Appendix D4

ESEM and SEM/EDS Data for Test-2 Day-30 Birdcage Fiberglass

List of Figures

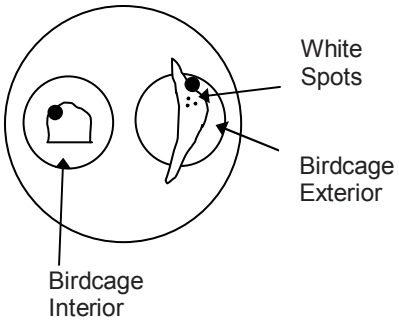
List of Tables

This appendix lists the ESEM/SEM/EDS results for the fiberglass samples within a birdcage submerged in the test solution. The purpose of this analysis was to determine the degree and the extent of particulate debris attached to the fiberglass. The fiberglass samples were extracted from the birdcage on March 7, 2005, the date Test 2 was shut down. Both exterior and interior fiberglass samples were examined. Microprobe SEM was used to examine the fiberglass samples after they were air dried and coated with Au/Pd. In addition to microprobe SEM, environmental SEM (ESEM) was employed to analyze the wet fiberglass samples without any coating and 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. Microprobe SEM/EDS and ESEM results of the Test-2 Day-30 birdcage fiberglass samples were obtained on March 9, 2005.

Transcribed Laboratory Log

Microprobe laboratory session from March 9, 2005

T2D30 Samples—NRC



Conditions: 15-kV, 1-nA beam current, Aperture = 2
Note: bold spots in sketch denote carbon glue used to secure samples.

Sample Birdcage Exterior

Image:	T2D30_BirdcageExt041	90 ×	BSE	Figure D4-1
EDS:	T2D30EDS21		Bright particle left side of image 041	Figure D4-2
Image:	T2D30_BirdcageExt042	150 ×	BSE	Figure D4-3
	T2D30_BirdcageExt043	150 ×	SE	Figure D4-4
	T2D30_BirdcageExt044	1000 ×	SE	Figure D4-5
EDS:	T2D30EDS22		EDS on lath-like crystal	Figure D4-6
	T2D30EDS23		Particles on fiberglass	Figure D4-7

Sample Birdcage Interior

Image:	T2D30_BirdcageInt045	90 ×	SE image	Figure D4-8
	T2D30_BirdcageInt046	150 ×	SE image same area	Figure D4-9
	T2D30_BirdcageInt047	150 ×	BSE same area	Figure D4-10
	T2D30_BirdcageInt048	1000 ×	SE same area	Figure D4-11
EDS:	T2D30EDS24		Globules on fiberglass	Figure D4-12

ESEM laboratory session from March 9, 2005

Birdcage Sample Exterior

Image:	T2D30BX9	95 ×	On debris/precipitate	Figure D4-13
	T2D30BX10	150 ×	On glass fibers	Figure D4-14
	T2D30BX11	1000 ×	On fibers	Figure D4-15
	T2D30BX12	700 ×	On particle	Figure D4-16

Birdcage Sample Interior

Image:	T2D30BI13	150 ×	On fibers	Figure D4-17
	T2D30BI14	1000 ×		Figure D4-18

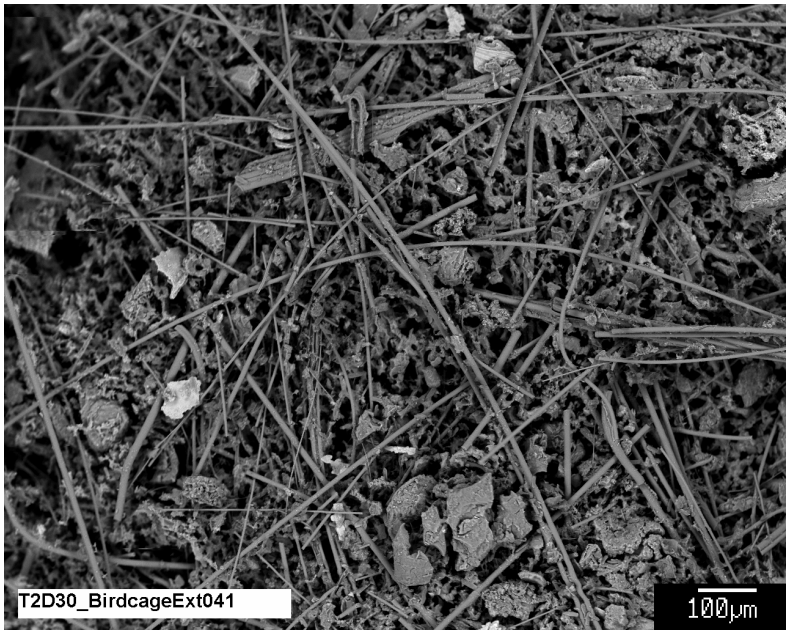


Figure D4-1. SEM image for a Test-2 Day-30 exterior fiberglass sample within the birdcage. Image shows particulate deposits or growth on fiberglass (TD30_BirdcageExt041).

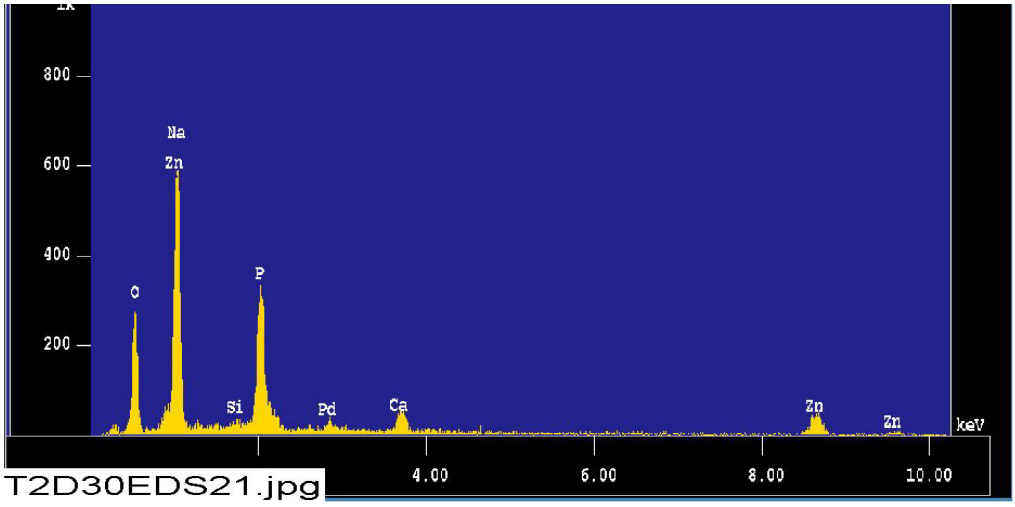


Figure D4-2. EDS counting spectrum for the bright particle on the lower left side of Figure D4-1 (T2D30EDS21).

The results from the chemical composition analysis for T2D30EDS21 are given in Table D4-1.

Table D4-1. The Chemical Composition for T2D30EDS21

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Group : NRC
Sample : T2D30 ID# : 21
Comment : bright particle
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 5.403E-10 A
Stage Point : X=36.780 Y=58.457 Z=10.627
Acq. Date : Wed Mar 9 17:26:42 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	20.3402	0.0022	2572 / 15
Na K	Normal	0.83- 1.28	0.7506	0.0151	298 / 23
P K	Normal	1.75- 2.38	11.8434	0.0034	3577 / 28
Ca K	Normal	3.40- 4.30	1.8504	0.0050	586 / 11
Zn K	Normal	8.22-10.03	19.6836	0.0071	719 / 2

Chi_square = 3.0137

Element	Mass%	Atomic%	ZAF	Z	A	F
O	35.828	61.5751	1.0317	0.9179	1.1240	1.0000
Na	2.043	2.4438	1.5945	0.9209	1.7289	1.0014
P	19.372	17.1970	0.9581	1.1032	0.8685	0.9999
Ca	3.013	2.0671	0.9537	0.9240	1.0329	0.9993
Zn	39.743	16.7170	1.1826	1.1840	0.9988	1.0000
Total	100.000	100.0000				

Normalization factor = 1.7073

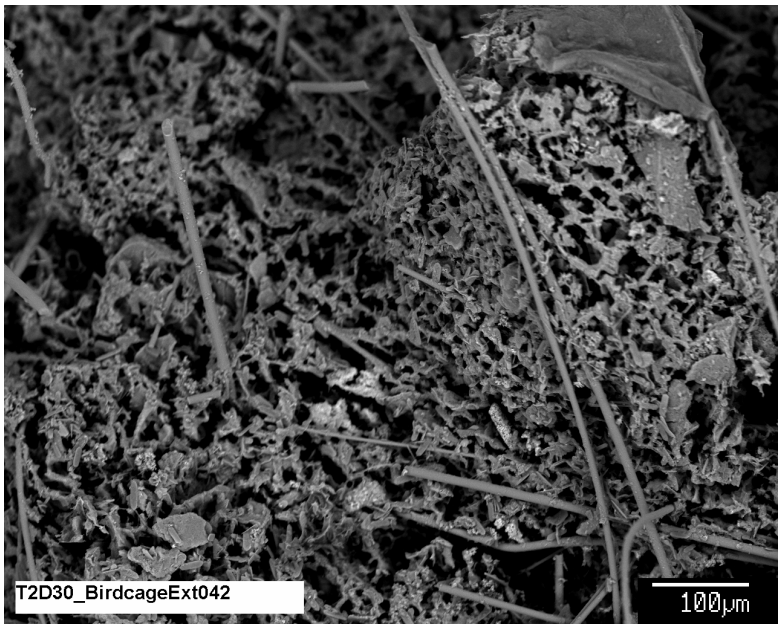

Figure D4-3. Backscatter SEM image for a Test-2 Day-30 exterior fiberglass sample within the birdcage (T2D30_BirdcageExt042).

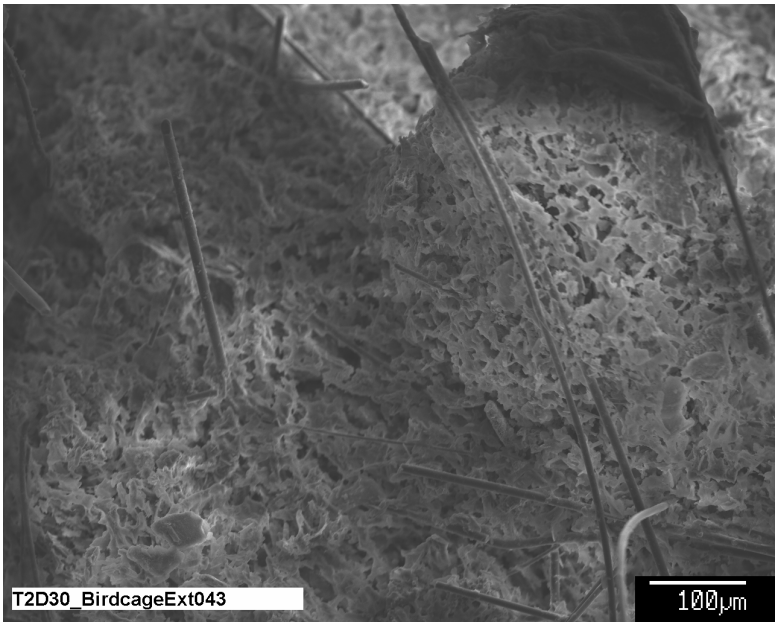

Figure D4-4. SEM image for a Test-2 Day-30 exterior fiberglass sample within the birdcage. Same field as that shown in Figure D4-3 (T2D30_BirdcageExt043).

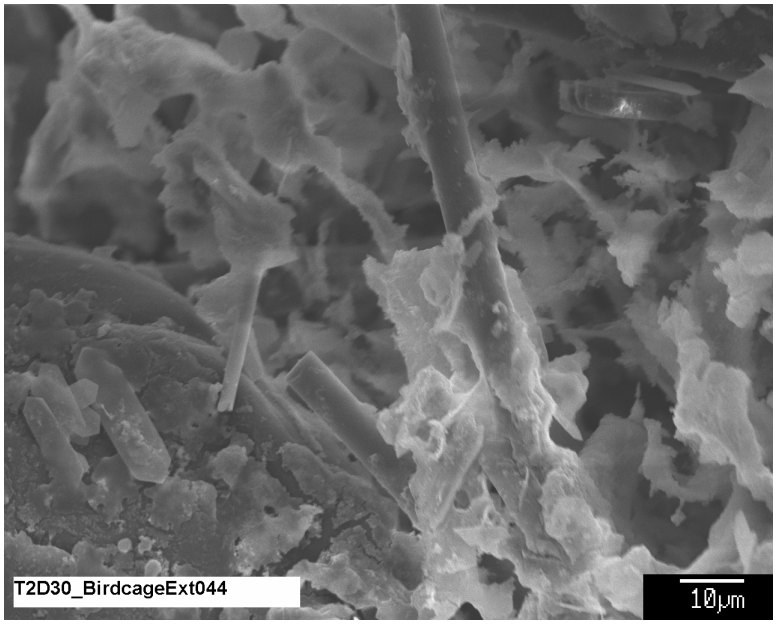

Figure D4-5. SEM image of a higher magnification for a Test-2 Day-30 exterior fiberglass sample within the birdcage (T2D30_BirdcageExt044).

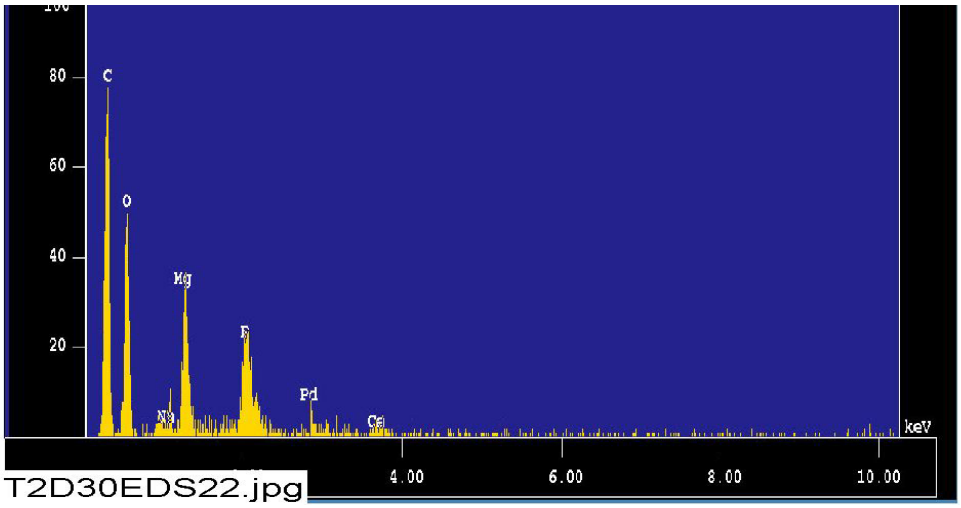

Figure D4-6. EDS counting spectrum for the crystal-shaped deposit at the lower left side of Figure D4-5 (T2D30EDS22).

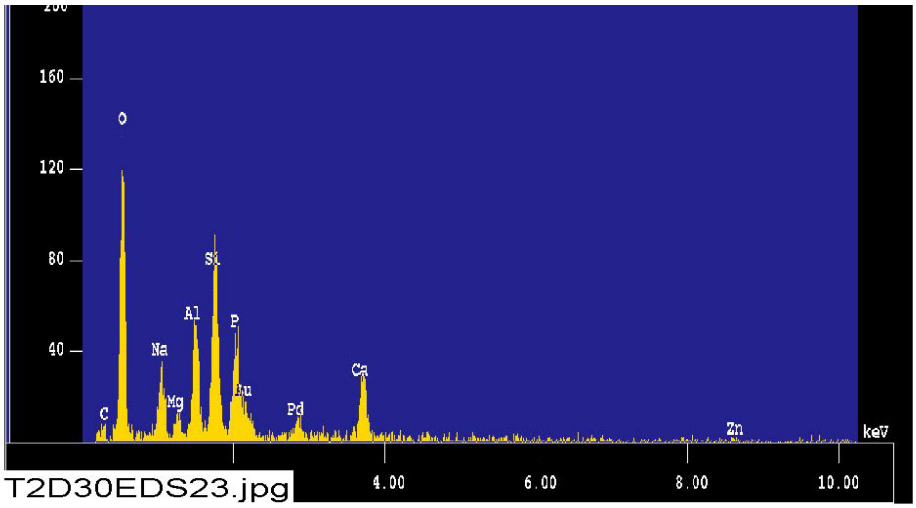

Figure D4-7. EDS counting spectrum for the deposits attached to fiberglass as shown in Figure D4-5 (T2D30EDS23).

The results from the chemical composition analysis for T2D30EDS23 are given in Table D4-2.

Table D4-2. The Chemical Composition for T2D30EDS23

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Group : NRC
Sample : T2D30 ID# : 23
Comment : particles on fiberglass
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 120.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.020E-10 A
Stage Point : X=35.661 Y=58.464 Z=10.627
Acq. Date : Wed Mar 9 17:47:47 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	23.8450	0.0014	1139 / 6
Na K	Normal	0.83- 1.28	1.5443	0.0028	232 / 10
Mg K	Normal	1.03- 1.52	0.4403	0.0005	84 / 38
Al K	Normal	1.26- 1.78	1.8853	0.0004	427 / 28
Si K	Normal	1.50- 2.07	3.3376	0.0006	755 / 38
P K	Normal	1.75- 2.38	3.3673	0.0014	384 / 43
Ca K	Normal	3.40- 4.30	2.8112	0.0031	336 / 2

Chi_square = 2.0568

Element	Mass%	Atomic%	ZAF	Z	A	F
O	62.741	75.5081	0.9977	0.9907	1.0070	1.0000
Na	4.658	3.9012	1.1437	0.9951	1.1485	1.0007
Mg	1.284	1.0170	1.1059	0.9978	1.1091	0.9993
Al	5.681	4.0539	1.1426	1.0033	1.1431	0.9963
Si	9.837	6.7440	1.1176	0.9910	1.1308	0.9973
P	8.399	5.2214	0.9458	1.1947	0.7920	0.9996
Ca	7.399	3.5544	0.9980	1.0045	0.9934	1.0001
Total	100.000	100.0000				

Normalization factor = 2.6373

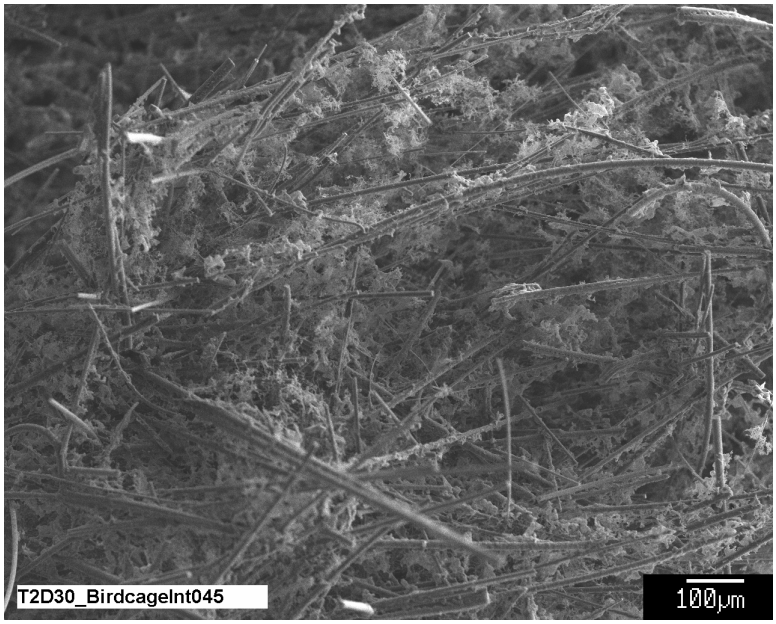

Figure D4-8. SEM image for a Test 2-Day-30 interior fiberglass sample within the birdcage. Image shows deposits or growth on fiberglass (T2D30_BirdcageInt045).

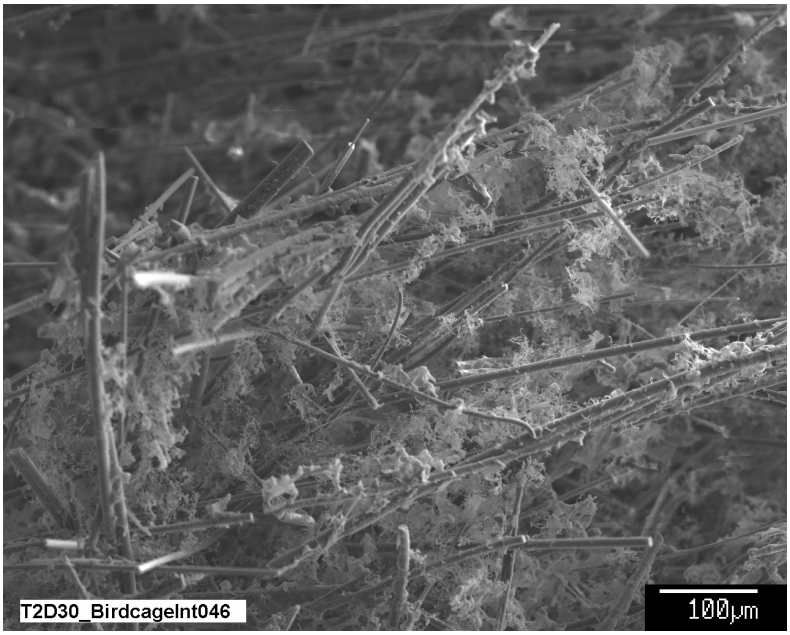

Figure D4-9. SEM image for a Test-2 Day-30 interior fiberglass sample within the birdcage. Image shows deposits or growth on fiberglass (T2D30_BirdcageInt046).

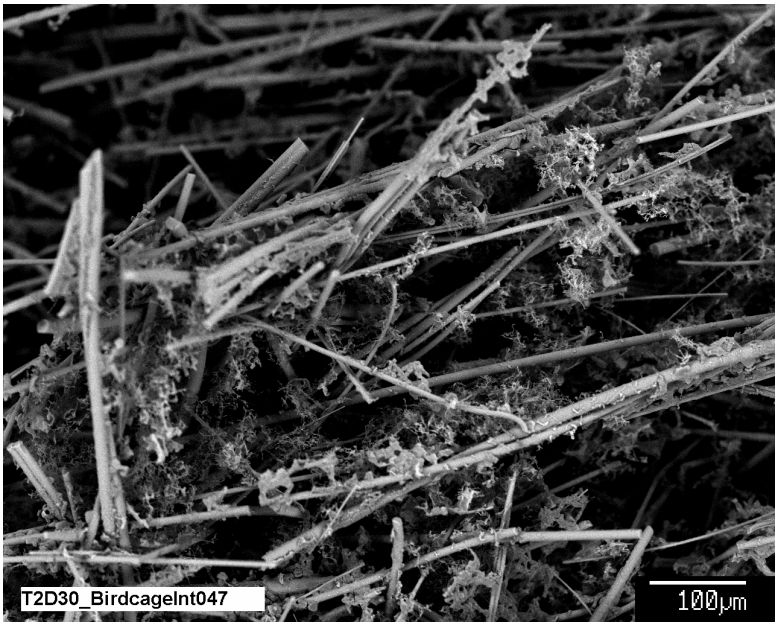

Figure D4-10. Backscattered SEM image for a Test-2 Day-30 interior fiberglass sample within the birdcage. The atomic number of the deposits is similar to fiberglass (T2D30_BirdcageInt047).

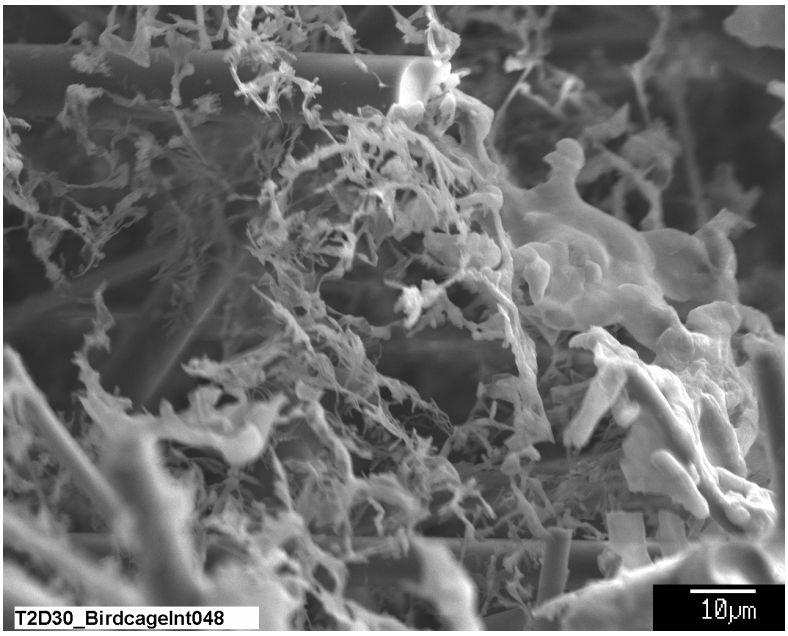

Figure D4-11. SEM image of a higher magnification for a Test-2 Day-30 interior fiberglass sample within the birdcage. Image shows deposits or growth on fiberglass (T2D30_BirdcageInt048).

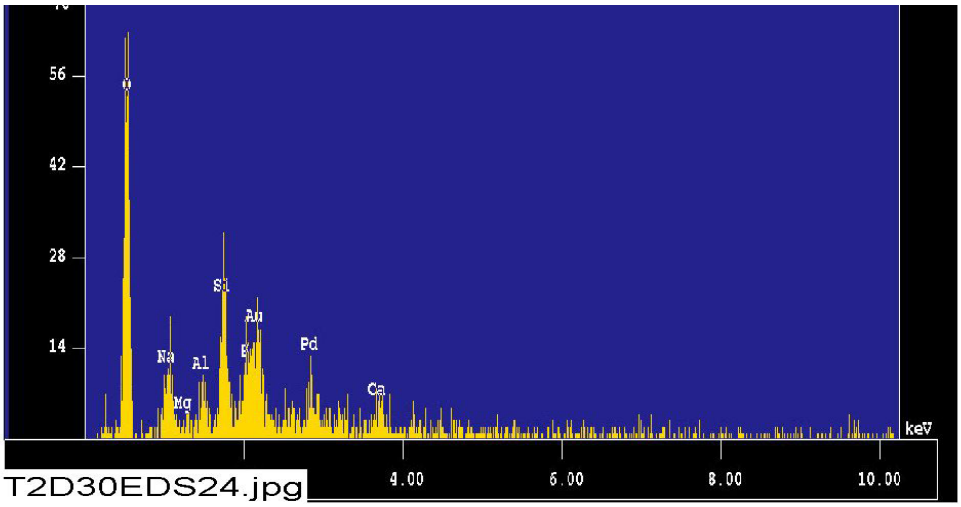

Figure D4-12. EDS counting spectrum for deposits on fiberglass as shown in Figure D4-11 (T2D30EDS24).

The results from the chemical composition analysis for T2D30EDS24 are given in Table D4-3.

Table D4-3. The Chemical Composition for T2D30EDS2

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Group : NRC
Sample : T2D30 ID# : 24
Comment : globules on fiberglass birdcage interior
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 120.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.019E-10 A
Stage Point : X=49.105 Y=62.519 Z=10.627
Acq. Date : Wed Mar 9 18:06:33 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	10.6755	0.0009	509 / 4
Na K	Normal	0.83- 1.28	0.6105	0.0018	92 / 3
Mg K	Normal	1.03- 1.52	0.0691	0.0003	13 / 10
Al K	Normal	1.26- 1.78	0.1947	0.0002	44 / 8
Si K	Normal	1.50- 2.07	0.8895	0.0004	201 / 12
P K	Normal	1.75- 2.38	0.6913	0.0009	79 / 14
Ca K	Normal	3.40- 4.30	0.4040	0.0018	48 / 2

Chi_square = 1.1748

Element	Mass%	Atomic%	ZAF	Z	A	F
O	73.306	82.8087	0.7875	0.9951	0.7914	1.0000
Na	6.327	4.9737	1.1884	0.9997	1.1875	1.0011
Mg	0.700	0.5202	1.1607	1.0024	1.1578	1.0001
Al	1.996	1.3369	1.1755	1.0080	1.1697	0.9969
Si	8.560	5.5082	1.1035	0.9959	1.1103	0.9981
P	5.609	3.2731	0.9305	1.2006	0.7752	0.9998
Ca	3.502	1.5793	0.9941	1.0101	0.9841	1.0001
Total	100.000	100.0000				

Normalization factor = 8.7200

Figure D4-13. ESEM image for a Test-2 Day-30 exterior fiberglass sample within the birdcage (T2D30BX9).

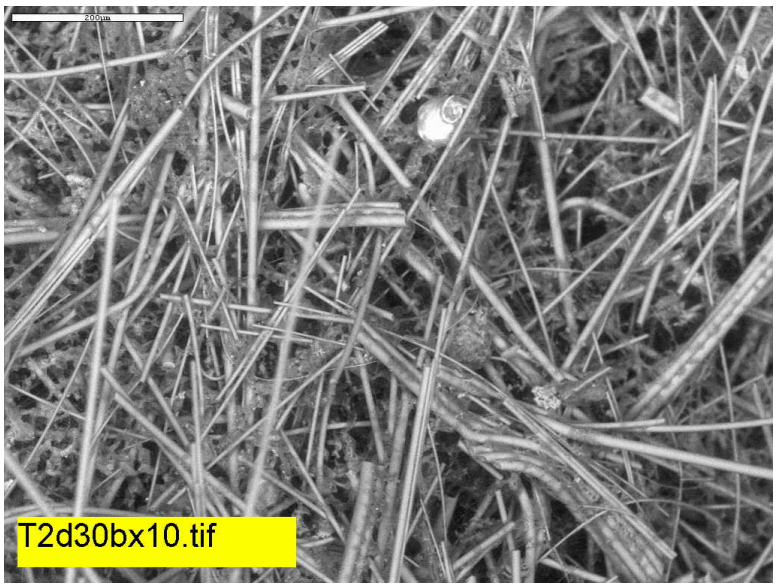

Figure D4-14. ESEM image for a Test-2 Day-30 exterior fiberglass sample within the birdcage (T2d30bx10).

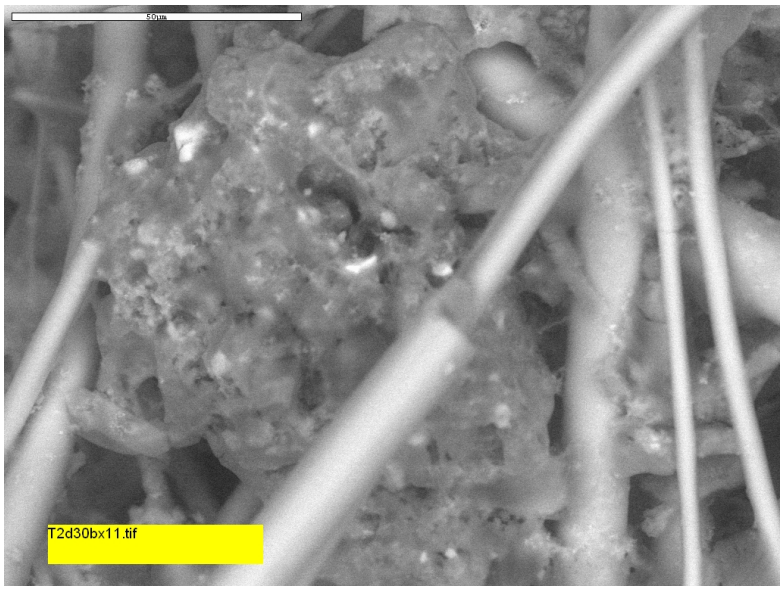

Figure D4-15. ESEM image of a higher magnification for a Test-2 Day-30 exterior fiberglass sample within the birdcage (T2d30bx11).

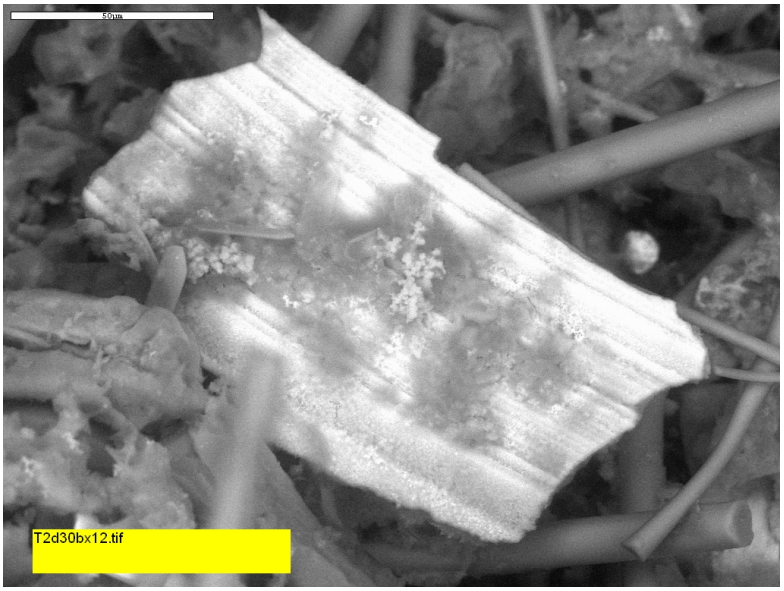

Figure D4-16. ESEM image of a higher magnification from another area for a Test-2 Day-30 exterior fiberglass sample within the birdcage (T2d30bx12).

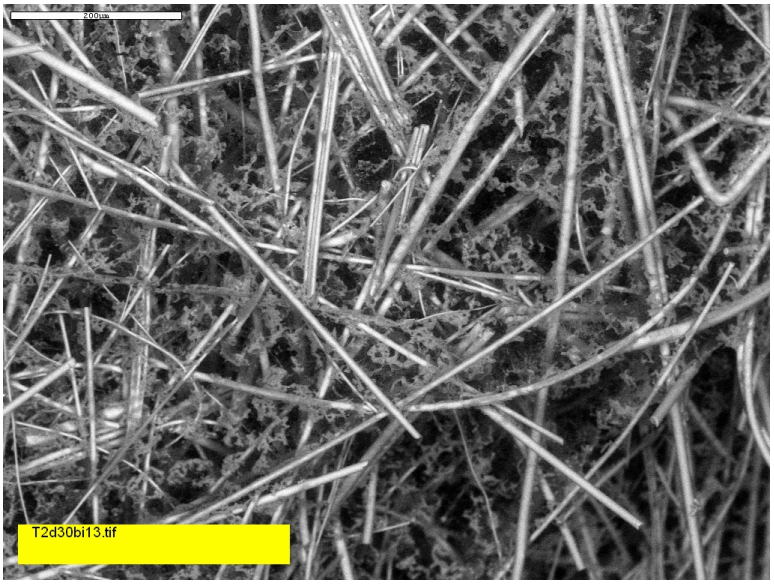

Figure D4-17. ESEM image for a Test-2 Day-30 interior fiberglass sample within the birdcage. Image shows deposits or growth on fiberglass (T2d30bi13).

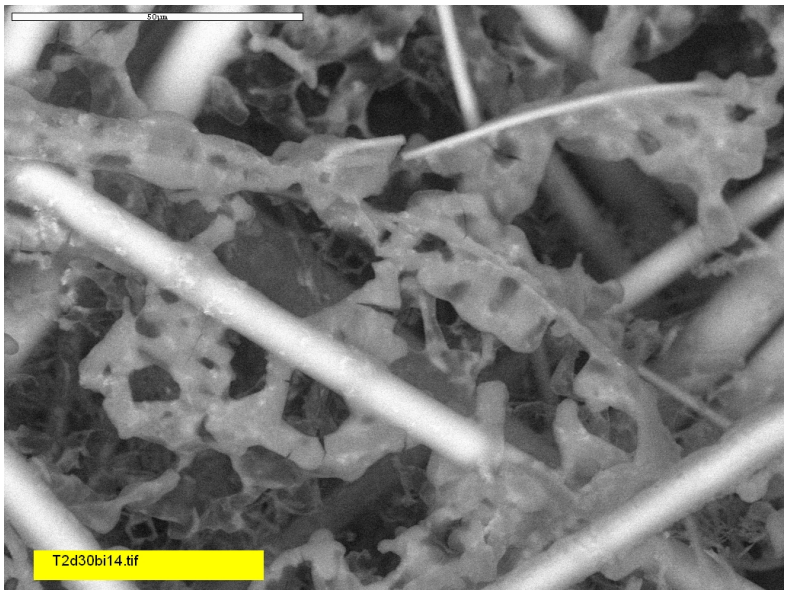

Figure D4-18. ESEM image of a higher magnification for a Test-2 Day-30 interior fiberglass sample within the birdcage. Image shows deposits or growth on fiberglass (T2d30bi14).