Appendix D3

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

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Chemical deposits accumulated on fiberglass during ICET testing are of great interest because they may cause additional head loss during recirculation of the coolant following a loss of coolant accident. The drain collar placed in the test tank represents an area of higher flow velocity where water continually flows through any accumulated debris. This condition of continual water flow is similar to that found on the face of a recirculation sump screen, so it is important to examine via ESEM and SEM/EDS the fiberglass samples recovered from the drain collar and compare the results with similar examinations of fiberglass from alternative flow regimes.

Fiberglass samples from the drain collar were extracted on the date that Test 2 was shut down (March 7, 2005). Samples located at the outside exterior (away from the drain screen), the inside exterior (next to the drain screen), and the interior of the collar were examined. Microprobe SEM was used to examine the fiberglass samples after they were dried in air at room temperature and coated with carbon. In addition to microprobe SEM, environmental SEM (ESEM) was employed to analyze the wet fiberglass samples. ESEM was performed without any required coating under a low-vacuum condition (80 Pa) to minimize any modification of the sample that might occur through the drying process. Microprobe SEM/EDS and ESEM results of the Test-2 Day-30 drain collar fiberglass samples were obtained on March 9, 2005.

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Microprobe laboratory session from March 9 2005 T2D30 Samples from fiberglass drain collar



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

Sample: Drain Screenside

Image:	T2D30_DrainScreen029	$150 \times$	SE	Figure D3-1
	T2D30_DrainScreen030	$150 \times$	BSE	Figure D3-2
	T2D30_DrainScreen031	90 ×	SE	Figure D3-3
	T2D30_DrainScreen032	1000×	SE	Figure D3-4
EDS:	T2D30EDS16		Center of image 032	Figure D3-5
	T2D30EDS17		Film on fiberglass	Figure D3-6

Sample: Drain Outside

Image:	T2D30_DrainOutside033	$90 \times$	SE	Figure D3-7
	T2D30_DrainOutside034	$150 \times$	SE same area	Figure D3-8
	T2D30_DrainOutside035	$150 \times$	BSE same area	Figure D3-9
	T2D30_DrainOutside036	$1000 \times$	SE same area	Figure D3-10
EDS:	T2D30EDS18		Particles on fiberglass, high C content	Figure D3-11
	T2D30EDS19		Particle on fiberglass, high C content	Figure D3-12

Note: *Very difficult to get EDS spectrum of particles. The particles are very thin and react under the beam. EDS18 and EDS19 are simply replicates at slightly different sample locations.

Sample: Drain Interior

Image:	T2D30_DrainInt037	$90 \times$	SE	Figure D3-13
	T2D30_DrainInt038	$150 \times$	SE new area	Figure D3-14
	T2D30_DrainInt039	$1000 \times$	SE same area	Figure D3-15
	T2D30_DrainInt040	$150 \times$	BSE same area	Figure D3-16
EDS:	T2D30EDS20		Particles on fiberglass	Figure D3-17

Transcribed Laboratory Log

ESEM laboratory session from March 9, 2005 T2D30 NRC - Fiberglass on Drain Collar ESEM



Conditions: 20-kV, 12-mm Working Distance, 80 Pa pressure

Outside Sample					
Image:	T2D30DO1	$150 \times$	BSE Overview	Figure D3-18	
	T2D30DO2	$1000 \times$	Same area	Figure D3-19	
	T2D30DO3	$90 \times$	Same area	Figure D3-20	

Screen-Side Sample

Image:	T2D30DS4	$90 \times$	Overview	Figure D3-21
	T2D30DS5	$150 \times$	Same area	Figure D3-22
	T2D30DS6	$1000 \times$	Same area as above	Figure D3-23

Interior Sample

Image:	T2D30DI7	$90 \times$	1 st area	Figure D3-24
	T2D30DI8	$150 \times$	Different area	Figure D3-25
	T2D30DI9	$1000 \times$	Same as above	Figure D3-26



Figure D3-1. SEM image for a Test-2 Day-30 fiberglass sample on the drain collar next to the drain screen (T2D30_DrainScreen029).



Figure D3-2. Backscattered SEM image for a Test-2 Day-30 fiberglass sample on the drain collar next to the drain screen, illustrating that deposits have a similar atomic number to fiberglass (T2D30_DrainScreen030).



Figure D3-3. SEM image for a Test-2 Day-30 fiberglass sample on the drain collar next to the drain screen (T2D30_DrainScreen031).



Figure D3-4. SEM image at 1000× magnification for a Test-2 Day-30 fiberglass sample on the drain collar next to the drain screen (T2D30_DrainScreen032).



Figure D3-5. EDS counting spectrum for the center of the image shown in Figure D3-4 (T2D30EDS16~Drain Screen Side Center of Image 032).

The results from the chemical composition analysis for T2D30EDS16 are given in Table D3-1.

Table D3-1. The Chemical Composition for T2D30EDS16 (Figure D3-5)

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Group Sample Comment Condition	: NRC : T2D3 : drai : Full Live Acc. Stag Acq.	0 ID# : 16 n screen side Scale : 20KeV(10eV/ch,2Kch) Time : 60.000 sec Aperture # : 1 Volt : 15.0 KV Probe Current : 1.004E-09 A e Point : X=75.582 Y=53.863 Z=10.627 Date : Wed Mar 9 16:16:45 2005	Ŧ
Element C K O K Na K Al K Si K P K Ca K Zn K	Mode Norma Norma Norma Norma Norma Norma Norma	ROI (KeV)K-ratio (%)+/-Net/Background10.09-0.460.00000.00000 /2710.25-0.7788.97960.00222775 /2210.83-1.282.13160.0068209 /1911.26-1.781.43060.0004211 /5311.50-2.073.51710.0007520 /7311.75-2.3813.86240.00241032 /4413.40-4.309.23720.0047722 /718.22-10.0310.88800.003598 /3	7 2 3 3 3 4 7 3
		Chi_square = 2.8663	
Element Ma C Na Al Si P Ca Zn 1	ass% 0.000 5.359 2.262 1.422 3.163 9.891 7.056 0.847	Atomic% ZAF Z A F 0.0000 4.3367 1.0203 4.2504 0.9999 81.5376 0.9412 0.9731 0.9672 1.0000 1.9637 1.3596 0.9772 1.3894 1.0013 1.0518 1.2735 0.9851 1.2952 0.9981 2.2478 1.1523 0.9730 1.1881 0.9968 6.3735 0.9142 1.1728 0.7798 0.9996 3.5137 0.9787 0.9855 0.9933 0.9998 3.3119 1.2765 1.2799 0.9973 1.0000	
Total 10 Normalizat	0.000 i ion fa	100.0000 ctor = 0.7805	



Figure D3-6. EDS counting spectrum for the film between fibers shown in Figure D3-4. The film is rich in Si, O, Al, Na, and Ca (T2D30EDS17~Drain Screen Side Film on Fiber Glass).

The results from the chemical composition analysis for T2D30EDS17 are given in Table D3-2.

Table D3-2. The Chemical Composition for T2D30EDS17 (Figure D3-6)

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Group Sample Comment Condition	: NRC : T2D30 : film on : Full Sca Live Tin Acc. Vo Stage Po Acq. Dat	ID# : 17 fiberglass ale : 20KeV me : 60.0 lt : 15.0 pint : X=75.0 te : Wed Ma	(10eV/ch,2K 00 sec A KV P 684 Y=53.97 ar 9 16:21	ch) perture ; robe Cur: 7 Z=10.62 ;30 2005	# : 1 rent : 1.001 27	E-09 A
Element O K Na K Al K Si K P K Ca K Fe K	Mode Normal Normal Normal Normal Normal Normal	ROI(KeV) 0.25- 0.77 0.83- 1.28 1.26- 1.78 1.50- 2.07 1.75- 2.38 3.40- 4.30 6.04- 7.40	K-ratio(%) 7.9629 0.4017 0.8589 2.1743 0.6172 0.6433 0.0946	+/- 0.0018 0.0037 0.0006 0.0011 0.0018 0.0037 0.0232	Net/Backgro 1866 / 296 / 954 / 2413 / 345 / 378 / 18 /	und 12 15 93 84 129 6 2
Element Ma O 5 Na Al Si 1 P Ca Fe	ss% Atc 9.959 73 3.492 2 7.333 5 8.694 12 4.794 3 4.994 2 0.734 0	Ch: 0617 0.9746 9608 1.1249 2984 1.1051 9763 1.1128 0176 1.0055 4289 1.0048 2562 1.0038	i_square = Z 0.9921 0.9 0.9964 1.1 1.0045 1.1 0.9923 1.1 1.1961 0.8 1.0056 0.9 1.0013 1.0	A I 824 1.000 283 1.000 064 0.994 231 0.998 408 0.999 992 1.000 018 1.000	F 00 06 44 86 98 00 08	
Total 10 Normalizat	0.000 100 ion factor	0000 = 7.7260				



Figure D3-7. SEM image for a Test-2 Day-30 exterior fiberglass sample on the drain collar (away from the drain screen) showing deposits or growth on fiberglass (T2D30_DrainOutside033).



Figure D3-8. SEM image for a Test-2 Day-30 exterior fiberglass sample on the drain collar showing deposits or growth on fiberglass (T2D30_DrainOutside034).



Figure D3-9. Backscattered SEM image for a Test-2 Day-30 exterior fiberglass sample on the drain collar (T2D30_DrainOutside035).



Figure D3-10. SEM image at a higher magnification for a Test-2 Day-30 exterior fiberglass sample on the drain collar showing deposits or growth on fiberglass (T2D30_DrainOutside036).



Figure D3-11. EDS counting spectrum for deposits or growth on fiberglass. The deposits contain a significant amount of C (T2D30EDS18~Drain Outside Particles High C).



Figure D3-12. EDS counting spectrum for a deposit or growth on fiberglass. The deposit contains a significant amount of C (T2D30EDS19).

The results from the chemical composition analysis for T2D30EDS19 are given in Table D3-3.

Table D3-3. The Chemical Composition for T2D30EDS19 (Figure D3-12).

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Group Sample Comment Condition	: NRC : T2D30 ID# : 19 : particles on fiberglass : Full Scale : 20KeV(10eV/ch,2Kch) Live Time : 120.000 sec Aperture # : 1 Acc. Volt : 15.0 KV Probe Current : 9.749E-09 A Stage Point : X=81.088 Y=63.027 Z=10.627 Acq. Date : Wed Mar 9 16:55:59 2005
Element C K O K Al K Si K Zn K Ca K	ModeROI(KeV)K-ratio(%)+/-Net/BackgroundNormal0.09-0.460.11380.0002771 /2Normal0.25-0.770.03590.0006164 /44Normal1.26-1.780.00480.0002104 /6Normal1.50-2.070.00330.000371 /11Normal8.22-10.030.10990.0036145 /2Normal3.40-4.300.00380.001844 /4
Element Mas C 59 O 12 Al 1 Si 0 Zn 25 Ca 0 Total 100 Normalizati	Chi_square = 3.5725 s% Atomic% ZAF Z A F .630 79.7022 2.7985 0.9722 2.8786 1.0000 .718 12.7615 1.8946 0.9274 2.0429 1.0000 .176 0.6998 1.3104 0.9398 1.3945 0.9998 .710 0.4060 1.1613 0.9286 1.2506 1.0000 .102 6.1648 1.2204 1.2263 0.9952 1.0000 .664 0.2658 0.9225 0.9421 0.9800 0.9991 .000 100.0000 On factor = 187 2002



Figure D3-13. SEM image for a Test-2 Day-30 interior fiberglass sample on the drain collar. Image shows deposits or growth on fiberglass (T2D30_DrainInt037).



Figure D3-14. SEM image for a Test-2 Day-30 interior fiberglass sample on the drain collar. Image shows deposits or growth on fiberglass (T2D30_DrainInt038).



Figure D3-15. SEM image of a higher magnification for a Test-2 Day-30 interior fiberglass sample on the drain collar. Image shows deposits or growth on fiberglass (T2D30_DrainInt039).



Figure D3-16. Backscattered SEM image for a Test-2 Day-30 interior fiberglass sample on the drain collar. Deposits appear to have a similar or lower atomic number than fiberglass (T2D30_DrainInt040).



Figure D3-17. EDS counting spectrum for the deposits or growth on fiberglass. The deposits are rich in Si, C, O, and Ca. The deposits are very thin, thus it was difficult to obtain high-quality EDS spectra (T2D30EDS20).

The results from the chemical composition analysis for T2D30EDS20 are given in Table D3-4.

Table D3-4. The Chemical Composition for T2D30EDS20 (Figure D3-17)

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Group : NRC Sample : T2D30 ID# : 20 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 : 1.375E-09 A Stage Point : X=68.013 Y=68.140 Z=10.627 Acq. Date : Wed Mar 9 17:14:10 2005	Ŧ
Element Mode ROI(KeV) K-ratio(%) +/- Net/Background C K Normal 0.09- 0.46 0.0420 0.0001 40 / 2 O K Normal 0.25- 0.77 0.1425 0.0005 92 / 3 Si K Normal 1.50- 2.07 0.0292 0.0002 89 / 2 Ca K Normal 3.40- 4.30 0.0225 0.0013 36 / 1	2 3 2 L
Chi_square = 0.7730 Element Mass% Atomic% ZAF Z A F C 30.132 39.0987 2.5156 1.0305 2.4412 0.9999 O 55.239 53.8107 1.3590 0.9832 1.3822 1.0000 Si 8.443 4.6849 1.0136 0.9850 1.0293 0.9998 Ca 6.187 2.4058 0.9650 1.0007 0.9642 1.0001	
Total 100.000 100.0000 Normalization factor = 285.2892	



Figure D3-18. ESEM image for a Test-2 Day-30 exterior fiberglass sample on the drain collar. Image shows particulate deposits or growth on fiberglass (T2D30DO1).



Figure D3-19. ESEM image of a higher magnification for a Test-2 Day-30 exterior fiberglass sample on the drain collar. Image shows particulate deposits or growth on fiberglass (T2D30DO2).



Figure D3-20. ESEM image for a Test-2 Day-30 exterior fiberglass sample on the drain collar. Image shows particulate deposits or growth on fiberglass (T2D30DO3).



Figure D3-21. ESEM image for a Test-2 Day-30 fiberglass sample on the drain collar next to the drain screen (T2D30DS4).



Figure D3-22. ESEM image for a Test-2 Day-30 fiberglass sample on the drain collar next to the drain screen (T2D30DS5).



Figure D3-23. ESEM image of a higher magnification for a Test-2 Day-30 fiberglass sample on the drain collar next to the drain screen (T2D30DS6).



Figure D3-24. ESEM image for a Test-2 Day-30 interior fiberglass sample on the drain collar. Image suggests that interior fiberglass was relatively clean compared to exterior (T2D30DI7).



Figure D3-25. ESEM image for a Test-2 Day-30 interior fiberglass sample on the drain collar (T2D30DI8).



Figure D3-26. ESEM image of a higher magnification for a Test-2 Day-30 interior fiberglass sample on the drain collar. Image shows deposits or growth on the interior fiberglass, although the quantity was less than for exterior samples (T2D30DI9).

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