GENERATION AND RECOVERY OF HIGH PURITY WATER USED DURING THE MANUFACTURE OF SEMICONDUCTOR PROCESS EQUIPMENT

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ABSTRACT

FSI International, a supplier of surface conditioning, microlithography and chemical management tools for the semiconductor industry, has undertaken a major project to improve the quality of the ultrapure water used at its production and laboratory facilities. The goal is to produce water that meets the SEMI/SEMATECH “acceptable” criteria recommended by Balazs Analytical Laboratory. Achievement of this goal is especially significant because more than 95% of the production water is recycled.

This paper explains how the purity of the water was improved by on-line monitoring, elimination of undesirable components and implementation of advanced water purification techniques. The improvements shown in the overall water quality were matched by an improvement in the rinsing time necessary for process tools to achieve required purity specifications before shipment.

INTRODUCTION

Semiconductor manufacturers recognize the importance of process cleanliness in improving microcircuit device yield. Purity requirements in all phases of microcircuit production, especially in water and chemicals, have increased dramatically. As a supplier of semiconductor manufacturing equipment, FSI International is taking steps to improve the cleanliness of the products it supplies to the semiconductor industry: surface conditioning, microlithography and chemical management tools. It has undertaken a major project to evaluate and improve the quality of ultrapure water used in its manufacturing facilities.

The manufacture of some semiconductor equipment products requires large volumes of high purity fluids. FSI surface conditioning equipment is tested with high purity water to ensure that all components function properly. The equipment is also operated for a minimum of 24 hours to verify the function and reliability of all process controls. Finally, the equipment is flushed with water until a required particle specification is achieved. The

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quality of the water used in these steps directly affects the final product cleanliness and the time required to reach the particle specification.

CONTAMINATION CONTROL TASK FORCE

A Contamination Control Task Force was formed in March, 1994 to improve the quality of the water and gases at its manufacturing and laboratory facilities. The water purity goals for the process laboratory were based on Balazs Analytical Laboratory's "acceptable" levels, while the goals for production water quality were set at the "alert" levels.[1] Since particle concentrations are not specified in the Balazs guidelines at the "alert" level, the task force set manufacturing goals that were double the "acceptable" level. The goals used by the task force for ultrapure water quality are detailed in Table I.

Table I: Water purity goals established by the Contamination Control Task Force

Contaminant	*Process Laboratory Goal*	*Manufacturing Goal*
Particles ≥ 0.1μm (N/l)	≤ 375	≤ 750
Particles ≥ 0.3μm (N/l)	≤ 25	≤ 50
TOC (ppb)	≤ 2	≤ 5
Resistivity (MΩ-cm)	≥ 18	≥ 18
Residue (ppb)	≤ 100	≤ 100
Silica (ppb)	≤ 1	≤ 3
Bacteria (counts/100 ml)	≤ 6	≤ 25

The initial task of the contamination control project was to collect water purity data to determine the status of the system. On-line instrumentation was selected to monitor most of the characteristics in Table I. PMS IMOLV particle counters, an Anatel TOC analyzer and a Hach silica monitor were installed on the water circulation loops past the last point of use (POU). Data collected at these locations represent worst case conditions. The TOC analyzer, residue monitor and silica monitor were time-shared, while the other instruments were dedicated. In addition, water samples were taken at various locations twice each month to determine bacteria levels and once each month to determine ion concentrations.

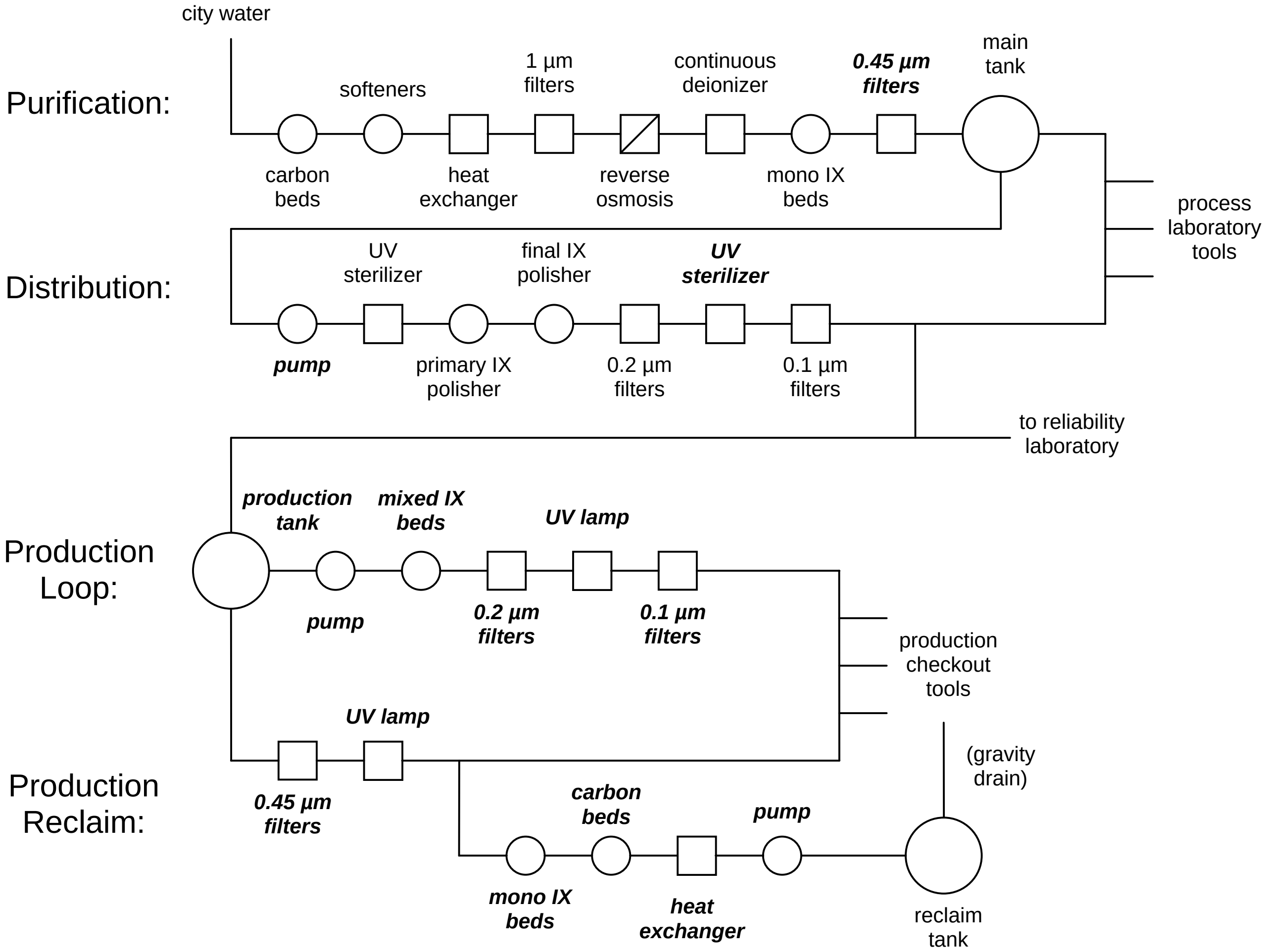
The second task of the project was to improve water quality. When the task force was formed, the process laboratory water was close to meeting the goals. However, the production water was far from meeting the goals for many parameters. Therefore, the task force concentrated on improving the quality of the production water. Numerous steps have been taken to improve water quality.

The third task was to develop procedures to ensure that surface conditioning equipment met particle specifications. All surface conditioning equipment is rinsed with ultrapure water prior to shipment. The specification for particle purity is <1 N/ml ≥ 0.3 μm in the tool effluent. The concentration of particles in the effluent is determined by diverting fluid entering the process chamber to a particle counter. The task force developed quality control procedures and collected data regarding the rinsing time required to meet specification. Improvements in water quality were evaluated for their effect on the rinsing time.

FACILITIES WATER SYSTEM DESCRIPTION

Figure 1 describes the major components in the facilities ultrapure water system. The upper purification and distribution elements in the figure are shared by several end users (process laboratory, production and reliability lab). The production system, which was the focus for improvement, is shown in the bottom portion of the diagram. All components in Figure 1 which are new or were upgraded during this project are labeled with bold letters.

Figure 1: Water Purification/Reclamation System



Initial purification of the city water consists of chlorine removal, softening, reverse osmosis and deionization. Ion removal is accomplished through continuous electro-deionization and ion exchange (IX). The water is then filtered and sent to a main storage tank. Water from the tank is sterilized, polished and filtered as it is pumped to the process laboratory. When this project was initiated, the quality of the water delivered to the process laboratory was near the Balazs acceptable levels, as required for the wafer processing experiments. Part of the water in this high purity loop is transferred to a storage tank for the production loop. It is pumped through additional mixed bed ion exchangers, an ultraviolet sterilizer and filters before reaching the tools on the production floor.

A reclaim system is used in the production water loop to reduce water consumption. This loop is isolated from the laboratory loop to avoid cross-contamination and accumulation of hard-to-remove contaminants. Most of the water used by production (> 95%) is captured by gravity in a storage tank. It is then pumped through a heat exchanger, carbon beds, ion exchange beds and filters before being sent back to the production storage tank. The heat exchanger is critical to the overall performance of the system because several surface conditioning tools discharge water at 95°C. This water must be cooled before it reaches the purification components, which can be damaged by elevated temperatures.

CONTAMINANT CONCENTRATION HISTORY

Data have been collected hourly on the concentrations of particles, TOC, resistivity and residue in the water since the task force was formed. Data for the production water loop are presented in Figures 2-6 as weekly means as a function of time. Target levels are shown as dashed lines for reference.

Changes made to the system in the first three months of the project resulted in a dramatic improvement in particle concentrations, resistivity and residue. Resistivity quickly increased from 5 to 16 MΩ-cm, concentrations of particles ≥ 0.3 µm decreased from more than 10,000 counts per liter (or N/l) to less than 1000 N/l, and residue decreased from 10 to 1 ppb.

As later improvements were made, the concentration of particles continued to decrease until the particles ≥ 0.3 µm reached the target level in September, 1994. Particles ≥ 0.1 µm stabilized at approximately twice the target level. TOC, however, has remained above the specification despite all the improvements made to the system through November, 1995. TOC concentrations only reached the target when production activities were moved to a new facility in November/December, 1995. The following sections describe the actions taken to achieve these improvements. They also detail plans to resolve the TOC issue.

Figure 2: Resistivity History

(measured with an Anatel A-100 TOC analyzer)

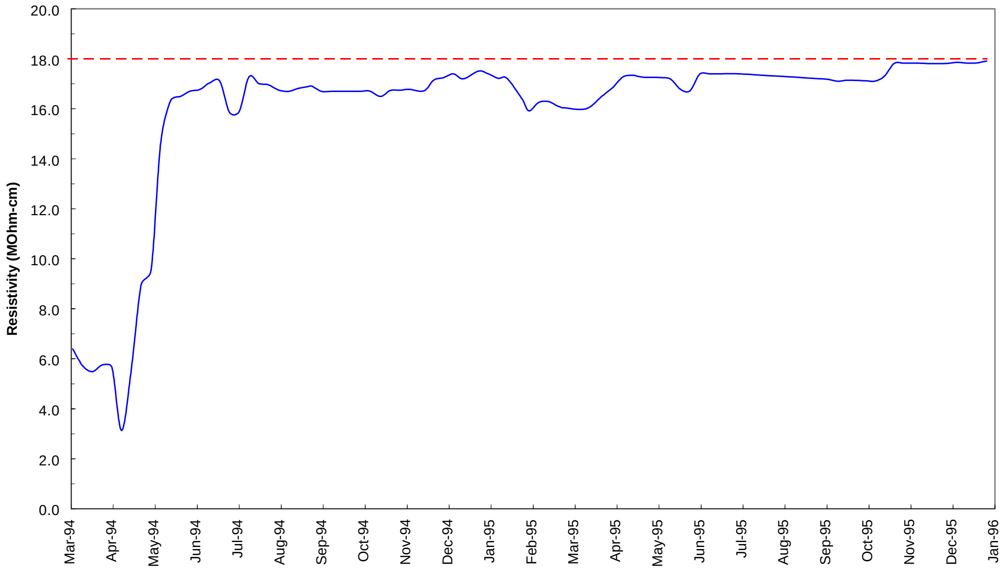


Figure 3: Residue History

(measured with a TSI 7761 nonvolatile residue monitor)

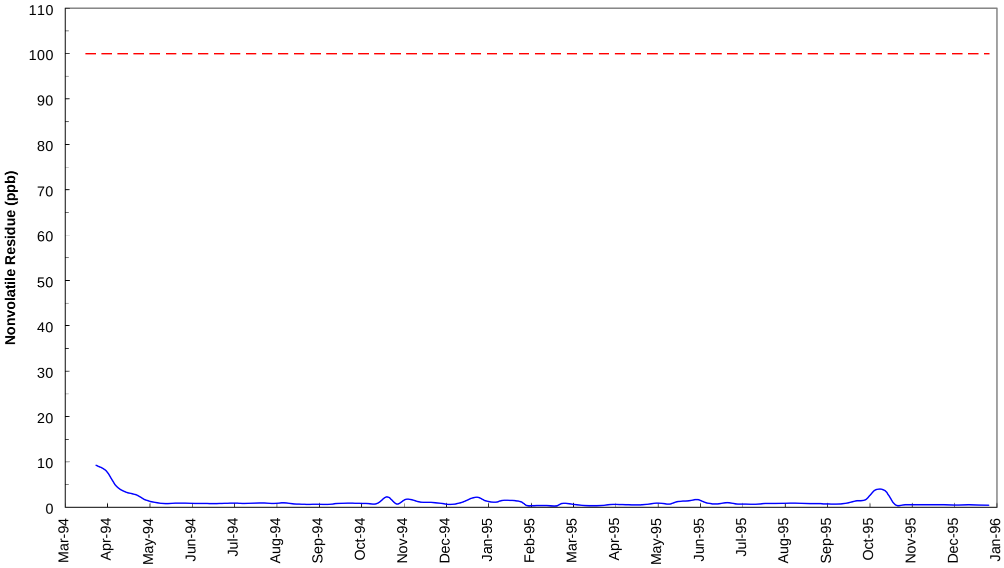


Figure 4: Total Organic Carbon History
(measured with a Anatel A-100 TOC analyzer)

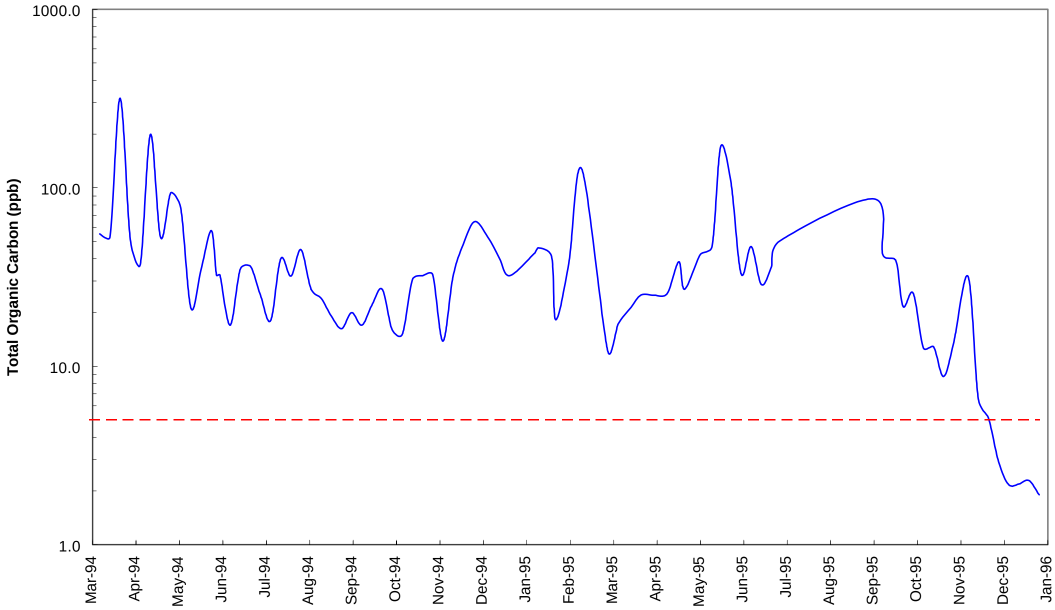


Figure 5: Particle Concentration History (≥ 0.3 µm)
(measured with a PMS IMOLV liquid particle counter)

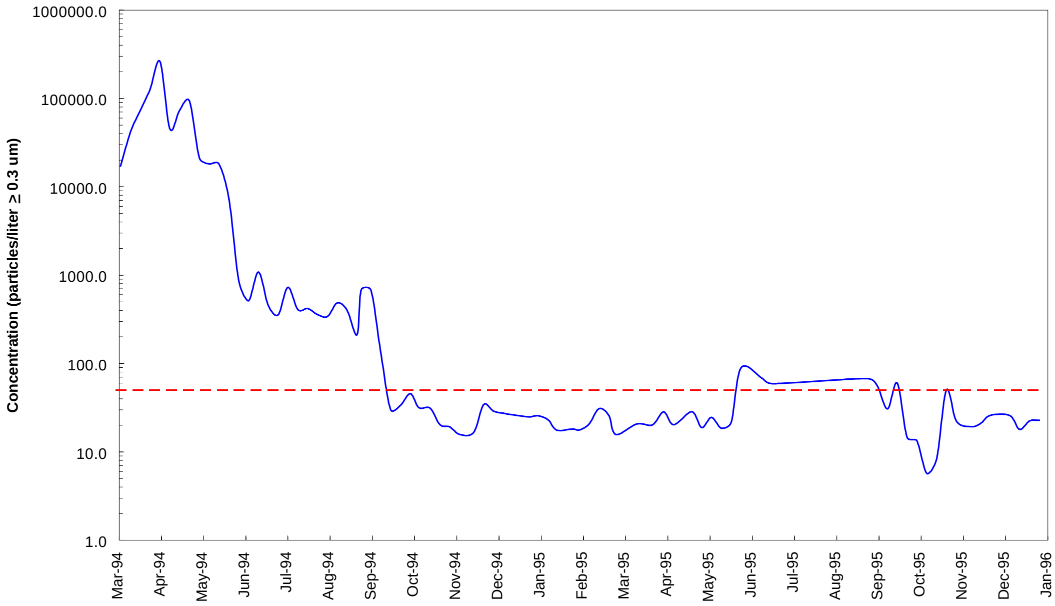
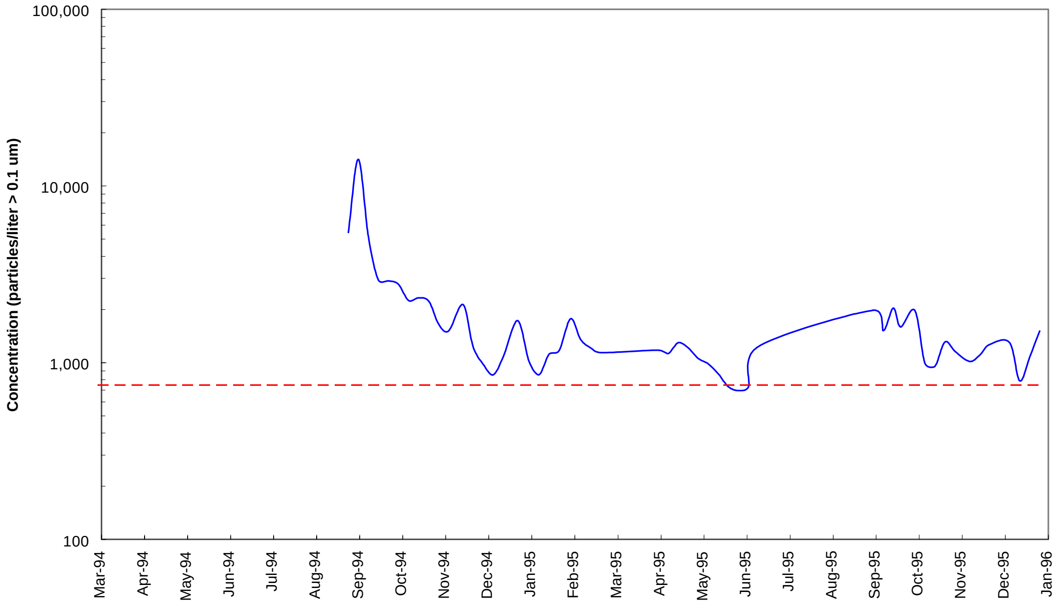


Figure 6: Particle Concentration History (≥ 0.1 μm)
(measured with a PMS HSLIS M100 liquid particle counter)



SYSTEM IMPROVEMENTS

The changes made to the production loop during the first three months of the project included the installation of mixed bed polishers and a UV sterilizing lamp. In addition, extra mono ion exchange beds and carbon beds were added to improve system capacity and performance. Final filters in the production loop were changed from 0.2 μm to 0.1 μm. Some system components were eliminated because their materials of construction were shown to shed particles or leach inorganic ions. Many of these changes were made after site visits and consultation with customers.

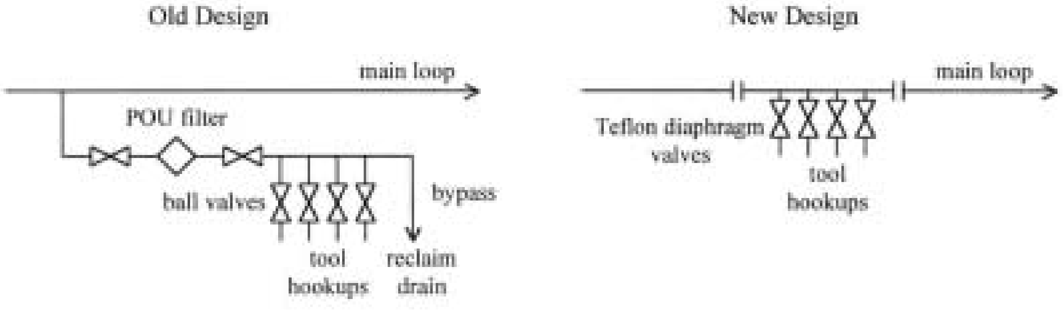
The rapid improvement in water **resistivity** was mostly due to the addition of the mixed bed ion exchangers in May, 1994 (Figure 2). Upgraded mono bed capacity in the production loop and reclaim system also assisted in increasing final loop resistivity and system maintainability. A nitrogen blanket was added to the storage tank to eliminate CO_2 absorption, which lowers water resistivity. After these changes, resistivity in the production loop depended mainly on the frequency of ion exchange bed replacement.

Decreases in **residue** in the system water matched improvements in resistivity over the same time period (Figure 3). This corroborates the reduction of dissolved ions and other contaminants indicated by the resistivity trend. Furthermore, the total concentrations of 11 anions and cations averaged less than 2.5 ppb for ten samples taken during 1994 at the end of the production loop. The ions measured were bromide, chloride, fluoride, nitrate, phosphate, sulfate, ammonium, potassium, sodium (analyzed by ICP-MS), calcium and magnesium (analyzed by GFAA).

During the next four months the task force focused on reducing **particle concentrations** (Figures 5 and 6). New particle counters (HSLIS M100 models from Particle Measuring Systems) capable of detecting 0.1µm particles were installed in August, 1994 at the end of the loop and in the production testing area. The water delivery manifolds were inspected carefully. Testing showed that the ball valves and POU filters in each manifold were releasing particles on flow initialization. Bypasses designed to stabilize the flow through the POU filters were inadequate to control particle concentrations and wasteful of power and system delivery capacity. The water quality from the production manifolds would often not meet the tool effluent specification for the first 10-30 minutes of usage. POU filters, which had previously been necessary because the particle concentrations in the main loop were excessive, had become a hindrance.

A new manifold was designed to eliminate the ball valves and POU filters. Diaphragm valves were assembled into manifolds with minimum deadlegs (≤ 2.5" or 2.5 pipe diameters) to replace the previous manifold design, as shown in Figure 7. These new manifolds were installed directly into the main loop in September/October, 1994. At the same time, the plumbing in the water system was consolidated into a single loop, eliminating some stagnant regions. Particle concentrations ≥ 0.3 µm in the production loop dropped below the target level for the first time. Water demand was nearly cut in half by the elimination of the manifold bypasses which were constantly sending high purity water to the reclaim drain.

Figure 7: Water Manifold Designs



Despite all the changes made to the production water loop, **TOC** levels remained above the target of 5 ppb (Figure 4). The fluctuations in TOC were correlated to water usage by the tools on the production floor, especially hot water functions. In general, the TOC levels increased during the day when water was being discharged from new tools and decreased at night and on weekends when there was little or no production activity. It is likely that the source of the TOC was the PVC reclaim piping system rather than the tools in production.

Although some components were not able to withstand long term ozone exposure, an experiment was performed to quantitate ozone destruction of TOC in the water system. Assuming a first order reaction for ozone destruction of TOC, the reaction constant in this experiment was 0.06 ppb per ppm $O_3 \bullet$min. This effect has been documented by others.[2,3,4] This reaction constant has been used to establish sizing capacity for ozonation of the reclaim water in the new FSI production facility.

DATA MANAGEMENT

Documentation of procedures to monitor and maintain the system according to ISO guidelines was completed in June, 1994. All warning and alarm levels were set and appropriate responses to them were documented. Procedures to produce monthly control charts were implemented.

To facilitate data management, a DataTrax monitoring system was installed in August 1995. It continuously monitors and stores fluid purity data from approximately 50 monitoring points in the high purity water and gas delivery systems. It automatically pages personnel when alarm limits are exceeded. Creation of monthly control charts was dramatically simplified. Previous procedures included compilation of comma-separated data files in spreadsheets from various independent instruments and computers. The DataTrax monitoring system also helped the task force recognize patterns and correlate contamination episodes with system events.

LESSONS LEARNED

The implementation of continuous data collection and analysis with control charts was a valuable part of the effort to improve water quality. It allowed the task force to quickly identify the effects of system parameter settings and maintenance procedures. Several of these examples are described below.

Mono ion exchange bed replacement. The first correlation noticed after installation of a continuous monitoring system concerned the mono ion exchange beds in the main water treatment system (the first set of mono beds in Figure 1). The effluent from the mono beds was monitored continuously by an on-line silica analyzer (Figure 8A). The mono beds were replaced when silica in the effluent reached 10-20 ppb. The interval for mono bed replacement was normally every 2-3 weeks. However, during a period when the continuous deionization system (CDI) was not functioning properly, the mono beds required replacement every 2-3 days. Figure 8B shows that TOC spikes and resistivity dips occurred whenever the mono beds were replaced. Varying degrees of TOC degradation were observed; one replacement did not cause any observable change. It was obvious that mono bed replacement had a detrimental effect on final water quality.

Figure 8A: Silica Content of Mono Bed Effluent

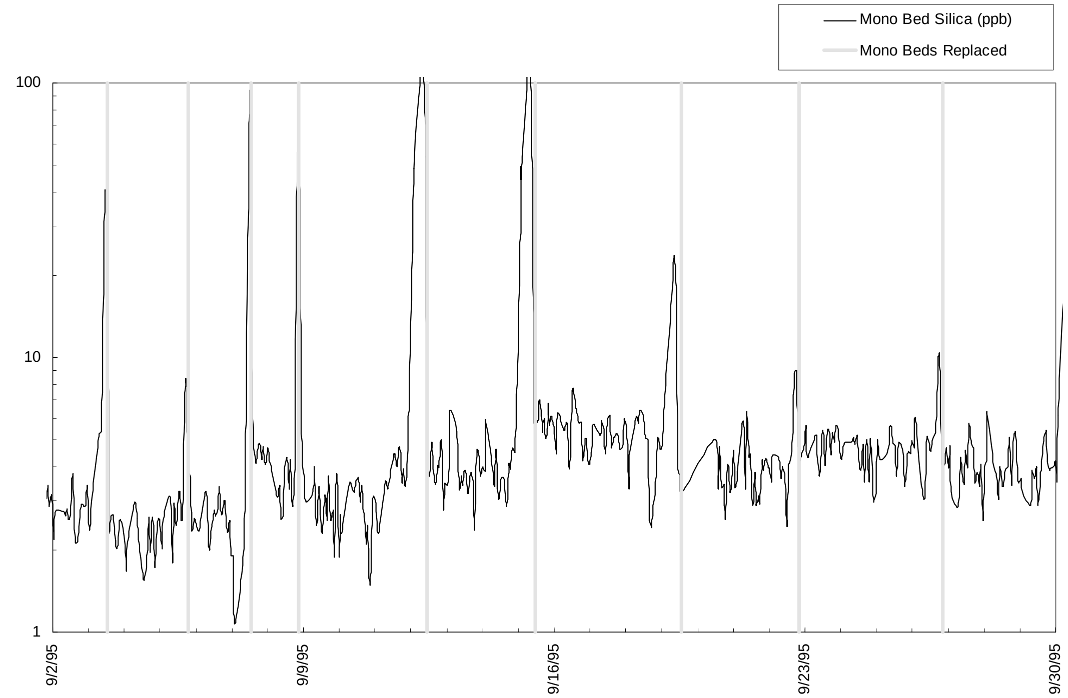
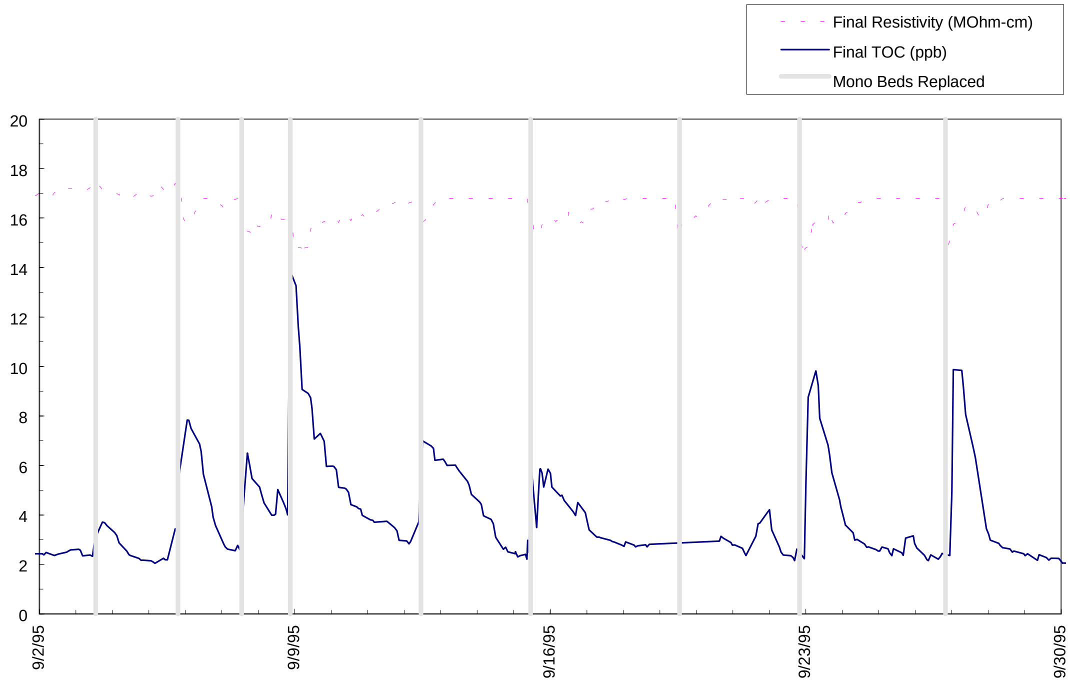


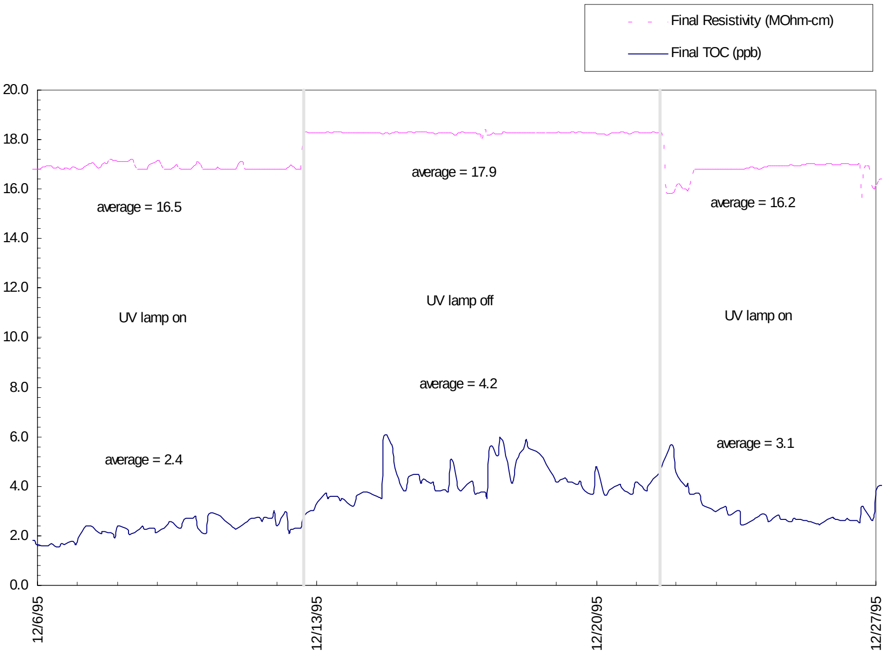
Figure 8B: Effect of Mono Bed Replacement on Final Resistivity and TOC



The upsets in final water quality occurred even though the mono beds were flushed to drain until their output was over 4 MΩ-cm before being placed on line, which usually required 700-900 gallons of ultrapure water. To minimize the effect of the upsets, the maintenance procedure was changed. Regardless of the silica output, the mono beds were replaced every
other weekend (unless critical processes were in progress). This allowed the system to fully recover from mono bed replacements before it was required by laboratory and production users. Another procedure being considered is to sample the effluent from each mono bed for TOC before putting it into service.

Ultraviolet light. The 185 nm UV lamp installed upstream of the final filter in the lab loop was found to reduce TOC, but at the expense of resistivity. Figure 9 shows that the resistivity was higher when the UV lamp was off during the period from 12/13 - 12/21. It is not clear whether lower TOC or higher resistivity is more important. The task force has chosen to leave the UV lamp on to control TOC and bacteria. Other authors have also found that UV light reduces TOC to very low levels through breakdown to organic acids, but resistivity worsens.[5]

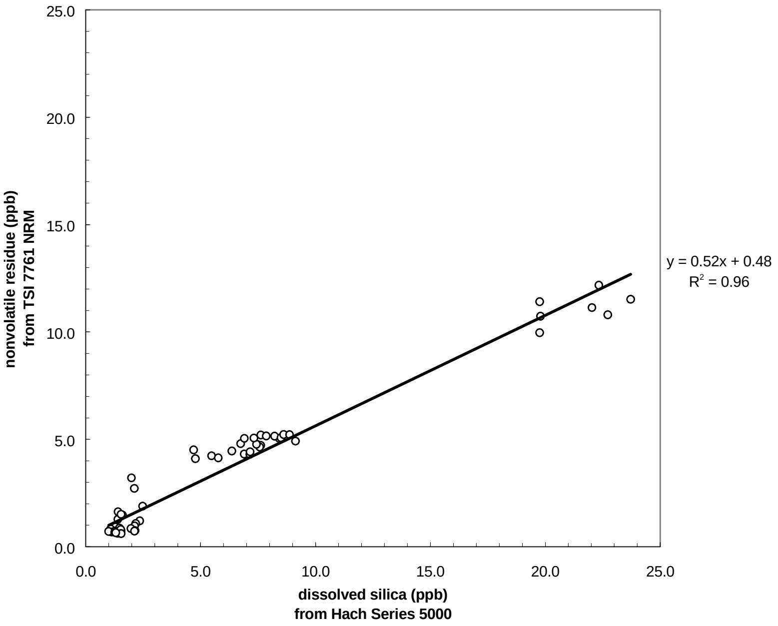
Figure 9: Effect of UV Sterilization on Final Resistivity and TOC



Dissolved Silica/Nonvolatile Residue Correlation. Data from a TSI 7761 nonvolatile residue monitor and a Hach Series 5000 silica analyzer were recorded for one month from the process laboratory and production water loops. Water purity varied widely during that time. Residue and silica levels were compared to determine whether they were dependent or independent, so that existing instrumentation could be utilized efficiently and so that system upsets could be understood more thoroughly. The residue and silica data are graphed in Figure 10. Each data point represents a stable average value for a 12-hour time period.

As Figure 10 shows, there is a strong correlation between silica and residue in the ultrapure water system. The linear correlation coefficient is 0.96 and the slope of the line is approximately 0.5. This means that the silica readings are always about twice as high as the residue readings. Other authors have found similar correlations with different coefficients.[6]

Figure 10: Residue/Silica Correlation Data



WATER QUALITY EFFECT ON PRODUCT CLEANLINESS

Improvements in water quality were expected to have a positive impact on the cleanliness of semiconductormanufacturing products. MERCURY® spray processors are tested for particle cleanliness after they have undergone a 24-hour burn-in period. HELIOS® water heaters are tested as soon as they have passed initial inspection and functionality checks. Figures 11 and 12 show particle concentrations after 30 minutes of water rinsing for MERCURY and HELIOS tools. Figures 13 and 14 show the flushing time required to meet the product specification for the same tools.

All four tool performance graphs include data regression lines, which show continuous improvement with time. In less than one year, the rinse time required to meet specification had decreased by as much as a factor of 10 and the majority of the tools were qualifying in less than 30 minutes of particle testing. Very few outlying data points have been recorded since the plumbing changes and installation of the new valve manifolds in September/ October, 1994. These changes caused particle concentrations to remain low and stable throughout the tool rinsing procedures.

Figure 11: MERCURY Spray Processor Particle Concentrations
After 30 Minutes of Rinsing

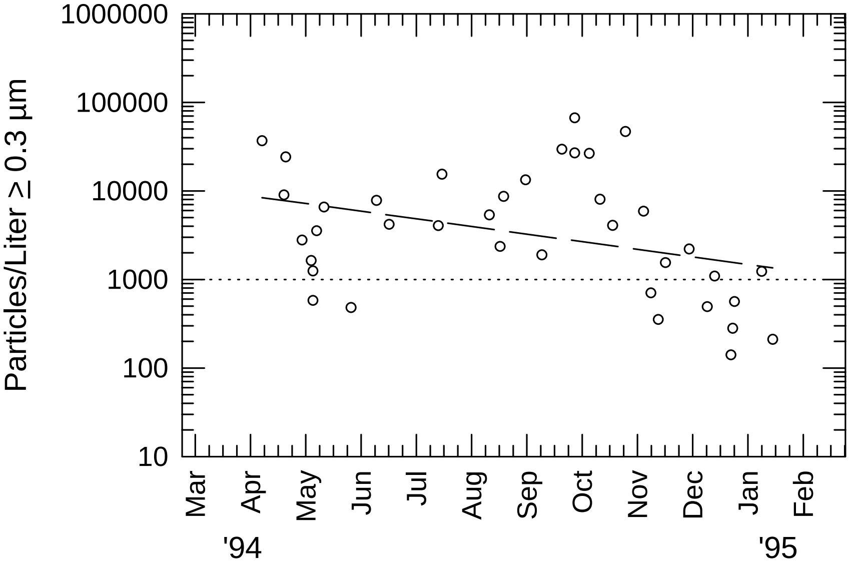


Figure 12: HELIOS Water Heater Particle Concentrations
After 30 Minutes of Rinsing

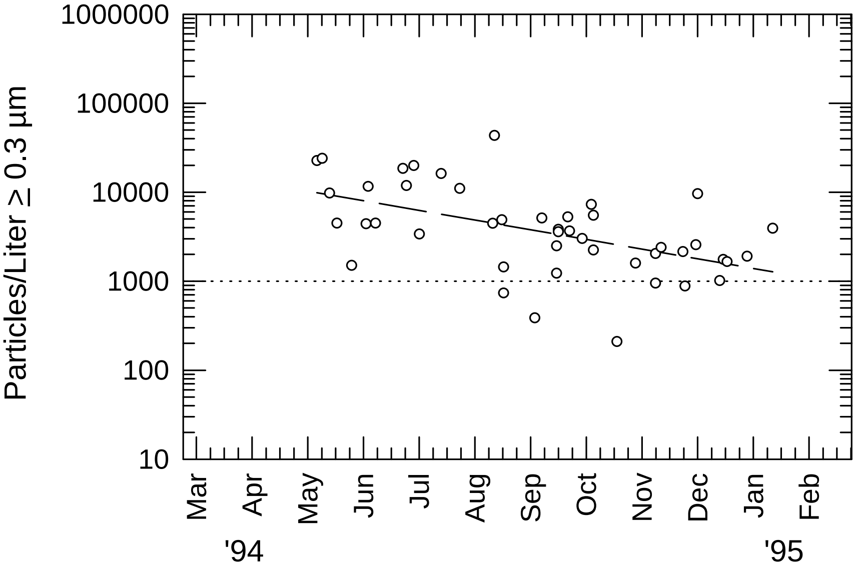


Figure 13: Time Required for MERCURY Spray Processors
to Achieve Particle Specification

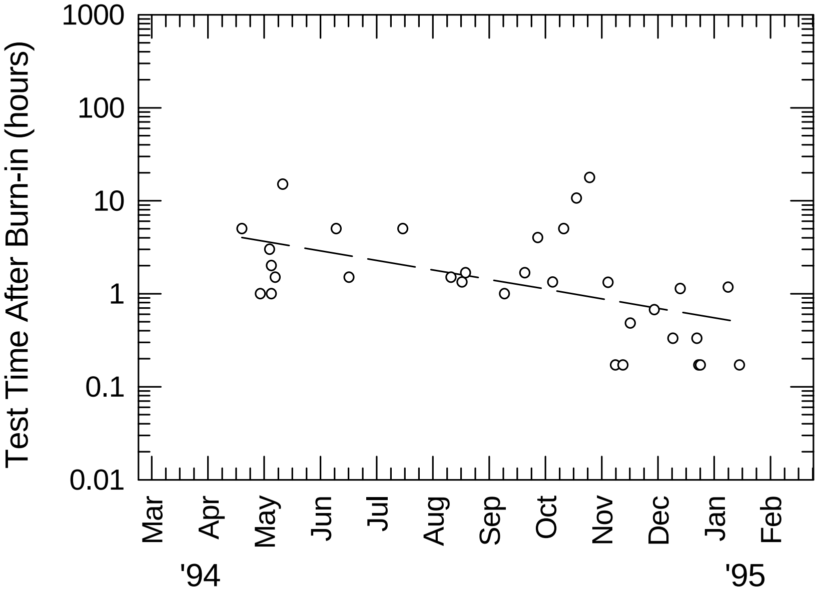
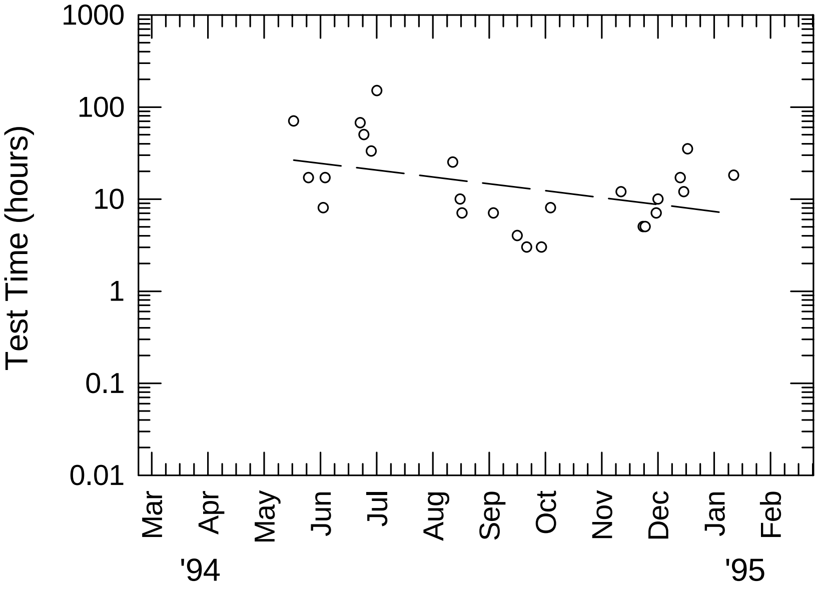


Figure 14: Time Required for HELIOS Water Heaters
to Achieve Particle Specification



WATER SYSTEM DESIGN IN THE NEW MANUFACTURING FACILITY

FSI International opened a new manufacturing facility in November, 1995. It features a new water system and a 40,000 square foot cleanroom for manufacturing, product checkout and packaging. The design of the new water system was influenced by the experience gained during the upgrade of the water system at the older facility. The system circulation loop is constructed from PVDF piping and Teflon® diaphragm valves, and contains no dead legs. Ozonation of the reclaim water is being evaluated for reducing TOC. New monitoring equipment and an expanded version of the DataTrax system have been included. Performance in terms of particle concentrations, resistivity, silica and bacteria load is expected to surpass the “acceptable” Balazs target levels (the process laboratory goal shown in Table I). Preliminary results indicate that this goal will be achieved.

FUTURE PLANS

The Contamination Control Task Force will continue its efforts to improve the quality of the facility water systems. Work is underway to assess the cleanliness of all new components used in the water systems and products. The task force is collaborating with vendors on material changes and manufacturing procedures so that vendors’ new components will meet the task force’s purity requirements. New procedures for purity measurement during production checkout will be developed and implemented for the remaining products. Also, purity specifications for outgoing equipment will be lowered as the semiconductor industry changes.

SUMMARY

The Contamination Control Task Force has improved the FSI water systems to produce the high purity water required to manufacture clean semiconductor process tools. The water systems provide water for functional and cleanliness checks of production equipment and for laboratory wafer cleaning experiments. Particle concentrations, resistivity, residue, silica content and bacteria levels are similar to those recommended by Balazs Laboratory. TOC was the most difficult parameterl to control. Water usage and cost have been reduced by reclaiming more than 95% of all the water used for production. Semiconductor process tools produced since the improvements reach product cleanliness specifications approximately 10 times faster.

Among the most important changes to the water system were increasing capacity of ion exchange and carbon adsorption beds, using 0.1 µm filters for particle control and replacing components that leached inorganic ions or particles. Plumbing changes included elimination of stagnant areas and redesign of manifolds. A continuous monitoring system facilitated data analysis and identification of trends and correlations. TOC destruction by ozone is recommended for systems with compatible materials of construction. Information learned during this system upgrade was used in the design of the water system in FSI’s new manufacturing facility.

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