



ASSESSING THE DESIGN AND PERFORMANCE OF A CENTRAL HOT ULTRAPURE WATER SYSTEM

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COMPENDIUM: A 75-gal/min electric central ultrapure water heating and distribution system was recently installed at the AT&T MOS IC production facility in Orlando, FL. This article describes the system's basic design and summarizes its operating performance. Generally, the system performed above design targets for temperature variation and induced no significant increases in dissolved chemical contaminants. Moderately elevated particle counts and changes in colloidal silica composition were, however, observed in the system product.

Demand for hot ultrapure water (HUPW) in semiconductor facilities has risen sharply as new processes substitute HUPW in steps traditionally requiring 20°C UPW. In recent AT&T submicron facility installations, typical HUPW design capacity has averaged 20–30% of total UPW capacity; several years ago, that percentage would have been near zero. Central HUPW systems present challenging facility-design problems, but they offer significant benefits over local HUPW generation, specifically in lowering costs and maintaining water quality.

Most wafer-processing equipment using HUPW requires a temperature of 70° to 80°C for optimum performance. Also, AT&T process specifications allow for few variables, so the delivered temperature must be tightly controlled. Further, reaction kinetics are faster in HUPW than in UPW. The quality of HUPW delivered to the wafer must, therefore, be at least as high as that of UPW to prevent water contaminants from causing device defects.

This presents a variety of problems, specifically:

- Many wetted materials have contaminant elution rates dramatically higher at 80°C than they do at 20°C in UPW.¹
- Many filters cannot tolerate 80°C, limiting particle control options.
- Traditional analytical instruments are not designed for use at high temperatures. Compensation algorithms or cooling loops must be employed, which could introduce monitoring errors.
- Distribution-system material choices are restricted,

	Range	Method
Indicated resistivity (MΩ/cm @ 25°C)	14–15	On-line temperature-compensated meter
Temperature	80°C +15° -40°	Thermocouple

Table 1: Typical local spot heater quality.

because most polymers suffer strength loss at high temperatures. Stainless steel is generally not used, because iron could leach into the hot water.

- Distribution systems require insulation, and conventional fiberglass cannot be used in cleanrooms.

Until recently, HUPW was commonly provided via “spot” heating: a local heater and filtration system for each process tool using HUPW. These systems remained at low flow while idle, requiring only a small amount of replacement heat. During demand periods, flow rates rapidly increased, requiring fast response by heating elements. As a result, temperature consistency in spot heaters was generally poor.

Material elution during low-flow conditions was another problem. Transient contamination spikes, although never documented directly, were inferred from the failure of wafers processed immediately after high flow began. Particle shedding also occurred as a result of rapid flow and temperature changes. Table I lists typical point-of-use performance of local heaters at AT&T-Orlando.

CENTRAL-SYSTEM DESIGN

The central HUPW system was designed to supply 10 new spray tools and 2 automatic wet stations in the Orlando OR-1 MOS cleanroom, to eliminate local heaters at existing process equipment, and to allow for future expansion. AT&T-Orlando already operated a high-quality UPW system, so cold makeup for HUPW was already available.²

Capital-cost analysis indicated that an upgradable central system could be installed for less than the cost of adding heaters. Also, operation and maintenance costs for the entire system would be less than those for just the existing local heaters. Because economics vary among sites (e.g., electric heaters were specified instead of steam because electric power rates are relatively low in central Florida) and cost-comparison data are readily available,³ this article focuses only on design and performance.

Our goal was to produce $80^{\circ}\text{C} \pm 10^{\circ}\text{C}$ HUPW at the wafer-process-equipment inlet and keep chemical contamination at or below levels in the makeup UPW. Further, we decided to employ the best-available technology in order to minimize particulate contamination.

Our final design includes two centrally located electric-heater stations, each with an independent distribution system. Particle counters, resistivity monitors, and temperature sensors are located downstream of the most distant wafer-processing equipment. Figure 1 shows the basic system configuration of the OR-1 West HUPW Station; an identical installation is located on the facility's east side. Each station is physically situated directly below the operating clean area. A small, nonrecycled drain flow at each end of the distribution grid prevents stagnation.

Ten design features of the HUPW system merit special attention.

Central Heating Stations. Because heat loss and elution are functions of retention time, the system has minimum internal volume, with less than 100 linear piping feet separating the final filter from the most distant user.

Electric Heaters. Each station includes five individually controlled 105-kW units. All internal surfaces other than seals are high-purity PTFE and PVDF. No purity or reliability problems arose during previous uses of these materials in local heaters.

Filters. Each station has one 40-in. custom four-cartridge filter housing located after the heaters and no filters at the process equipment connection. All internal housing surfaces except seals are PVDF, Ra-50 maximum. Cartridges are 0.05- μm polycarbonate membrane with polypropylene support and hardware. Polycarbonate membranes had been used in local heater installations.

Piping. All grid piping and fittings are high-purity PVDF with internal microfinish.

Joints. All joints are welded by a modified bead-and-crevice-free technique, which produces a smooth internal finish.

Vibration Isolation. To avoid sharp impacts that could create particle events, all piping and filters are vibration-isolated at hangers and filter pads.

Process Equipment Taps. The taps have a high-purity PVDF body and PTFE diaphragm-isolation valves sized at the supply header diameter, and include a PVDF sanitary union with sanitary PFA tubing and fittings to the process equipment. Very low particle contributions had resulted from this configuration in previous UPW applications.

Insulation. All nonclean-area insulation is 1.5-in.-

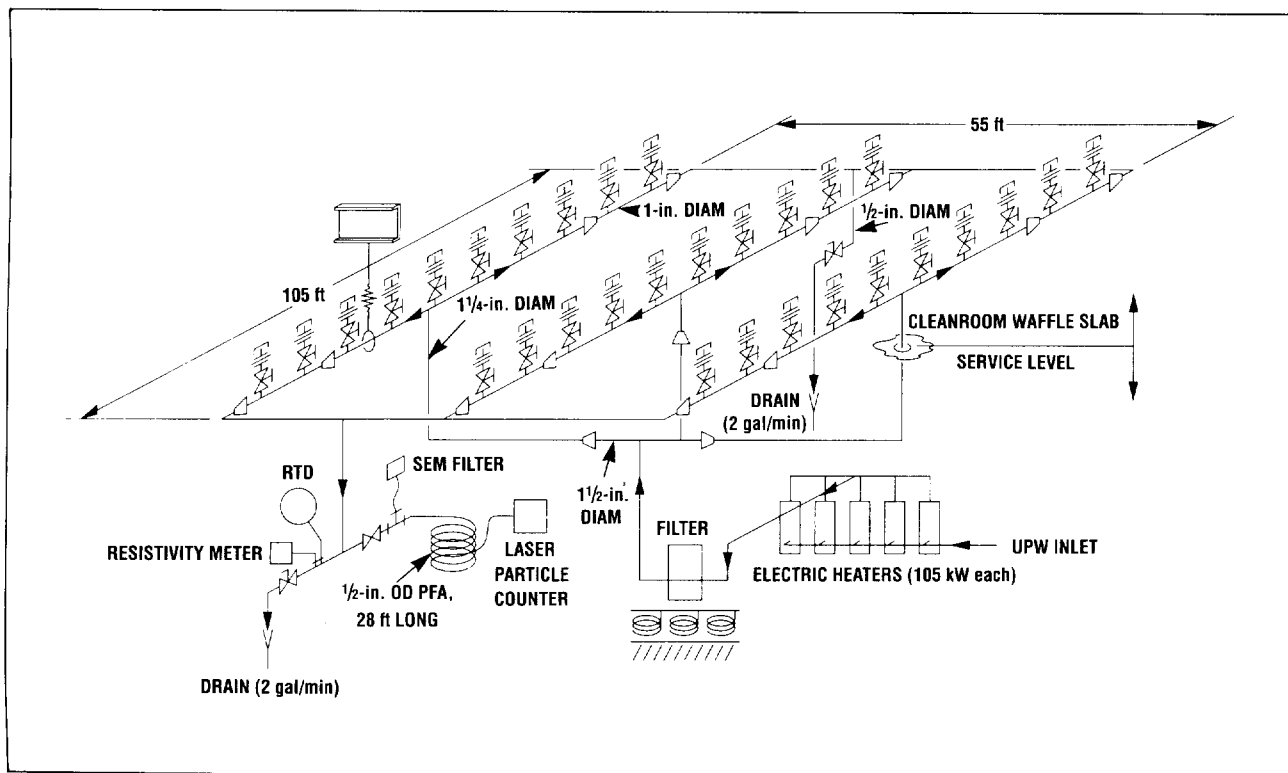


Figure 1: Layout of OR-1 West HUPW Station.

Table II: Typical UPW versus HUPW performance.

	UPW	HUPW	Alert Limit (HUPW)	Method
Indicated resistivity (MΩ/cm @ 25°C)	18.1–18.2	18.1–18.6 ^a	18.0	On-line temperature-compensated meter
TOC (ppb)	3–5	3–5	7.5	On-line meter
Soluble silica (ppb, as SiO ₂)	<1	<1	>1	Heteropoly blue ^b
Total silicon (ppb)	5–6	6–7	None	ICP-AES ^b
Bacteria (per 500 L)	0	0	None	SEM count ^b
Particles (per ml, >0.05 μm, 8-hr average)	1.0	3.5	5.0	Laser particle counter
Particles (per L, >0.05 μm, >1440 L filtered)	35	111	None	SEM ^b
Temperature	79°F ± 3°F	78°C ± 2°C	<70°C and >90°C	RTD
Sodium (ppb)	<0.05	<0.05	None	Ion chromatography ^b
Chloride (ppb)	0.05–0.07	0.05–0.07	None	Ion chromatography ^b
Fluoride (ppb)	<0.05	<0.05	None	Ion chromatography ^b
Potassium (ppb)	<0.05	<0.05	None	Ion chromatography ^b
Total calcium (ppb)	0.3–0.6	0.3–0.6	None	ICP-AES ^b

^a Certified meter with sample cooler shows resistivity at 56°–60°C; reading reflects error introduced by elevated and slightly fluctuating sample temperature.
^b AT&T Analytical Facility in Allentown performed all tests.

thick fiberglass. Cleanroom piping is wrapped with elastomeric, closed-cell, flexible insulation.

O-Rings. Each heater and filter housing has several silicone O-rings. Sanitary unions use FEP-coated Viton, and filter cartridges use PFA-coated Viton.

Analytical Instrumentation. Resistivity is measured with a temperature-compensated, microprocessor-based resistivity probe preceded by a sample cooling loop. Temperature is monitored by a precision 100-Ω resistance temperature device (RTD) with a time constant of <1 minute. Particles are tabulated by a laser counter with a sensitivity of 0.05 μm. Because the monitoring point is downstream of the final process tool, the readings represent the minimum quality supplied to the equipment.

CENTRAL-SYSTEM PERFORMANCE

The installed system controls temperature considerably better than the targeted ±10°C. Each heater has a heat-controlling microprocessor set at 82°C, which yields an average 78°C downstream of the last user. The West HUPW Station, which supplies eight spray tools and one robotic sink, consistently runs ±2°C at the RTD over a 24-hour period. Slight piping and

controller differences among the five station heaters cause individual heater outlet temperatures to vary, but the overall reading is extremely stable. When one station heater was recently removed from service for a day, RTD fluctuation rose to ±4°C, but returned to ±2°C after the unit was returned to service.

Six-month data show that HUPW quality matches UPW closely for nearly all parameters. Table II summarizes measured performance and HUPW alert levels. (Figure 1 gives monitoring locations.)

Previous confined-water leaching studies have predicted substantial elution of fluoride and organics from PVDF in HUPW,¹ but our experience did not support that finding. Analyses of product HUPW and supply UPW did not demonstrate any significant differences in TOC or fluoride concentrations. Only the initial sample contained any detectable fluoride (150 ppt); subsequent samples were below the detection limit of 50 ppt.

Two on-line TOC monitors characterized organic elution. Before this, both monitors had been verifying relative accuracies at the same UPW location. Simultaneous analyses performed over 9 hours provided agreement within 0.2 ppb for average TOC concentrations. One monitor subsequently installed on

the HUPW system for similar 9-hour analyses of the UPW supply and HUPW product showed results agreeing within 0.4 ppb. Actually, the HUPW instrument yielded a lower result in both cases, but the difference is not statistically significant, especially since the hot sample was cooled on-line using a PFA-tubing loop, and there was still a 13°C average difference in sample temperature between instruments. Table III summarizes the TOC results.

Measuring HUPW resistivity is challenging, because most high-quality meters are not as precise or accurate at 80°C as at 25°C. Microprocessor-based resistivity monitors almost always include a temperature-compensation algorithm to correct for deviations from 25°C; results are then displayed in MΩ/cm at 25°C. Although that is acceptable when temperature variations are small, corrections at high temperatures are at best approximate because the equation expressing resistivity in water as a function of temperature is a very complex polynomial.

Resistivity data obtained using a high-quality meter preceded by a sample cooling loop ranged between 18.1 and 18.6 MΩ/cm as displayed, corrected to 25°C. That value exceeds the theoretical maximum of 18.2 MΩ/cm, so the data's accuracy is questionable. Because the total ionic concentration as measured by ultratrace ion chromatography consistently demonstrated no increases in any ionic species, however, it is presumed that HUPW resistivity is unaffected by system elution.

PARTICLES AND COLLOIDAL SILICA IN HUPW

A laser particle counter, calibrated to 0.05 μm, was used to study particle performance. Figure 1 shows the particle counter at the end of the distribution grid; a 28-ft cooling loop of 0.5-in.-OD PFA tubing was installed upstream. A collection filter was placed in the same location for concurrent evaluation of particles by scanning electron microscopy (SEM), direct count, and energy-dispersive x-ray spectroscopy (EDX). Discrete samples were collected periodically for elemental analysis by inductively coupled plasma-mass spectroscopy (ICP-MS) and ultratrace ion chromatography.

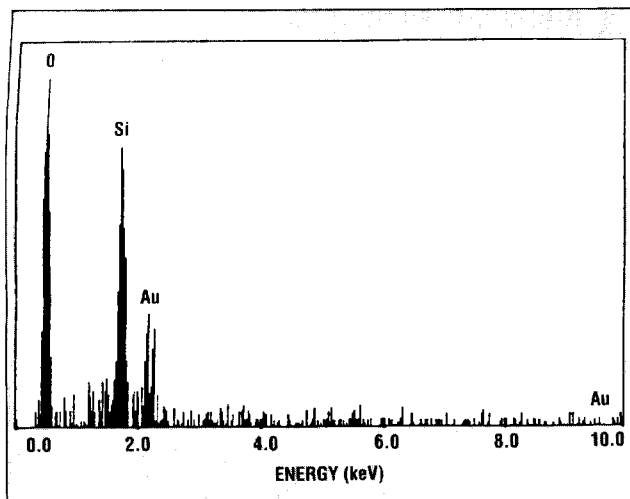


Figure 2: Elemental analysis of typical HUPW system particle.

	Panel Mount Unit (ppb)	Portable Unit (ppb)	Δ (ppb)
Precision check: UPW	3.97	3.84	-0.13
Actual measurement: UPW	4.45	—	—
HUPW	—	4.06	-0.39

Table III: TOC in OR-1 UPW and HUPW by on-line meters (9-hour average).

	UPW (Return)	HUPW ^c
Particles, laser count (per ml, >0.05 μm)	1.0	3.5
Particles SEM ^a (per L, >0.05 μm)	35	111
Total silicon ^b (as Si, ppb)	5.1	6.0
Soluble silica ^b (as SiO ₂ , ppb)	0.63	0.57

^a HUPW taken at 70°C SEM temperature and UPW at 27°C SEM temperature during February–April 1992.

^b Average of three samples taken in March and April 1992.

^c See Figure 1 for sampling location.

Table IV: Particle, total silicon and soluble silica counts in HUPW versus UPW.

Samples were also analyzed for total iron, calcium, and silicon by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). The total-silicon samples were analyzed for soluble (molybdate reactive) silica by a modified heteropoly blue colorimetric procedure. The difference between the total and reactive silicon concentrations was colloidal (molybdate nonreactive) silica. The ICP-AES silica method provides more-complete recoveries than traditional silicon methods, such as graphite-furnace atomic absorption (GFAA) and hydrofluoric acid digestion/molybdate colorimetry. Silicon carbide interference typically causes low GFAA results. The loss of volatile silicon fluoride compounds during sample preparation generally leads to low fluoride digestion results. Both traditional methods further underestimate silicon concentration in UPW because they use external analytical water blanks containing trace levels of silicon.

Tables II and IV summarize results for critical ionic species. No ions or metals were detected in either the UPW feed or the HUPW system product. Total and soluble—and hence colloidal—average silica concentrations also remained unchanged between feed and product. Particle counts, however, were higher in the HUPW than in the UPW feed. Count averages at 0.05 μm over 8 hours were 3.5/ml and 1.0/ml, respectively. This trend was confirmed by SEM direct particle counts of 111/L and 35/L, respectively.

Filter particles were then examined by SEM/EDX. Elemental analysis of HUPW particles revealed silicon

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