Testing liquid-handling system components to ensure purity and reliability

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While awaiting industrywide specifications, a component supplier has adopted a three-pronged test program.

Because the processes used to produce state-of-the-art microcircuits are extremely sensitive to contamination, the cleanliness of the systems that supply process chemicals and other fluids is a critical concern to semiconductor manufacturers. Contaminants from equipment subsystems and components can and do lead to process defects and lower-yielding wafers. For example, particulate contamination can cause open or short circuits, structural defects, altered electrical properties, and unreliable photolithographic reproduction. Metallic contaminants on the wafer surface can diffuse into the substrate during subsequent heat treatments, causing drifts in surface potential, current leakage, structural defects in vapor-grown epitaxial layers, and reduced breakdown voltage of gate oxides.

While the need for ultraclean equipment has long been clear, cleanliness guidelines for the manufacturers who produce such systems have been slow in coming. SEMI has recently released specifications for particulate and metallic contamination in components.[1] Not included, however, are specifications for particle release, while requirements for metallic extractables are based on extraction into water. Section 3.7 states that the relative leach-out performance of polymer components in actual use with other chemicals (e.g., acids and bases) cannot be

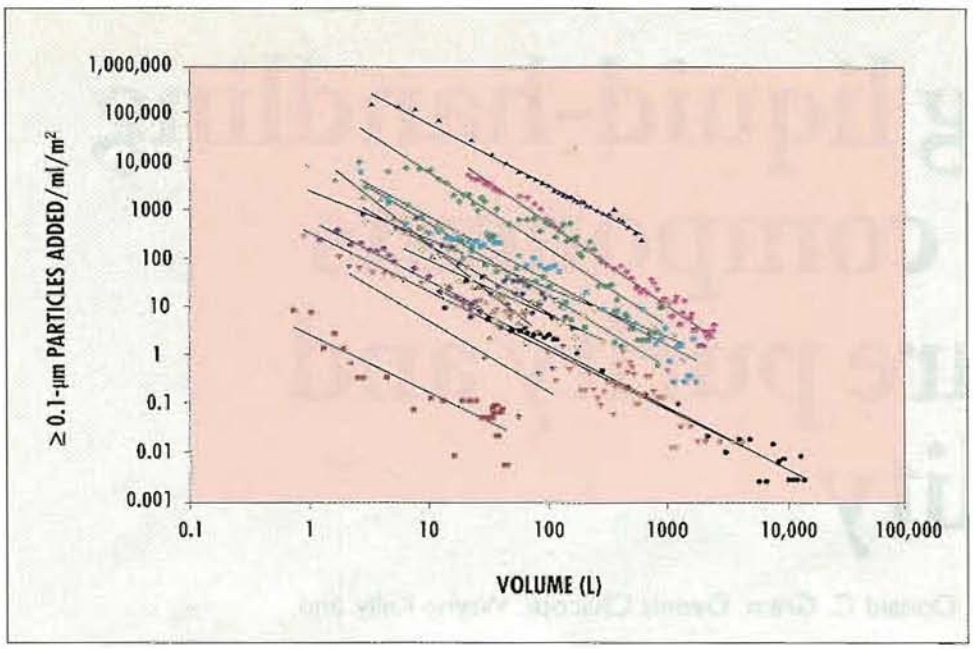

Figure 1: Particle addition by a variety of fluid-handling components under steady-flow conditions.[7]

directly derived by using the ultrapure water (UPW) data. Hence, companies must set their own specifications for their products and components used to transport process chemicals. The chemical-management division of BOC Edwards (Santa Clara, CA), for example, has adopted specifications for particle release and metal extraction from the components used in its chemical delivery systems.

Another major concern for semiconductor manufacturers is component reliability, since equipment failures result in costly fab downtime. Often a component's resistance to damage is dependent on the chemical it contacts. For example, diaphragm valves have two main modes of failure. Internal metal components such as springs usually fail because of metal corrosion caused by exposure to acids that permeate the diaphragm. Hydrochloric acid (HCl) is thought to be the greatest contributor to this type of failure. The other major failure mode is diaphragm fatigue, possibly exacerbated by environmental stress cracking (ESC). In ESC, crack propagation through plastics subjected to stress is accelerated by a weak plastic-chemical interaction. Hydrofluoric acid (HF) is believed to be the chemical most responsible for fatigue failure of fluoropolymer diaphragms.

In response to these industry concerns, component supplier Saint-Gobain Performance Plastics (Garden Grove, CA) has adopted a testing program for its components used in high-purity semiconductor applications. This article describes the testing program that was conducted by CT Associates (Bloomington, MN), the program's goals, the experimental and analytical procedures used, and examples of the test results.

Testing Strategy

A component-testing program offers several benefits. Because it measures and verifies component performance, it increases customer confidence in the company's products. In addition, it enables the component manufacturer to make informed decisions about process changes. For example, new materials or cleaning processes can be evaluated to ensure that they maintain or improve component quality.

The program adopted by Saint-Gobain includes separate protocols for particle release, metal extraction, and reliability. The component cleanliness goals are based on the specifications set by BOC Edwards. These specifications mandate that components must release <2 ≥0.1-µm particles/ml/m² within 1000 L of flushing. Active components—that is, those with moving parts—are exempt from this requirement if they add <0.1 ≥0.1-µm particles/ml to water flowing through the component within 300 L of flushing. However, active components must meet a second specification limiting particle release during operation. For example, valves must release <100 ≥0.1-µm particles per actuation cycle within 500 cycles. The test program measures particle shedding by monitoring particle release into flowing UPW. Active components, such as valves and pumps, are also tested for particle release into UPW during operation.

The BOC Edwards specifications also stipulate that its systems must not contribute more than 20% of the metallic contaminant concentrations allowed by the Semiconductor Industry Association roadmap for process chemicals.[2] System specifications are normalized for surface area so that the contamination limit for individual components can be calculated.[3] The respective component-purity specifications for surface contamination and bulk extraction rate are ≤20 ng/cm² and ≤0.5 ng/cm²/day after 7 days of chemical exposure. The company also requires that component testing be performed in 35–37% HCl, 49% HF, or 70% nitric acid because these chemicals aggressively extract metals present in the polymers.[4]

The test program under discussion uses BOC Edwards' DyconEX dynamic extraction procedure to determine the rate of metal extraction over time.[5] (Theory predicts, and test results have demonstrated, that the rate of metal extraction from components decreases over time.) The extraction tests are performed in HCl because it is one of the most effective acids for extracting metals from fluorinated polymers.[4]

The dynamic extraction procedure has several advantages over techniques in which metal concentrations in an extractant are measured at the end of a static soak period. Unlike those conventional methods, dynamic extraction can measure and predict any change in extraction rate. Its use of a small chemical volume and multiple sample points over time also

makes the method sensitive enough to detect very low levels of extractables. Results can be used to predict a component's contribution to contamination in a process chemical stream.[6] Finally, the technique can be used to distinguish between surface and bulk contamination, which enables engineers to understand how a component will affect a system at start-up and over time.

The program's third protocol addresses component reliability under operating conditions. Active components, such as valves and pumps, are evaluated in both 37% HCl and 49% HF, ensuring that their potential for failure via different mechanisms will be detected. The tests are continued until 70% or more of the test components have failed, which provides an adequate basis for statistical analysis.

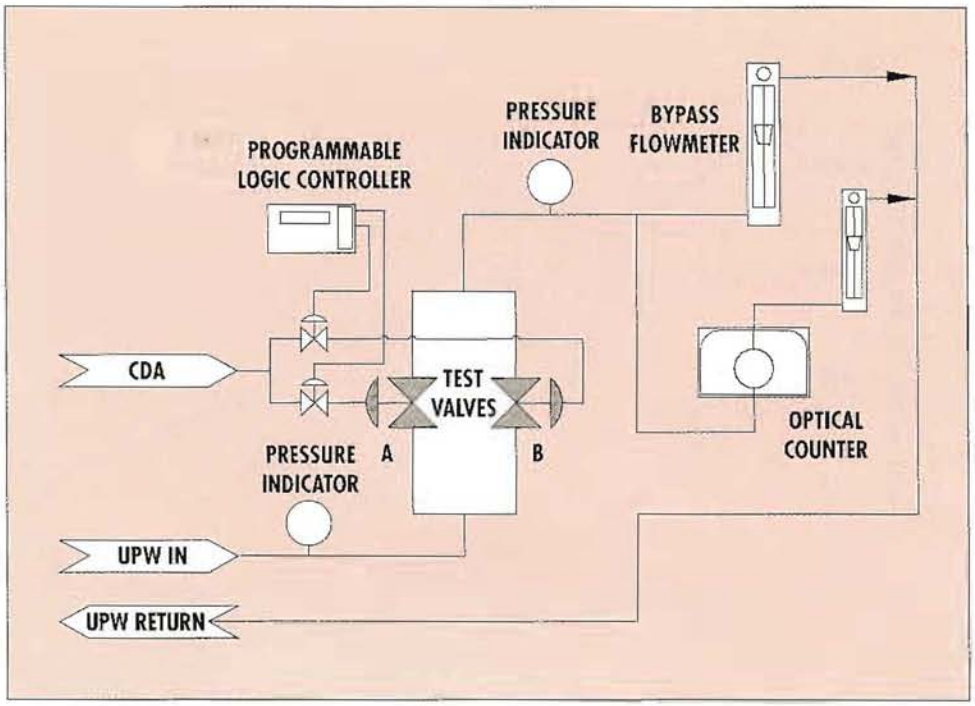

Figure 2: Schematic of a typical test system used to measure passive and active particle shedding from components.

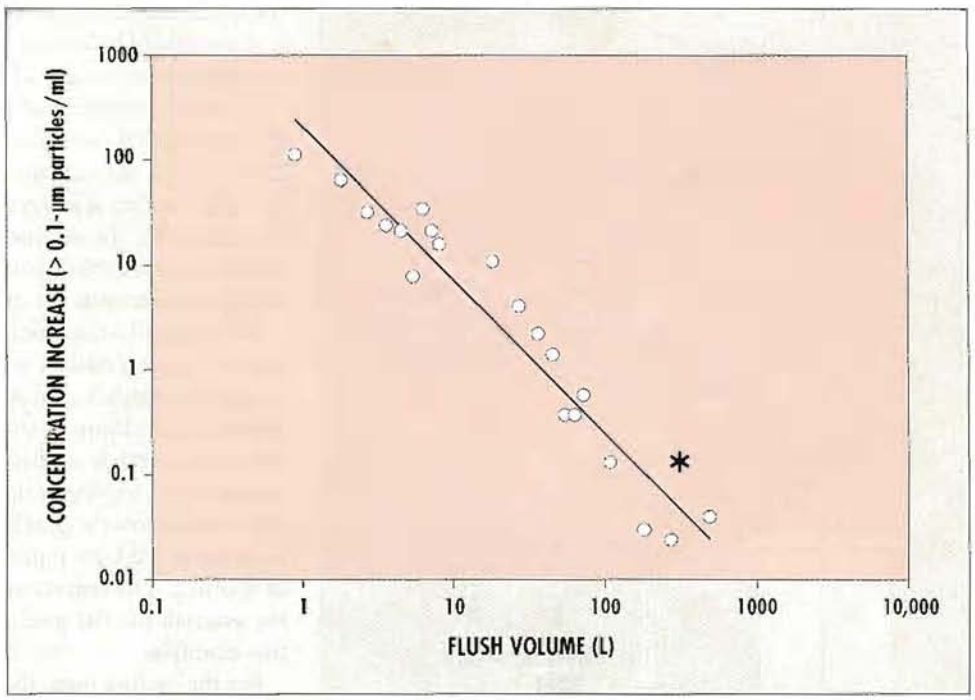

Figure 3: Particle shedding from one type of valve under steady-flow conditions.

Experimental and Analytical Procedures

Particle Release Testing. Component cleanliness is tested by measuring particles shed into UPW, which meets the following specifications: >18 MΩ/cm resistivity, <5 ppb total organic carbon, and <0.1 ≥0.1-µm particles/ml. Both active and passive components, such as tubing and tanks, are evaluated for passive shedding under steady-flow conditions, and active components are also tested under operating conditions, which can generate particle excursions. For example, valve actuation can cause a burst of particles to be released into the fluid stream.

Before the program began, the steady-flow test for particle shedding had been used with many types of components from different manufacturers. That work revealed that in tests run at face velocities ranging in Reynolds number from 100 to 30,000, shedding is independent of velocity and linear on a log-log plot when plotted as the concentration of particles added versus flush volume (see Figure 1).[7]

A typical system used to measure particle shedding is shown schematically in Figure 2. After entering the system, UPW flows through the component(s) to be tested at a flow rate calculated to yield a Reynolds number of approximately 1000. The pressure of the UPW is regulated by a bypass flowmeter and measured both upstream and downstream of the test equipment. A portion of the downstream UPW then flows

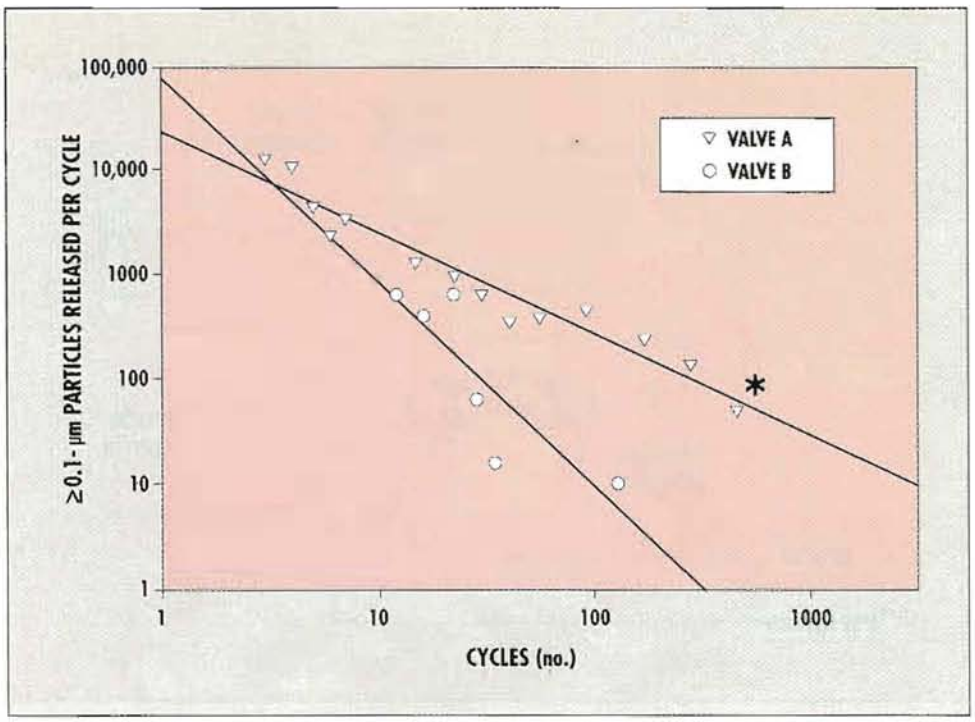

Figure 4: Particles released from two types of valves during cycling.

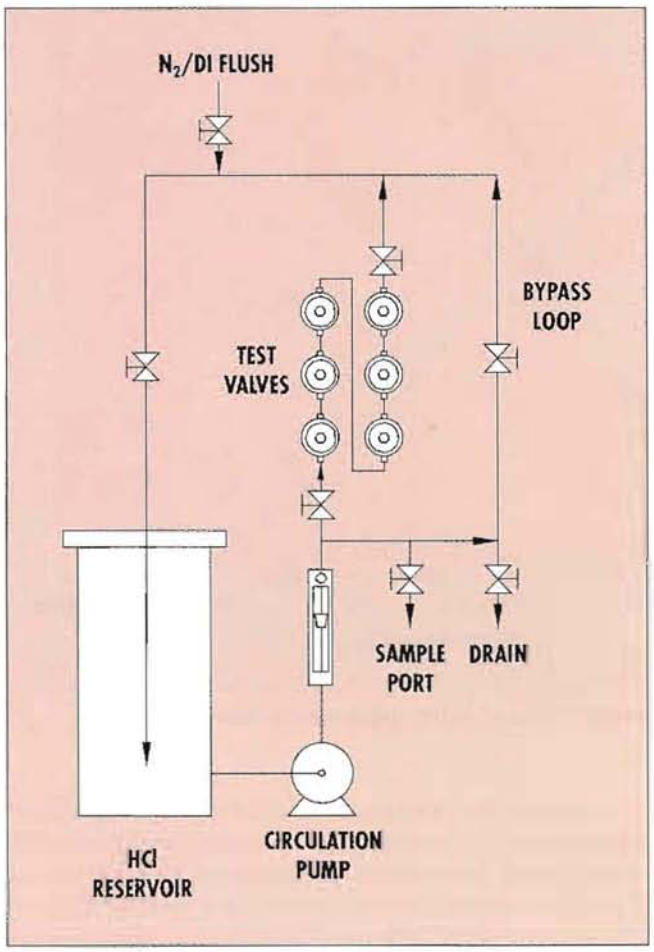

Figure 5: Schematic of a test system used to measure metal extraction from components.

through an optical particle counter (an HSLIS M50 from Particle Measuring Systems of Boulder, CO, in this example), which measures the concentrations of ≥0.05-µm particles in a steady 100-ml/min flow stream. Valves can also be tested under operating conditions in this apparatus. In such tests, the valves are actuated in a repeating sequence with compressed dry air (CDA) using a programmable logic controller.

Prior to each test, the background particle concentrations of the system are measured using a spool piece in place of the test components. For steady-flow, passive particle-shedding tests, a single component is then installed in the system and flushed with UPW as described above, either until background particle concentrations are achieved or until the flush volume reaches 1000 L. Concentration data from two to four parts of each type are averaged to determine component cleanliness.

To measure active shedding, two valves are installed in parallel and cycled two times per minute using an alternating pattern of 16 seconds open and 14 seconds closed. Because one of the valves is always open, this pattern minimizes system hydraulic shocks and ensures a constant pressure and flow rate at the particle counter. The valves are cycled through the open/close cycle a minimum of 2000 times.

For the steady-flow tests, particle shedding from each component is calculated by subtracting the system background from each sample's concentration. The data are then plotted versus flush volume, as shown in the example in Figure 3. In this case, particle addition decreased linearly with flush volume on a log-log scale, as expected. The asterisk in the figure represents the goal for particle release from active components of <0.1 ≥0.1-µm particles/ml added within 300 L of flushing. The regression line for the data must fall below the asterisk for the goal to be achieved, which it does in this example.

For the cycling tests, the number of particles released per valve cycle is plotted versus total valve cycles, as shown in Figure 4. The goal of <100 ≥0.1-µm particles added per cycle within the first 500 cycles is represented by the asterisk. In this example, two types of test valves surpassed this goal: particle release fell below 100 particles per cycle in just 280 and 30 cycles, respectively.

Metallic Extraction Testing. In any test for metallic contaminants from components, it is important to distinguish between surface and bulk contamination. Failure to do so can lead to incorrect conclusions about the cleanliness of a

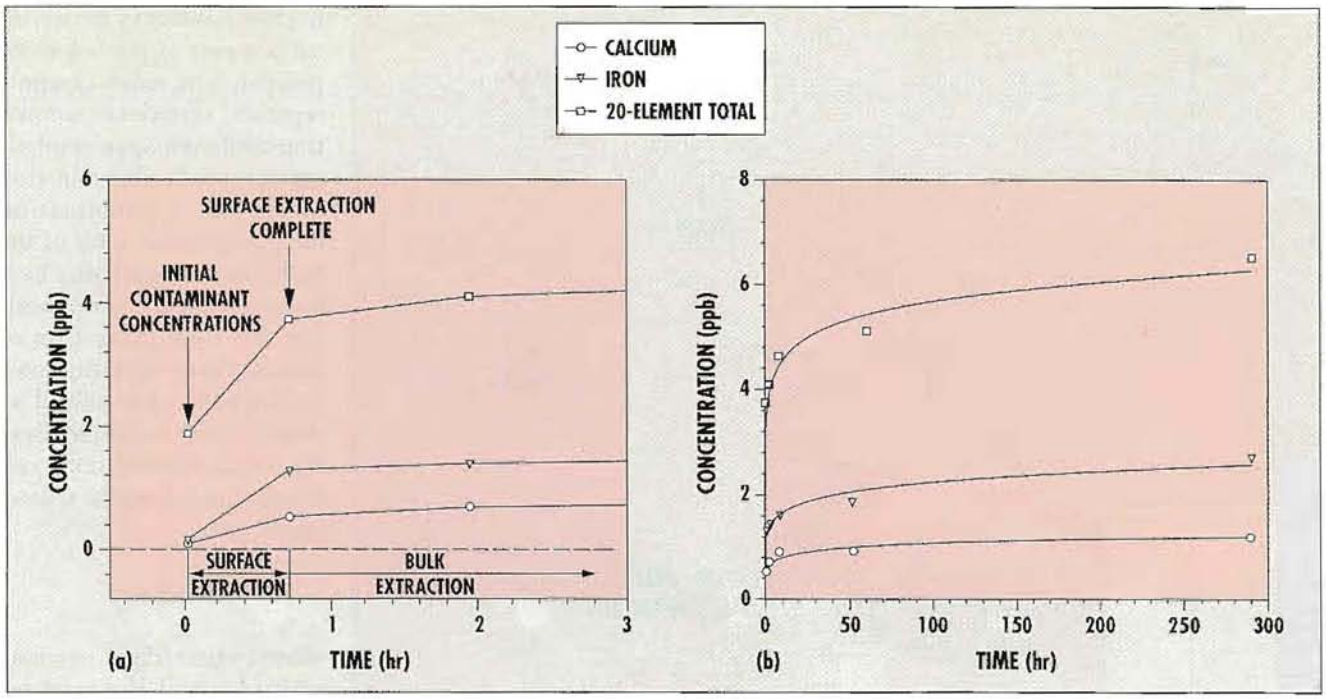

Figure 6: Extraction of selected metals from one type of cleaned valve: (a) data from the first 3 hours of a test, and (b) data from the entire 290-hour test period.

component or the efficacy of a cleaning process. *Surface contamination* in this context has been defined as the mass of contaminants removed from the component within 40 minutes of exposure to the extracting chemical. Any subsequent contaminant removal is considered extraction from the bulk of the component material. Bulk contaminants must be quantitated because they can continue to leach from components during microcircuit production.

The DyconE^X dynamic extraction system used in the test program to determine both surface contamination and bulk extraction is shown in Figure 5. All of the system's wetted components are made of fluoropolymers. Located in a Class 100 cleanroom, the system has been preconditioned in concentrated (35 to 37%) HCl to eliminate its potential contribution to measurable metal extraction. The number of test components plumbed into the apparatus for each test run depends on the components' internal surface area. Enough parts are included to ensure a minimum of 300 cm^2 of wetted surface area. Trace-metal-quality, concentrated HCl is used as the extractant.

To maximize the sensitivity of the test, the amount of

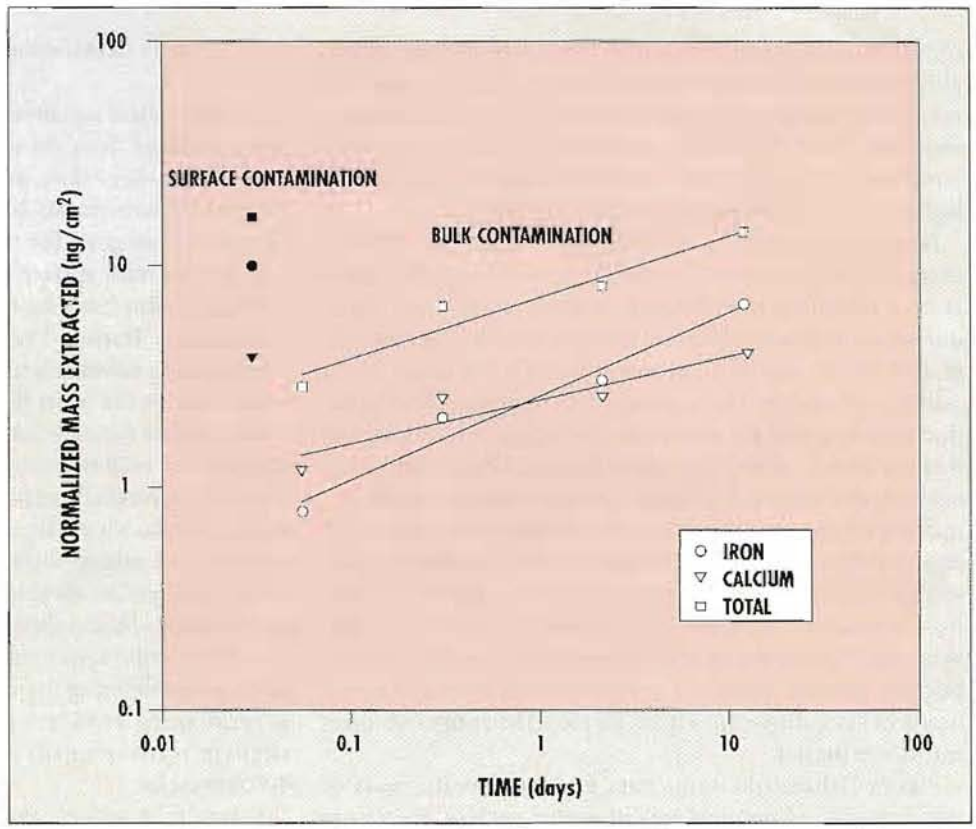

Figure 7: Data from Figure 6 expressed as the masses of metals extracted from the cleaned valves.

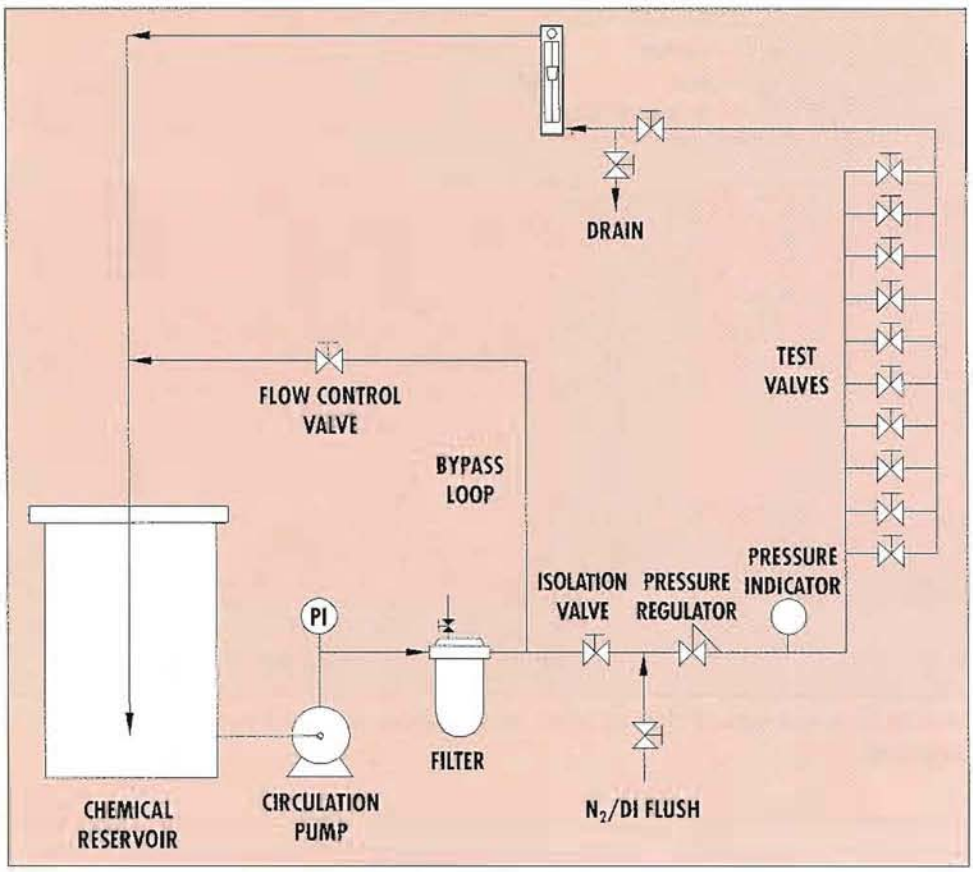

Figure 8: Schematic of the system used for reliability testing.

chemical used is limited to that needed to fill the system plumbing and test components and permit the drawing of 750 ml of chemical samples. Approximately 1 L of HCl is usually required. The chemical flow through the test component(s) is maintained at 300 ml/min throughout most tests, although higher flow rates are used for large components.

Just prior to initiating chemical circulation, the system is charged with the extractant and a background sample is taken from a sampling port located in the bypass loop. Then, during the test run, extractant samples are taken at approximately evenly spaced time intervals on a log scale. These samples are analyzed for a group of 20 trace metals using inductively coupled plasma–mass spectroscopy and graphite furnace atomic absorption spectroscopy. The results of the analyses are converted to cumulative mass extracted and normalized for the wetted surface area of each component. The concentration increases attributable to bulk contamination are calculated by subtracting surface metal concentrations from subsequent samples. The examples of analytical plots shown in Figure 6 depict results from valves cleaned by a proprietary process. Figure 6a shows the data from the first 3 hours of extraction, and Figure 6b plots the entire 290-hour extraction period.

Figure 7 shows the same data expressed as the mass of metals extracted per unit area of wetted surface, the values of which are determined by multiplying the concentration data by the volume of chemical in the test system and dividing that product by the wetted surface area of the test component. The solid symbols represent surface contamination while the open symbols represent bulk contamination of the bulk contamination data. Regression lines of the bulk contamination data have been added to the plot, revealing that bulk extraction of metals is linear with time on a log-log scale, as predicted by theory.[5] The mass of metal extracted at different times can be calculated using the following formula:

$$m = k \times t^n$$

where t = time (days), m = normalized cumulative mass extracted (ng/cm²) at time t, k = a proportionality constant, and n = an exponent. In addition, the rate of extraction can be determined using the following derivative of the equation with respect to time:

$$\text{Rate of extraction} = dm/dt = n \times k \times t^{n-1}$$

Based on these equations, the normalized mass extraction rate calculated from the valve data in the example was 0.49 ng/cm²/day after 7 days, and the total surface contamination for the 20 elements was 16.5 ng/cm². Thus, these valves met the cleanliness goals for metal extraction from active components for both surface contamination (≤20 ng/cm²) and bulk extraction (≤0.5 ng/cm²/day after 7 days).

Reliability Testing. The third part of the test program involves testing valves in both HCl and HF, which are known to contribute to the most likely failure mechanisms. To have sufficient data for statistical analysis, at least 10 valves must be evaluated in each test run. Shown schematically in Figure 8, the test apparatus allows parallel flows of chemical through all 10 test valves, which are opened and closed approximately six times per minute with an actuator pressure equal to the lower of 70 psi or maximum valve rating. The cleanroom-grade chemical is supplied to the valves from a reservoir using a double-diaphragm pump and passes through a 0.45-µm filter before entering the valve manifold. Chemical pressure is maintained at 65 ± 5 psi. To ensure that full chemical strength is maintained, the reservoir is replenished every 400,000 cycles.

Valves that leak or show other visible signs of failure during the test cycling are removed from the apparatus immediately, and valves that exhibit no visible signs of failure

every 200,000 cycles are removed to undergo cracking-pressure and port-to-port integrity tests. Using the test stand shown in Figure 9, each valve's cracking pressure is determined by applying air pressure to the closed valve and measuring the pressure at which it opens.

The test is conducted on both the inlet and outlet of the valve, and the maximum pressure applied is 125 psi. If a valve's measured cracking pressure is >120% of its rated pressure, the valve is returned to the test manifold. If its cracking pressure is ≤120% of its rated pressure, it is also tested for port-to-port integrity. This test measures the rise in pressure downstream of the closed valve when its rated pressure is applied. Valves fail if the pressure rise corresponds to a leak rate in water of ≥0.0001 ml/min or ≥0.14 ml/day. Valves that pass this test are returned to the test manifold. The open/close cycling then continues until at least 70% of the valves have failed.

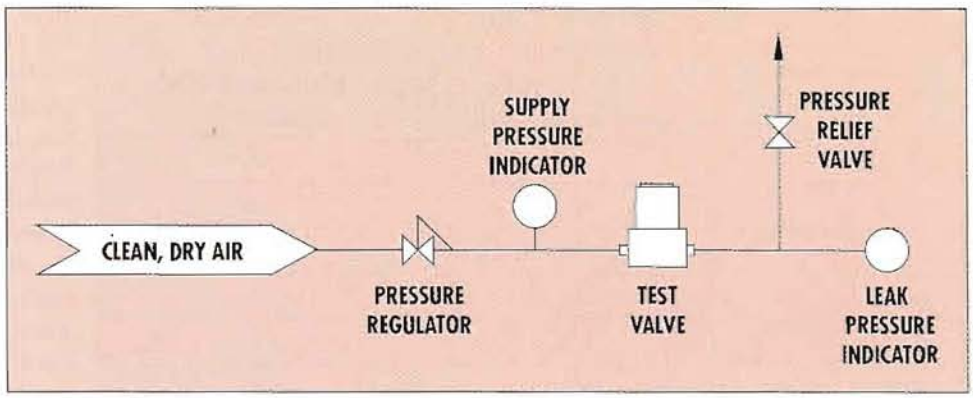

Figure 9: Schematic of the test stand used for measuring cracking pressure and port-to-port leakage.

The resulting data are analyzed for median cycles to failure and the number of cycles at which 5% of the valves have failed. The percent failure data indicate the valve type's probability of failure. For example, Figure 10, which is based on a test using 49% HF, plots the probability that the test valve will fail as a function of millions of open/close cycles. The reliability testing has revealed that failures are lognormally distributed (a statistical concept that is explained in detail elsewhere[8]), and the figure is based on that assumption. The x-axis is a probability scale, the y-axis is a log scale, and the solid line represents a fit of the data to a lognormal distribution. The dashed lines show the number of cycles that had been completed when 5 and 50% of the valves failed. Specifically, these lines indicate that there is a 5% probability of a valve of this type failing in HF before it completes 830,000 cycles, and a 50% probability of its failure before 2.3 million cycles.

Test Result Summaries

The three test protocols have been used to evaluate a variety of components including piping, unions, and valves. The results of five particle-shedding tests are presented in Table I, along with the cleanliness goal for active components. The ½-in. tubing, the only passive component included in this testing, has a different goal, as noted in the table. Comparison of the test data with the goals shows that the tubing met its single goal, and valve A met the goals for both steady-flow and cycling tests. The other three valves each met one of the two relevant goals.

Component reliability is a significant concern for semiconductor fabs, since equipment failures result in costly fab downtime.

Table II summarizes the results of several metal extraction

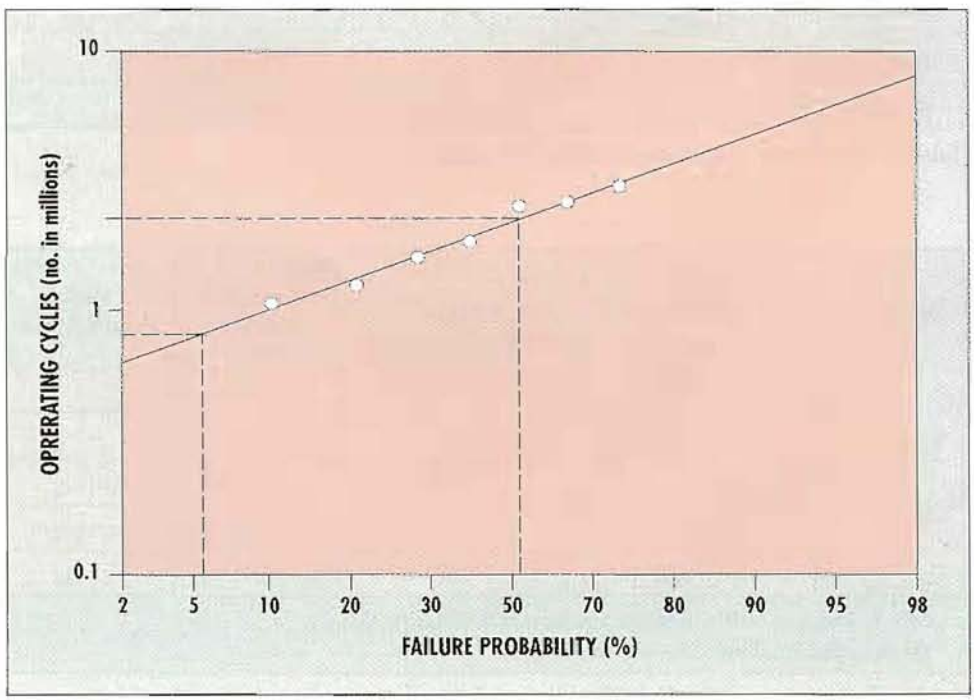

Figure 10: Results of an analysis of one valve type's reliability in 49% HF.

Component	Steady-Flow Tests (volume in liters to <0.1 ≥0.1-µm particles/ml added)	Cycling Tests (cycles to <100 ≥0.1-µm particles released per cycle)
½-in. tubing	60	NA
Valve A	250	280
Valve B	940	<100
Valve C	190	>2000
Valve D	850	310
Cleanliness goal[a]	≤300	≤500

[a] The goal for passive components (tubing, piping, tanks, etc.) is <2 ≥0.1-µm particles/ml/m² added within 1000 L.

Table I: Summary of particle release test data.

Component	Mass Extracted (ng/cm²)			Extraction Rate at 7 Days (ng/cm²/day)
	Surface	Bulk	Total	
½-in. tubing	0.29	0.48	0.77	0.02
¼-in. union	7.1	2.0	9.1	0.04
Valve A	3.6	3.3	6.9	0.11
Valve B	180.0	36.0	216.0	0.61
Valve C	14.0	7.8	22.0	0.28
Valve D	19.0	26.0	45.0	0.87
Check valves	12.0	9.5	22	0.35
Cleanliness goal	≤2.0	—	—	≤0.50
SEMI specification[a]	—	—	≤12.0	—

[a] For parts in UPW at 85° ± 5°C.

Table II: Summary of metal extraction test data.

Valve Type	Test Fluid	Statistics of Failure		Main Failure Mode
		Median (millions of cycles)	Geometric Standard Deviation	
A	37% HC	>1.0[a]	—	—
B	37% HCl	7.0[b]	—	—
C	37% HCl	2.90	1.14	Spring
C	49% HF	2.28	1.85	Diaphragm
D	37% HCl	1.40	1.45	Spring

[a] Both ¼- and ½-in. valves tested to 1 million cycles with no failures.
[b] Valves cycled 7 million times with no failures.

Table III: Summary of reliability testing data, expressed as statistics of failure and failure mode.

tests. The SEMI specification given in the table refers to total metal extraction under other, presumably less stringent, conditions and is included for information only. All of the components tested met the cleanliness goals except valves B and D. Because the type B valves failed to meet both the metal extraction and the steady-flow particle release test goals, Saint-Gobain began investigating a new cleaning process that would reduce contamination levels. In recent testing, valves cleaned with this process met all cleanliness goals, and the process is now being implemented.

Reliability test results for the four types of valves tested are summarized in Tables III and IV. As the statistical data in Table III show, type A valves showed no failures after 1 million cycles, and valves D and C had median cycles to failure of 1.4 million and approximately 2.5 million cycles, respectively. Although the median cycles to failure for the type C valves tested in HF and HCl were similar, the failure modes were different and failure times were more variable in HF. Tests of the type B valves are continuing, with no failures after 7 million cycles. Additional testing of type A valves is planned to determine failure statistics.

Table IV presents reliability data in terms of the number of cycles run before 5 and 50% of the test valves failed. This method of expressing the test results is useful in deciding how frequently the various types of valves should be replaced. All of the valves tested reached the 5% failure rate after more than 0.7 million cycles, while valve B had a 5% failure rate of >6 million cycles. (It should be noted that some of the numbers in this table were estimated.)

Conclusion

To protect against wafer defects and low device yields, the

components used in high-purity chemical distribution systems must release very low levels of contaminants and be highly reliable. Rigorous component testing for particle release, metal extraction, and reliability can help ensure that these goals are met. A test program established by Saint-Gobain Performance Plastics includes particle-shedding tests that measure particle release in ultrapure water under steady-flow and open/close cycling conditions,

The use of the three test protocols has led to manufacturing improvements.

Valve Type	Test Fluid	Failure Rate (millions of cycles to failure)	
		5% Failure	50% Failure
A[a]	37% HCl	>0.86	>1.7
B[a]	37% HCl	>6	>11
C[b]	37% HCl	2.3	2.9
C	49% HF	0.82	2.3
D	37% HCl	0.76	1.4

[a] Failure rate was estimated because no failures were observed during testing. Estimates were made assuming that failures are lognormally distributed with a geometric standard deviation of 1.5 and that one of 10 valves failed one cycle after the last test point. The value of 1.5 assumed for the geometric standard deviation was based on historical data.

[b] Failure rate was estimated assuming that a failure at 800,000 cycles was not representative of the general valve population.

Table IV: Summary of reliability testing data, expressed as valve cycles to a 5 and 50% probability of failure.

a dynamic test that measures the rate of metal extraction in concentrated acid over time, and reliability tests in which components are exposed to 37% HCl or 49% HF under operating conditions. In order to provide an adequate basis for statistical analysis, the reliability tests are continued until 70% or more of the components have failed. The use of these tests has led to both product and manufacturing improvements, including the development of a cleaning procedure that reduces contamination in components.

Acknowledgment

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