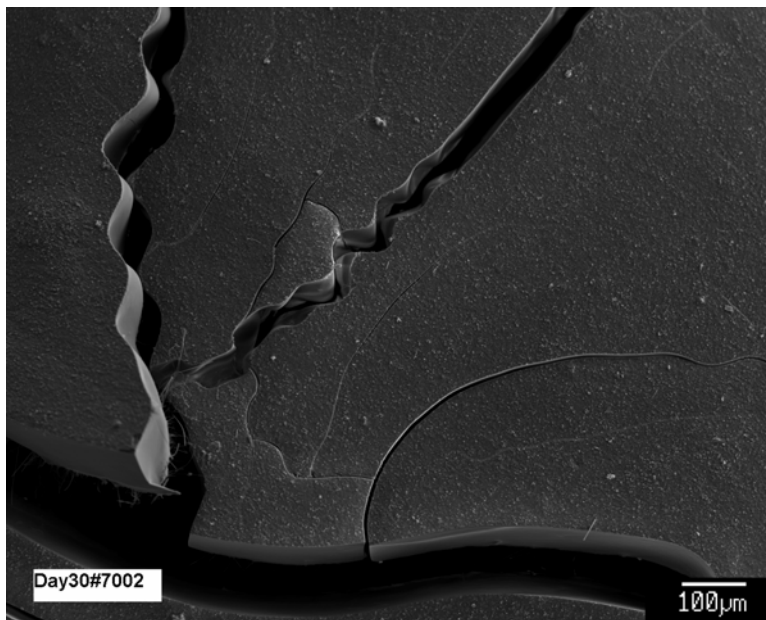


Figure C-74. Day 30, sample #7 SEM image (#7002), magnified 100 times; close-up of precipitate layer.

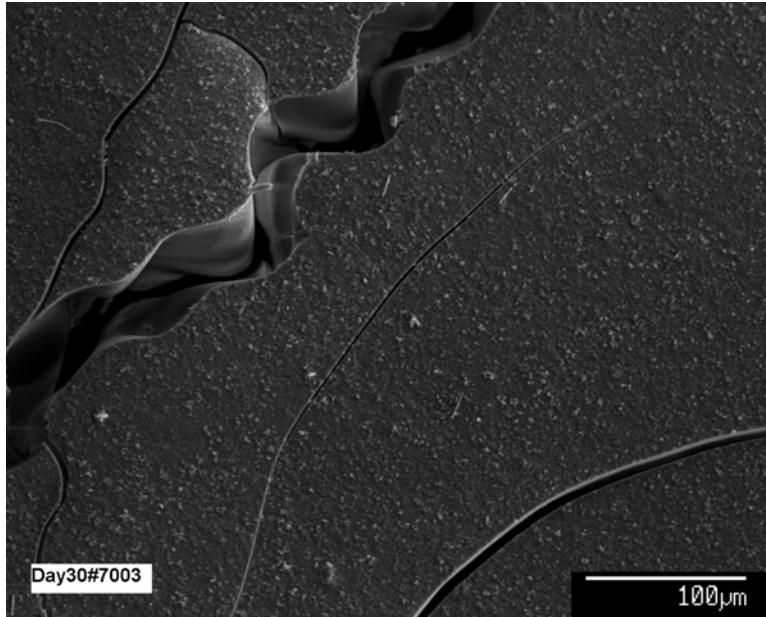


Figure C-75. Day-30, sample #7 SEM image (#7003) magnified 250 times; close-up of precipitate layer.

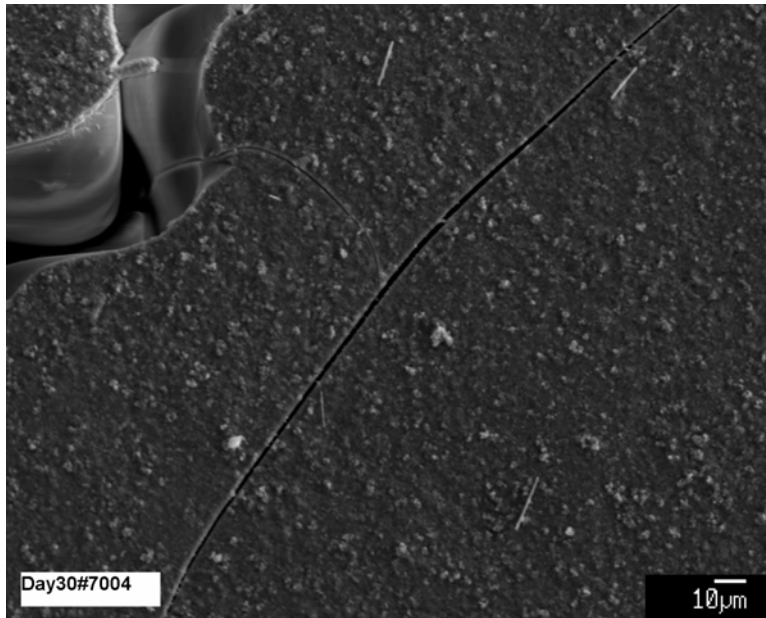


Figure C-76. Day 30, sample #7 SEM image (#7004) magnified 500 times on the crust on fiberglass; close-up of precipitate layer.