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# **Pilot-Scale Testing of a Rotary Microfilter with Irradiated Filter Disks and Simulated SRS Waste**

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## SUMMARY

The processing rate of the Actinide Removal Project (ARP) is limited by the flow rate of the solid-liquid separation process. If the Department of Energy (DOE) could identify and develop a solid-liquid separation technology with a higher filter flux, they could increase the throughput of the Actinide Removal Project and complete treating that fraction of the waste stream in a shorter time, with a significant reduction in life-cycle cost. SRTC personnel identified the rotary microfilter as a technology that could significantly increase filter flux, with improvements of as much as 10X over the 0.5 micron crossflow filter and 5X over the 0.1 micron crossflow filter. The Savannah River Technology Center (SRTC) received funding from the DOE-HQ, Office of Cleanup Technologies, via the National Energy Technology Laboratory (NETL), to evaluate and develop the rotary microfilter for radioactive service at the Savannah River Site (SRS).

The authors performed pilot-scale simulant filtration tests with irradiated filter disks. They employed three types of filter disks for the tests (0.5  $\mu$  stainless steel, 0.1  $\mu$  stainless steel, and 0.1  $\mu$  ceramic/stainless steel). They analyzed the filter's structural material, Ryton<sup>®</sup>, for hardness, and irradiated the entire disk with an estimated 2.5 - 5 year (83- 165 MRad) radiation dose. They measured the hardness of the Ryton<sup>®</sup> after the irradiation of the disk. Following irradiation, they placed the filters in the pilot-scale rotary microfilter unit and tested them with feed slurries containing 0.29 and 4.5 wt % solids.

The conclusions from this work follow.

- None of the nine disks tested experienced a catastrophic failure from radiation exposure, but some evidence of delamination existed. Separation started to occur between the epoxy and the base Ryton<sup>®</sup> material.
- The flux with the irradiated filter disks was 35 – 40% lower than the flux measured with unirradiated filter disks. A likely cause of this difference is the feed to the irradiated filter disks had a smaller particle size than the feed to the unirradiated filter disks. Another plausible cause is that personnel did not thoroughly clean the filter disks following the 2002 test.
- The 0.1 micron ceramic/stainless steel filter produced the highest flux.
- The 0.1 micron stainless steel filter produced higher flux than the 0.5 micron stainless steel filter at the lower solids loading, and the same flux at the higher solids loading. Scanning electron microscope pictures show particles filling the pores of the 0.5 micron filter, and solid particles on the surface of the 0.1 micron filter.
- With the exception of one sample, all filtrate samples showed turbidity less than 5 NTU. The sample with high turbidity occurred during the test with the 0.1 micron stainless steel filter. We are uncertain of the cause. Subsequent samples with that filter showed filtrate turbidity less than 5 NTU, and all samples with a larger pore size filter showed turbidity less than 5 NTU. We believe this high turbidity sample was an anomaly.

## INTRODUCTION

The processing rate of the Actinide Removal Project is limited by the flow rate of the solid-liquid separation process. Similarly, the size of the planned Salt Waste Processing Facility (SWPF) depends heavily upon the filtration rate achievable. The baseline filtration flux rate for the

0.5 micron crossflow filter is 0.02 gpm/ft<sup>2</sup> under the anticipated operating conditions. If the DOE could identify and develop a solid-liquid separation technology with a higher filter flux, they could increase the throughput of the Actinide Removal Project and complete treating that fraction of the waste in a shorter time, with a significant reduction in life-cycle cost.

SRTC personnel identified the rotary microfilter as a technology that could significantly increase filter flux, with improvements of as much as 10X over the 0.5 micron crossflow filter and 5X over the 0.1 micron crossflow filter.<sup>1,2,3,4,5,6,13</sup> The rotary system combines centrifugation with membrane filtration. The system contains a group of flat disks with filter media on both sides. The feed slurry flows across the surface of the disks, and a pressure gradient forces the liquid through the filter. Solids are removed at the membrane surface, and the centrifugal force acts to keep the surface clean, minimizing the formation of a filter cake.

The rotary microfilter disks can be constructed with most commercially available filter media (i.e., filter disks could be fabricated using 0.1 or 0.5 micron porous metal filter sheets that are similar to the Mott crossflow filters in the current design bases, or could use filter media produced by other manufacturers). Centrifugal filter systems are commercially available (Spintek, ASPECT USA, Pall, Canzler) and have been used in radioactive service both at Los Alamos National Laboratory (LANL)<sup>7</sup> (i.e., for Low-Level Waste) and in Russia (for High-Level Waste).<sup>16</sup>

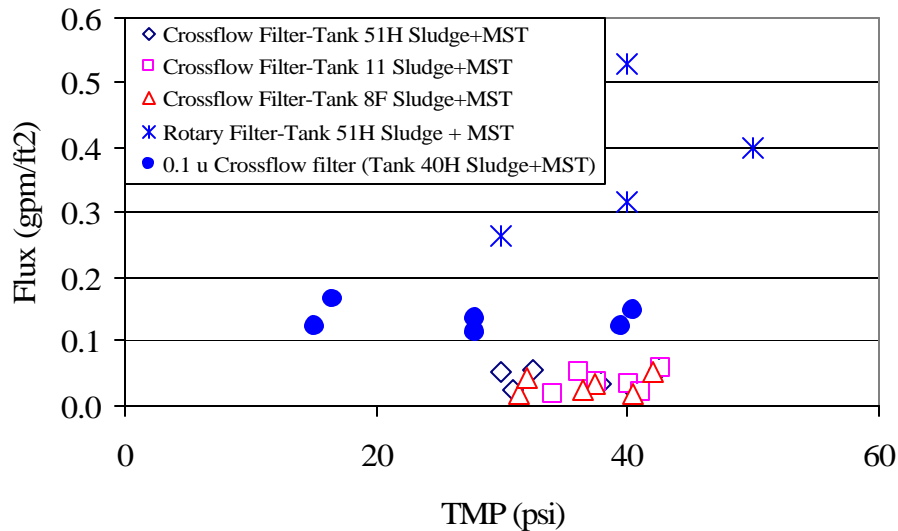
SRTC researchers tested the rotary microfilter as an alternative to the crossflow filters in the current baseline of the Salt Waste Processing Project and the Actinide Removal Project. Table 1 summarizes the results of scoping testing and Figures 1 and 2 show some of the data from the actual waste testing and pilot scale testing.<sup>2,3,4</sup> The data show significant improvement in filter flux with the rotary microfilter over the 0.5 micron crossflow filter (2.5 – 6.5 X during the scoping tests, up to 10 X in the actual waste tests, and approximately 2 X in the pilot-scale tests). The rotary filter also produced higher flux than the 0.1 micron crossflow filter (1.5 – 2 X during the scoping tests, and 2 – 5 X in the actual waste tests).

**Table 1. Comparison of SpinTek Filter with Conventional Crossflow Filter from Vendor Scoping Tests<sup>2</sup>**

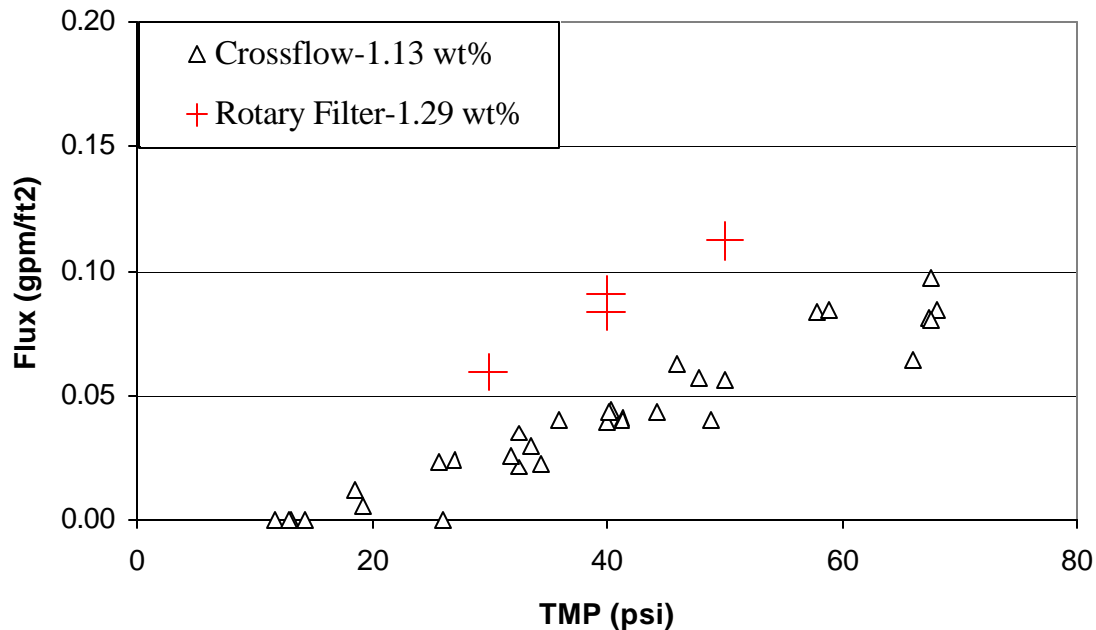
Solids (wt %)	Rotary (gpm/ft <sup>2</sup> , measured)	0.5 μ Crossflow (gpm/ft <sup>2</sup> , predicted)	Ratio	0.1 μ Crossflow (gpm/ft <sup>2</sup> , measured)	Ratio
0.05	0.21	0.08	2.6	0.13	1.6
0.22	0.19	0.07	2.7		
1.0	0.15	0.04	3.8		
4.8	0.13	0.02	6.5	0.069	1.9

The SRTC received funding from the DOE-HQ, Office of Cleanup Technologies, via the NETL, to continue developing the rotary microfilter for SRS high level waste applications. As part of this task, the authors performed pilot-scale simulant filtration tests with irradiated filter disks. We conducted the tests with filter disks irradiated with an estimated 2.5 - 5 year (83- 165 MRad) radiation dose. The tests provide additional operating data, allow us to evaluate the impact of

radiation on the rotary microfilter, and help us assess feasibility of using SpinTek's existing filter disk design in radioactive waste applications.



**Figure 1. Rotary Microfilter Actual Waste Test Results<sup>3</sup>**



**Figure 2. Rotary Microfilter Pilot-Scale Test Results<sup>4</sup>**

While the rotary filter produced higher flux than the 0.1 and 0.5 micron crossflow filters, the largest currently available rotary filter contains 25 ft<sup>2</sup> of filter area. Two 25 disk units would contain 50 ft<sup>2</sup> of filter area versus 230 ft<sup>2</sup> for the crossflow filter currently installed in 512-S.

The authors are currently working with the rotary filter manufacturer to develop and test a 50 ft<sup>2</sup> filter.

## TESTING

### Equipment

The SpinTek rotary microfilter unit at the University of South Carolina's Filtration Research Engineering Demonstration (FRED) is a Model ST-II-3, Laboratory Test Unit with three membrane disks for a total of 3 ft<sup>2</sup> active membrane area (see Figure 3). The disks spin inside a pressurized vessel with spoked turbulence promoters above and below each disk. Personnel can manually adjust the speed of the disk rotation between 500 and 1400 rpm. Increasing the rotational speed increases the shear forces at the surface of the disk. For the purpose of this test, we kept the disk rotational speed at  $1170 \pm 20$  rpm, except where noted.



**Figure 3. Pilot-Scale SpinTek Rotary Microfilter**

A valve on the concentrate exit automatically controls the pressure inside the filter housing. This pressure provides the transmembrane pressure required to force filtrate through the filter membranes. For the purpose of this test, we controlled the pressure at 40 psi. The FRED personnel added pressure sensors to the feed inlet and filtrate lines so they could collect data and calculate transmembrane pressures.

The feed slurry flows across the surface of the filter disks. A differential pressure drives the supernate through the filter membrane and into the center of the disks. The filtrate moves to the center of the disk and collects in the shaft holding the disks. The equipment provides no pressure

control on the filtrate line, with only a solenoid valve to stop filtrate flow when desired. We measured filtrate flow by use of a magnetic flow meter.

Personnel manually controlled feed flow by adjusting the speed of the feed pump. We measured feed flow with a magnetic flow meter. For the purposes of this testing, we maintained feed flow between 3.8 and 4.2 gpm.

The feed tank has a working capacity of 115 L. The agitator in the feed tank operates at a variable speed with a single marine blade. The feed tank includes a sensor for the Lasentec<sup>®</sup> particle size analyzer.

We provided automatic temperature control for the system with a heat exchanger located on the line from the feed pump to the filter housing. Personnel supplied cooling water from a remote source and maintained the temperature with the control valve on the skid.

Materials of construction for the unit are all corrosion resistant (i.e. stainless steel, Teflon<sup>™</sup>, etc.)

The onboard Programmable Logic Controller (PLC) performs automatic control with data passed to the facility Data Control System for logging.

## **Test Protocol**

Personnel conducted the pilot-scale simulant rotary microfilter tests as follows. We selected three types of filter disks for these tests (0.5 micron stainless steel, 0.1 micron stainless steel, and 0.1 micron ceramic/stainless steel). All filters incorporated the stainless steel permeate carrier mesh. The ceramic membrane is SpinTek's standard filter media. Personnel analyzed the filter structural material, Ryton<sup>®</sup>, for hardness, and irradiated the entire disk in a Co-60 source. We also measured the hardness of the Ryton<sup>®</sup> after the irradiation of the disk to determine if a change in material properties occurred. The stainless steel filters received an estimated 5-year radiation dose, and the ceramic filters received a 2.5 year radiation dose. Following the irradiation, USC personnel placed the filters in the pilot-scale rotary microfilter unit and tested them with feed slurries containing 0.29 and 4.5 wt % solids. We tested each set of filter disks for at least one week. These loadings allow comparison with previous crossflow filter test data.<sup>8,9,10,11,12</sup>

Personnel conducted the tests with sludge plus MST slurry. They prepared the feed from previously used test slurries.<sup>4,7,9,11</sup> We selected these slurries to match the solids used in previous crossflow filter tests.<sup>3,4,8,9,10,11,13,14</sup>

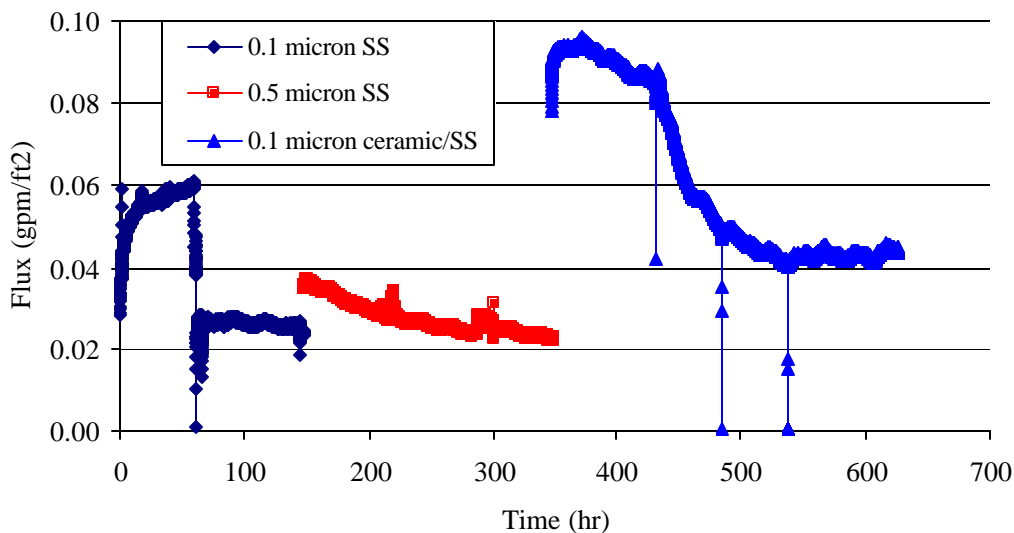
We prepared the sludge plus MST slurry in the following manner. Personnel decanted the supernate (i.e., 5.6 M sodium, "average" salt solution) from drums containing sludge and MST. We analyzed the supernate for insoluble solids to determine the mass of solids needed to achieve the target concentrations. We analyzed settled solids from the drums for insoluble solids concentration and added them to the feed tank to achieve the target solids loading.

Once the feed tank contained the desired slurry (nominal 0.29 wt % insoluble solids), personnel started the rotary microfilter and circulated the feed. They set the transmembrane pressure to 40 psi. After a pre-determined operating time, personnel increased the solids loading to nominal 4.5 wt % and operated the unit with the new feed slurry.

## RESULTS

### Filter Flux

Figure 4 shows the filter flux plotted as a function of time during the test. Personnel performed the first test with 0.1 micron stainless steel filter disks irradiated with 165 MRad (estimated 5 year dose). The initial feed slurry contained 0.29 wt % insoluble solids. They operated the rotary microfilter for 60 hours. The flux averaged 0.060 gpm/ft<sup>2</sup> over the last hour. Personnel then increased the solids loading to 4.5 wt % and operated the filter for an additional 88 hours. The filter flux averaged 0.024 gpm/ft<sup>2</sup> over the last hour.



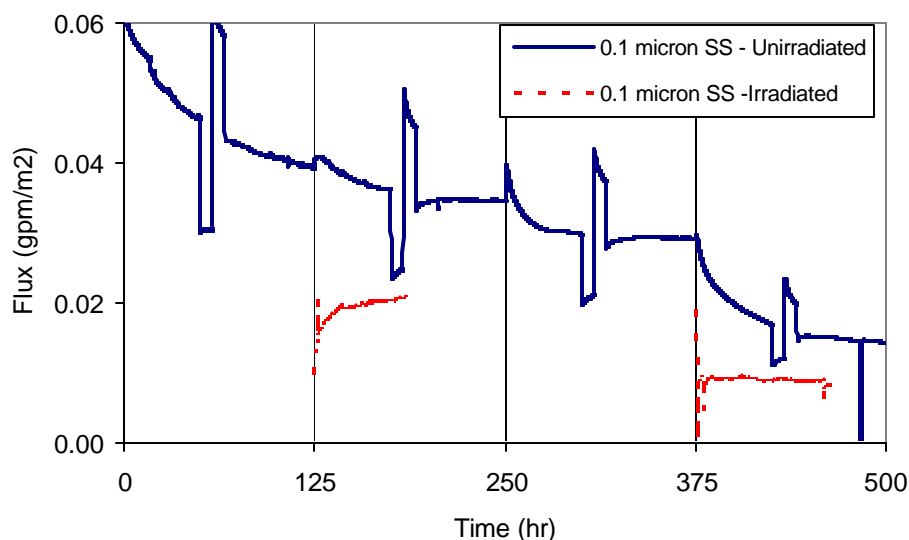
**Figure 4. Rotary Microfilter Performance with Irradiated Filter Disks**

Personnel flushed the filter unit with 0.01 M NaOH, removed the 0.1 micron filter disks, and installed 0.5 micron stainless steel filter disks irradiated with 165 MRad (estimated 5 year dose). The initial feed slurry contained 0.29 wt % insoluble solids. They operated the rotary microfilter for 152 hours. The flux averaged 0.026 gpm/ft<sup>2</sup> over the last hour. Personnel then increased the solids loading to 4.5 wt % and operated the filter for an additional 48 hours. The filter flux averaged 0.023 gpm/ft<sup>2</sup> over the last hour.

Personnel flushed the filter unit with 0.01 M NaOH, removed the 0.5 micron filter disks, and installed 0.1 micron ceramic/stainless steel filter disks that had been irradiated with 83 MRad (estimated 2.5 year dose). The initial feed slurry contained 0.29 wt % insoluble solids. They operated the rotary microfilter for 84 hours. The flux averaged 0.085 gpm/ft<sup>2</sup> over the last hour.

Personnel then increased the solids loading to 4.5 wt % and operated the filter for an additional 194 hours. The filter flux averaged 0.045 gpm/ft<sup>2</sup> over the last hour.

Figure 5 compares filter flux of the irradiated 0.1 micron stainless steel filter with the flux of an unirradiated filter from a previous test.<sup>4</sup> With 0.1 micron stainless steel irradiated filter disks, filter flux over the last hour averaged 0.06 gpm/ft<sup>2</sup> at 0.29 wt % solids compared to 0.10 gpm/ft<sup>2</sup> with unirradiated filter disks. At 4.5 wt % solids, the filter flux over the last hour averaged 0.024 gpm/ft<sup>2</sup> using irradiated filter disks compared with 0.04 gpm/ft<sup>2</sup> using unirradiated filter disks. The flux with the irradiated filter disks proved 35 – 40% lower than the flux measured with unirradiated filter disks.

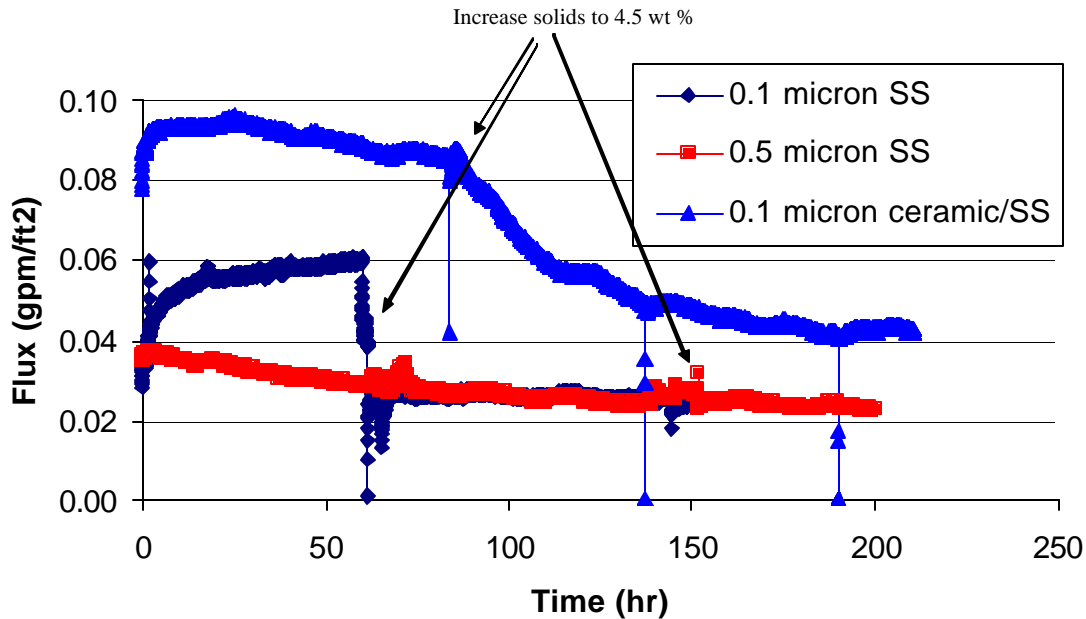


**Figure 5. Comparison of Filter Flux between Irradiated and Unirradiated Filter Disks**

This result is surprising, because radiation should not affect the stainless steel filter media. A likely cause of the difference is that personnel did not thoroughly clean the filter disks following the 2002 rotary microfilter test.<sup>4</sup> The sludge and manganese dioxide solids that adhered to the filter in that test likely dried and set in the filter. Presence of these solids may have lowered the flux in the more recent tests.

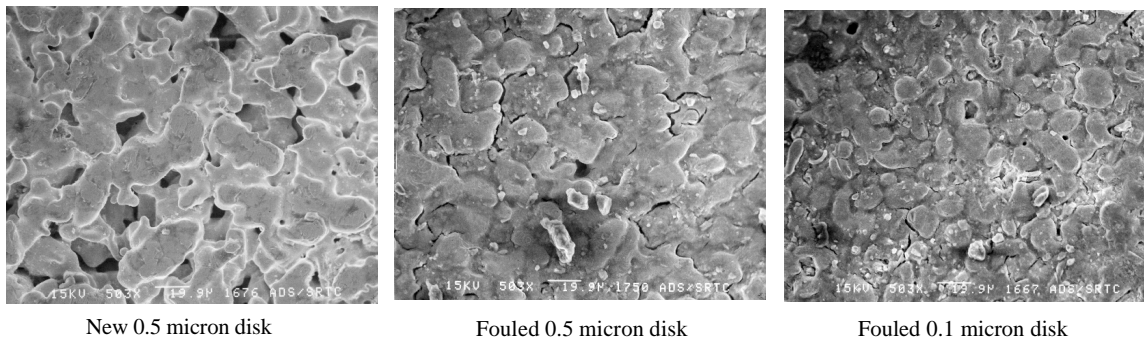
Another plausible explanation for the difference in flux between the irradiated and unirradiated filters is differences in particle size distribution for the two feeds. We will discuss this hypothesis later.

Figure 6 compares the flux measured with the different filter media. The 0.1 micron ceramic/stainless steel filter produced the highest flux at both solids loadings. The 0.1 micron stainless steel filter produced higher flux than the 0.5 micron stainless steel filter at the lower solids loading, and the same flux at the higher solids loading. The result is consistent with previous crossflow filter test results that showed a 0.1 micron stainless steel filter produces higher flux than a 0.5 micron stainless steel filter.<sup>8,13</sup>



**Figure 6. Comparison of Filter Media**

Figure 7 shows Scanning Electron Microscope (SEM) pictures of the “new” 0.5 micron filter, the “fouled” 0.5 micron filter, and the “fouled” 0.1 micron filter. The “new” 0.5 micron filter shows a very open pore structure. After testing, the 0.5 micron filter SEM picture shows a less open structure with many particles filling the pores. The SEM picture of the 0.1 micron filter after testing shows solid particles located on the filter surface. If the particles form a filter cake rather than accumulating in the filter pores, the resistance is lower and filter flux remains higher.

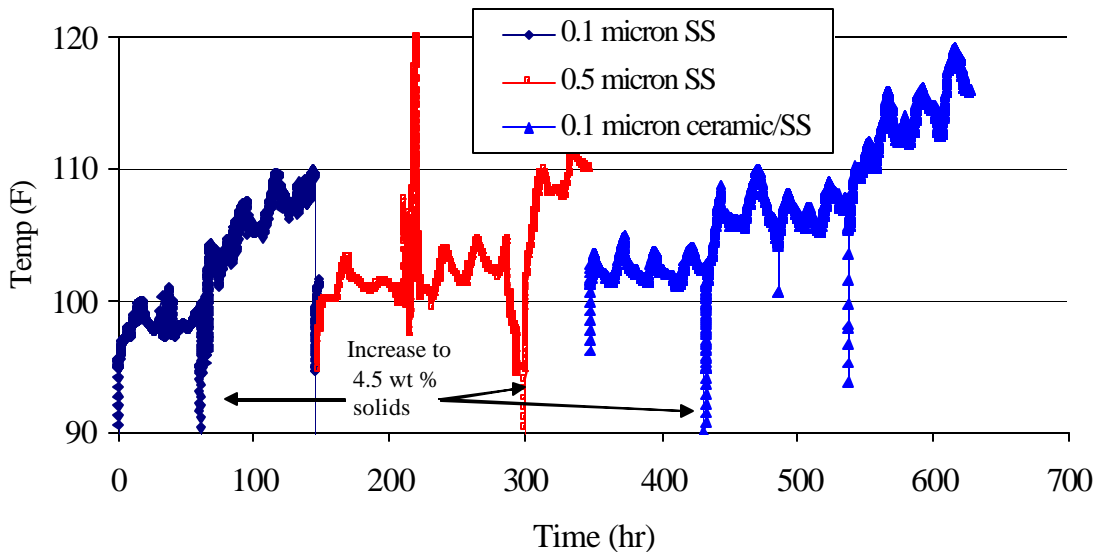


**Figure 7. SEM Photos of Stainless Steel Filter Media**

### Feed Temperature

Figure 8 shows the slurry temperature during the rotary filter testing. Operation targeted a temperature of  $95 \pm 5$  °F. The data show we had difficulty maintaining the slurry temperature during the test. The tests occurred during August, when ambient temperatures can exceed 100 °F. After reviewing the operating experience from the test, personnel judged the heat

exchanger as undersized for this application, and procured a larger heat exchanger for future testing.



**Figure 8. Feed Slurry Temperature**

### Particle Size Data

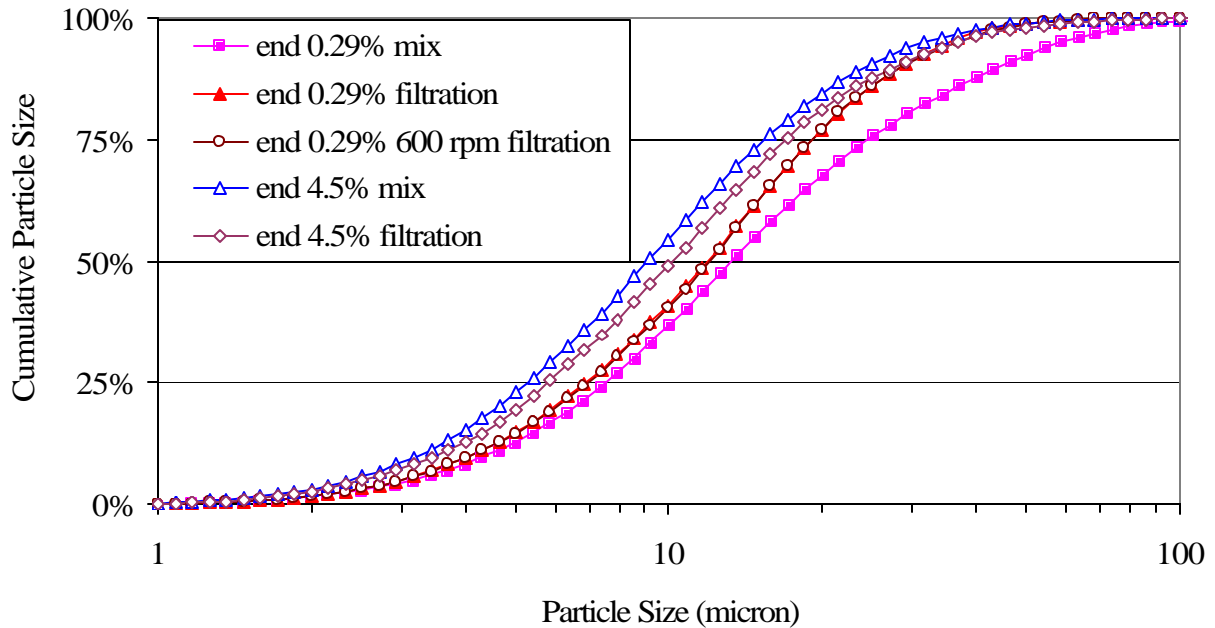
Personnel collected particle measurements with a Focused Beam Reflectance Measurement (FBRM) probe (Lasentec®). The probe works in the following manner. Personnel installed the probe in the feed tank. The laser beam projects through the window of the FBRM probe and focuses just outside the window surface. This focused beam follows a path around the circumference of the probe window. As particles pass by the window surface, the focused beam will intersect the edge of a particle. The particle will backscatter laser light. The particle will continue to backscatter the light until the focused beam reaches the opposite edge of the particle. The instrument collects the backscattered light and converts it into an electronic signal.

The FBRM isolates the time of backscatter from one edge of an individual particle to its opposite edge. The software records the product of the time multiplied by the scan speed as a chord length. A chord length is a straight line between any two points on the edge of a particle or particle structure (agglomerate). FBRM typically measures tens of thousands of chords per second, resulting in a robust number-by-chord-length distribution.

The chord-length distribution provides a means of tracking changes in both particle dimension and particle population. The calculations do not assume a particle shape. The chord-length distribution is essentially unique for any given particle size and shape distribution. Assuming the average particle shape remains constant over millions of particles, changes to the chord-length distribution reflect solely a function of the change in particle dimension and particle number.

The authors will present the FBRM data as particle size (i.e., chord length) distributions and as calculated mean chord lengths (un-weighted). Figure 9 shows the particle size distribution

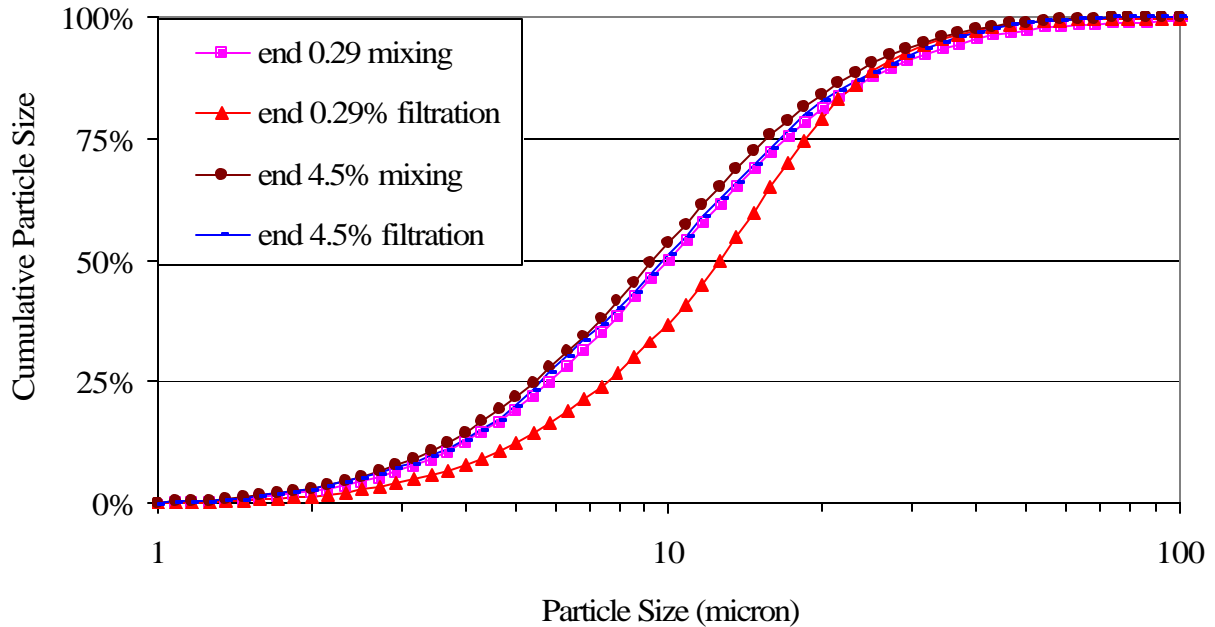
during the test with the 0.5 micron stainless steel filter. The initial 0.29 wt % feed slurry had a median particle size of 13 micron. Following 60 hours of filtration, the median particle size decreased to 12 micron. The figure shows a reduction in the number of large particles, which is likely due to the shearing caused by the filter feed pump. Reducing the rotor speed from 1170 rpm to 600 rpm did not impact the particle size. The curves for those data sets overlap. The median particle size of the initial 4.5 wt % slurry equals 9 micron, which is less than the median particle size of the 0.29 wt % slurry. After 88 hours of filtration, the median particle size increased to 10 micron.



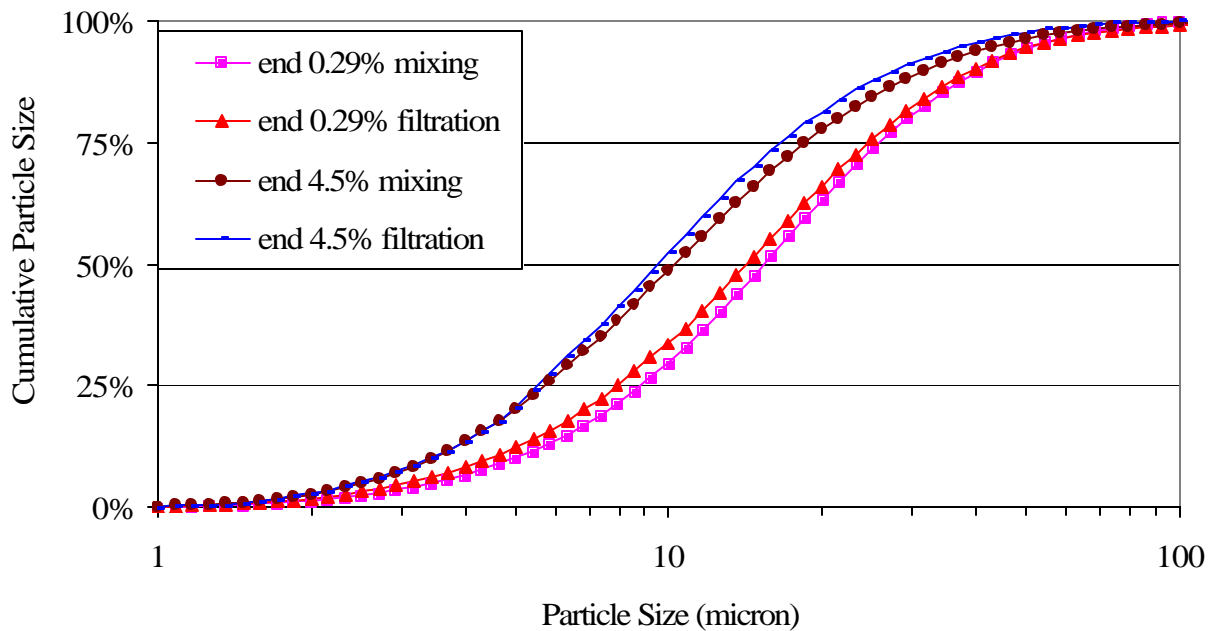
**Figure 9. Particle size data from the SpinTek Test with 0.5 Micron SS Filter**

Figure 10 shows the particle size distribution during the test with the 0.1 micron stainless steel filter. The initial 0.29 wt % feed slurry had a median particle size of 10 micron. Following 151 hours of filtration, the median particle size increased to 13 micron. The figure shows no significant change in the fraction of large particles. The median particle size of the initial 4.5 wt % slurry equals 9 micron, which is less than the median particle size of the 0.29 wt % slurry. After 49 hours of filtration, the median particle size increased to 10 micron.

Figure 11 shows the particle size distribution during the test with the 0.1 micron ceramic/stainless steel filter. The initial 0.29 wt % feed slurry had a median particle size of 15 micron. Following 84 hours of filtration, the median particle size decreased to 14 micron. The median particle size of the 4.5 wt % slurry equals 10 micron, which is less than the median particle size of the 0.29 wt % slurry. After 194 hours of filtration, the median particle size remained 10 micron.



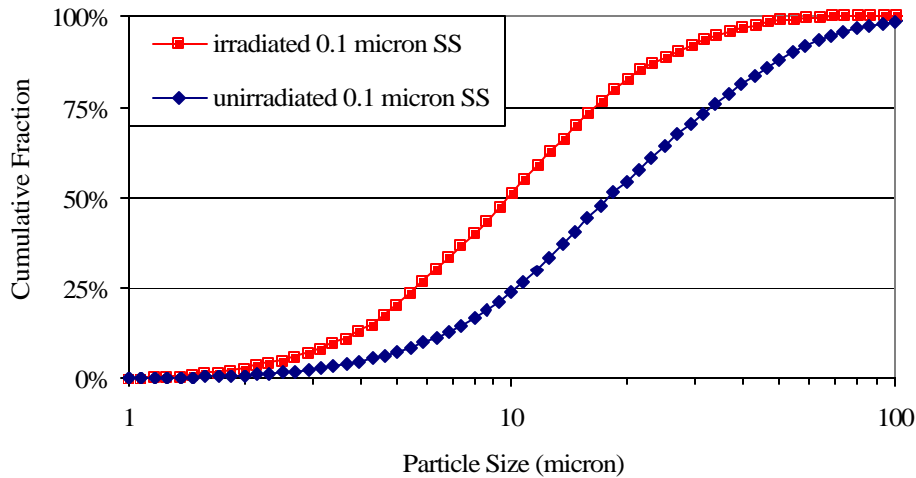
**Figure 10. Particle size data from the SpinTek Test with 0.1 Micron SS Filter**



**Figure 11. Particle size data from the SpinTek Test with 0.1 Micron Ceramic/SS Filter**

Figure 12 shows the particles size distribution of the feed slurry following the tests with 0.1 micron stainless steel filter disks. One test used disks irradiated with 165 MRad, and the other test used unirradiated disks.<sup>4</sup> The median particle size in the test with the irradiated filter disks equals 10 micron compared with 17 micron in the test with unirradiated filter disks. This

difference in particle size could explain the difference in flux observed in the two tests with the same filter media.



**Figure 12. Particle Size Following Tests with 0.1 Micron Filter Disks**

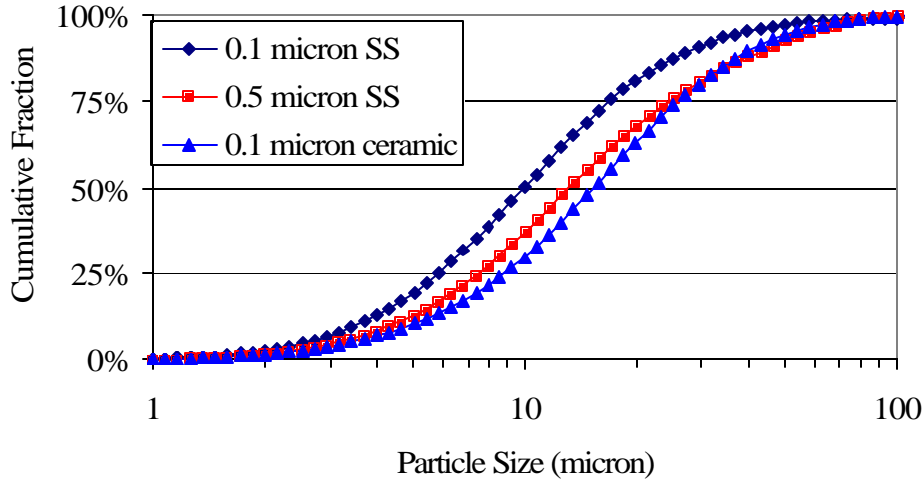
Table 2 and Figure 13 compare the particle size distributions of the initial feed slurries (0.29 wt % solids) used in each of the filter tests. The mean particle sizes in Table 2 differ slightly from those obtained from the particle size distribution plots. Table 2 shows the mean (i.e. average) particle size, and the figures provide data to calculate the median (50<sup>th</sup> percentile) particle size. The feed slurry in the 0.1 micron stainless steel filter test had the smallest median particle size, and the feed slurry in the 0.1 micron ceramic/stainless steel filter test had the largest median particle size. Even though the initial feed to the 0.1 micron stainless steel filter had a smaller median particle size than the feed to the 0.5 micron stainless steel filter, it produced a higher filter flux. The 0.1 micron ceramic/stainless steel filter produced the highest flux.

**Table 2. Mean Particle Size of Feed Slurries**

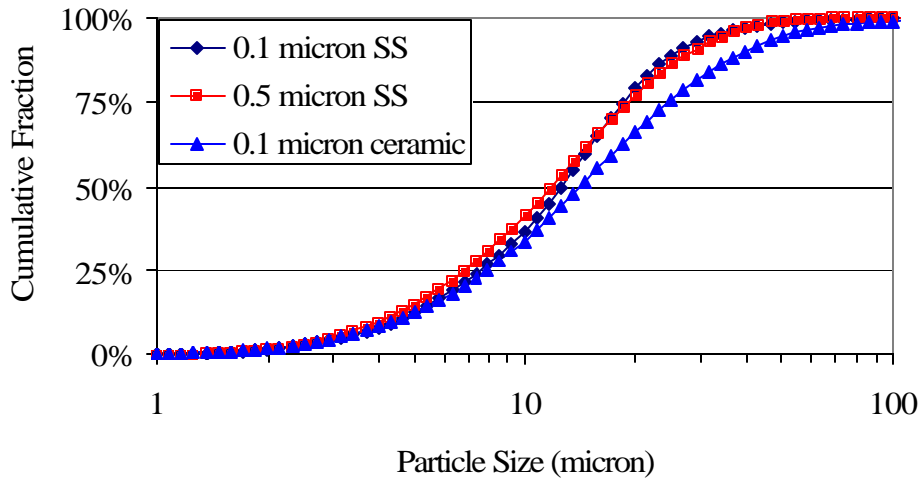
<u>Filter</u>	<u>Insoluble Solids (wt. %)</u>	<u>Time (start/finish)</u>	<u>Mean Particle Size (micron)</u>
0.5 SS	0.29	Start	13.8 ± 0.4
0.5 SS	0.29	Finish	13.2 ± 0.1
0.5 SS	4.5	Start	10.7 ± 0.6
0.5 SS	4.5	Finish	11.1 ± 0.1
0.1 SS	0.29	Start	13.2 ± 0.3
0.1 SS	0.29	Finish	13.2 ± 0.2
0.1 SS	4.5	Start	10.2 ± 0.3
0.1 SS	4.5	Finish	10.5 ± 0.2
0.1 ceramic	0.29	Start	17.0 ± 0.6
0.1 ceramic	0.29	Finish	15.3 ± 0.9
0.1 ceramic	4.5	Start	10.0 ± 0.4
0.1 ceramic	4.5	Finish	10.1 ± 0.1
0.1 SS-unirrad.	0.06	Start	15.4 ± 0.5
0.1 SS-unirrad.	4.5	Finish	13.0 ± 0.2

Figure 14 compares the particle size distributions of the 0.29 wt % feed slurries following filtration. The feed slurries in the 0.5 micron and 0.1 micron stainless steel filter tests had

approximately the same median particle distribution. The feed slurry in the 0.1 micron ceramic/stainless steel test had a slightly larger median particle size and a larger fraction of large particles.

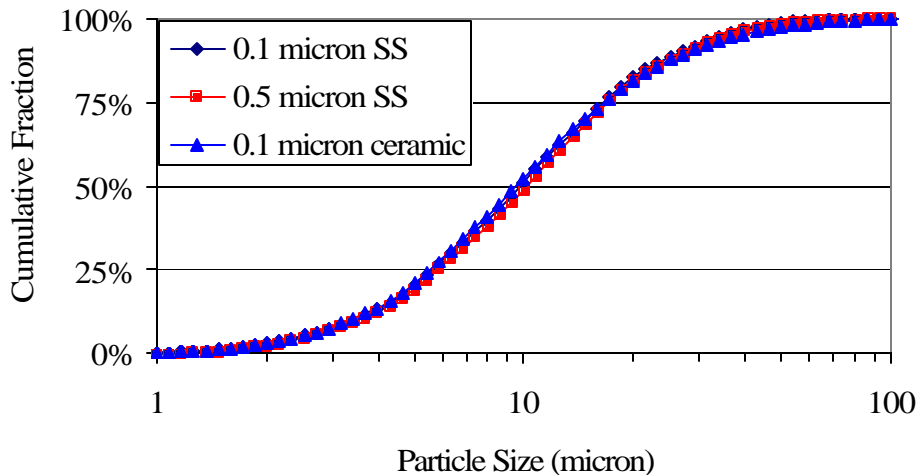


**Figure 13. Comparison of Initial Feed Slurries for Filter Tests**



**Figure 14. Comparison of 0.29 wt % Feed Slurries Following Filtration**

Figure 15 compares the particle size distributions of the feed slurries (4.5 wt % solids) at the conclusion of each of the filter tests. The distributions are essentially the same. Coincidentally, at the conclusion of the tests, the 0.1 micron and 0.5 micron stainless steel filters produced approximately the same flux (0.026 gpm/ft<sup>2</sup> versus 0.023 gpm/ft<sup>2</sup>). The ceramic/stainless steel filter produced a higher flux (0.042 gpm/ft<sup>2</sup>).



**Figure 15. Comparison of Feed Slurries at Conclusion of Filter Tests**

Figures 13 – 15 show that the initial feed in the tests with the 0.1 micron stainless steel filter had a smaller median particle size than the initial feed in the tests with the 0.5 micron stainless steel filter. A smaller median particle size produces a filter cake with higher resistance, which reduces filter flux. However, the 0.1 micron stainless steel filter produced a higher initial flux than the 0.5 micron stainless steel filter. In addition, by the end of the tests, the particle size distributions appeared the same for the three tests. After reviewing this data, as well as the scanning electron microscope data, we believe the reason for the lower flux with the 0.5 micron stainless steel filter is its larger pores allowed fine particles to enter the pores where they became trapped. This result is consistent with results from crossflow filter testing with similar feed slurries.<sup>13</sup>

### Reliability

During the testing, personnel collected filtrate samples periodically and analyzed them for turbidity (target < 5 NTU). During testing with the 0.1 micron SS disk, one sample showed high turbidity (> 200 NTU). Subsequent samples, collected every six hours, showed turbidity less than 5 NTU. We are uncertain of the cause of the high turbidity, but it did not persist through the test. All filtrate samples from tests with the 0.5 micron SS filters and the 0.1 micron ceramic filters showed turbidity less than 5 NTU. We believe this high turbidity sample was an anomaly.

Results indicate the filter continued to perform satisfactorily, though evidence of degradation is observed on some of the disks after a 5-year equivalent radiation dose. Therefore, the 2-year lifetime assumed in the risk assessment is conservative.<sup>15</sup> However, the irradiation occurred in air and does not include the effect of irradiation in a caustic environment.

None of the nine disks tested experienced a catastrophic failure, but some evidence of delamination exists. Separation started to occur between the epoxy and the base Ryton<sup>®</sup> material. Personnel sectioned the disks and observed that the joint remained sealed. Figure 16 illustrates the damage to the disk.

We tested the hardness of the Ryton<sup>®</sup> support plate as received and after exposure in the cobalt source. Hardness measurements used the Rockwell B scale with a 100 kg load and 1/16" indenter. A representative test block occurred after each set of tests and all tested within range.



**Figure 16. Delamination of Filter Disk**

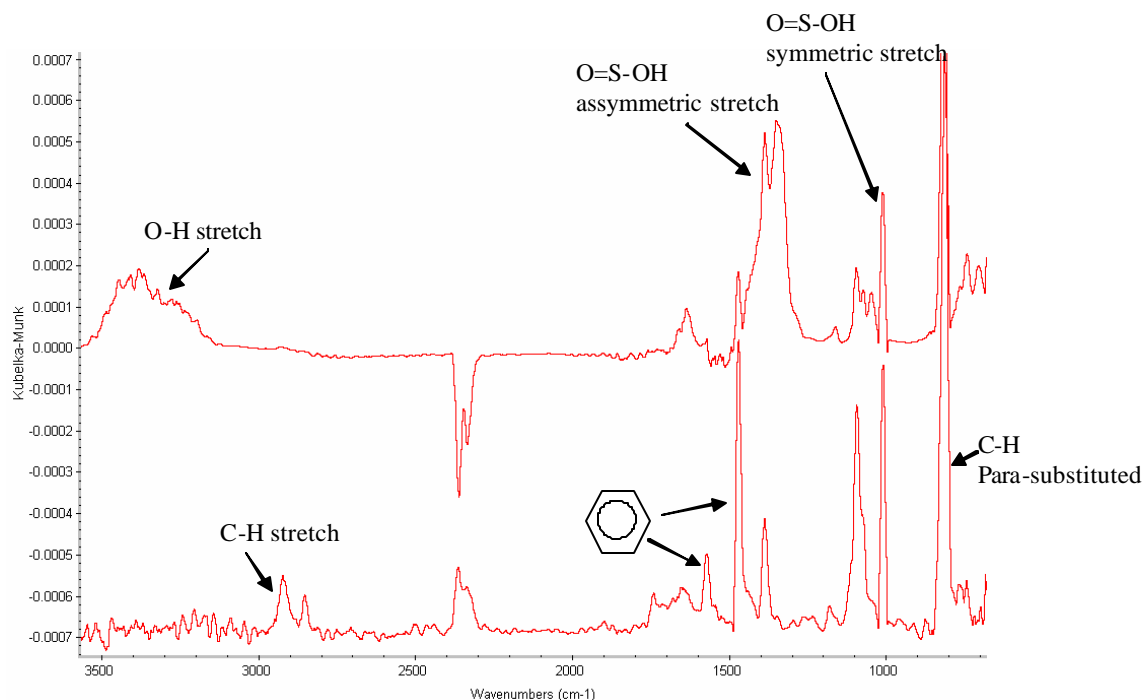
Table 3 shows the measurements and average hardness of the exposed Ryton<sup>®</sup>. The results show no pattern to the measurements. This result indicates that the damage to the Ryton<sup>®</sup> is on the surface of the Ryton<sup>®</sup> plate whereas the hardness measurement measures the bulk property.

**Table 3. Hardness Measurements of Ryton<sup>®</sup> Using Rockwell B Scale**

Disk	As Received	8.25E8 Rad Equivalent Dose	1.65E8 Rad Equivalent Dose
0.1 SS-1	8.7	9.2	7.8
0.1 SS-2	9.7	8.8	8.3
0.1 SS-3	9.0	6.3	6.2
Average	9.1 ± 0.5	8.1 ± 1.6	7.4 ± 1.1
CER-1	4.7	8.7	NA
CER-2	6.8	10.2	NA
CER-3	7.7	7.7	NA
Average	6.4 ± 1.5	8.9 ± 1.3	NA

An FTIR analysis of the Ryton<sup>®</sup> (polyphenylene sulfide) exposed to the five year equivalent dose showed the Ryton<sup>®</sup> degrading, weakening the bond between the Ryton<sup>®</sup> and the epoxy.

Comparisons of the disk material before and after irradiation from the FTIR analysis are shown as Figure 17. The unirradiated sample shows carbon-hydrogen stretches and phenyl groups, which are indicative of polyphenylene sulfide. Following irradiation, the sample shows an O-H stretch, an asymmetric O=S-OH stretch, and a symmetric O=S-OH stretch. These peaks indicate that the phenyl ring groups are destroyed (note the disappearance of the phenyl ring bands) while new sulfonic acid groups (oxidation and hydration of the sulfur atoms) are created on the surface.



**Figure 17. FTIR Analysis of Ryton**

It should be again noted that irradiation of the disks occurred in air and not caustic. The caustic could potentially accelerate the degradation of the disks due to radiation damage. A five year life is anticipated for the off the shelf vendor filter disks, and the two year life assumed in the risk assessment and cost/benefit analysis is probably conservative.

## FINAL COMMENTS

The testing conducted thus far shows the rotary filter produces higher flux than the baseline crossflow filter. Parallel work shows increased throughput in ARP process occurs by placing rotary filters in 512-S.<sup>17</sup> Details are contained in the referenced document. Because of the long cycle time (24 hours) required for the MST strike, the rotary microfilter benefits increase further when combined with methods to reduce the MST strike time or increase ARP filter utility.

## CONCLUSIONS

The conclusions from this work follow.

- None of the nine disks tested experienced a catastrophic failure from radiation exposure, but some evidence of delamination existed. Separation started to occur between the epoxy and the base Ryton<sup>®</sup> material.
- The flux with the irradiated filter disks was 35 – 40% lower than the flux measured with unirradiated filter disks. A likely cause of this difference is the feed to the irradiated filter disks had a smaller particle size than the feed to the unirradiated filter disks. Another plausible cause is that personnel did not thoroughly clean the filter disks following the 2002 test.
- The 0.1 micron ceramic/stainless steel filter produced the highest flux.
- The 0.1 micron stainless steel filter produced higher flux than the 0.5 micron stainless steel filter at the lower solids loading, and the same flux at the higher solids loading. Scanning electron microscope pictures show particles filling the pores of the 0.5 micron filter, and solid particles on the surface of the 0.1 micron filter.
- With the exception of one sample, all filtrate samples showed turbidity less than 5 NTU. The sample with high turbidity occurred during the test with the 0.1 micron stainless steel filter. We are uncertain of the cause. Subsequent samples with that filter showed filtrate turbidity less than 5 NTU, and all samples with a larger pore size filter showed turbidity less than 5 NTU. We believe this high turbidity sample was an anomaly.

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