

**Hydrogen Production via a Commercially Ready
Inorganic membrane Reactor**

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Abstract

The porous stainless steel substrate commercially available from Pall offers great potential for large-scale membrane based high temperature gas separations. Our proposed project involves the deposition of the M&P carbon molecular sieve-based hydrogen membrane on AccuSep substrate as a membrane to reactor water-gas-shift reaction. However, the AccuSep substrate was originally designed for liquid phase applications. During the 1st half, this commercial substrate has been modified and improved with regard to its surface topography and end seals. The substrate is now suitable for the deposition of the CMS membrane for hydrogen separation according to the characterization we performed. In addition, 40Å Al₂O₃ membrane layers have been deposited on the improved AccuSep substrate successfully. The SEM, EDX and pore size distribution analysis indicate that the 40Å membrane is extremely thin, and defect free with a narrow pore size distribution around 40Å primarily. As the above results suggest, we have made significant progress in preparing a high quality nominal 40Å (actually 50Å) layer on the Pall substrate. During the 2nd half of Year I, we will (i) continue this development work with a focus on eliminating the high pore size peak and (ii) begin the CMS layer deposition on the 40Å deposited AccuSep.

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1. Introduction

In the first six months of Year I, we have concentrated on the cursory evaluation of Pall's commercial stainless steel membranes as substrates for our proposed carbon molecular sieve (CMS) based hydrogen selective membrane. Although the Pall stainless steel substrate is commercially available (under trade name "AccuSep") in pore size as small as $0.1\mu\text{m}$, it is currently designed for liquid phase applications. To be qualified as a gas separation membrane for high temperature applications, Pall has had invested significant effort in working with us during this period to (i) eliminate leaks from the end seal, and (ii) improve the topography of the surface suitable for gas separation membranes. In addition, we have performed CMS thin film deposition on Pall's existing substrate. Although the selectivity obtained is reasonable, the permeance obtained is lower than that obtained from ceramic substrate. Hence, during this period, we have also concentrated on the improvement of the Pall substrate, including (i) smooth the surface of Pall's substrate, and (ii) deposition of a 40\AA Al_2O_3 sub layer. This semiannual report summarizes our findings in these areas.

2. Executive Summary

The porous stainless steel substrate commercially available from Pall (under Trade Name AccuSep) offers great potential for large-scale membrane-based high temperature gas separations, such as power generation applications. Our proposed project involves the deposition of the M&P Carbon Molecular Sieve (CMS)-based hydrogen selective membrane on AccuSep substrate for the water-gas shift reaction. The AccuSep substrate was originally designed for liquid phase applications. During the first half of Year I, this commercial substrate was modified and improved with regard to its surface topography and end seals. Then, the 40\AA Al_2O_3 layer was deposited on the improved AccuSep substrate successfully. The SEM, EDX and pore size distribution analysis indicate that the 40\AA membrane is extremely thin, and defect free with a narrow pore size distribution around 40\AA primarily. As the above results suggest, we have made significant progress in preparing a high quality nominal 40\AA layer on the AccuSep substrate ready for the deposition of the CMS hydrogen selective membrane. During the 2nd half of Year I, we will (i) continue this development work with a focus on eliminating the high pore size peak and (ii) begin the CMS layer deposition on the 40\AA deposited AccuSep.

3. Experimental

Experimental methods employed during this period include:

- Stainless Steel Substrate Qualification

Two experimental techniques have been employed by us to qualify Pall's stainless steel membrane as substrate. Bubble point of the substrate was measured to determine leaks of the end seals and defects of the substrate. The morphology of the stainless steel substrate was characterized using SEM and EDX.

- 40Å Al₂O₃ Membrane Characterization
The 40Å Al₂O₃ membrane deposited on Pall's substrate was characterized by (i) the selectivity of He vs. nitrogen, (ii) % initial flow to measure defects of the 40Å membrane, (iii) pore size distribution via a flow weighted pore size distribution analyzer. In order to prove the good attachment of this layer on the base stainless steel substrate, we have performed two tests: (i) thermal cycling, and (ii) boiling the membrane in water.
- CMS Membrane Characterization
Hydrogen and nitrogen single component permeances were measured at the temperature ranging from 25 to 300°C to characterize the hydrogen separation efficiency of the membrane. In addition, helium permeance was performed sometimes as a surrogate gas for hydrogen, while nitrogen was used to represent CH₄ and CO, commonly found in the WGS reaction.

4. Results and Discussions

4.1. Cursory Evaluation of Pall's Substrates for preparation of Gas Separations Membranes

The stainless steel substrate as received from Pall Corp (AccuSep) was used for the deposition of the precursor of the CMS membrane, which was then fired according to the protocol we have developed previously for the ceramic substrate. During October to December 2003, we began to transfer the technology from the ceramic substrate to the AccuSep. We fabricated 3 membranes initially with the first generation of AccuSep we received. The results are presented below:

Figure 1 shows the performance of Part Pall #1013 in terms of the hydrogen and nitrogen permeance and selectivity plotted as a function of temperature to ca. 415°C.

Several important points to consider from this data are as follows.

First, the hydrogen permeance is about an order of magnitude less than that which would be required for economical hydrogen recovery. Typical permeances on ceramic substrates are ca. 1 to 3 m³/m²/hr/bar at 180°C. The deposition procedure was modified for the next two parts (Pall #1014 and #1015) in an attempt to improve the hydrogen permeance as highlighted below. Second, the selectivity is good to temperatures above 400°C. Again, modifying the firing conditions should yield improved performance at these higher temperatures. Pall #1014 and 1015, were prepared using the same deposition procedure as that of #1013 but with progressively lower carbon precursor concentrations in an attempt to improve the membrane permeance. As can be seen, the H₂ permeance is improved considerably (yet still slightly below the target of at least 1 m³/m²/hr/bar.) However, the selectivity is reduced significantly.

As the table shows, increasing the firing temperature appears to improve the membrane selectivity. However, additional work is likely necessary, since the N₂ permeance appears to be increasing with temperature. This implies the pore size is still not tight enough. Another problem also appears to be the substrate tips. Following epoxy work, both the #1014 and #1015 membranes show significantly improved selectivity, but much reduced permeance. Several reasons for this effect are (i) a defect in the end seal, (ii) delamination of the carbon layer from the weld, and/or (iii) some effect of the epoxy “vapor” during curing on the membrane performance. These problems have been corrected later by Pall Corp and M&P (see Sec. 3.2 & 3.3).

4.2. Defects of Substrate and Leaks of End Seals

Our previous experience concludes that defect of the substrate can be identified easily once a membrane layer (e.g., 40Å) was deposited thereon. Based upon our experience with the 40Å membrane preparation, the %IF represents flow through defects (cracks, pinholes, etc.) in the 40Å layer that simply expose the underlying substrate. The %IF reflects the percentage of gas flow in the part through pores larger than approximately 100 to 150Å. Based upon the initial success with 40Å slip we used, several batches of membranes were prepared to characterize the substrate via the % IF measurement. Each series (2 through 5) was prepared using a new/fresh batch of solution. In general, the results were good with %IF results typically less than 10% on initial testing and below about 4% after repair of the end seal and/or cutting of the parts to remove obvious flaws in the layer.

Table 1 highlights these various test results and also describes testing conducted to assess the source of the high defect levels observed in several of the parts. In general, flaws in either the end seal and/or the layer contributed significantly to the high defect levels seen in the membranes. A short description of the results for several of the tubes is discussed below to highlight the general defect analysis testing that was conducted.

Parts 5B and 5C: A 40Å deposition was conducted on “old style” Zr.001 substrates. Very poor results were obtained. No further work was conducted with the Zr.001 substrates.

Part 5E: This part shows a number of different flaws that appeared in the membrane yielding very high defects in the 40Å membrane. In general two specific areas are targeted, (i) the end seal region and (ii) the center of the part. Both visual inspection and bubble point testing was conducted with this part (and several others). As the table indicates, following 40Å deposition, this part displayed a defect level on the order of ca. 29%. Visual inspection of the part (looking down the center of the tube) showed a number of flaws in the surface layer. Further, bubble point testing of the part (pressurize the outside of the part, fill the inside of the part with water, and look for bubbles) revealed a large number of bubbles from the center of the part with several flaws also apparent in the end seal. Based upon these results, it was apparent that significant level of flaws in the part contributed to the high defect level in the 40Å layer. This was verified in successive %IF tests conducted with part after:

- (i) Removal of a large section of the part that showed substantial defects in the layer thereby yielding a %IF of 9.7%, and
- (ii) Subsequent removal of the Pall end cap yielding a further reduction in the %IF to ~5%.

Based upon these results, significant flaws in the substrate contributed substantially to the defect level in the 40Å membrane layer.

Part 5F: Similar to 5E, the 5F part initially showed very high %IF. But visual and bubble point inspection of the part showed a large number of flaws in the substrate layer and end seal. By progressively removing these sections from the part, the %IF improved substantially from ca. 15% to ~2%.

Part 5D: This part showed no visual flaws in the layer. Bubble point testing revealed a few flaws in the end seal and also in the center of the part. Patching the end seal with epoxy improved the %IF from 11.6 to 8%. No additional work was conducted with this part. However, it is expected that the %IF would improve further if the obvious flaws in the surface layer were removed from the part.

Part 5A: Similar to 5F. Even after complete removal of the end seal still showed relatively high %IF at ca. ~11%. Did not attempt to cut the part to remove any defects evident in the surface of the layer.

Part 5H: This 40Å membrane was initially better than the 5F membrane. This could be traced directly to the apparent higher quality of the substrate. Both visual and bubble point inspection of the substrate showed few flaws.

Part 5I: Similar to 5H. The 40% %IF is very good at 4.1 and 3.0% before and after end seal epoxy patching. This data is consistent with the apparent higher quality of the substrate.

Substrate Inspection: The substrate was also inspected on several membranes including a few prior to deposition of the 40Å layer deposition. In general, flaws observed in the substrate translated directly into flaws in the 40Å membrane.

In general, the poor %IF performance of several of the membranes described above can be directly traced to flaws in the end seal and/or substrate surface layer. This was demonstrated by progressively removing these sections from the parts and retesting the %IF. SEM analysis of the surface of a 40Å deposited membrane and a substrate in the attached figure further supports this conclusion. As can be seen from these photomicrographs, obvious flaws in the substrate surface contribute directly to defects in the 40Å membrane. Further, these flaws were observed visually in the part prior to cutting for SEM inspection, suggesting that additional defects are present in the parts that are not easily identifiable by the crude visual inspection technique utilized.

Overall, it appears that relatively high quality 40Å membranes can be prepared on Pall AccuSep substrates. Low %IF results, <3 to 4%, could be obtained on many of the parts either initially or following removal of suspected flawed sections of the membrane. Of

the seven Series 5 parts (prepared on the 1st generation substrate), two showed good performance following only minor touch up of the end seal with some epoxy. The remaining five parts showed various levels of flaws in the surface of the layer, either by visual or bubble point inspection. These flaws in the 40Å layer, based upon SEM analysis and/or examination of several bare substrates, could in general be traced to flaws in the substrate. In the parts that were cut to remove these flawed regions, significant improvement in the membrane performance was obtained (e.g.: 5E and 5F). Defects and end seals of AccuSep substrates were corrected by Pall based upon the above findings. A good 40Å membrane layer can be prepared with the improved substrates (i.e., 2nd generation) as presented in Sec. 3.3.

4.3. 40Å Membrane Development

Nine improved substrates (2nd generation) based upon the result obtained from Sec. 3.2 were received from Pall Corp. Preliminary bubble testing of two of the parts showed no obvious problems, either from the center of the part or at the membrane to end seal interface. This appears to be a significant improvement over the previous end seal design. The bubble point of the part was consistent with in house our results with ca. 0.2µm pore size ceramic substrates, indicating the pore size of the substrate in the range of 0.2µm.

The 40Å membrane was deposited on one of these membranes using the formula established during our previous study (Sec. 3.2). This is a continuation of work conducted over the last several months with the objective to improve (decrease) the pore size of the Pall substrate from ca. 0.1 to 0.2µm to 40Å in an attempt to improve the CMS layer quality. We have successfully deposited a ca. 50Å pore size membrane on the Pall substrate. This pore size is intermediate to our 40 and 100Å pore size ceramic membranes. All of the Pall 40Å membranes display a bimodal distribution, with a second peak appearing in the 100 to 200Å range. At the moment, it appears that the source of this larger peak can be attributed to the surface roughness of the Pall substrate. Through optimization of the deposition strategy during this period, the contribution of this peak to the overall flow of the part has been successfully reduced from ca. 40 to 50% to 10 to 15%. Additional optimization is expected to further reduce the contribution of this peak. In preliminary challenge tests, the layer integrity appears to be good. CMS deposition is underway on one of the parts but no results are available as yet.

Figure 3 shows the pore size distribution of **all** of the nominal 40Å membranes prepared on Pall substrates at various deposition conditions. The data is compared with our 40 and 100Å ceramic membranes. (We have successfully prepared CMS membranes on these 40 and 100Å ceramic substrates).

Although Figure 1 shows a significant quantity of data, we have included all of the data to stress a particular point noted concerning the nominal 40Å Pall membranes. Specifically, all of these membranes showed a bimodal distribution with peaks at ca. 50Å (for all of the membranes) and an additional peak between 100 and 200Å. This is compared with the monomodal distribution of the M&P 40 and 100Å membranes

prepared using a similar deposition strategy. We are unsure of the source of the high pore size peak in these membranes but it appears to be related to the surface roughness of the Pall substrate. This is based upon the fact that the Pall 40Å-10 membrane (shown in Figure 3 and in Figure 4) displays the smallest high pore size peak. The Pall 40Å-10 membrane was the best of the batch. About 10-15% of the total flow through this membrane occurs through these larger pores, compared to up to 50% for several of the other membranes.

Both Figure 1 and Figure 2 show that the “lower” pore size developed on the Pall substrate is ca. 50Å. This pore size should be adequate for the CMS layer deposition, since we have successfully prepared high quality CMS membranes on our 40 and 100Å ceramic substrates.

Figure 4 shows the pore size distributions of the Pall 40Å-10 substrate before and after challenge testing in boiling water, compared with the M&P ceramic membranes. This challenge test is the first of a series that will be conducted on this membrane to verify its layer stability and attachment.

As the results suggest, we have made significant progress in preparing a high quality nominal 40Å (actually 50Å) layer on the Pall substrate. During the 2nd half year of Year I, we will (i) continue this development work with a focus on eliminating the high pore size peak and (ii) begin CMS layer deposition on the various membranes.

SEM photomicrograph of the 40Å layer is presented in Figure 5 to 10. Figure 5 exhibits the inside surface coated with the 40Å layer along with the cross section of the stainless steel substrate. The cross section may have been melted during the cutting; its morphology was changed and should not be paid attention to. According to our experience, the inner tubular surface of the Pall's substrate after the 40Å layer deposition is observed at the bottom part of the picture. In comparison, Figure 6 presents the top surface of the bare stainless steel substrate. Because the 40Å membrane on our ceramic substrate shows a smooth surface with no grain boundary recognized, evidently the uneven surface observed here in Figure 1 is the result of the AccuSep substrate. The 40Å layer is very thin (1-3 μm, according to our experience with the ceramic substrates) and coated on the surface evenly. Figure 7 shows a similar top surface with the 40Å layer under a higher magnification. Figure 8 to 10 present the EDX probe analysis along the cross section of the membrane. No alumina was detected in Figure 8 and 9, which represent the cross section near the surface, but not the top surface of the membrane. Figure 10 representing the location on the top surface shows significant presence of alumina. Thus, the SEM and EDX examination conclude that the AccuSep substrate surface topography remains uneven although this particular batch (2nd generation) of samples is "smoothed" by Pall. However, the pore size distribution, and EDX and SEM analysis all conclude that a thin and nearly defect free 40Å membrane layer has been deposited on this substrate.

Table 1 Performance of the #1013, 1014, and 1016 membranes

Part ID	Temperature [°C]	H₂ Permeance [m³/m²/hr/bar]	N₂ Permeance [m³/m²/hr/bar]	H₂/N₂ [-]
1013	180	0.0656	0.00114	57
1014	180	0.513	0.0384	13.4
1014, T _{fire 1}	RT	0.549	0.024	22.5
	140	0.514	0.038	16
1014, T _{fire 2}	RT	0.215	0.0128	16
	144	0.305	0.0122	25
1014, epoxy tip	RT	0.0298	0.00074	40
	186	0.0397	0.00083	48
1015	RT	2.49	0.699	3.56
1015, epoxy tip	RT	0.137	0.00059	232
	78	0.217	0.00192	113
	140	0.261	0.0059	44.2

Table 2 Characterization of 40Å Membranes and their Substrates

Part ID	Substrate ID	%F	epoxy end	Substrate Quality			Finished 40A Membrane		
				Visual Flaw	End Bubble	Center Bubble	Visual Flaw	End Bubble	Center Bubble
40A Formula #1									
std	Zr.002	35	Yes	Poor performance of these membranes is related to the 40A deposition					
1/3	Zr.002	76	Yes						
1/10	Zr.002	93	Yes						
40A Formula #2									
	1.1 Zr.002	5.7	Y						
	1.2 Zr.002	2.5	Y						
	1.5 Zr.002	8.5	Y						
2B	Zr.002	1.01	Y						
2A	Zr.002	0.77	Y						
	Thermal cycling	3.92	Y	3x550C					
		3.79	Y	6x550C					
		4.76	Y	3x400C					
		6.01	Y	3x400C					
Series 3									
3A		33	Y						
3B		16.2	Y						
Series 4									
4A		4.6	Y	Part sent to R. Kleiner, no visual/bubble point inspection conducted.					
4B		5.4	Y	Part sent to R. Kleiner, no visual/bubble point inspection conducted.					
4C		0.56	Y	Part sent to R. Kleiner, no visual/bubble point inspection conducted.					
Series 5									
5A		14.2	N				None obvious	Yes, few	Yes, few.
		10.9	Y				None obvious	Still a few	Yes, few.
		8.9	Y	Remove ends completely and replace with epoxy			None obvious	None	Yes, few.
5B	Zr.001	38.6	Y	This part was a Zr.001 substrate.					
5C	Zr.001	39.8	Y	This part was a Zr.001 substrate.					
5D		11.6	N				None obvious	Yes, few	Yes, few.
		8.1	Y	Add epoxy over the ends			None obvious	None	Yes, few.
5E		29.1	Y				No data	No data	Yes, many in center
		9.7	Y	Cut away section of tube that showed significant visual flaws/defects.			Few	Many bubbles	
		5.02	Y	Cut off Pall endseals and retest.					
5F		15.4	N				No data	No data	Yes, many.
		7.8	Y	Add epoxy endseal			Yes, many.	Few	Many bubbles
		2.9	Y	Cut away section of part showing visual defects			None	None	Very few.
5G	H-30	19.7	N				No data	No data	Many
5H	H-151	7.2	N				None obvious	Several	Very few, small
		7.2	Y				None obvious	Several	Very few, small
		3.4	Y	Remove ends completely and replace with epoxy			None obvious	None	Very few, small
5I	H-166	4.1	N				1 bare spot	Few	Very few, small
		3.02	Y	Add endseal					
Substrates									
	G-187						Several	Not obvious	Large number
	H-166						1 bare spot	Few	None obvious
	H-219						None.	Few	None obvious
	G-121						None.	Few	None obvious

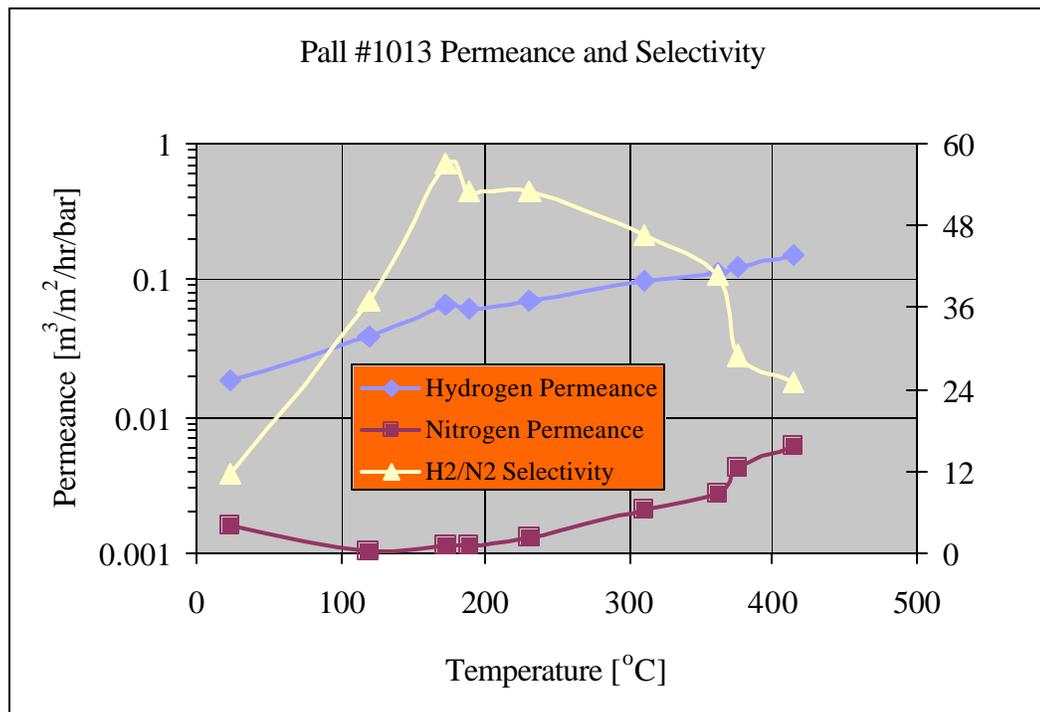


Figure 1: Permeance and selectivity of a carbon membrane prepared on a Pall stainless steel substrate (1st generation.)

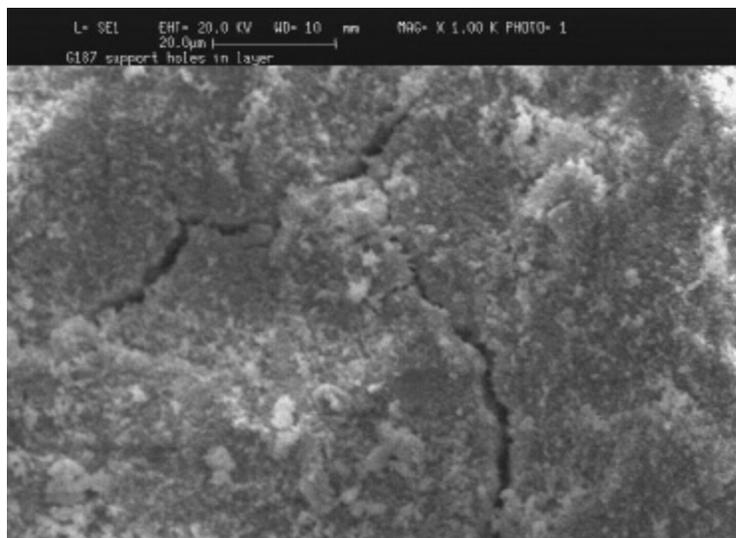
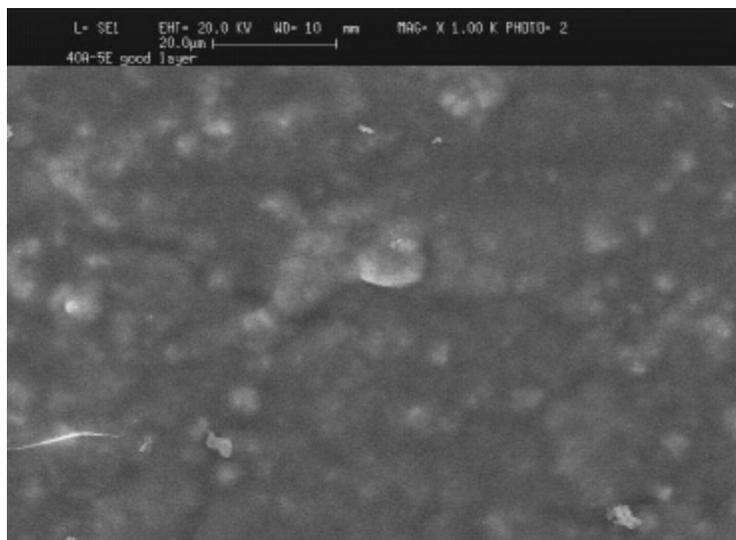
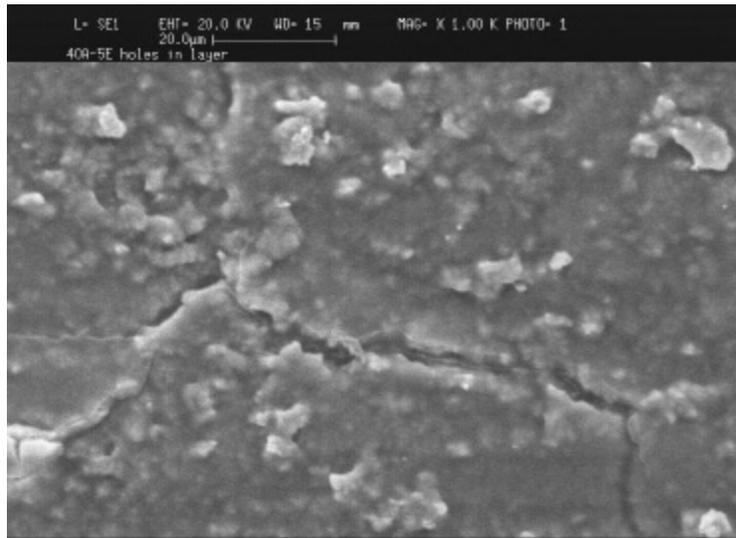


Figure 2 SEM Photomicrograph of AccuSep Substrate deposited with 40Å Al₂O₃ Membrane, cross section at 300X.

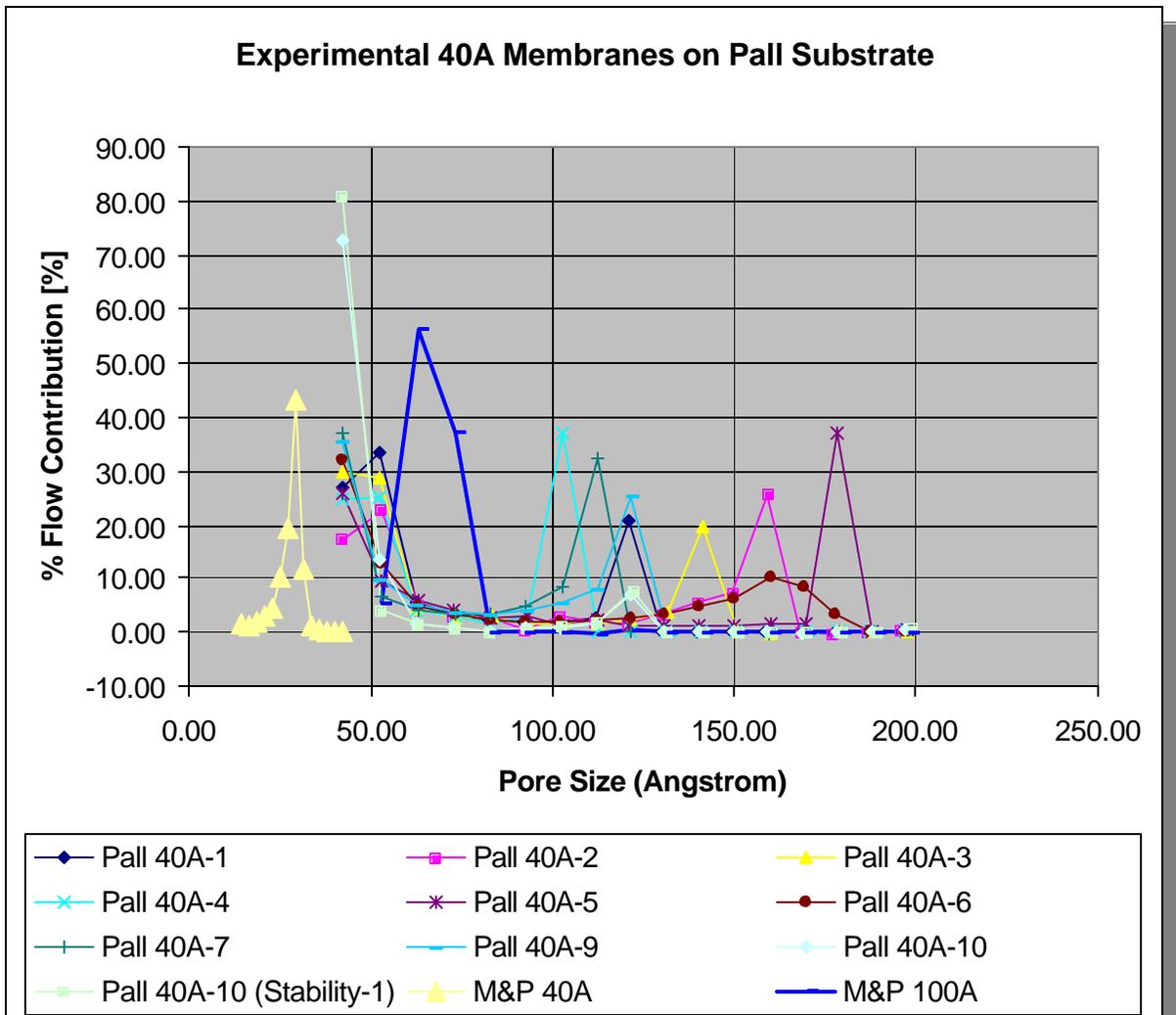


Figure 3: Pore size distributions of the Pall 40Å membranes prepared with “2nd generation” stainless substrate.

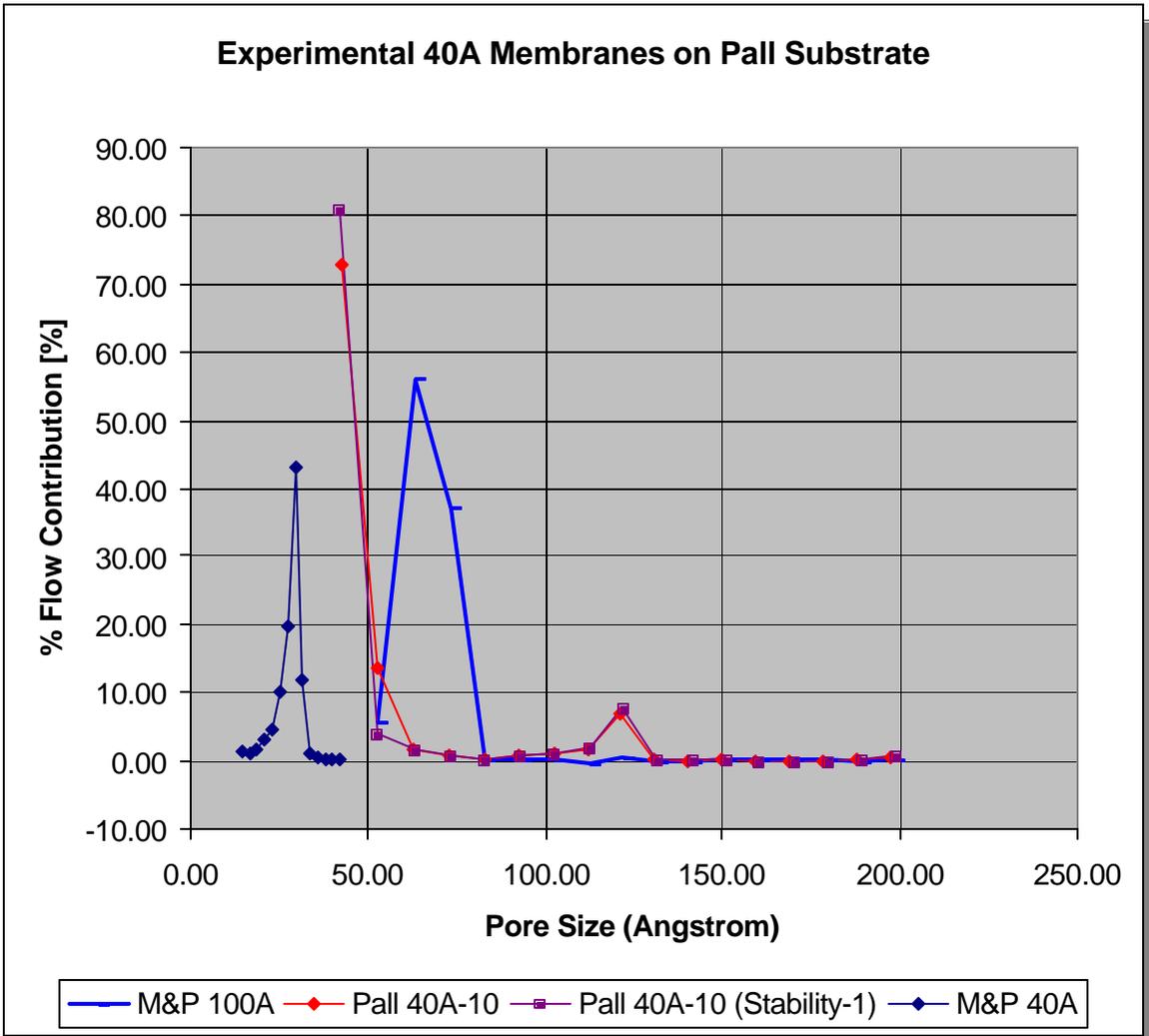


Figure 4: Pore size distributions of the Pall 40Å-10 membrane before and after challenge testing (boiling water) compared with M&P 40Å commercial ceramic membranes.

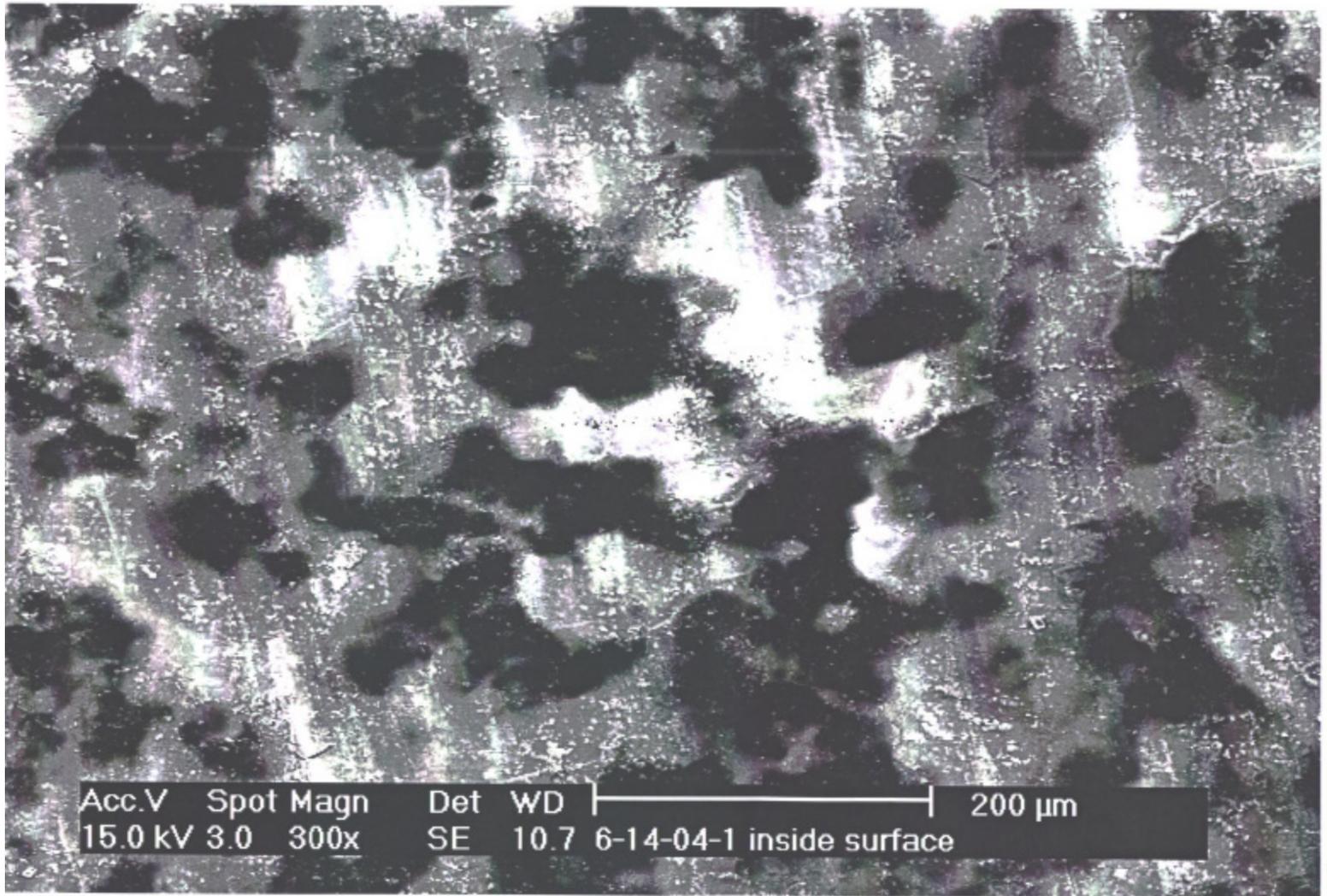


Figure 5 SEM Photomicrograph of AccuSep Substrate deposited with 40Å Al₂O₃ Membrane, cross section at 300X.

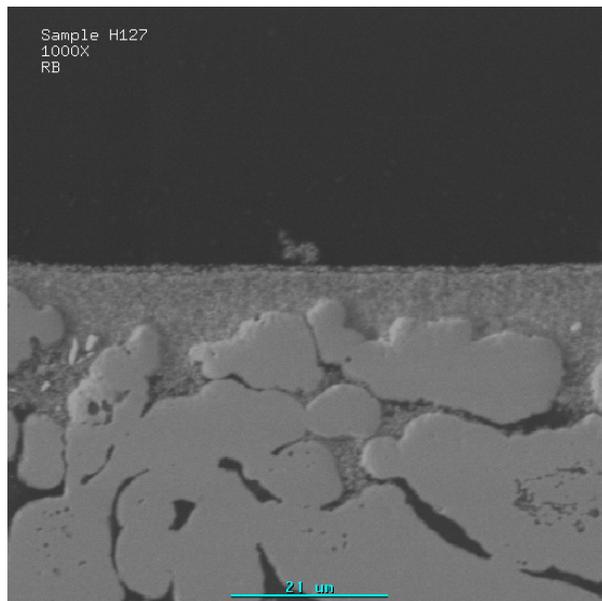
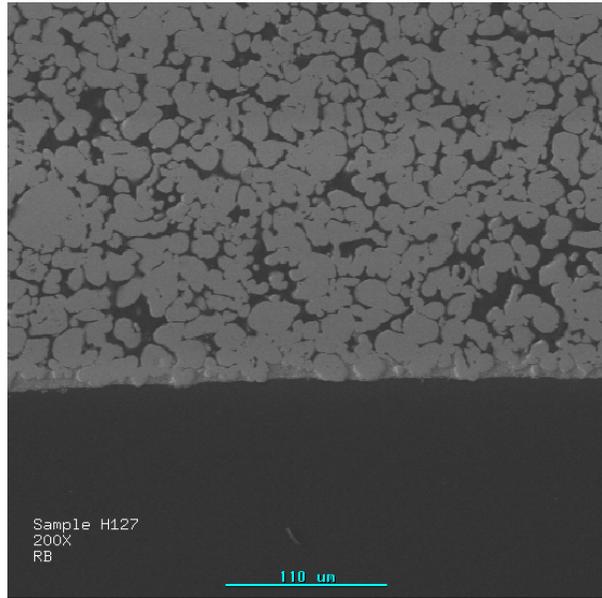


Figure 6 SEM Photomicrograph of AccuSep Substrate

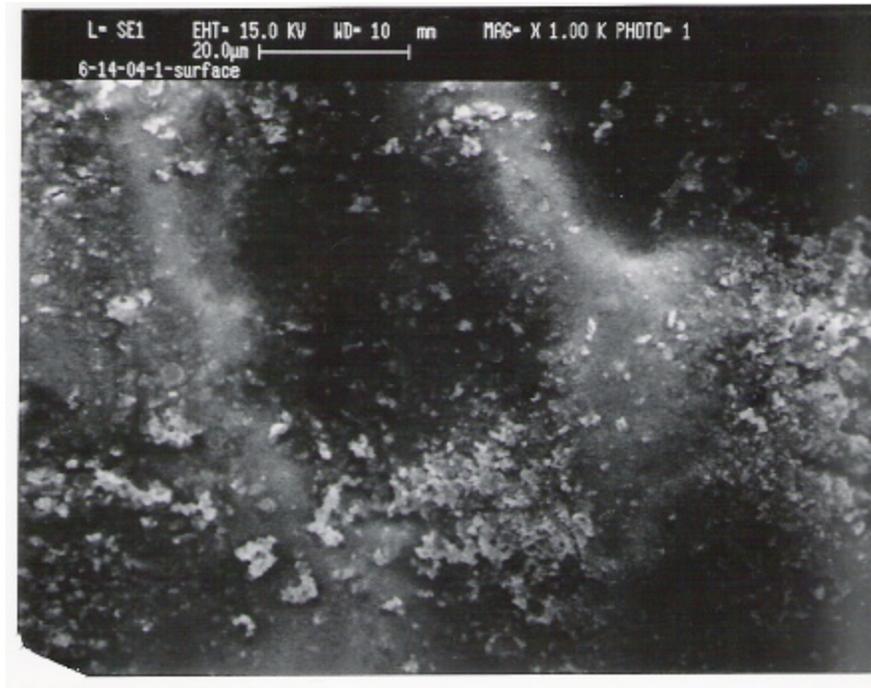


Figure 7 SEM Photomicrograph of AccuSep Substrate deposited with 40Å Al₂O₃ Membrane, cross section at 1000X

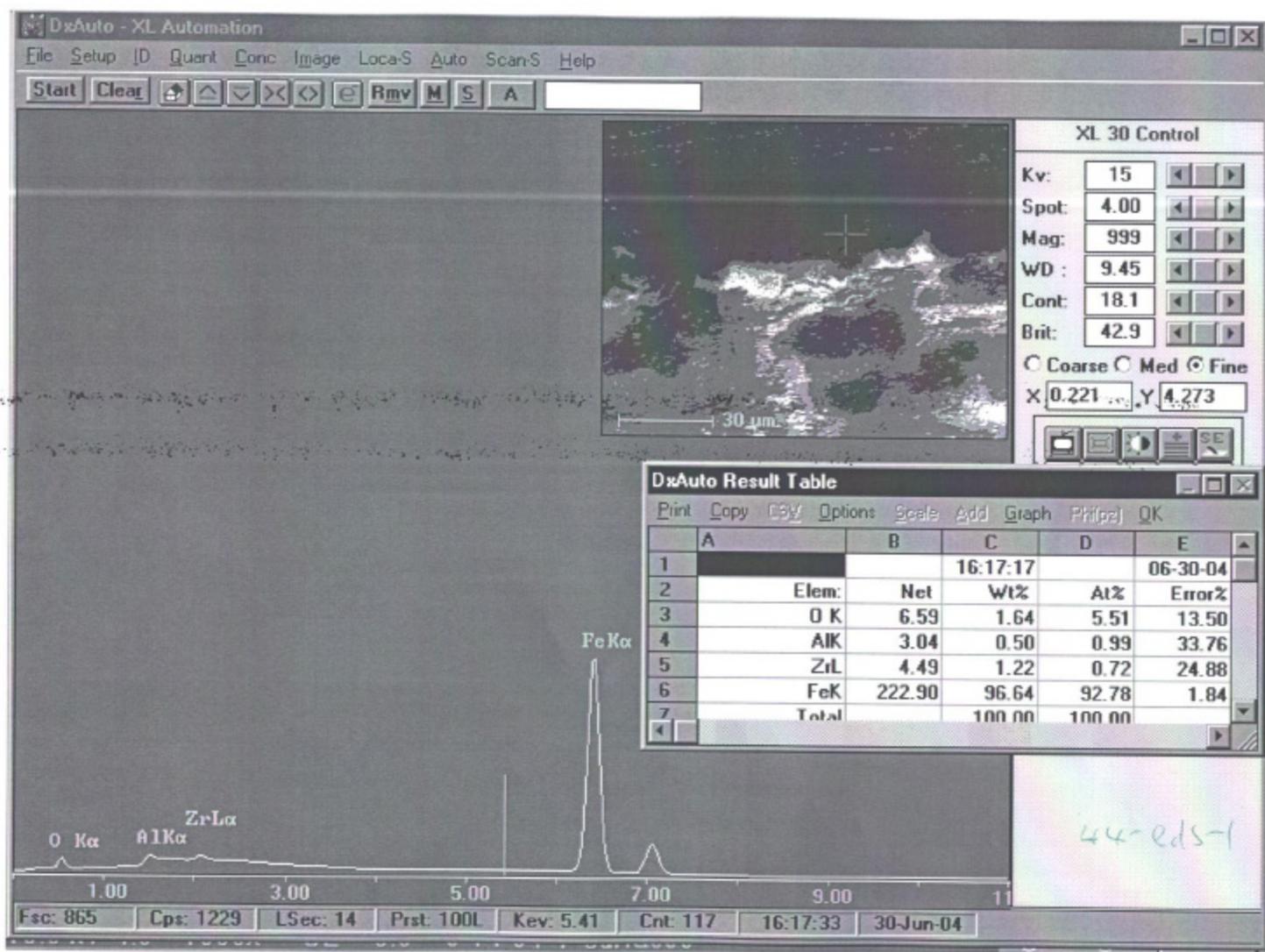


Figure 8 EDX Analysis of Cross Section of AccuSep Substrate deposited with 40Å Al₂O₃ Membrane, position 1.

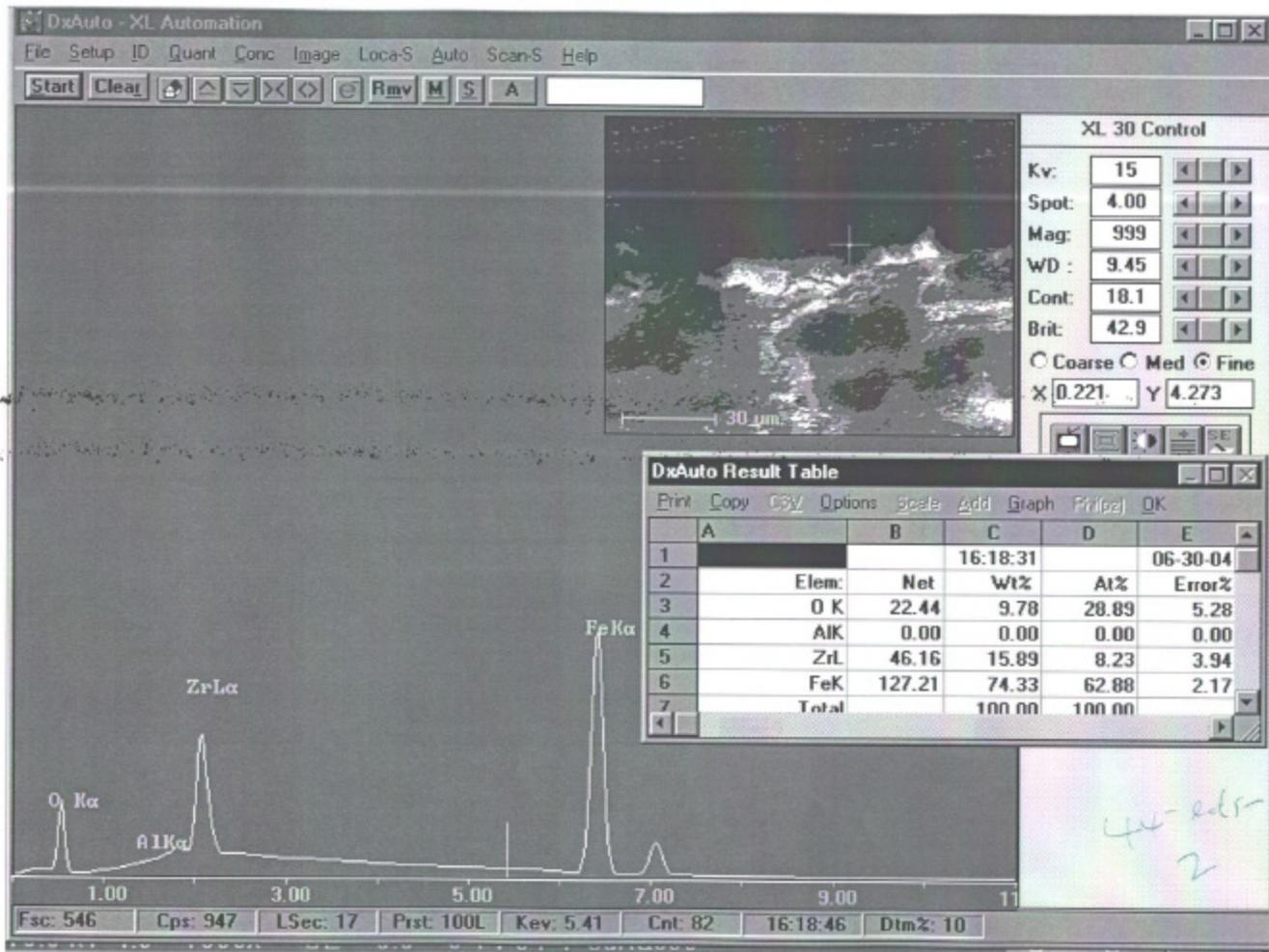


Figure 9 EDX Analysis of Cross Section of AccuSep Substrate deposited with 40Å Al₂O₃ Membrane, position 2.

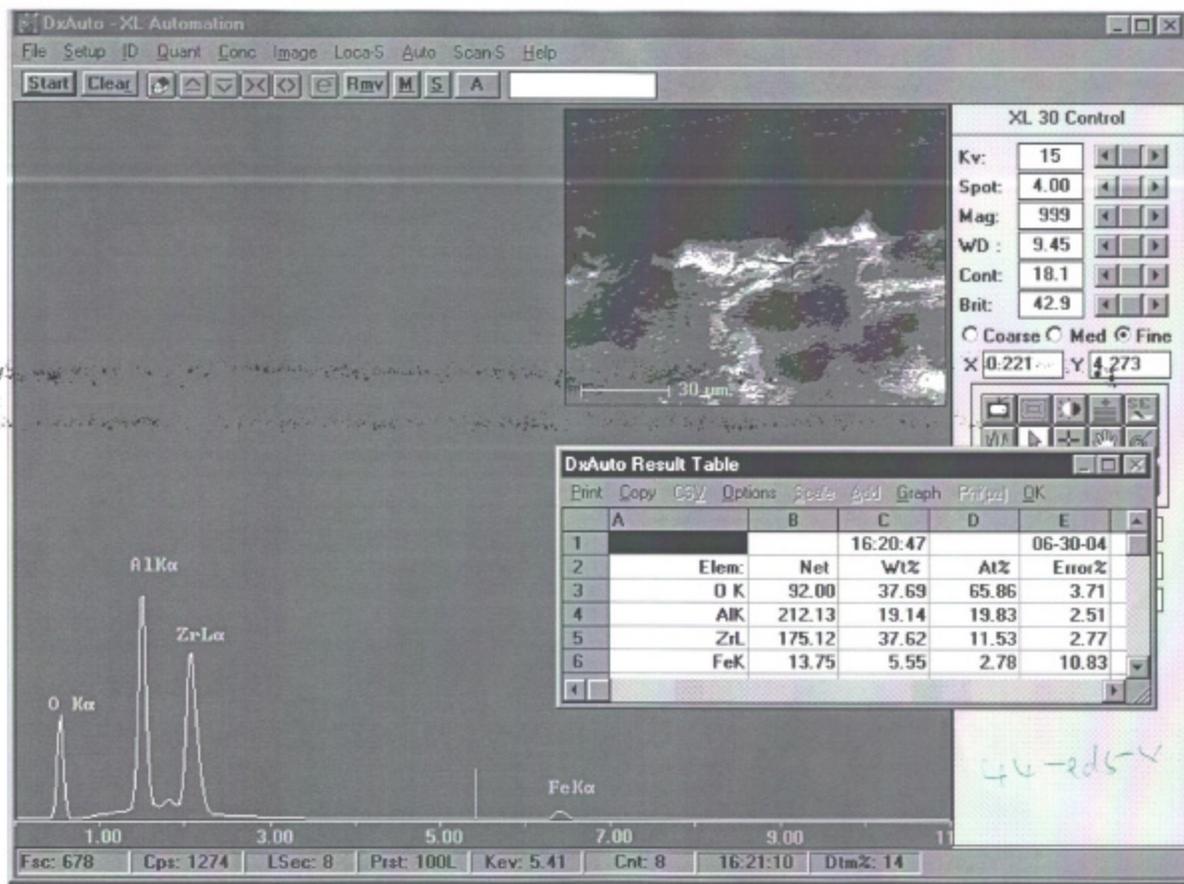


Figure 10 EDX Analysis of Cross Section of AccuSep Substrate deposited with 40Å Al₂O₃ Membrane, position 3 (top surface).

5. Conclusions

During the 1st half of Year I, we have concentrated on the evaluation of the Pall's commercial stainless steel substrate (AccuSep) and the improvement required for the deposition of M&P Carbon Molecular Sieve (CMS) hydrogen selective membrane. Our study has included:

- The AccuSep stainless steel substrate commercially available from Pall Corp for liquid phase applications has been modified and improved with regard to its surface topography and end seals. Based upon the characterization of the 40Å Al₂O₃ layer deposited thereon, we conclude that the substrate is suitable for the deposition of the CMS membrane for hydrogen separation.
- Our preliminary evaluation of the CMS membrane deposited on the substrate indicates that the selectivity of the CMS membrane meets the minimum selectivity requirement. However, the membrane permeance is reduced to an extremely low value in order to meet the selectivity requirement. We believe that the deposition of the 40Å membrane sub layer on top of the existing AccuSep substrate could correct this deficiency.
- Defects of substrate, rough surface topography, and leaks of the end seals were identified for existing AccuSep membranes (1st generation.) Several iterative studies between M&P and Pall were performed to correct these problems. A 2nd generation AccuSep substrate was prepared, which was successfully deposited with the 40Å Al₂O₃ layer (see below.)
- 40Å Al₂O₃ membrane layers have been deposited on the 2nd generation AccuSep substrate successfully. The SEM, EDX and pore size distribution analysis indicate that the 40Å membrane is extremely thin, and defect free with a narrow pore size distribution around 40Å. A minor 2nd pore size distribution around 100Å has been observed, possibly due to the uneven surface of the substrate. We believe that this minor contribution from the larger pore size is manageable for the purpose for the CMS membrane deposition.

As the results suggest, we have made significant progress in preparing a high quality nominal 40Å (actually 50Å) layer on the Pall substrate. During the 2nd half year of Year I, we will (i) continue this development work with a focus on eliminating the high pore size peak and (ii) begin CMS layer deposition on the various membranes.

Bibliography

None

Acronyms

SEM	Scanning Electro microscopic
EDX	Energy Dispersive x-ray analysis
M&P	Media and Process Technology, Inc.
AccuSep	Trade name of Pall's Stainless Steel Membranes
CMS	Carbon Molecular Sieve
% IF	Flow of the Membrane contributed by defects