The Value of SEM Particle Counting for Monitoring D.I. Water

by

Theresa Chu, Balazs Laboratory and
Barbara Stanczyk, IBM Corporation

Presented at the Semiconductor Pure Water Conference (SPWC)
January 1987

Reprinted with permission from SPWC

THE VALUE OF SEM PARTICLE COUNTING FOR MONITORING D.I. WATER

BARBARA STANCZYK
IBM CORPORATION
and
THERESA CHU
BALAZS ANALYTICAL LABORATORY

The quality of D.I. water in terms of particle contamination is extremely crucial for most of the products manufactured by the electronic industry. The SEM (Scanning Electron Microscope) analysis of particles and bacteria in D.I. water has been used by the semiconductor industry for only a few years. The results of this monitoring are utilized for submicron filtration control. SEM analyses revealed several problems in filtration and distribution systems, which were not detectable until the SEM method was employed.

The SEM analysis helps to select efficient and economical filtration systems. The method is extremely important in system troubleshooting due to the fact that the SEM photograph provides visual information about the contaminants. This makes the SEM analysis an important tool in maintaining high quality D.I. water.

Introduction

There are several available methods of particle counting in D.I. water such as light scattering, light absorption, ultrasonic technique and image replication. There are two basic ways to obtain water samples for particle analyses: batch sampling and in-line sampling. A detailed description of these methods and techniques can be found in the paper "Counting and Identifying Particles in High Purity Water".[1]

A new method, the direct count of particles using SEM, was developed jointly by Roy Hango from IBM Burlington and Industrial & Environmental Analysts, Inc.[2] This technique involves passing water through an in-line 0.2 micron filter, coating the filter with a conductive material and examining it with a scanning electron microscope. The advantages of this method are that it can detect all particulate matter as small as 0.2 micron (Picture 1), all species of bacteria, live and dead (Picture 2). The SEM with EDS (energy dispersive spectroscopy) analysis provides physical data as well as information about chemical composition of the contaminants. Consequently, this method allows for determination of the source of contaminants. The disadvantage of the SEM method is that the results are based on the statistical calculations. A minimum of 200 liters of water sample is required in order to obtain good data. This results in a long sampling time.

The SEM testing of particles in D.I. water was started at IBM San Jose in 1984. Since then several problems related to the particulate contamination have been solved successfully. These problems are presented in the following cases.

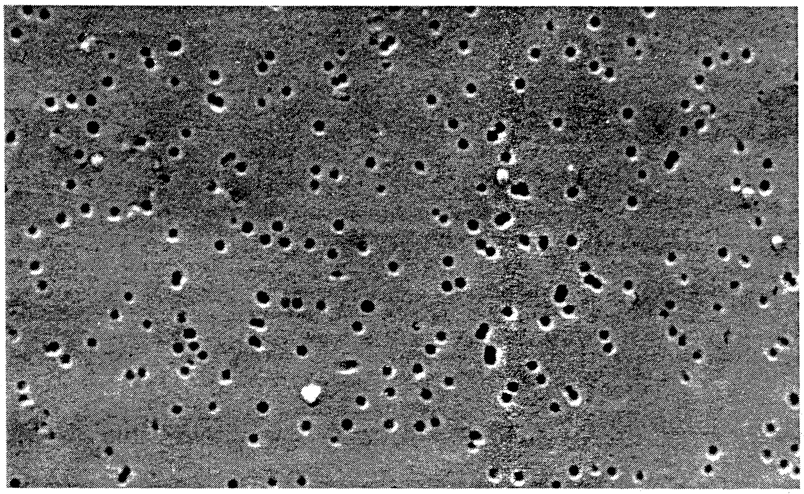
Case 1

This case describes the applicability of the SEM particle testing in a troubleshooting of the filtration process in one of the IBM San Jose D.I. water polishing stations, Station A. The D.I. water polishing station consisted of the following equipment: storage tank, pumps, two banks of polishing bottles, ultraviolet sterilizers and two banks of 0.2 micron cartridge filters (the bank of the prefilters and the bank of the final filters). See Drawing 1.

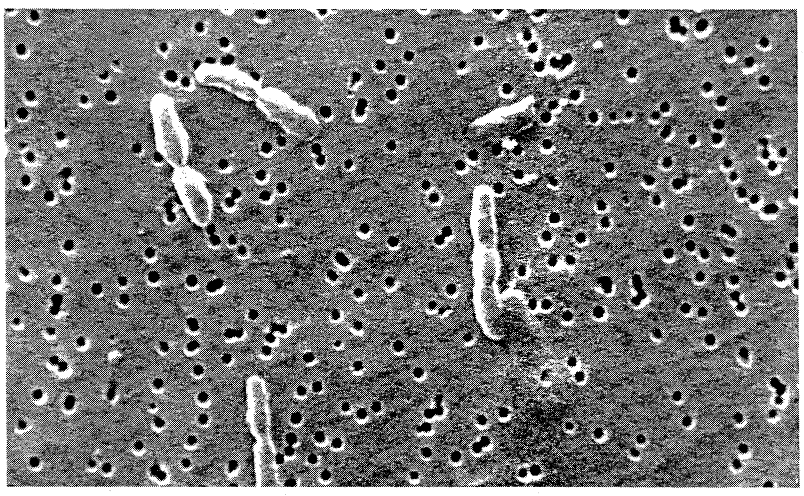
Before SEM testing was started, particles in D.I. water were analyzed by the direct visual microscope method and bacteria were checked by the standard bacteria culture count method. The direct visual microscope method only allows for the detection of particles larger than 1 micron and standard bacteria culture count method reflects the presence of live organisms only.

It was obvious that these methods were insufficient for checking the effectiveness of 0.2 micron filtration.

The filter cartridges were replaced every 6 months on a preventative maintenance basis in spite of the fact that the pressure drop across the filter did not reach 15 psi. The frequency of the cartridge replacement was based on the assumption that bacteria might develop on the filter being in use for an extended period of time.



PICTURE 1



PICTURE 2

The SEM samples were taken once a month. The time of the sample collection was approximately 48 hours, the volume of the samples between 300 and 800 liters. The areas labeled "Stage I" on graphs 1, 2, 3 and 4 present the data for this period of time. The results are inconsistent and high, in particular for small sizes of particles and bacteria.

An in-line particulate counter was connected in an effort to find correlation between the data. The area labeled "Stage I" on graph 5 presents the data obtained for this period of time. Average counts per day of particles larger than 0.5 micron are shown on the graph (the smallest size of particles the instrument can measure is 0.5 micron).

No correlation between the SEM results and the particle counter was found. The counts of particles given by the particle counter were in general lower. At the same time live bacteria counts were very low. See Table 1, Stage II.

Only the SEM data showed clearly that a problem in the polishing station existed. This fact was confirmed when the results for station B, another polishing station at IBM San Jose, were obtained. Table 2 shows the comparison of the SEM data for the different polishing stations. The better results for station B were surprising since a single bank of the filters was installed in this station.

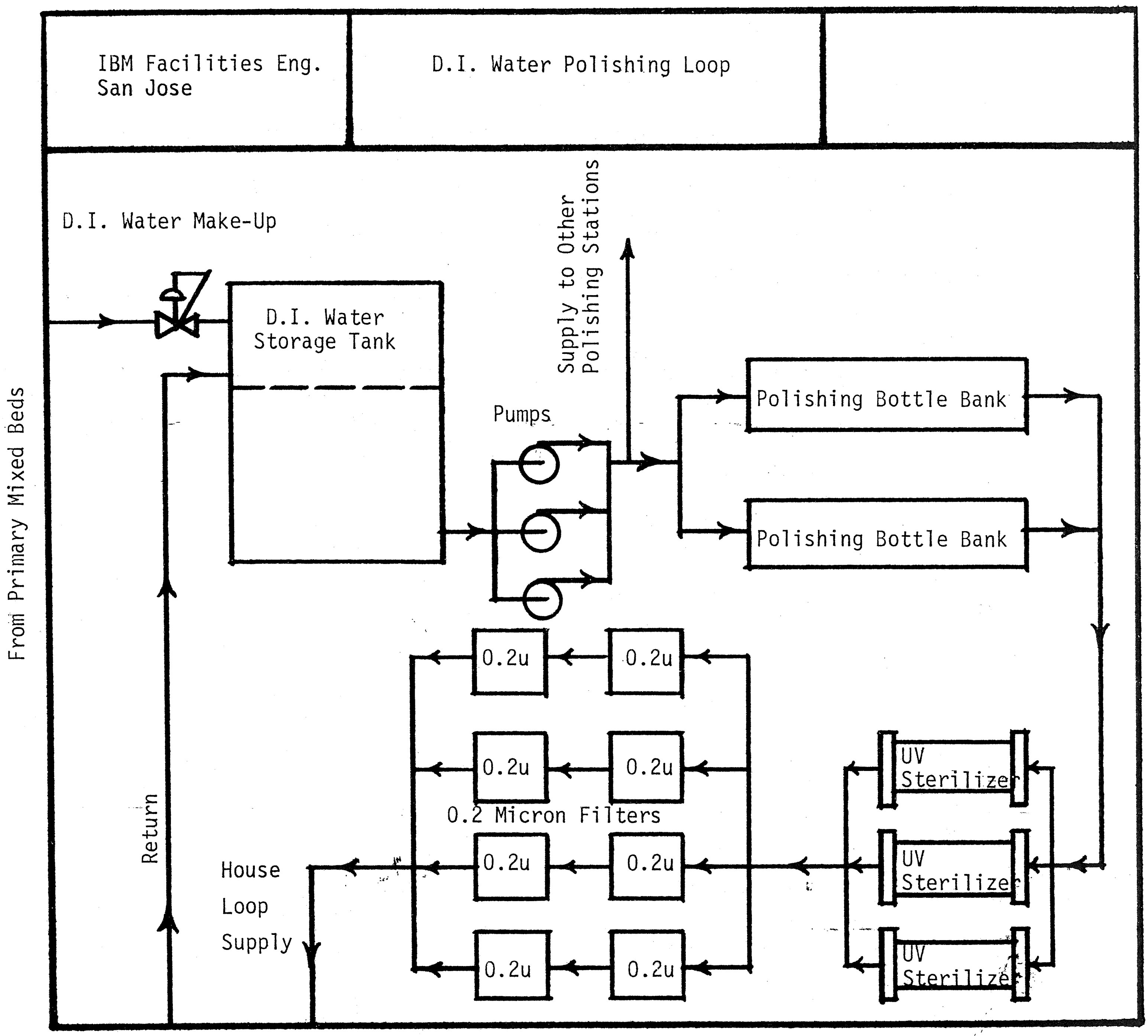
The design and the equipment of the polishing station was reviewed thoroughly. At first, high pressure fluctuations in the loop were noticed in Station A. The pressure fluctuations were caused by two facts:

1. The common pump supplied water to the loop in station A and to the other buildings located on the site.
2. The back pressure in the return line was not controlled.

This problem was solved by separating the loop supply from the site supply and installing a back pressure control valve.

Further testing of D.I. water by SEM showed some improvement (see Graphs 1, 2, 3, and 4, Stage II). The results obtained from the particle counter were inconsistent but in general lower than in Stage II (see Graph 5). However, more improvement was expected.

Additional investigation of the problem was conducted and it was found that the old type of the filter housings in the station A could only accommodate the cartridges with the flat gaskets. The new housings with double "O" ring cartridges were installed and the bank of the prefilters eliminated. The SEM results for this period of time are presented on Graphs 1, 2, 3, and 4 in the areas marked Stage III. The section labeled Stage III on Graph 5 shows the



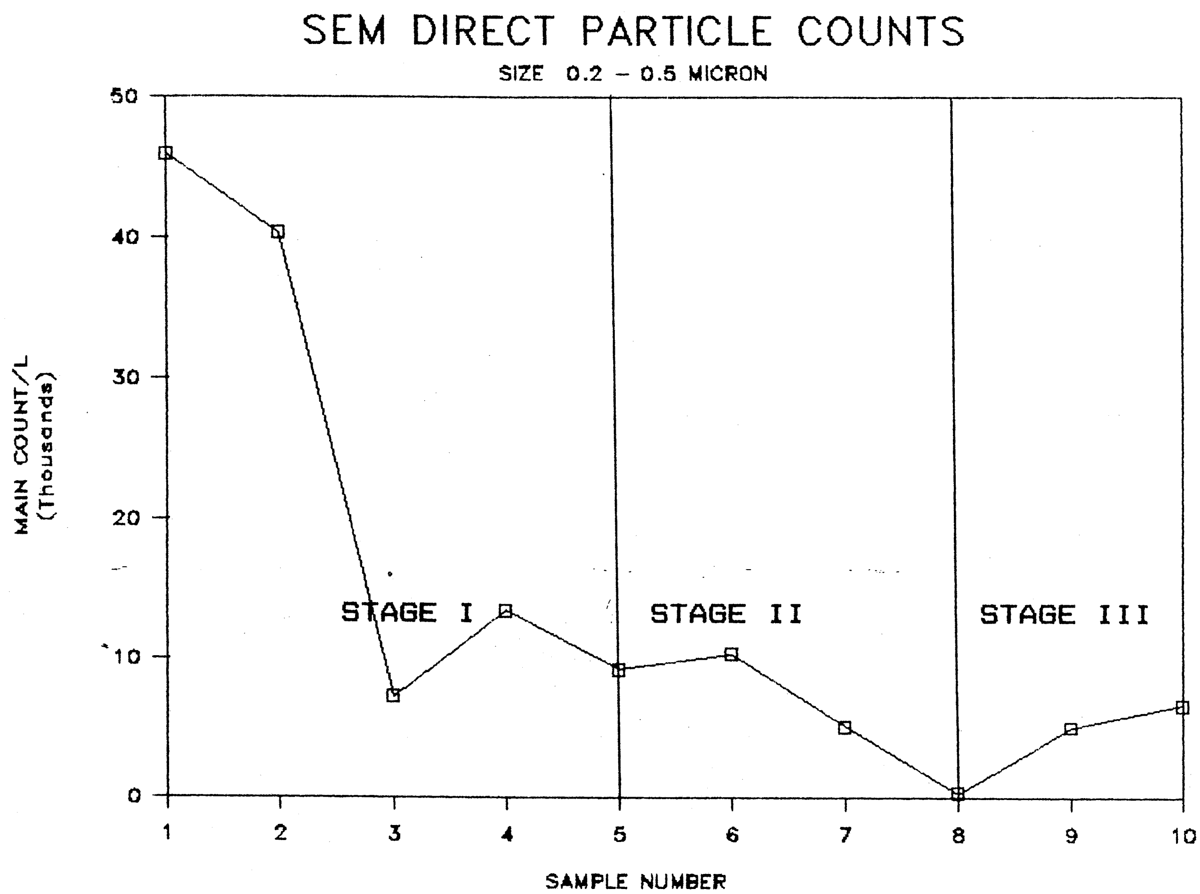
IBM Facilities Eng.
San Jose
D.I. Water Polishing Loop
D.I. Water Make-Up
From Primary Mixed Beds
D.I. Water
Storage Tank
Supply to Other
Polishing Stations
Pumps
Polishing Bottle Bank
Polishing Bottle Bank
0.2u
0.2u
0.2u
0.2u
0.2 Micron Filters
0.2u
0.2u
0.2u
0.2u
UV
Sterilizer
UV
Sterilizer
UV
Sterilizer
Return
House
Loop
Supply

DRAWING 1

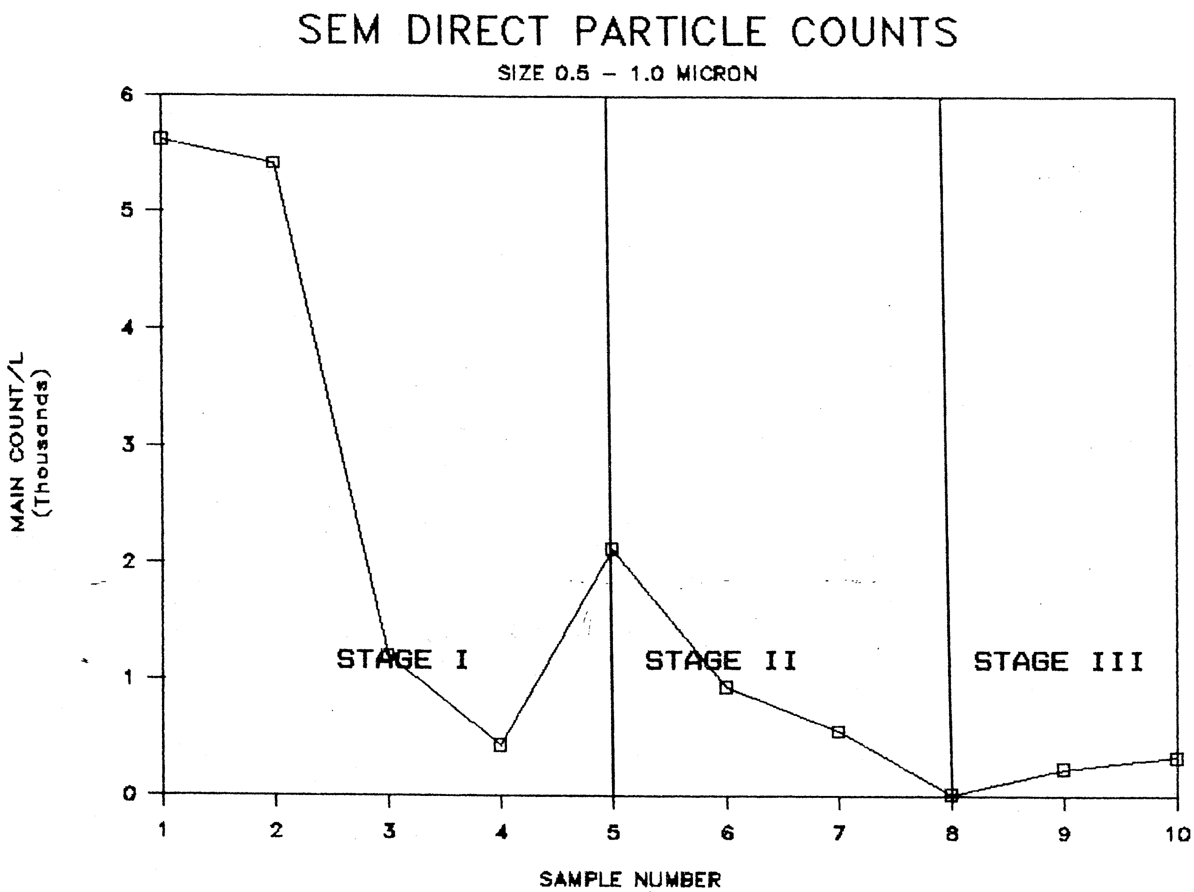
particle counter data. The improvement of the results is obvious.

Presently, the particles and bacteria are monitored on a regular basis, along with the pressure difference across the filter. The filters have been in operation for over 8 months, making high quality D.I. water. The pressure drop across the filter is equal to 8 psi only.

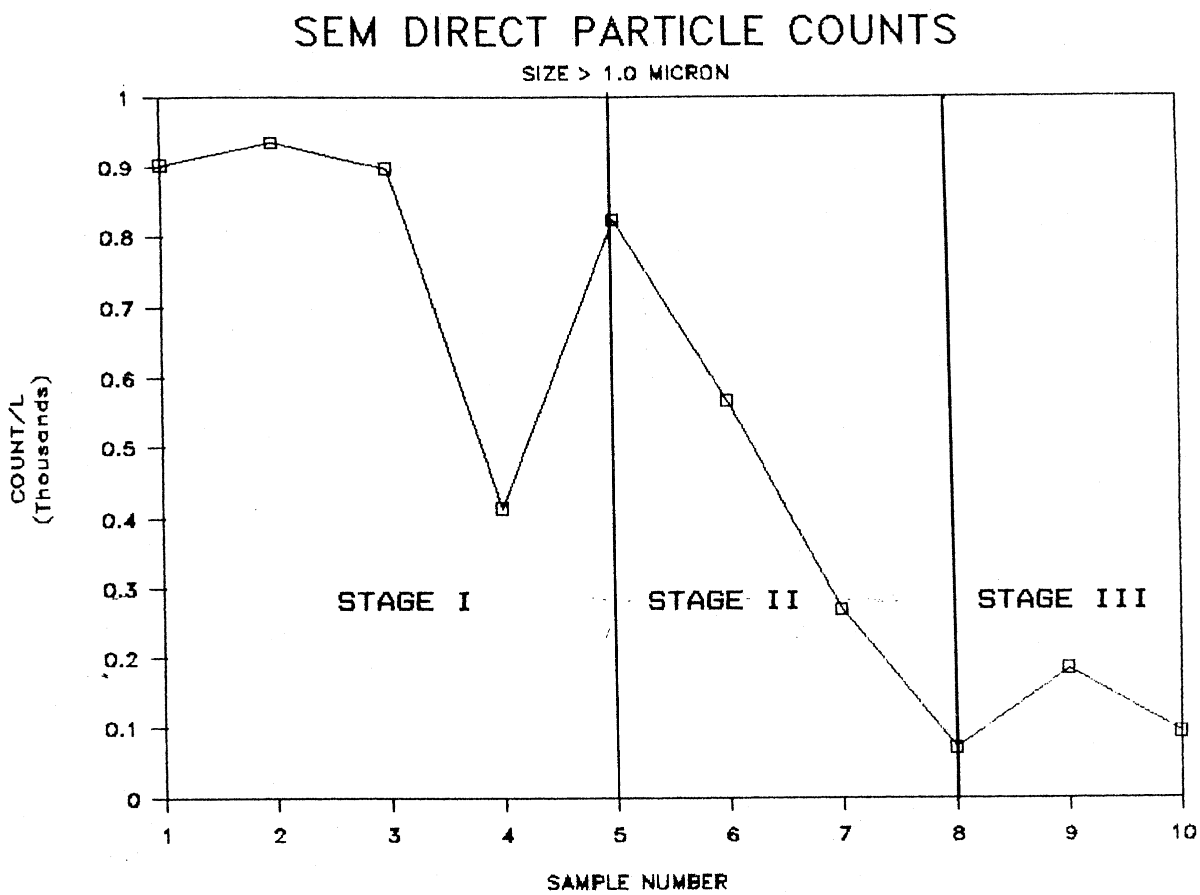
The important fact to note is that besides the quality improvement, a significant reduction of the filtration cost was accomplished.


SEM DIRECT PARTICLE COUNTS
SIZE 0.2 – 0.5 MICRON
MAIN COUNT/L
(Thousands)
50
40
30
20
10
0
STAGE I
STAGE II
STAGE III
1
2
3
4
5
6
7
8
9
10
SAMPLE NUMBER

GRAPH 1

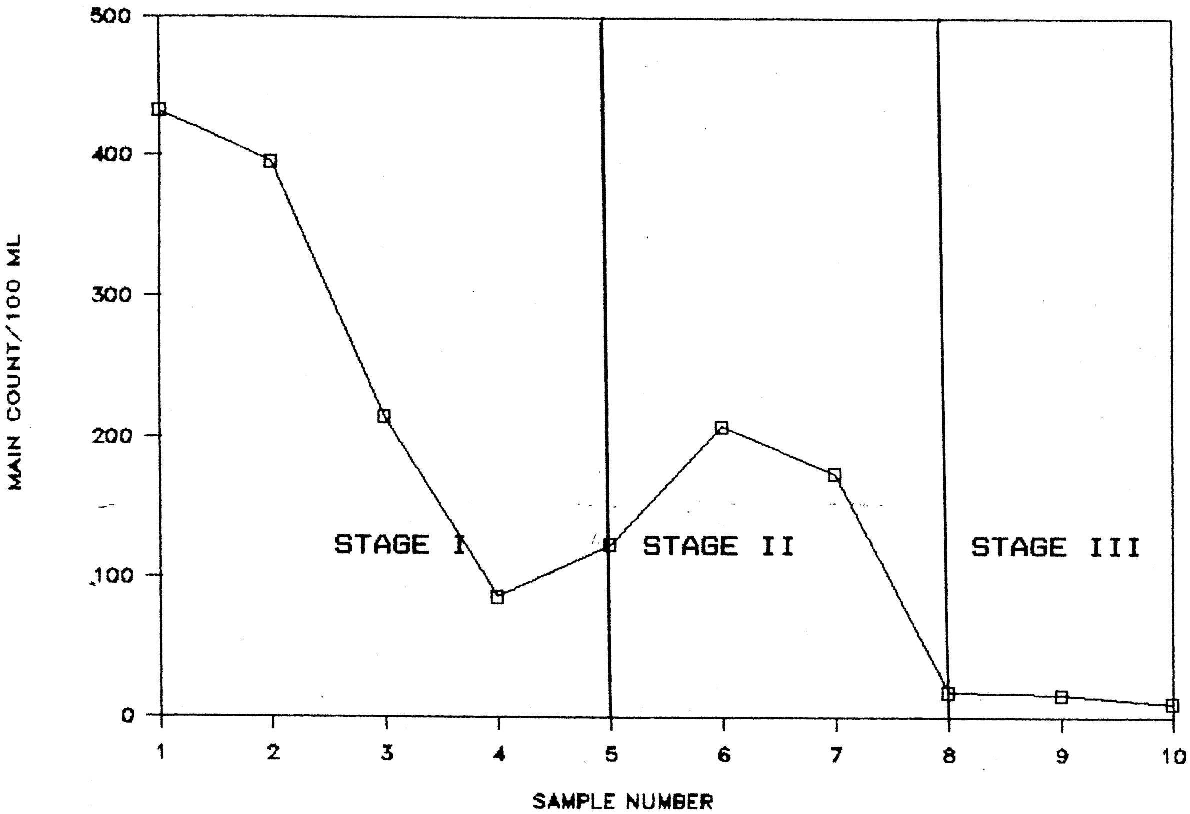

SEM DIRECT PARTICLE COUNTS
SIZE 0.5 – 1.0 MICRON
MAIN COUNT/L
(Thousands)
0
1
2
3
4
5
6
STAGE I
STAGE II
STAGE III
1
2
3
4
5
6
7
8
9
10
SAMPLE NUMBER

GRAPH 2

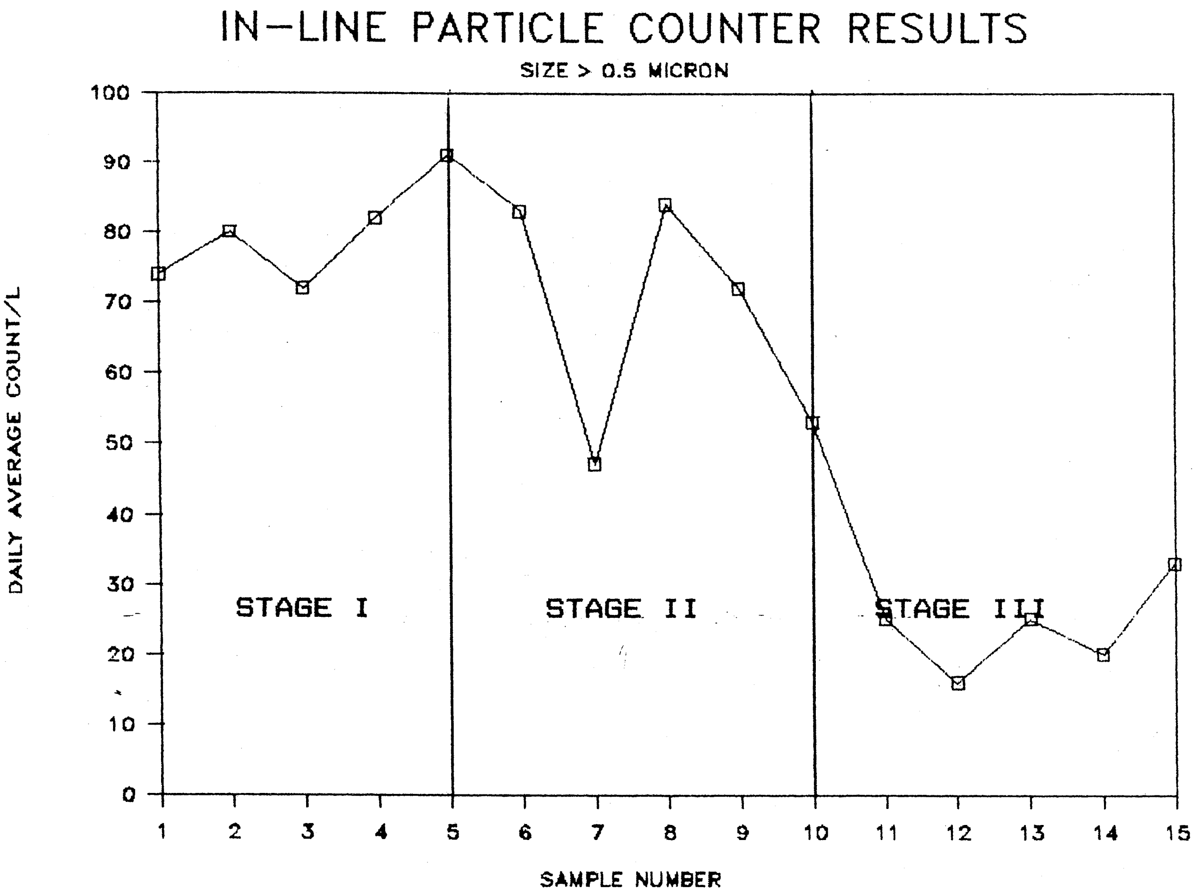

SEM DIRECT PARTICLE COUNTS
SIZE > 1.0 MICRON
COUNT/L
(Thousands)
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
STAGE I
STAGE II
STAGE III
1
2
3
4
5
6
7
8
9
10
SAMPLE NUMBER

GRAPH 3

SEM TOTAL BACTERIA COUNTS



GRAPH 4


IN-LINE PARTICLE COUNTER RESULTS
SIZE > 0.5 MICRON
DAILY AVERAGE COUNT/L
SAMPLE NUMBER
STAGE I
STAGE II
STAGE III
0
10
20
30
40
50
60
70
80
90
100
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15

GRAPH 5

TABLE 1

TOTAL LIVE BACTERIA COUNT (COL/100 ML)		
STAGE I	STAGE II	STAGE III
2	2	6
0	0	3
1	2	1
0	-	-

TIME OF INCUBATION 48 HRS.

TEMP OF INCUBATION 28° C

TABLE 2

	STATION A	STATION B
TOTAL SAMPLE VOLUME	325 L	601 L
PARTICLE COUNTS/L		
0.2 - 0.5 MICRON	7220 ± 1010	4960 ± 571
0.5 - 1.0 MICRON	1210 ± 504	460 ± 185
> 1.0 MICRON	896 ± 398	365 ± 156
TOTAL > 0.2 MICRON	9340 ± 1160	5790 ± 626
TOTAL BACTERIA/100 ML	214 ± 57	56 ± 18

NOTE: ± VALUES ARE AT 95% CONFIDENCE LEVEL

Case 2

This case even more clearly proves the practical value of the SEM method in the monitoring and troubleshooting of the D.I. water systems.

Poor quality of D.I. water was suspected to affect a product at the point of use (in one of the wet stations). At the same time the quality of D.I. water at the point of supply (after 0.2 micron filter in the polishing station) was good and there was not doubt that a contamination was coming from another source than D.I. water polishing station.

The testing of D.I. water by SEM was performed in order to determine a cause for the problem. Additional samples were analyzed by live bacteria count. Samples were taken from the following points:

1. The point of supply, after the final filter in the polishing station (see Picture 3).
2. Before point of use filter (see Picture 4).
3. After point of use filter (see Picture 5).

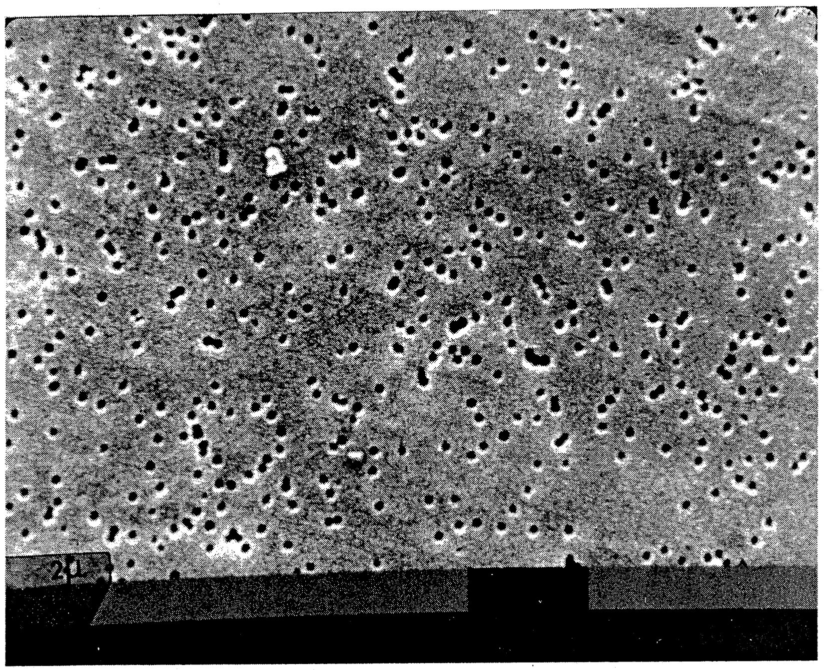
The results are presented in Table 3.

TABLE 3

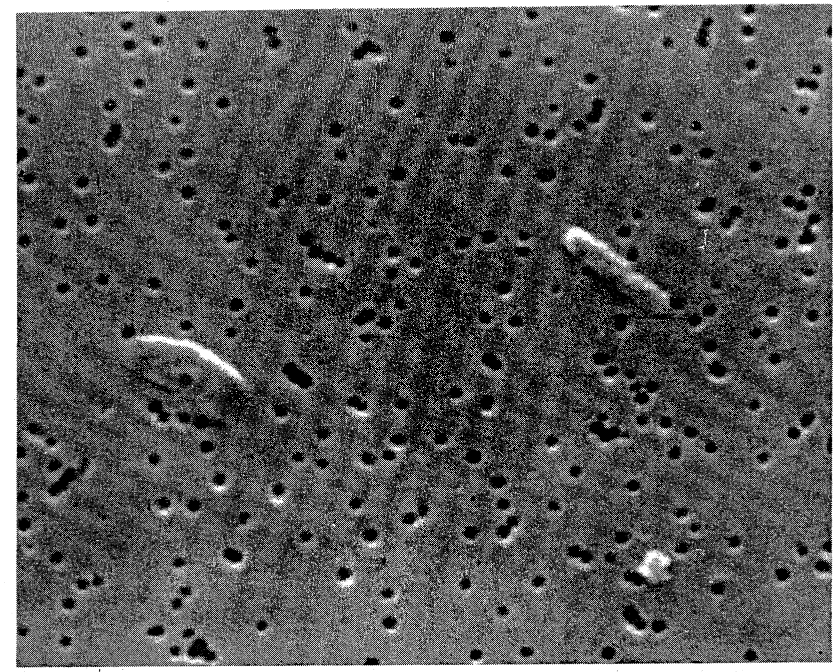
	A (POS)	B (PRE POU)	C (POST POU)
TOTAL SAMPLE VOLUME	670 L	360 L	360 L
PARTICLE COUNTS/L			
0.2 - 0.5 MICRON	352	2182	2095
0.5 - 1.0 MICRON	34	374	698
1.0 - 2.0 MICRON	0	187	44
>2.0 MICRON	34	62	44
BACTERIA COUNTS/100 ML	7	94	>5000
LIVE BACTERIA COUNTS (48 HOUR INCUBATION, 28^{o}C)	6/100 ML	130/100 ML	325/100 ML

The particle counts, as well as bacteria, increase before the point of use filter. This should be expected considering the large distance between point A and point B (approximately 500 feet). No reduction of particle counts at point C (after the point of use filter) shows complete inefficiency of the filter. The tremendous bacteria increase at point C was caused by the fact that the filter had not been replaced for almost three years. The large load of bacteria made the counting very difficult. It is quite possible that certain bacteria fragments were mistaken for particles.

The live bacteria counting method indicated also bacteria growth on the filter but SEM really reveals the magnitude of the problem.



PICTURE 3


PICTURE 4

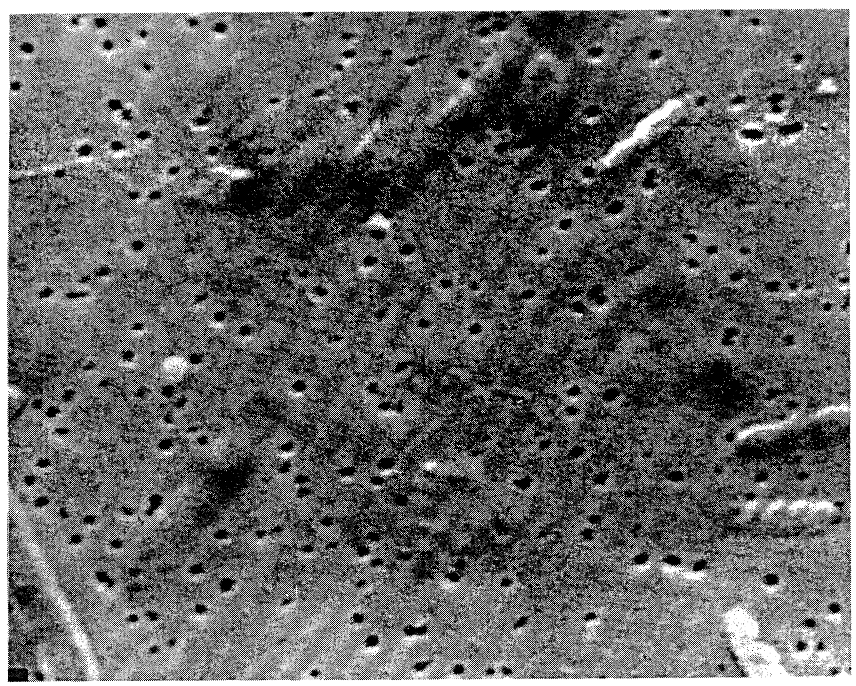

PICTURE 5

TABLE 4

MAIN COUNT/L	STATION A			STATION B			STATION C		
SAMPLE NO.	1	2	3	1	2	3	1	2	3
PARTICLE SIZE (MICRON)									
0.2 - 0.5	263	5000	6600	158	490	237	305	60	140
0.5 - 1.0	18	236	330	79	265	157	81	75	69
> 1.0	71	183	94	39	52	13	40	31	30
BACT./100 ML	18	16	10	90	25	55	6	6	-

As it can be easily noticed from Table 4, the best results were obtained for station C, where two banks of 0.2 micron filters Type B are installed. Obviously, the cost of double filtration is much higher and therefore this type of filtration should be installed if cost is justified. At this particular facility double filtration seems to be impractical due to the long distance (500 feet) between point of supply and point of use. As it was presented in Case 2, the quality of D.I. water in terms of particles and bacteria deteriorates with the increase of distance.

Case 3

This case shows the usefulness of the SEM testing in selection of an efficient filtration system. Evaluation of filters is a complicated and time consuming job. Such evaluation is not included in this study. However, working with various filters and regular testing of particles by the SEM method allows differences in the performance of these filters. A comparison led to conclusions that assisted in the selection of an efficient and economical type of filtration system.

Table 4 presents the SEM results obtained for three different polishing stations. Each of the stations has a different type of filtration:

Station A has one bank of the filters which contain 0.2 micron cartidges Type A.

Station B has one bank of the filters also, but they contain 0.2 micron cartridges Type B.

Station C has two banks of the filters (prefilters and final filters), both contain 0.2 micron cartridges Type B.

All the counts of the particles for station A are consistently higher than for station B. The difference is the most visible for small size particles. Both stations contain the same number of the filters and are designed in the same manner. The difference in water quality can only be explained by the better performance of the cartridge Type B than the cartridge Type A.

Cartridge A was tested by another user and the results were similar (see Table 5). It appears that the cartridge Type A sheds material from the membrane or its structure allows small particles to pass through. The first theory is more probable in view of the fact that the first results obtained for the newly installed cartridges were very low. The degradation of the performance was observed after two months of operation.

EDS analysis was performed in an attempt to determine the chemical composition of the particles but it was unsuccessful, because the particles were too small or organic in nature. Further investigation is being conducted in order to identify the source of the particles.

The bacteria counts in station B, higher than in station A, does not necessarily mean that the filter type B is less efficient in bacteria removal than the filter type A. Other factors may affect bacteria counts. For example, bacteria may be high in the building loop growing against the water flow back into the system. Further work is necessary to determine the source of bacteria.

TABLE 5

	STATION 1	STATION 2
TOTAL SAMPLE VOLUME	645 L	200 L
PARTICLE COUNTS/L		
0.2 - 0.5 MICRON	5000 ± 1200	5000 ± 1183
0.5 - 1.0 MICRON	115 ± 106	345 ± 332
>1.0	34 ± 36	141 ± 160
BACTERIA/100 ML	<1	7 ± 1

Note: ± values are at 95% confidence level

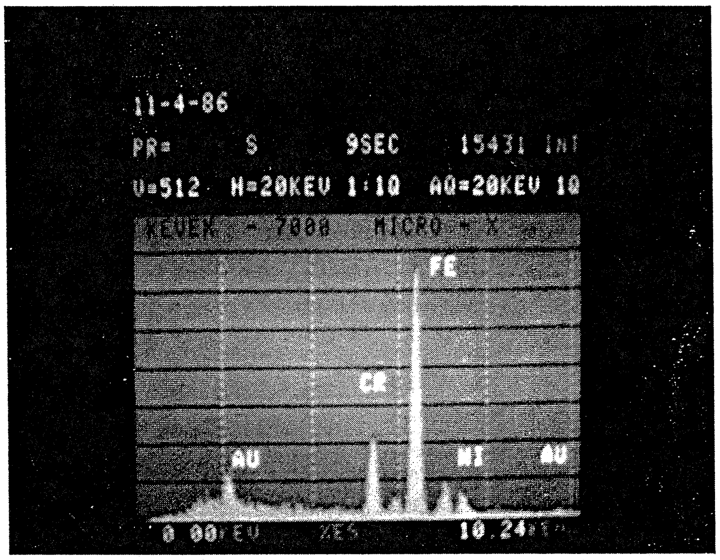
Case 4

This last case describes how SEM was utilized for identification of a source of the contamination.

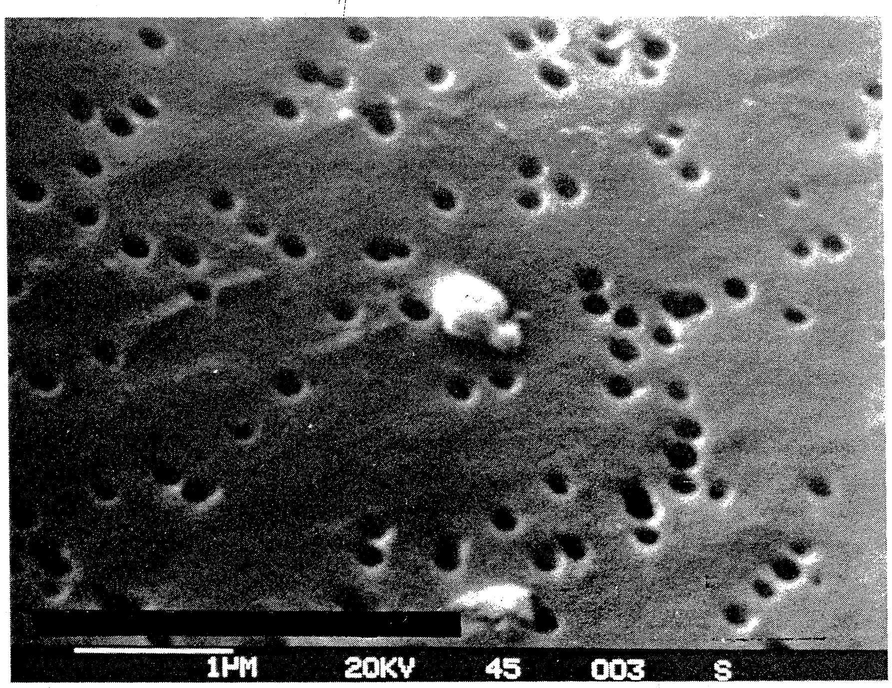
One user had contamination problem on wafers. The SEM revealed high particle counts in D.I. water. Subsequently, EDS revealed the presence of iron, nickel and chromium. The ratio of the detected metals indicated that the particles were 304 stainless steel. The source of these contaminants was found to be a corroded stainless steel filter housing. See Pictures 6 and 7.

CONCLUSIONS

1. The SEM testing of D.I. water for particles and total bacteria plays an important role in filtration process control.
   - It allows the user to select the most efficient and economical filtration system.
   - It helps to troubleshoot D.I. water filtration and distribution systems.
   - It enables determination of source of contamination.
2. At this time, SEM is the only available, proven method which detects particles down to 0.2 micron size.
3. The SEM testing should be performed periodically in spite of the time it takes and its cost.



PICTURE 6



PICTURE 7

REFERENCES

1. Balazs, Marjorie K. and Susan Walker, "Counting and Identifying Particles in High Purity Water", Semiconductor International Magazine, April 1982.

2. Roy Hango, Clifford Frith, Brad Eldred, "D.I.Water: A Common Sense Approach", presented at the Third Annual Semiconductor Pure Water Conference, January, 1984.