Evaluation of particle shedding and trace metal extraction from high purity pumps

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ABSTRACT

The production of semiconductor devices continues to be extremely sensitive to particulate and metallic contamination. As feature sizes continue to decrease, the need for purity will continue to increase. Various types of pumps are used in bulk chemical delivery systems, recirculating etch baths, and other high purity process applications. Many of these pumps shed significant quantities of particles that may reduce product yield or impact the performance or lifetime of filters used in the process loop. Furthermore, metallic contamination in process chemicals can cause a variety of yield-related issues. This paper evaluates the levels of trace metal extraction and particle shedding under different operating conditions using two high purity pump types from three manufacturers.

Introduction

The pumps being used for this study (positive displacement pumps from manufacturers A and B; magnetically levitated centrifugal pumps from manufacturer C) were evaluated for particle shedding in ultra pure water and trace metal extraction in 35% hydrochloric acid. Three pumps sizes (low, intermediate, and high capacity) from each manufacturer were evaluated for particle shedding over a range of operating conditions (i.e., varying flow rates and pressures). Pumps with similar capacity were tested under common operating conditions that were chosen for each group of pumps such that all pumps in the group could achieve each test condition. A subset of the pumps was also evaluated for trace metal extraction using a dynamic extraction method. Both surface and bulk contamination were determined. In addition, the type and rate of trace metal extraction over time were determined for each pump.

Experimental procedure

Pumps tested

Table 1 shows the breakdown of pump type evaluated in this study. Because the pumps from manufacturers A and B are positive displacement pumps, the operating curves of these pumps are significantly different from those of the magnetically levitated centrifugal pumps. While the flow rate of a centrifugal pump varies with system pressure, a positive displacement pump has relatively constant flow rate regardless of system pressure. In general, the positive displacement pumps deliver fluid at lower flow rates and higher pressures, while the magnetically levitated

centrifugal pumps deliver higher flow rates at lower pressures.

Particle shedding

A schematic of the particle shedding test system is presented in Figure 1. The pump being evaluated drew water from a filtered circulating loop. A pulse

dampener – used to reduce pulsations from the diaphragm pumps – was employed during each pump test (even though no pulse dampener was necessary for the magnetically levitated centrifugal pumps) and was located immediately downstream of the test pump. The desired operating conditions were achieved by
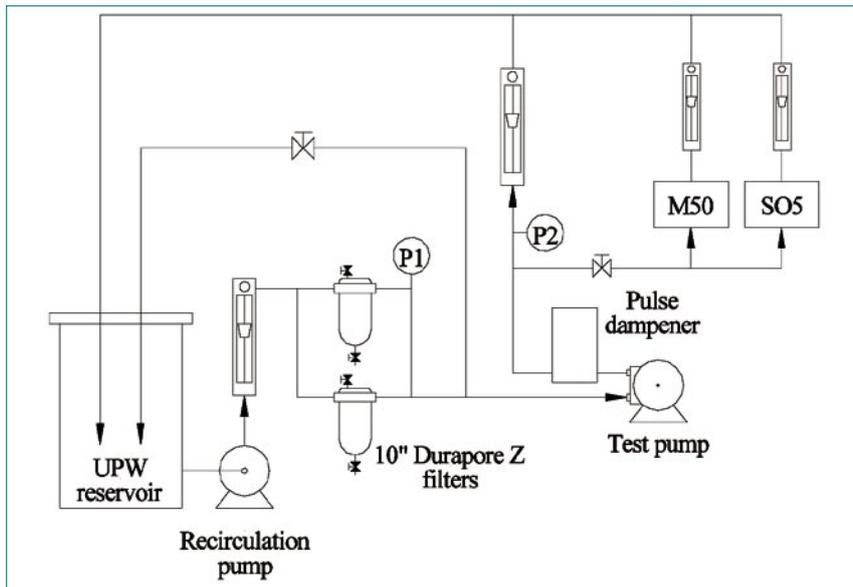

Figure 1. Particle shedding test system schematic.

TABLE 1: COMPARISON OF THE PUMPS EVALUATED			
Manufacturer	**Pump Type**	**Pump ID**	**Capacity**
A	diaphragm	A1	low
		A2	intermediate
		A3	high
B	diaphragm	B1	low
		B2	intermediate
		B3	high
C	magnetically levitated centrifugal	C1	low
		C2	intermediate
		C3	high

either adjusting the pump rotational speed (magnetically levitated centrifugal pumps) or air pressure (diaphragm pumps) to the pump and the pump outlet pressure with a control valve located downstream of the test pump.

A Particle Measuring Systems HSLIS-M50 particle monitor and a Particle Measuring Systems LiQuilaz-S05 liquid particle counter were used to measure particle sizes ranging from 0.05μm to larger than 10μm.

Each pump was run under typical operating conditions for a minimum of three days prior to the start of this test program to ensure that any particles being shed were not simply due to the fact that the pump was new. Following this break-in period, quasi steady-state particle shedding from each pump was monitored over a range of operating conditions. A minimum of two hours of data was averaged once particle concentrations had stabilized following each change in operating conditions.

Trace metal extraction

The pumps were tested for trace metal extraction using the dynamic extraction test apparatus shown in Figure 2.
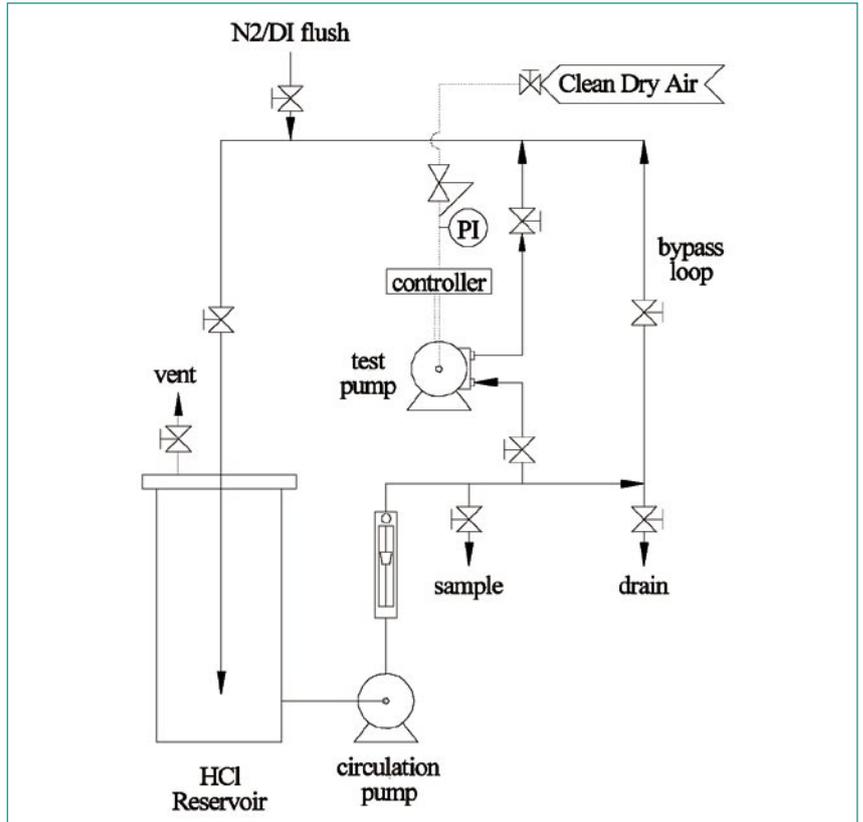

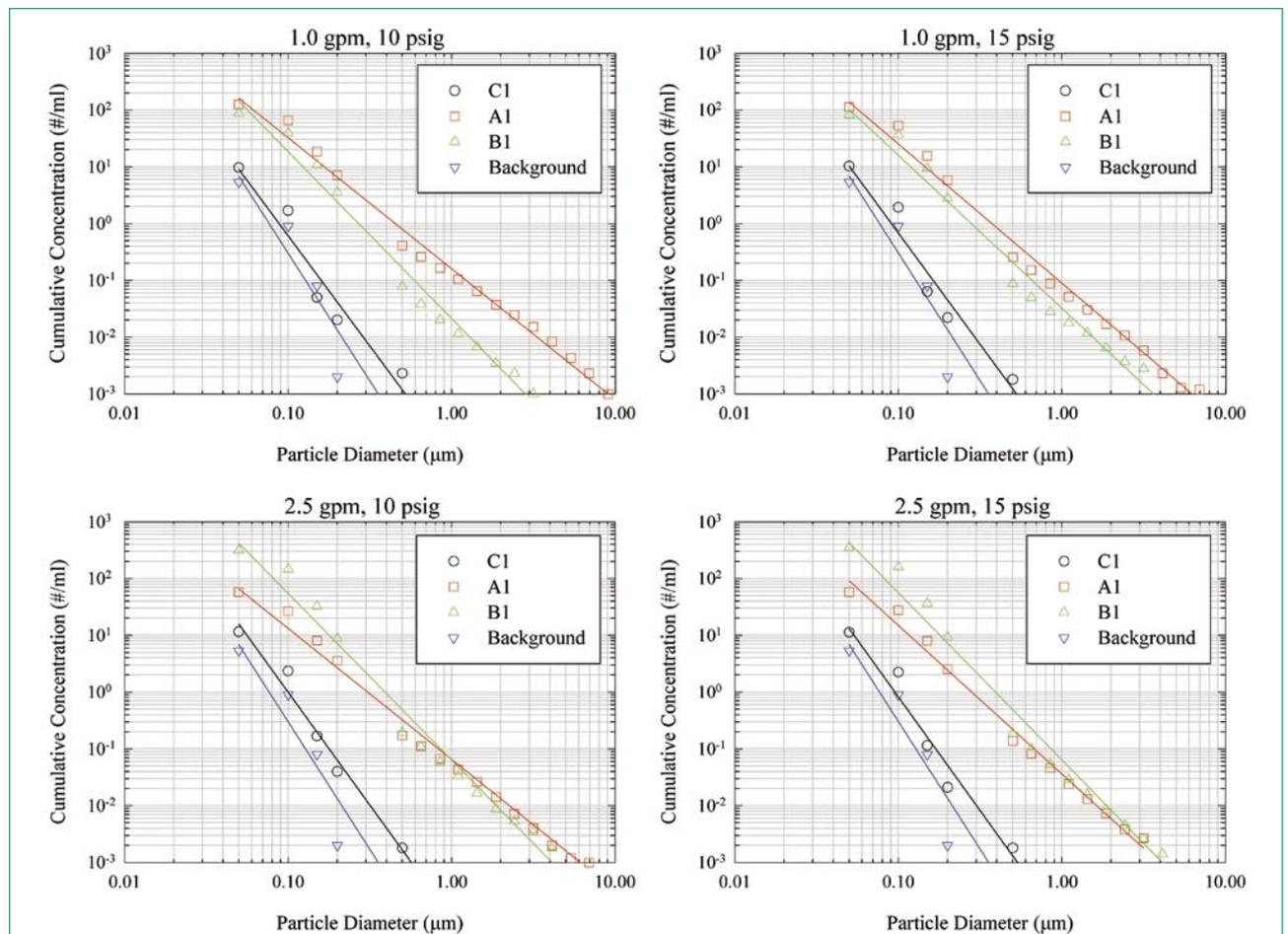**Figure 2. Trace metal extraction system schematic.**

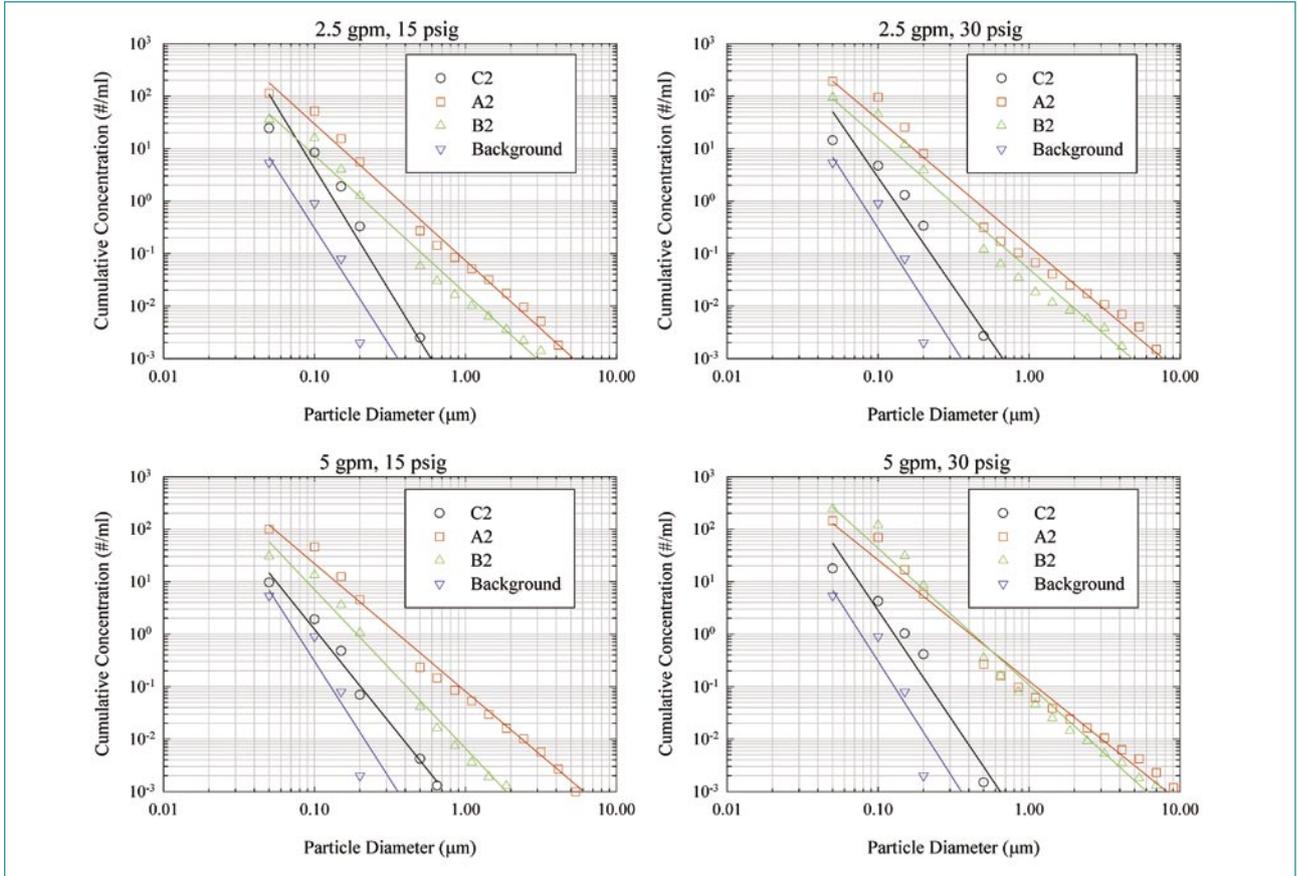**Figure 3. PSDs measured downstream of the low capacity pumps at selected operating conditions.**

Figure 4. PSDs measured downstream of the intermediate capacity pumps at selected operating conditions.
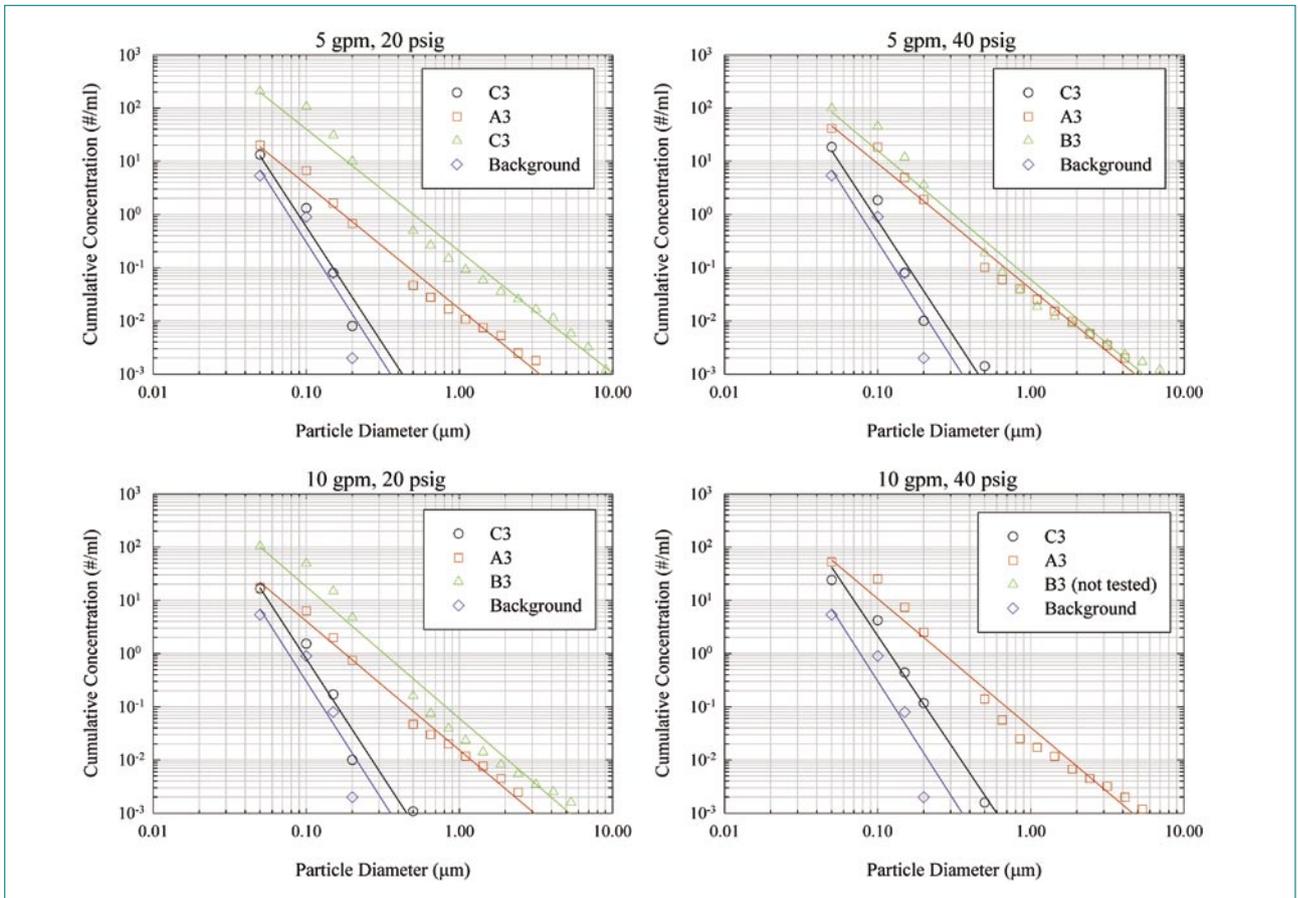

Figure 5. PSDs measured downstream of the high capacity pumps at selected operating conditions.

This method allows measurement of both surface contamination, which is removed within a few minutes of exposure to chemical, and contamination from the bulk material. The total contamination extracted from the component is the sum of the surface and bulk contamination. Since the contaminants are measured over time, the rate of extraction may also be determined [1]. High purity 35% HCl was used as the extractant during each test. The test pumps were operated during the test to ensure that the acid was well mixed in each system. Prior to each test, a background sample was taken; subsequent samples were taken at time intervals evenly spaced on a logarithmic scale from a sample port located in the circulation loop. The chemical samples were analyzed for 37 metallic elements, while the results of these analyses were converted to cumulative mass extracted.

Figure 6. Surface, bulk, and total contamination measured during five pump tests.

Results and discussion

Particle shedding

An example of the quasi steady–state particle concentrations measured downstream of each pump is presented for selected operating conditions in Figures 3, 4, and 5. System background particle concentrations are also included. Each graph presents the cumulative particle size distribution (PSD) at common operating conditions (flow rate and pump outlet pressure) denoted above each graph. The results from both particle counters were combined and presented in each graph.

The PSDs presented were linear when plotted on a log-log scale, which is typical of particle shedding from components in liquids [2]. A linear regression through each set of data is also included in each graph. The PSDs tend to flatten out at the smallest size
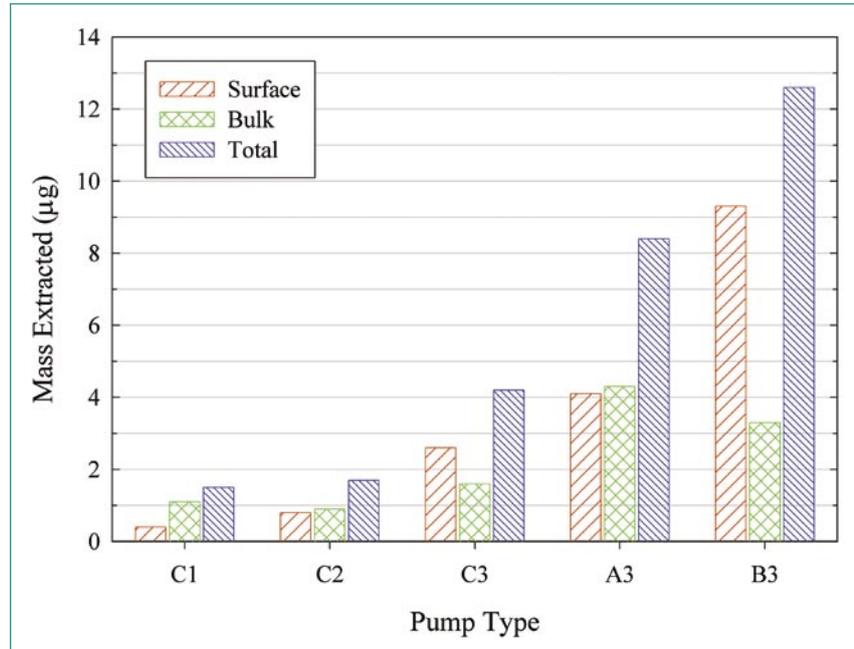
channel, ≥ 0.05μm. The detection limit of the M50 particle counter is believed to be ~0.07-0.08μm, rather than 0.05μm. This is not due to the fact that the M50 was not calibrated, but because how the manufacturer defines the sensitivity at 0.05μm.

The magnetically levitated centrifugal (C1-C3) pumps consistently shed the fewest particles of the pumps tested, regardless of operating conditions. Furthermore, the particle concentrations measured downstream of the centrifugal pumps were often close to (within a factor of 5) the system background concentrations.

Typically, the slopes of the PSDs measured downstream of the centrifugal pumps were steeper than the other pumps and were similar to the slope of the background PSD. The steeper slopes measured during the centrifugal pump tests resulted in much larger differences in particle concentrations between the

centrifugal pumps and the other pumps at larger particle sizes and smaller differences at smaller particle sizes.

Table 2 presents a comparison of the particle shedding results presented in Figures 3-5. The ratios of the particle concentrations downstream of positive displacement pumps to the particle concentrations downstream of comparably sized centrifugal pumps are presented for two particle size channels, ≥ 0.1μm and ≥ 0.5μm. The geometric mean and geometric standard deviation (GSD) are presented rather than arithmetic mean since the concentration ratios appear to be lognormally distributed rather than normally distributed. Pumps from manufacturers A and B typically shed approximately 5-30 times as many particles ≥ 0.1μm as a comparable centrifugal pump, and 40-275 times as many particles ≥ 0.5μm.

Operating conditions had little overall effect on particle concentrations

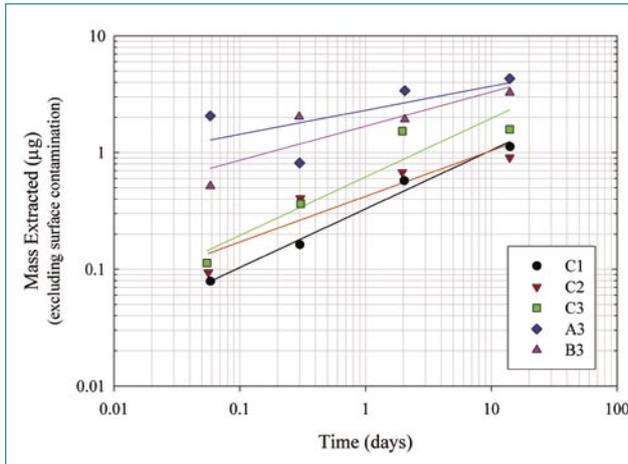| TABLE 2: COMPARISON OF THE PUMP MEAN PARTICLE CONCENTRATION RATIOS					
		Geometric Mean and GSD of the Concentration Ratios of Pumps from Manufacturers A and B to the Comparable Sized Pump from Manufacturer C			
		≥ 0.1 μm		≥ 0.5 μm	
Pump Manufacturer	Pump ID	Geometric Mean	GSD	Geometric Mean	GSD
A	A1	17	1.6	115	1.4
	A2	18	1.8	133	1.5
	A3	6	1.4	72	1.7
B	B1	23	1.9	48	1.7
	B2	7	3.1	40	3.5
	B3	32	2.0	277	2.4

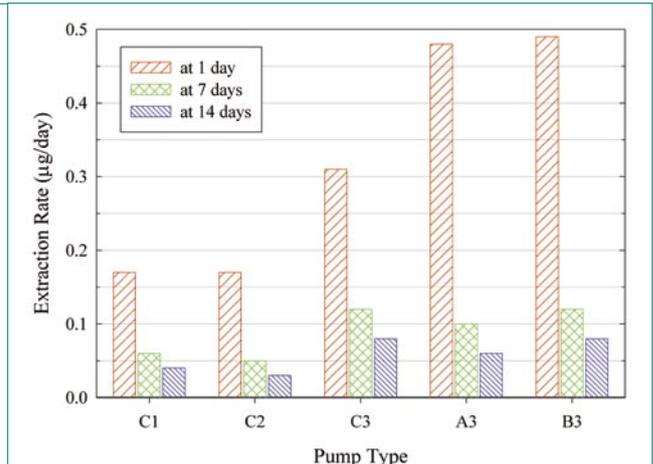Figure 7. Mass extracted from the bulk material of five pumps over time.

Figure 8. Mass extraction rates from five pumps at selected times.

downstream of the pumps [3]. Pump outlet pressure had a greater effect on particle concentrations than flow rate, with particle concentrations typically remaining the same or increasing with increasing outlet pressure. Meanwhile, flow rate had a minimal effect on particle concentrations at most operating conditions tested; as a result, the number of particles shed typically increased linearly with increasing flow rate. Under some operating conditions, particle concentrations increased with increasing flow rate. Differences among the pumps were similar under all operating conditions.

Trace metal extraction

Figure 6 compares the surface, bulk, and total metallic contamination extracted from each pump tested. All of the pumps showed relatively slow extraction with the total mass extracted varying from 1 to 13μg. The three pumps supplied by manufacturer C showed the lowest level of surface contamination; however, surface contamination is less important than extraction rate as it can be removed by pre–extracting a component prior to its use.

Figure 7 shows the mass extracted from the bulk material of each pump over time for all measured elements. Since theory predicts that the extraction data can be fit by a power law equation, the data should follow a straight line when plotted on a log–log scale [1]. The rate of trace metal extraction can be determined from the slope of these data. The mass extraction rates for each pump at selected times are presented in Figure 8. The rates of extraction were lowest for the C1 and C2 centrifugal pumps. Extraction rates from A3, B3, and C3 pumps were similar.

Summary

Particle shedding from nine high purity pumps from three different manufacturers was measured under typical operating conditions. The three magnetically levitated centrifugal pumps shed the fewest particles of the nine pumps evaluated, regardless of operating conditions. The particle concentrations measured downstream of the centrifugal pumps were often very close to system background concentrations. The slopes of the PSDs downstream of the centrifugal pumps were steeper than the other pumps and similar to the slope of the system background. As a result, the difference in particle concentrations increased as particle size increased. Operating conditions had little overall effect on particle concentrations downstream of the pumps. Particle shedding from the two types of positive displacement pumps were often similar.

The metallic extraction from all of the pumps was considered to be relatively low. The magnetically levitated centrifugal pumps had lower surface, bulk, and total metallic contamination than the other pumps (although two of these pumps were considerably smaller than the other pumps). The rates of extraction were lowest for the C1 and C2 magnetically levitated centrifugal pumps, while extraction rates from A3, B3, and C3 pumps were similar.

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ABOUT THE AUTHORS

Mark Litchy works as a Research Engineer for CT Associates, Inc., a contract research, development and testing services company for contamination control, particle measurement and control, filtration, permeation and chemical engineering. He has more than 12 years of experience in particle measurement and control in high purity liquid chemicals and gases and CMP slurry characterization. He is the author or co-author of more than 30 publications and presentations. He has an M.S. degree in mechanical engineering from the University of Minnesota (Minneapolis, MN) and a B.S. degree in physics from St. John's University (Collegeville, MN).

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