Appendix 7.1.2-4

Disassembling Analysis and Evaluation of Fould Hollow Fine Fiber Membrane

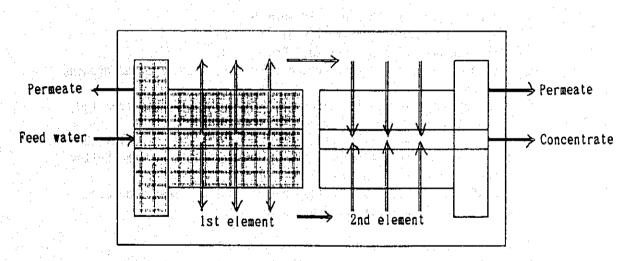
Disassembling, Analysis and Evaluation of Fouled Membrane

1. Objectives

One of the factors contributing to the deterioration of the performance of the module used for a long time is the adhesion of fouling substances to the membrane surfaces. Performance and life of the membrane, therefore, may be improved by analyzing those fouling substances and understanding their generating mechanism. Effective methods for analyzing fouling substances include (1) surface observation by SEM, (2) chemical element identification by EDX, and (3) qualitative analysis by IR. In the following, we would like to show the way we disassembled modules, took samples, and analyzed and evaluated them.

2. Sample Module

2-1 JD RO-1 E TRAIN Module No. 91 Serial No. HM10155 712103 1st element



2-2 Element profile

Start of service:	March 25, 1989
End of service :	August 22, 1993
	(after care: preserved in a sealed
	container filled with formalin)
Duration of servic	e: 38,666 hrs. (approx 4 years and 5 months)

3. Sampling Locations

Sample 1: A hollow fiber membrane in the inner layer of the element (near the feed tube)

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Sample 2: A hollow fiber membrane in the middle layer of the element (about 28 mm away from the surface layer)

Sample 3: A hollow fiber membrane in the outer layer of the element (layer immediately below the surface layer)

Sample 4: Foul deposits on the feed tube

- 4. Disassembling the Module
- 4-1. Tools and Materials
 - ① Olfa cutter
 - ② Sealed bag for preserving samples
 - 3 Formalin solution (5%)

4-2. Disassembling Procedure

<For the analysis by SEM and FT-IR methods>

- ① Extract the module from the vessel.
- ② Place the module on a sheet (see Photo.).
- 3 Make a notch on the circumference about 5 cm away from the opening (see Photo.)
- (4) Make another notch on the circumference about 3 cm away from the end plate (see Photo,).
- (5) Make a notch gradually along the shaft, and remove carefully the layers of the protection net, the protection cloth and the hollow fiber membrane (see Photo.).
- 6 Repeat the step 3 5 and take out the hollow fiber membranes of the specified locations (see Photo.).

<Sampling locations>

- (i) A hollow fiber membrane in the layer immediately below the surface layer
- (ii) A hollow fiber membrane in the middle layer (about a half of the module radius away from the surface layer).
- (iii) A hollow fiber membrane near the feed tube
- ⑦ Put the removed hollow fiber membranes in the sealed bag while they remain wet.
- (8) Add the 5% formalin solution in the bag and keep the bag sealed.

<For the evaluation of the mini-module>

- ① Extract the module from the vessel.
- 2) Put the module in a way that the opening comes at the bottom (see Photo.).

- ③ Make a notch on the circumference about 5 cm away from the opening (see Photo.).
- ④ Make another notch on the circumference about 3 cm away from the end plate, and remove the protection net and the protection cloth (see Photo.).
- (5) Make a notch gradually along the shaft, and unbind the hollow fiber membrane gradually (see Photo.).
- © Cut off the upper end of the hollow fiber membrane and take out the hollow fiber membrane of the specified location.

<Sampling locations>

- (i) A hollow fiber membrane in the layer immediately below the surface layer
- (ii) A ballow fibor mombrane in the
- (ii) A hollow fiber membrane in the middle layer (about a half of the module radius away from the surface layer)
- (iii) A hollow fiber membrane near the feed tube
- ⑦ Put the removed hollow fiber membranes in the sealed bag while they remain wet.
- (3) Add the 5% formalin solution in the bag and keep the bag sealed. (The structure of the hollow fiber membrane changes when it is dried, resulting in inaccurate measurement. So, be careful to keep it wet. Performance measurement must be taken immediately after sampling.)
- * For the evaluation of the mini~module, the equipment shown in Fig. can be used.

5. SEM-EDX Analysis

5-1 Tools and equipment for analysis

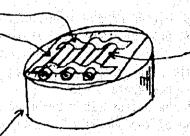
① Ion sputter
 ② Carbon vapor depositor
 ③ Scan electron microscope
 ④ Computer for analyzing
 ⑤ Stub (stage to place samples)
 ⑥ Desiccator
 ⑦ Case for preserving samples

5-1 Preparation of the sample for SEM-EDX analysis

- ① Dry a sample hollow fiber for 24 hours in a draft.
- 2 Stick pieces of carbon double coated adhesive tape on the stub.
- ③ Cut the hollow fiber into pieces (the length of each piece being
- 80% of the stub diameter each), and stick 5 10 pieces in a row on
- the stub. (If the ends of the fibers tend to charge up, cover them by pieces of carbon double coated adhesive tape.)

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Carbon double coated adhesive tape $(50 \ \Omega/inch^2)$



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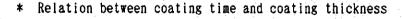
Aluminum block

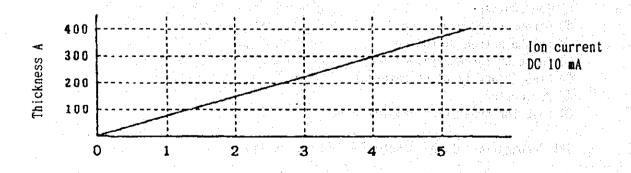
(Carry out sputter coating with conductive metal.

Target thickness	Application	Coating
C	Excellent conductivity Suitable for complicated shapes	10 nm (100 A)
Au-Pd	Coating particle diameters are small Suitable for highly magnified SEM observation	10 nm (100 A)

Note 1: The hollow fibers stuck on the stub have uneven shapes. In addition, observation of more than 10000 magnification is not needed. We, therefore, recommend to apply C-coating.

Note 2: If charge up is great, or if SEM observation is to be carried out for the second time, some 10 A re-coating is recommended.





Coating time (min.)

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← Environment protection
Prevention of charge up →

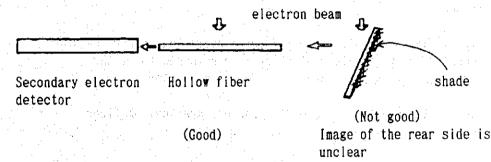
⑤ Put the samples into a plastic case, and keep it in a desiccator.

- 6 Conduct the SEM and EDX analysis.
 - The conditions for the analysis of hollow fiber membranes are shown in the following table.

	Sample current	Accelerating voltage	Remarks
SEM	10 -10 A	5 - 10 kV	To obtain surface data of fibers Damage to fibers is small
EDX	10 -12A	20 kV	X-ray detection amount (information) is large

* Location of the sample (hollow fiber membrane)

Set the axis of the sample to the detection end of the secondary electron detector as shown below.



5-2 Results of analysis

① Fig. 1 shows the surface of unused hollow fiber membrane. The membrane surface is smooth.

② Fig. 2-4 shows the surface of an inner layer hollow fiber membrane. As seen in Fig., foul deposits have accumulated over the entire membrane surface. (The lower the applied voltage, the greater the amount of secondary electron signals (despite the low resolution) and hence the clearer the information about the membrane surface. This can be confirmed by comparing Fig. 2 and Fig. 3 that are taken from the same area).

Fig. 4 is a magnified image of the foul deposits.

Fig. 5 and 6 are the charts resulting form EDX analysis of inner layer foul deposits.

Fig. 5 is the result of X-ray irradiation on foul deposits spread over a wide area, showing mainly Fe-based oxides but also NaCl as well as S and Si (coming from sand in sea water) that are abundant in sea. ③ Fig. 7-8 show the surface of a middle layer hollow fiber membrane.

As seen in these figures, foul deposits are localized and far fewer than on the inner layer membrane. A dent observed on the exposed deposit-free part of the membrane surface may have been caused during the sampling procedure.

(As is the case for the inner layer, the lower the applied voltage, the greater the amount of secondary electron signals (despite the low resolution) and hence the clearer the information about the membrane surface. This can be confirmed by comparing Fig. 7 and Fig. 8 that are taken from the same area).

Figs. 10 and 11 are the charts resulting form EDX analysis of middle layer foul deposits.

Fig. 10 is the result of X-ray irradiation on foul deposits spread over a wide area, showing mainly Fe-based oxides but also NaCl as well as S and Si (coming from sand in sea water) that are abundant in sea.

Fig. 11 is the result of spot X-ray irradiation on particular deposits, showing mainly Si which is considered to be the deposition of crushed sand particles in sea water.

④ Fig. 12-13 show the surface of an outer layer hollow fiber membrane.

As seen in the figures, foul deposits are even fewer than on the middle layer membrane. They are scattered in various parts. As in the middle layer, a dent observed on the exposed deposit-free part of the membrane surface may have been caused during the sampling procedure.

Fig. 14 and 15 are the charts resulting form EDX analysis of outer layer foul deposits.

Fig. 14 is the result of X-ray irradiation on foul deposits spread over a wide area, showing mainly Fe-based oxides but also NaCl as well as Si, Al (coming from sand in sea water) and S that is abundant in sea.

Fig. 15 is the result of spot X-ray irradiation on particular deposits, showing mainly Si which is considered to be the deposition of crushed sand particles in sea water.

(5) Fig. 16 shows foul deposits on the feed tube. As in the case of the deposits on the inner layer membrane, barlike aggregates of flaky substances (10 μm diameter) are observed. Fig. 17 and 18 are the charts resulting form EDX analysis of feed tube foul deposits.

Fig. 17 is the result of X-ray irradiation on foul deposits spread over a wide area, showing mainly Fe-based oxides but also NaCl as well as Si, AI (coming from sand in sea water) and S that is abundant in sea.

6 Table 2 summarizes the these results.

6. Results of FT-IR Analysis

6-1 Tools and equipment

① FT-IR 1725X (made by Perkin Elmar)

② Constant-temperature drier

(3) Mortar

④ Pestle

⑤ Tablet generator B KBr crystalØ Ge prism

③ Attachment for transparent method

Attachment for ATR method

10 Pure water

1 Desiccator

(Diffuse reflection method and microscopic method)

① Attachment for diffuse reflection method)

2 Optical microscope

③ Infrared microscope

④ Glass plate

6-2 Sampling of foul deposits and Measurement

① Method (m-1)

Sampling : Dry the hollow fiber membrane with foul deposits in a constant-temperature drier (6 hours at 110°C). After cooling it in a desiccator for 45 minutes, absorb the foul deposits with KBr powder. Then remove the membrane. Measuring: Diffuse reflection method or tablet method (transparent method)

Ø Method (m-2)

Sampling : Wash the foul hollow fiber membrane with pure water, dry the removed substances, and mix them in KBr powder. Measuring: Diffuse reflection method, tablet method (transparent method), or mircroscopic method

③ Method (m-3)

Sampling : Take a small amount of foul deposits using an optical microscope (by slicing), analyze them by irradiating with an infrared microscope.
Measuring: Microscopic method

(4) Method (m-4)

Sampling : Wind the hollow fiber membrane around a Ge prism, making the interval as small as possible. Measuring: ATR method

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(5) See Table for further detail.

- Note 1: When mixing foul deposits with KBr powder, be careful about humidity and take a sample quickly lest KBr powder should absorb moisture and OH absorption spectrum appears on the chart.
- * At present, SWCC R&D is equipped with facilities for the tablet method and ATR method. For the analysis of hollow fiber membranes, we recommend the diffuse reflection method (the attachment should be purchased).

6-3 Results of analysis

① Fig. 19 shows the results of the analysis by the diffuse reflection method (m-1).

From the inner to middle layer and to outer layer, absorption peaks become increasingly broad. This indirectly shows that the amount of foul deposits is at maximum in the inner layer but become fewer and fewer in the middle and outer layers. The result agrees with the fact that foul deposits are most likely to be trapped within the inner layer as feed water flows outwards from the feed tube to the inner, middle and outer layers (shown in the figure on P.1).

② Fig. 20 shows the results of the analysis of the diffuse reflection method and the microscopic method. As shown in Fig. 20-1, the microscopic method (m-3) was applied to the middle layer where foul deposits is not relatively few, result being compared with that in the inner layer. Compared to the middle layer, the inner layer shows the fall of deposit amount at the point of around 1100 cm⁻¹, which indicates a reduced presence of SiO₂ of silicates. This agrees with the result of the SEM-based observation.

③ Fig. 21 shows the results of the analysis of the composition analysis of foul deposits on the feed tube. It is estimated that the dark brown substances shown in Fig. 21-1 contains mainly metallic oxides(from peaks at 700 cm⁻¹ or below), carboxylates (from peaks between 1350 and 1450 cm⁻¹ and at 1650 cm⁻¹), and silicates (from peaks between 1000 and 1100 cm⁻¹ and at 3700 cm⁻¹9).

It is also estimated that the sticky gray substances shown in Fig. 21-2 contains SiOn (with peaks at 800 and 1100 cm⁻¹), or silicates (with peaks between 1000 and 1100 cm⁻¹ and at 3700 cm⁻¹) and alkyl groups (oil components, with peaks at 1380, 2850 and 2950 cm⁻¹). The black rubbery matter shown in Fig. 21-3 is considered to contain elastic substances resembling ethylene or propylene rubber (with peaks at 1350, 1420, 2850 and 2950 cm⁻¹). The black mass shown in Fig. 21-4 is considered to contain metallic oxides and silicates with alkyl groups (oil components, with peaks at 1380, 1450, 2850 and 2950 cm⁻¹).

④ Fig. 22 shows the results of the microscopic method using the sampling method m-2. As in Fig. 19, from the inner to middle layer and to outer layer, absorption peaks become increasingly broad. This indirectly shows that the amount of foul deposits is at maximum in the inner layer but become fewer and fewer in the middle and outer layers. Because of the use of the microscopic method, the charts have sharper peaks than those obtained by the diffuse refection method.

7. Conclusion

① The amount of foul deposits is the greatest in the inner layer of the hollow fiber membrane which is the first place to come in contact with feed water. Foul deposits becomes fewer in the middle and outer layers. It seems that hollow fiber membranes in the inner layer function as a filter to protect membranes in the subsequent layers.

② Foul deposits on fiber membranes contain mainly metallic oxides (corroded iron and sandy substances in sea water), silicates, and foreign substances in sea water (rubbery and oily matters).

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	Overall assessment	- - - -	·Fe-based •Sea sand	- Fe-based - Sea sand	•Fe-base Sea sat	Fe-based o Sea sand Rubbery an substances deposited sea water	2950 cm ⁻ 20. 2850 1 C00
· · · · · · · · · · · · · · · · · · ·	IR analysis		•Metallic oxides •Silicates •Carbohydrates	<pre>•Metallic oxides: metallic hydroxides •Silicates •SiO_a •Carboxylätes •Carboxylätes *Microscopic method used to small quantities</pre>	-Metallic oxides: metallic hydroxides -Silicates -SiO ₂ -Carboxylates -Microscopic method used to small quantities	<pre>DThick sticky substance (gray): silicates (SiO₂ family) Alkyl group(oily matter) Aropylene rubber Black mass: Metallic oxides: aromatic poly- ester (PET) Alkyl groups (oily matter): silicates</pre>	W.B. Silicates: 1000 -1100. 3700 cm ⁻¹ SiO _a : 800. 1100 cm ⁻¹ Alkyl groups: 1380. 1450. 2850 and 2950 cm ⁻¹ Ethylene/propylene rubber: 1350. 1420. 2850. 2 Carboxylates: 1350 - 1450. 1650 cm ⁻¹ C00-
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Results		Na	0	c1e Cle	C le	0	
2 Hollow Fiber Membrane Analysis	SEM analysis		•Entire membrane surface covered by foulants •Aggregate of fibrous and flaky substances	•Sparsely deposited •Particulate matter present •Fibrous matter present •Damage to the membrane surface -	-Sparsely deposited -Particulate matter present -Fibrous matter present -Damage to the membrane surface -	-Aggregate of fibrous and flaky substances	
Table		Sampling location	Inner layer	Middle layer	Outer layer	Deposits	
Ţ		Sampling loc.	Inner lay	Middle la	Outer lay	Deposits	

Pre-treatment Method	Single Crystal	Sample Preparation Method	Neasuring Method	Remarks
Dry hollow fiber membranes with foulant deposits in thermostatic dehydrator. (110°C x 6 Hrs.) -Let it cool in a desiccator (45min.)	Ż	Ocut holior fiber membranes with foutant deposits to 3cm lengths. CPPuiverize KBr crystal in mortar. (KBr is highly absorbed: If some KBr is leftover store in desiccator. Do not store it for a long lime.) Dut holior fiber membranes with foutant	crablet Method> OPrepare sample tablets with tablet forming machine. Pput sample into transission method attuchment. OUSe 1k spectrum measuring method.	*Make the grain sizes in each sample uniform, as the IR spectrum is heavily effected by different grain sizes of KBr powder.
		deposits into KBr porder and scrape off foulant deposits with pestle. (ARemove hollow fiber membranes.	Chiffuse Reflectance Spectroscopy (D'Fill mixed povder of KBr and foulant deposits into sample cup. Cup. Cuttach sample to diffuse reflectance spectroscopy attachent. Bethod.	
	3	Ocut hollor fiber membranes with foulant deposits to 5cm lengths. STind hollow fiber acebranes around Gerprise covering whole prise. (Try to minimize space between follow fiber and prise.)		
		OPut hollor fiber membranes with foulant deposits onto glide slide. Ouse optical microscope on portion you want to analyze for sampling.	<pre>cMicroscopy> OFfocus on stample on glass stide vith infrared microscope. OUse 1R spectrum measuring method.</pre>	
Tash hollow fiber membranes containing foulant deposits with pure vater and dry the foulants dislodged from vashing.	XBr	Othoroughly dry the foutants which were dislodged from washing hollow fiber membranes. Whutverize KBr crystal in mortar. (BF is highly absorbent. If some KBr is leftover, store in desiccator. bo. not store it for a long time.)	crablet Method> CPrepare sample tablets with tablet forming machine. 2Put sample into transmission method attachment. 3Dus 1R spectrum mcasuring method.	
		(ONIX KBr crystal and above dried foutants thoroughly.	 cDiffuse Reflectance Spectroscopy cDifil = xcd powder of XBr and foulant deposits into sample cup. CDAtiant deposits into sample reflectance spectroscopy reflectance spectroscopy allachaott. CDUse IR spectrum measuring acthod. 	
	۱. 	OPut hollor fiber membranes with foulant deposits onto glide slide. (2)Use optical microscope on portion you want to analyze for sampling.	<pre><hi>CHicroscony> OFocus on stapple on glass slide with infrared microscope. OUse IR spectrum measuring method.</hi></pre>	

Appendices

- Module dismantling photograph
- FT-IR sample preparation and analysis method
- Table summery fo hollow fiber membrane analysis results (Table 2)
- SEM photograph of an unused hollow fiber
- SEM photograph of foulant deposits on an inner layer hollow fiber membrane

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- EDX analysis results for foulant deposits on an inner layer p. hollow fiber membrane
- SEM photograph of foulant deposits on a middle layer hollow p. fiber membrane
- EDX analysis results for foulant deposits on a middle layer hollow fiber membrane
- SEM photograph of foulant deposits on an outer layer hollow fiber membrane
- EDX analysis results for foulant deposits on an outer layer hollow fiber membrane
- SEM photograph of the feed tube
- EDX analysis results for the feed tube
- IR analysis of foulant deposits on inner, middle and outer layer hollow fiber membranes based on diffuse reflectance spectroscopy
- IR analysis of foulant deposits on inner and middle layer hollow fiber membranes based on diffuse reflectance spectroscopy and microscopy
- · IR analysis of foulant deposits on the feed tube
- IR microscopic analysis of foulants dislodged from hollow fiber membranes during washing

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Photo. 1



Photo. 2

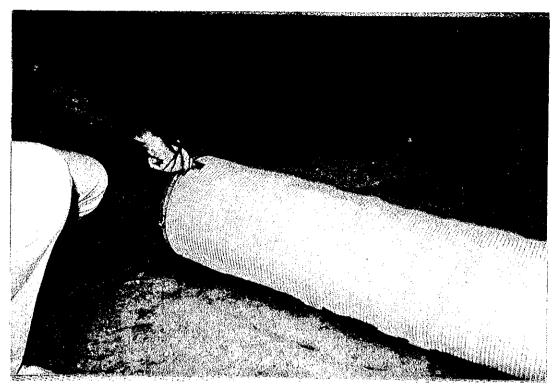


Photo. 3

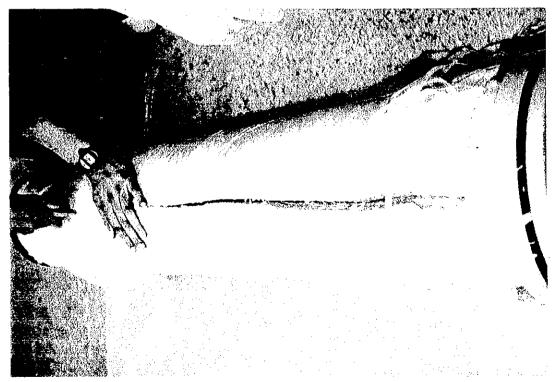


Photo. 4

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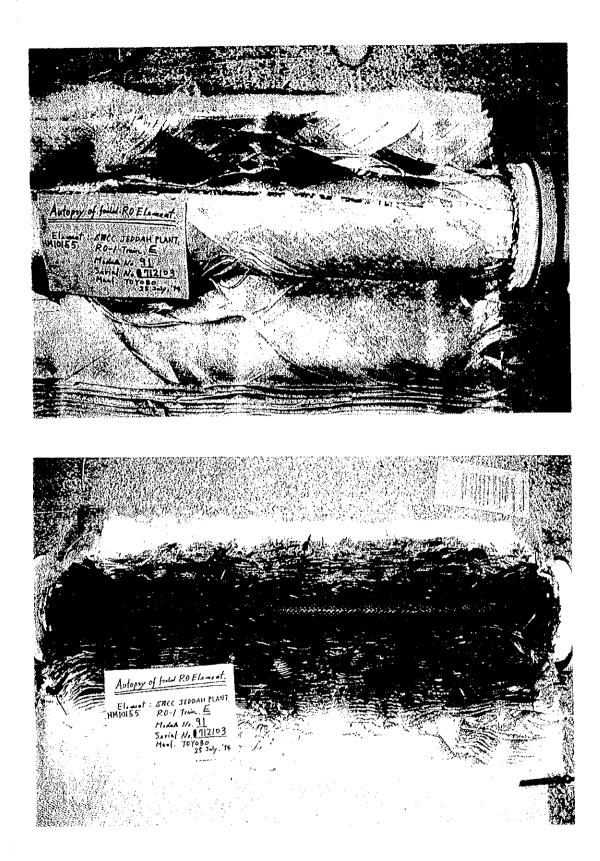


Photo. 5

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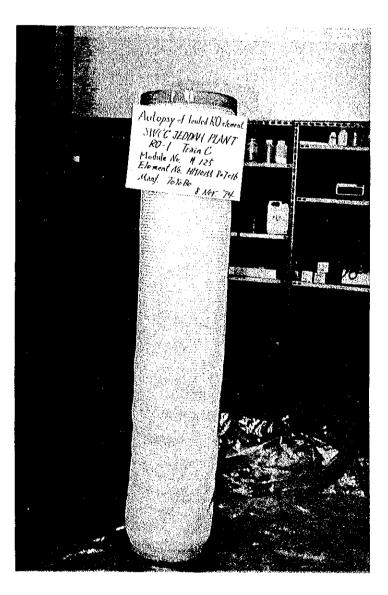


Photo. 6

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Photo.7

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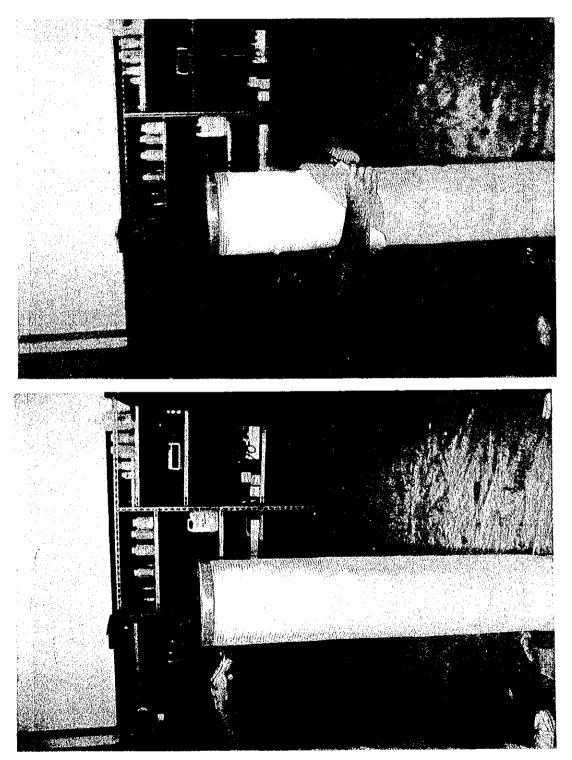


Photo.8

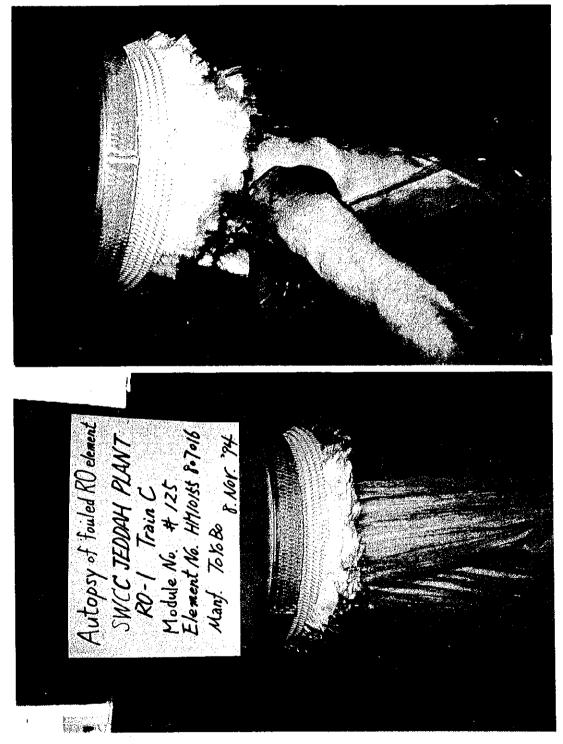
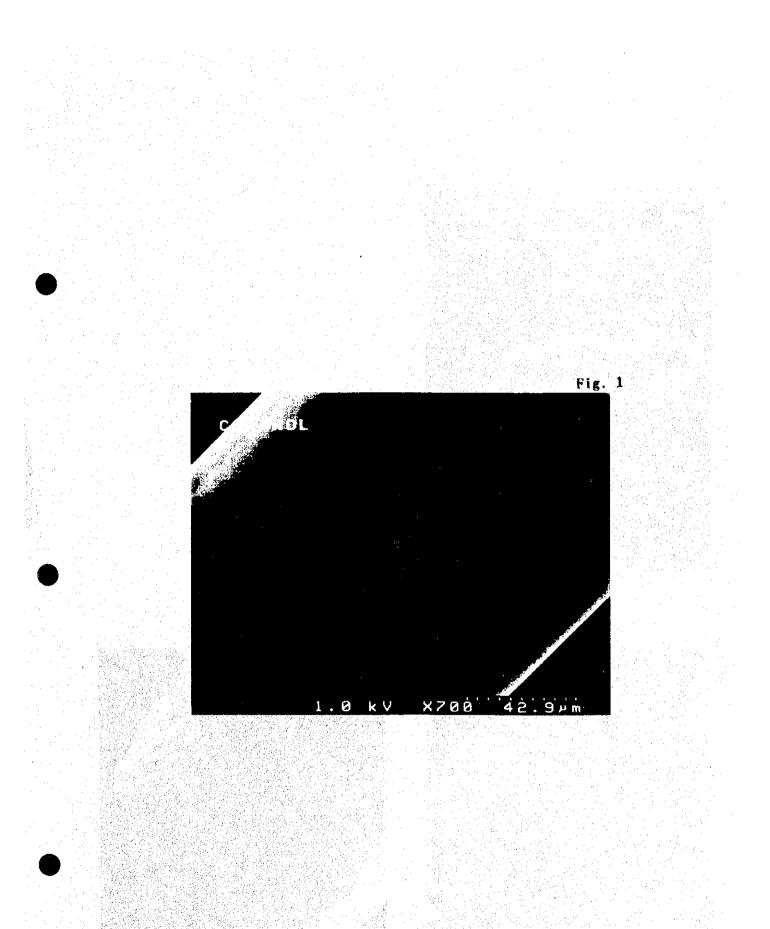
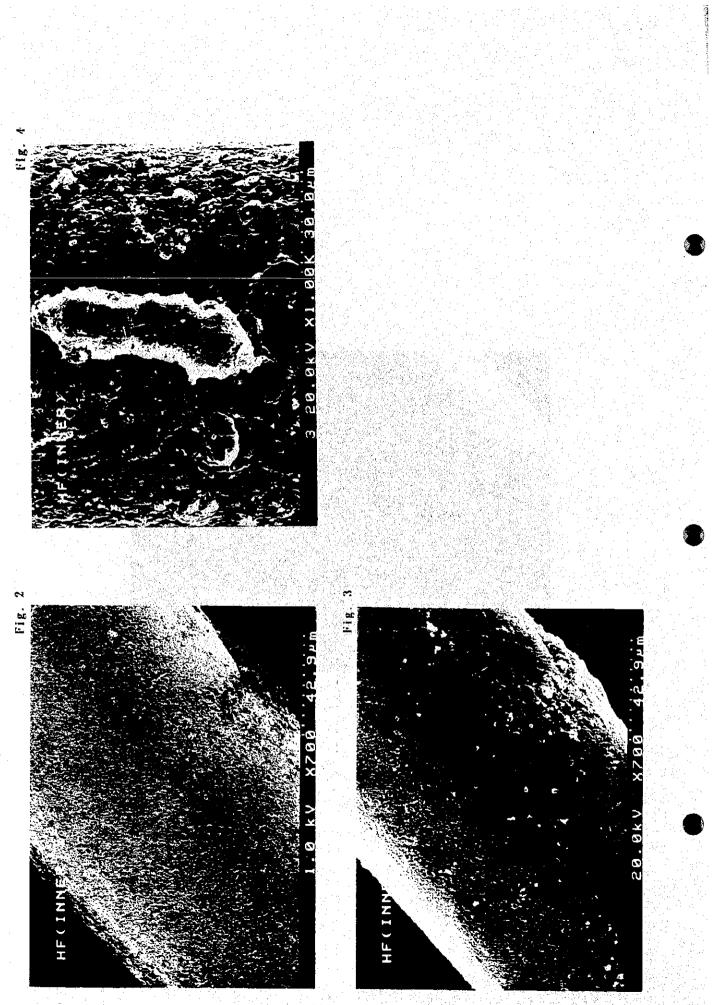
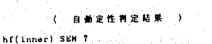


Photo.9





内層中空糸のEDX分析結果



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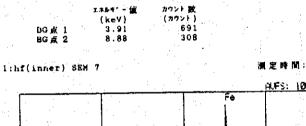
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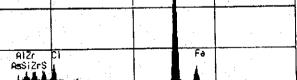
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【BGサーチ結果】

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Fig. 6 [15×15µm範囲分析(付着物のみ)







中層中空糸のEDX分析結果

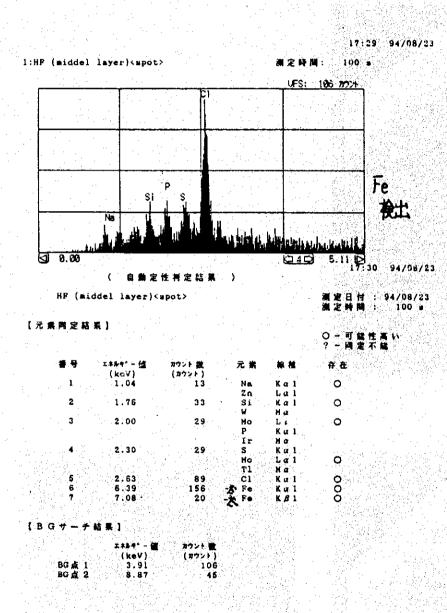
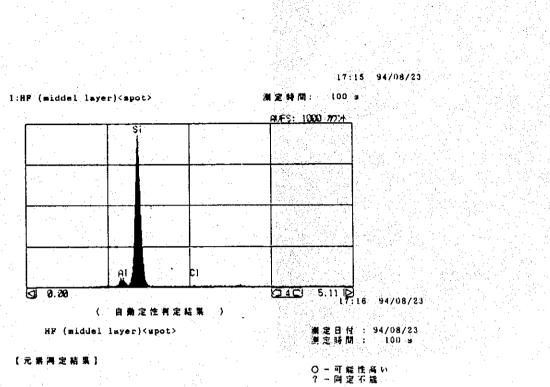


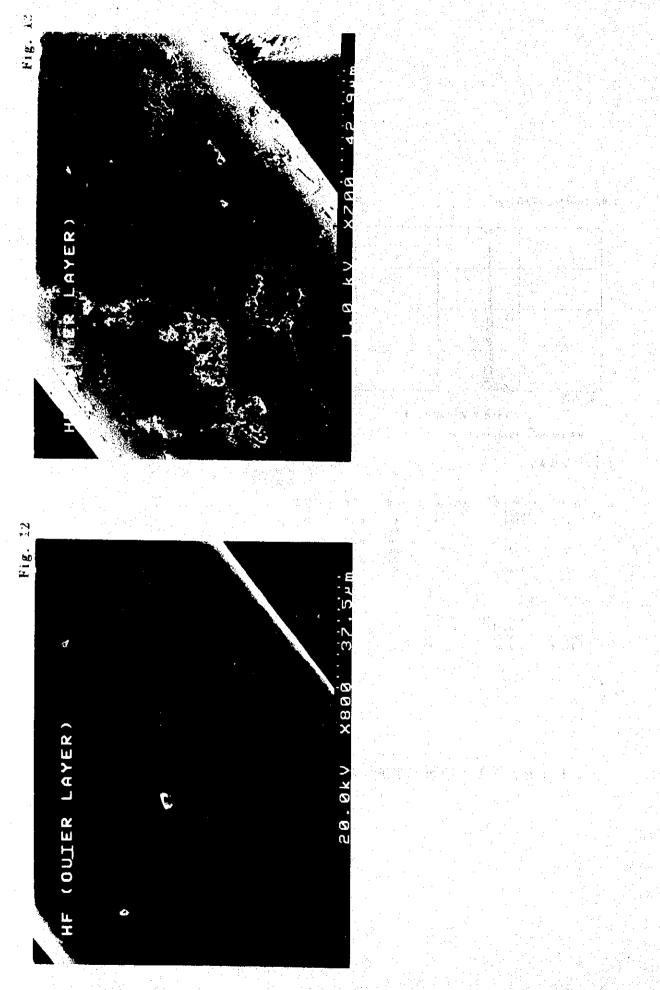
Fig.10 中層中空糸(付着物全体)



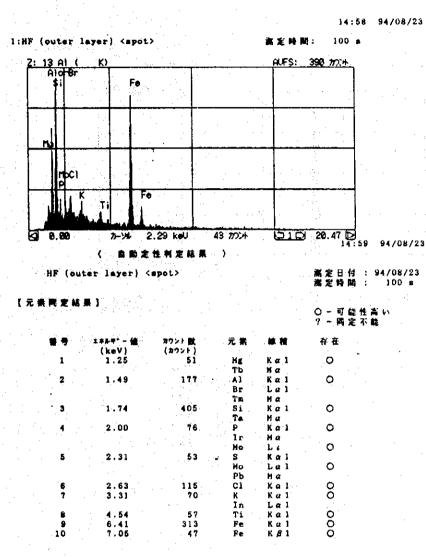
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Fig.11 中層中空糸(粒子状付着物)



外層中空糸のEDX分析結果



【BGサーチ結果】

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BG 点 2	15.00	55

Fig.14 外層中空糸(付着物全体)

美国著位人口可以来忽然就被

15:19 94/08/23

1:HF (outer layer) (spot) 测定终期: 100 .

A Contract of the second s		 AUFS: 2780 7774	
S			
AI	21 21	an chinan ta buta di Tana sa atao	

0.90

 $\langle \cdot \rangle$ 自動定性判定結果 •)

HF (outer layer) <spot>

D 5:20 94/08/23

〇一可能性高い ?一與定不错

祥 在

0 0

0

5.11

(DAD)

潮定日付: 94/08/23 潮定時間: 100 s

2

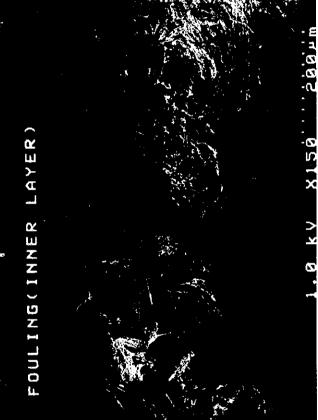
1 1.50 65 41 K Br L Tm H Yb H 2 1.75 2451 Si K	書号 □	エネ₽ キ* - 绪 (keV)	おウント 戦 (おウント)	元 業	新社
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	2	1.75	2451		Ha Kal
	: 3 -		1	Ť	Ma Kal

【BGサーチ結果】

	工**** - 値	カウント 数
	(keV)	(2021)
96 貞 1	3.91	278
BG点 2	8.50	131

Fig. 外層中空糸(粒子状付着物) 15





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