Effect of pump type on the health of various CMP slurries

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ABSTRACT

Delivery systems used to supply slurry to CMP planarization tools can damage slurry. In this experiment, four slurries were circulated in a simulated slurry delivery loop at a fixed flow rate and pressure using a variety of pumps (bellows, diaphragm, and magnetically levitated centrifugal) to determine the effect of circulation on the slurry health. During each test, a number of slurry health parameters were monitored including the size distribution of the particles in the slurry. Most slurry health parameters were unaffected during the tests. However, significant changes in the large particle tail (particles ≥ 0.5μm) of the slurry particle size distributions (PSD) were observed. Both the pump and slurry type played important roles in the magnitude of the change. In some slurries, large increases in the large particle concentrations were observed during circulation with diaphragm and bellows pumps, while in other slurries increases were not observed. With the magnetically levitated centrifugal pumps, minimal changes were observed, regardless of the type of slurry tested.

Introduction

Delivery systems are often used to supply slurry during wafer planarization. These systems pressurize and circulate the slurry to deliver it to the tools and keep the particles in suspension. Pressurization and circulation are accomplished by various means including pumping and pressure-vacuum technology. Typically, the slurry passes through the equipment providing the motive force approximately 100 times before it is used to polish wafers, i.e. the slurry is 'turned-over' approximately 100 times [1]. Some CMP slurries are susceptible to damage caused by mechanical handling. For example, particle agglomerates may form that can limit the life of filters or reduce yield by causing wafer defects.

Diaphragm and bellows pumps have been commonly used for bulk CMP slurry delivery. These pumps are accepted as low shear devices due to their relatively low speeds of operation. Centrifugal pumps have not been widely used for delivery of agglomeration-sensitive slurries since they are perceived as high shear devices, due to their relatively high speeds of operation.

Previous studies have shown significant increases in large particle concentrations when certain types of pumps (diaphragm and bellows) were used to circulate slurry [2–5]. However, these tests were performed using a slurry known to be sensitive to particle agglomeration. In practice, there are many CMP planarization applications incorporating many different types of slurries.

While it is not feasible to evaluate all available slurries with all types of pumps, this set of experiments was undertaken to determine the effect of pump type on the health of a small number, but wide variety, of slurry types. The slurries selected for this experiment include a fumed silica slurry, a colloidal silica slurry, an alumina slurry, and a ceria slurry. The pumps in this experiment included a diaphragm pump, a bellows pump, and two magnetically levitated centrifugal pumps. The sizes of the pumps were chosen such that each was capable of delivering slurry at a flow rate of 30 lpm at an outlet pressure of 30 psig. Manufacturer-recommended pulse dampeners were installed downstream of both the bellows and diaphragm pumps to minimize pulsation.

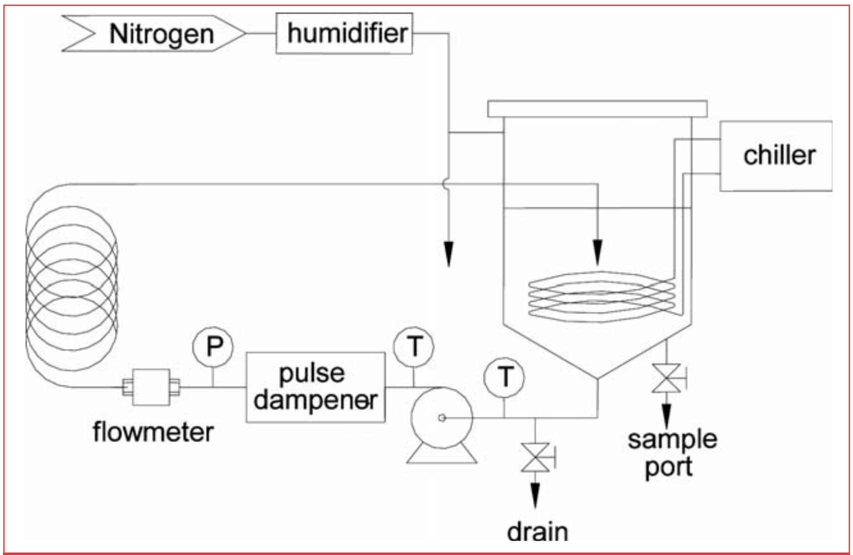

Figure 1. Test system schematic.

Experimental procedure

A schematic of the test system used in this evaluation is shown in Figure 1. Each pump was used to circulate 12 liters of slurry at a flow rate of approximately 26-30 lpm (7-8 gpm) and outlet pressure of approximately 30 psig (2.1 bars). Settling of the slurry in the tank was minimized by drawing from the bottom of a conical bottom tank and by turning the volume of slurry in the tank over in less than 30 seconds. The return line to the slurry tank was submerged below the liquid level of the slurry to avoid entraining gas into the slurry. The return line was also positioned to minimize the formation of a large vortex in the tank, which can entrain gas into the slurry. No valves were used to generate backpressure at the outlet of the pump. Instead, a long length of ½" PFA tubing was used to gradually reduce the pressure at the pump outlet to ambient pressure at the end of the return line to the tank.

The air pressure supplied to the diaphragm and bellows pumps was adjusted to achieve the desired flow rate and outlet pressure. Meanwhile, the speed of the small and large centrifugal pumps was varied to achieve the desired flow rate and pressure. In each test, the slurry was circulated until approximately 1,000 tank turnovers were achieved. The test system was constructed of PFA, except for the conical bottom tank that was constructed of polyethylene. The

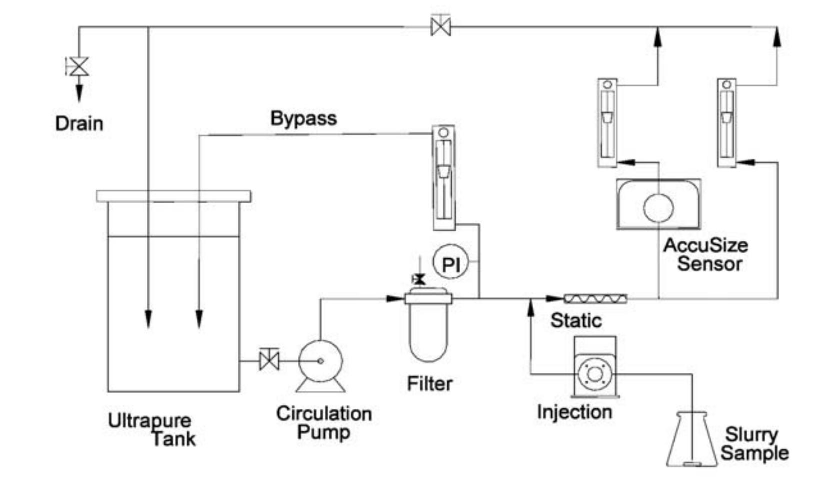

Figure 2. Slurry dilution system schematic.

slurry used in each test was taken from the same lot of slurry.

The tank holding the slurry was blanketed with nitrogen to prevent absorption of carbon dioxide from the air, which can change the pH of the slurry. The nitrogen was humidified to prevent dehydration of the slurry. The relative humidity in the tank was > 90% throughout the test. Shifts in the pH and dehydration can both result in particle agglomeration in the slurry. A chiller and stainless steel coil were used to maintain the slurry at 22 ± 2°C during the test.

Samples were drawn from the system at selected times for analysis. The PSD was measured using two techniques. The size of the 'working' particles (typically ~ 0.05-0.5µm) was measured using Particle Sizing Systems' NICOMP 380ZLS (Santa Barbara, CA) that determines particle size by dynamic light scattering. The size distribution of the large particle tail was measured using a Particle Sizing Systems AccuSizer 780 sensor.

The AccuSizer 780 sensor uses a combination of light scattering and light extinction to measure the size distribution of particles ≥ 0.56µm. The size measurements were performed by injecting the slurry sample into a flowing stream of filtered deionized water using the test system shown in Figure 2. The dilution ratio was varied by adjusting the slurry injection rate. The slurry types required different dilution ratios, which varied from approximately 800:1 to 32,000:1. Prior to starting the pump tests, each slurry was thoroughly analyzed to determine the proper dilution ratio for accurate measurement of the large particle tail of the slurry PSD. Each slurry contains a very high concentration of the 'working' particles, which are responsible for the mechanical portion of the polishing. Care must be taken to ensure that each slurry is diluted sufficiently such that scattering from particles < 0.56µm does not interfere with the particle size analysis.

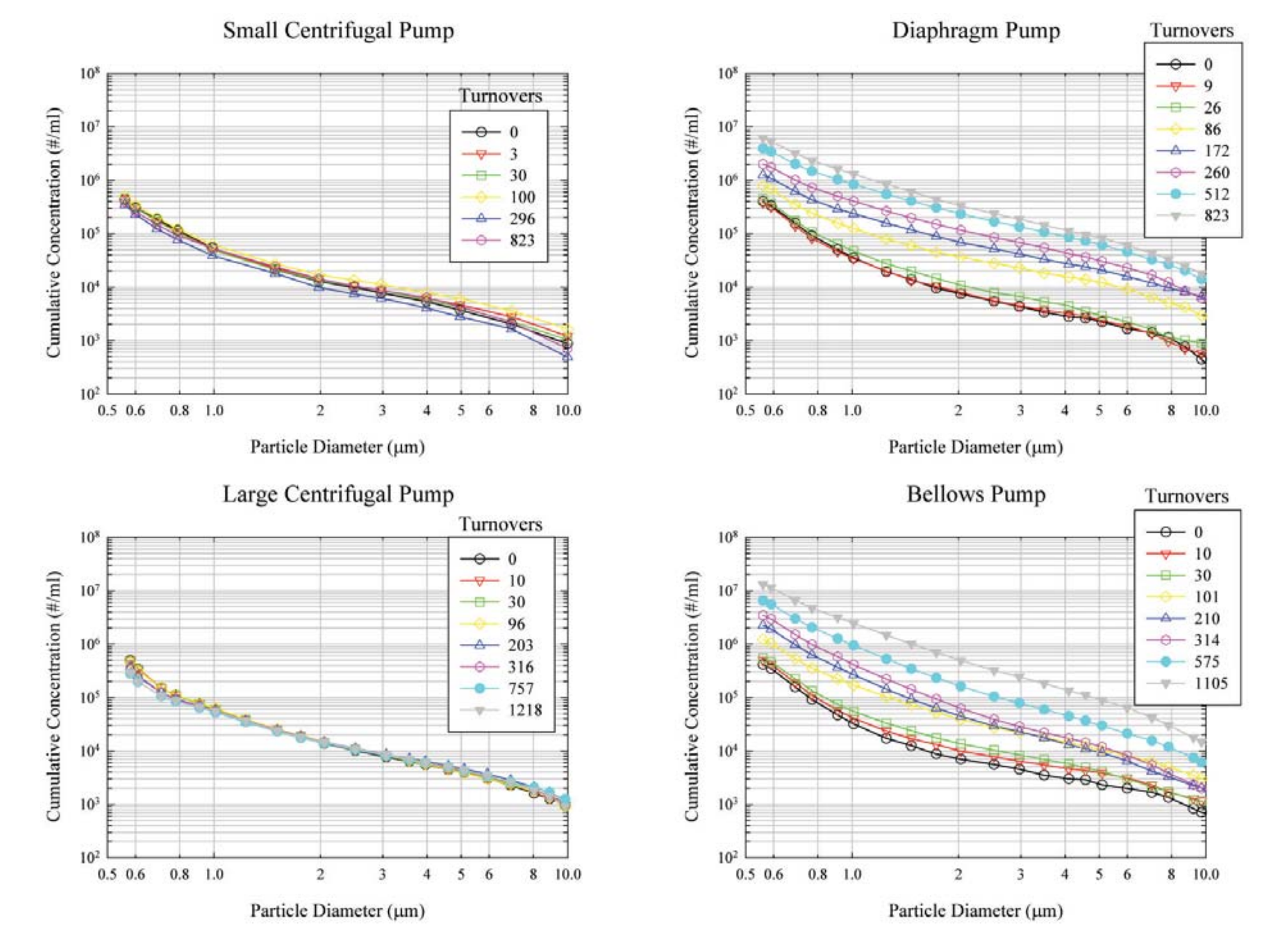

Figure 3. Cumulative PSDs measured in the fumed silica slurry.

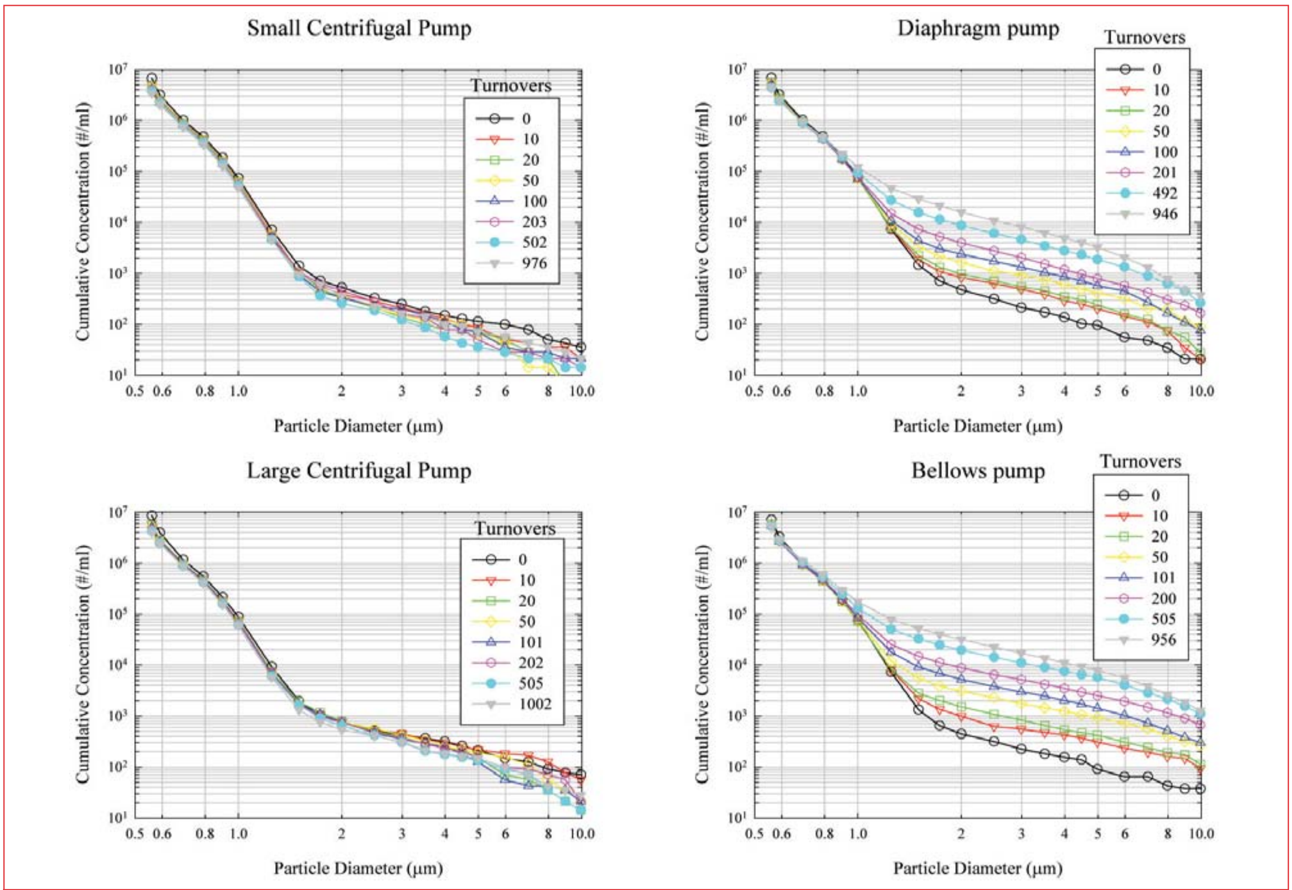

Figure 4. Cumulative PSDs measured in the alumina slurry.

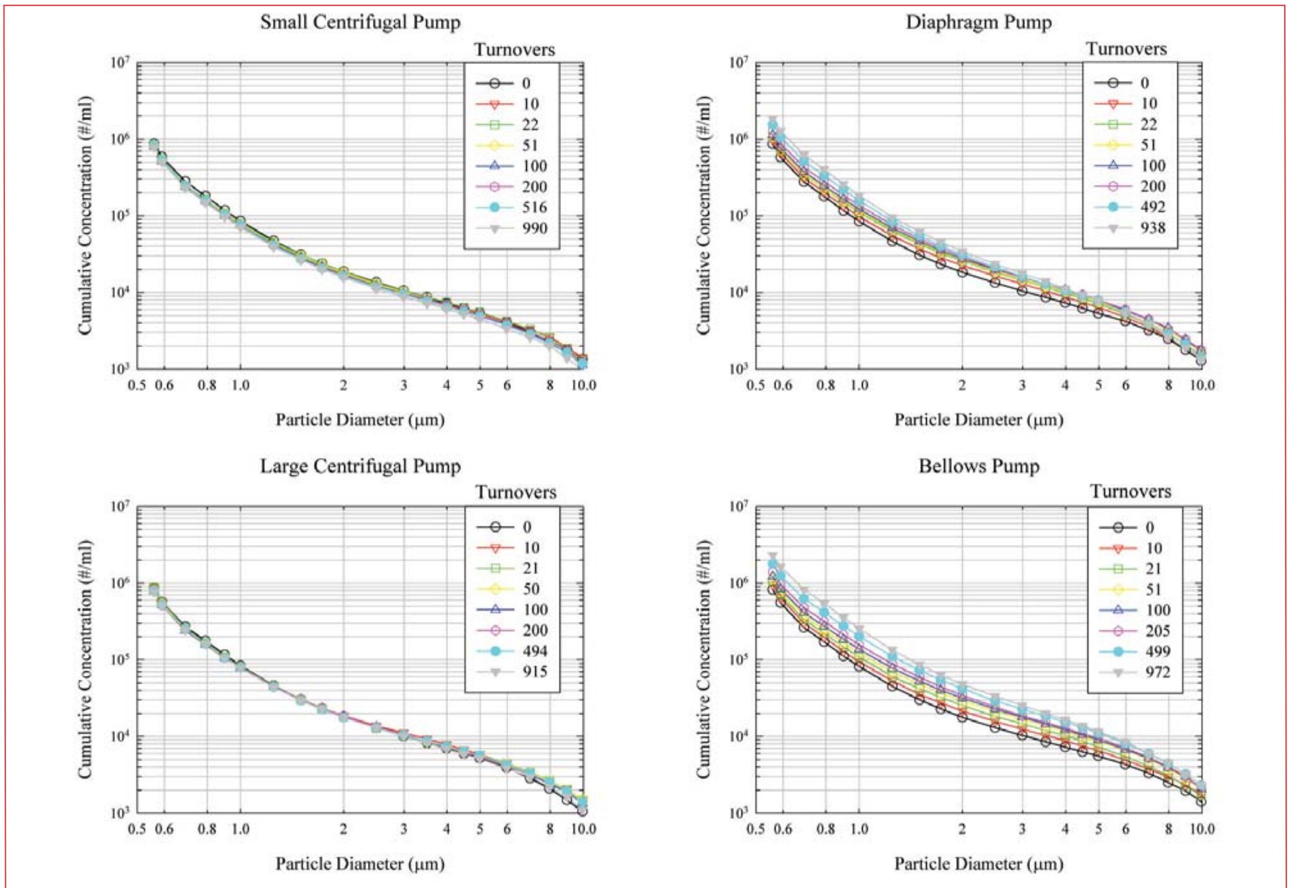

Figure 5. Cumulative PSDs measured in the colloidal silica slurry.

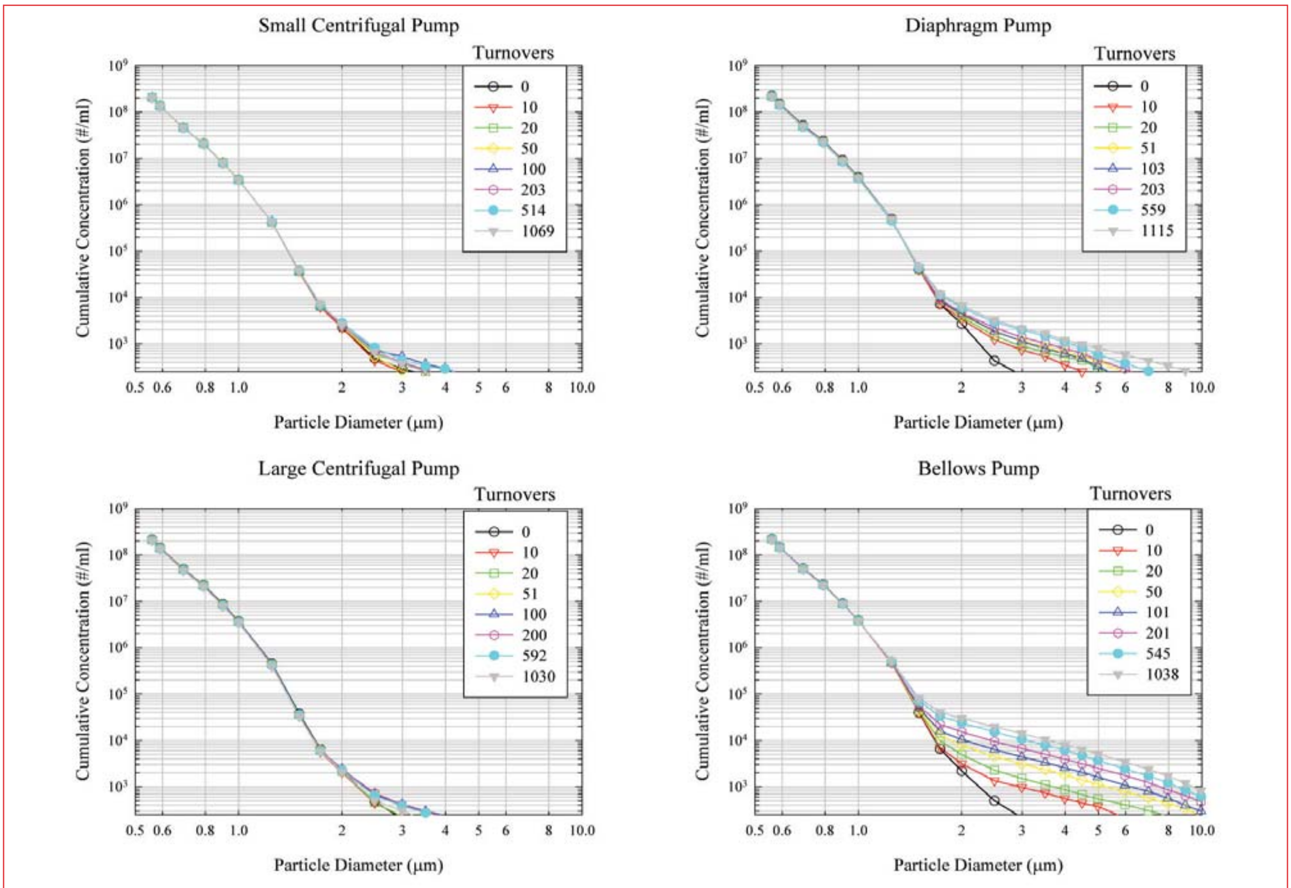

Figure 6. Cumulative PSDs measured in the ceria slurry.

The working PSD and zeta potential measurements were both made using the NICOMP 380ZLS. The samples were diluted approximately 40:1 into deionized water and analyzed at 23°C. Each PSD measurement was made over a period of 10 minutes, while each zeta potential measurement was made over 2 minutes. The PSD and zeta potential measurements of each sample were performed in triplicate and quintuplicate, respectively. The size measurement data were analyzed using the instrument's gaussian distribution assumption.

Other slurry health parameters measured included zeta potential, total percent solids, specific gravity, and pH. Measurements of each sample were performed in triplicate.

Results and discussion

The results presented will focus on the measurements of the slurry large particle tail since substantial changes were observed in the tail during these experiments. Results for the other parameters were not included due to the fact that none of those parameters changed significantly during the 1,000-turnover test.

Figures 3-6 present the cumulative PSDs of the slurry large particle tail measured with each slurry. The four graphs in each figure present the results from the four pumps tested. The initial PSD, measured prior to the start of each test, is presented in each graph as well as the PSDs after selected numbers of turnovers.

The fumed silica slurry, which has been known to be sensitive to particle agglomeration [2-5], exhibited large increases in the concentration of large particles when subjected to multiple passes through the diaphragm or bellows pumps (Figure 3). The concentration increases occurred over a wide particle size range from ≥ 0.56µm to more than 10µm. However, when this slurry was subjected to many passes through the magnetically levitated centrifugal pumps, little change in the concentration of large particles was observed.

Similar to the fumed silica slurry, little change was observed during the centrifugal pump tests in the alumina slurry as shown in Figure 4. However, large increases in the particle concentration, particularly for particles > 1µm, were observed during the diaphragm and bellows pump tests.

For the colloidal silica slurry (Figure 5), little change was observed during the centrifugal pump tests, while the concentration of particles ≥ 0.56µm increased during the diaphragm and bellows pump tests, but to a lesser extent than during tests with the fumed silica and alumina slurries.

As with the previous slurries, no changes were observed during the centrifugal pump tests in the ceria slurry (Figure 6), while significant increases in the concentration of particles larger than about 2µm in size were observed during the diaphragm and bellows pump tests.

To better visualize the changes in the PSDs during the tests, the ratios of the particle concentrations at each test point to the corresponding particle concentration at the start of each test were plotted. An example of the results is presented in Figure 7 for the alumina slurry tests. The four graphs in Figure 7 show concentration ratios for selected size channels as a function of tank turnovers during each pump test. The results for the other three slurry tests are included in reference [6]. In this slurry, significant changes in the large particle tail, particularly for particles ≥ 1.5µm in size, were observed during the diaphragm and bellows pump tests in less than 10 turnovers. The concentration of particles ≥ 1.5µm tended to increase

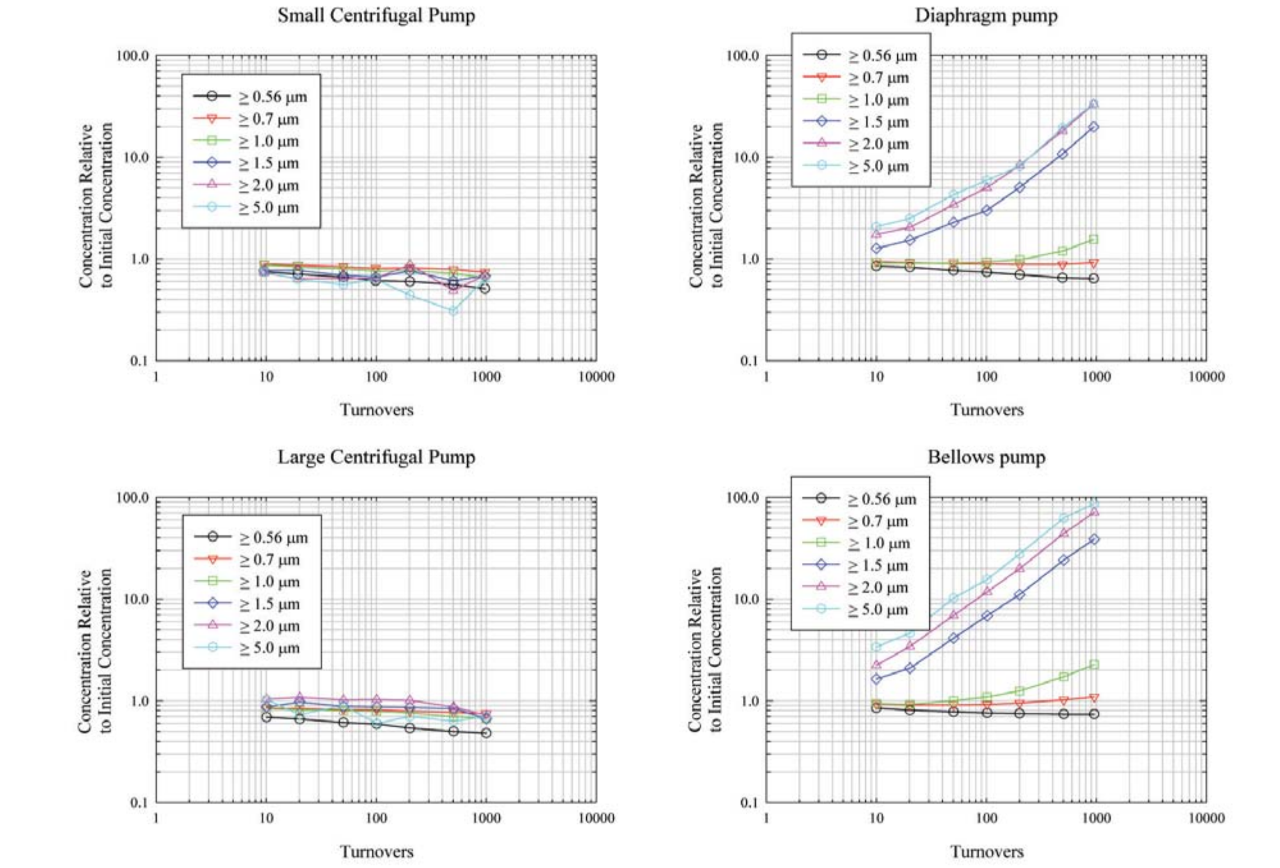

Figure 7. Concentrations relative to initial concentration during the alumina slurry tests.

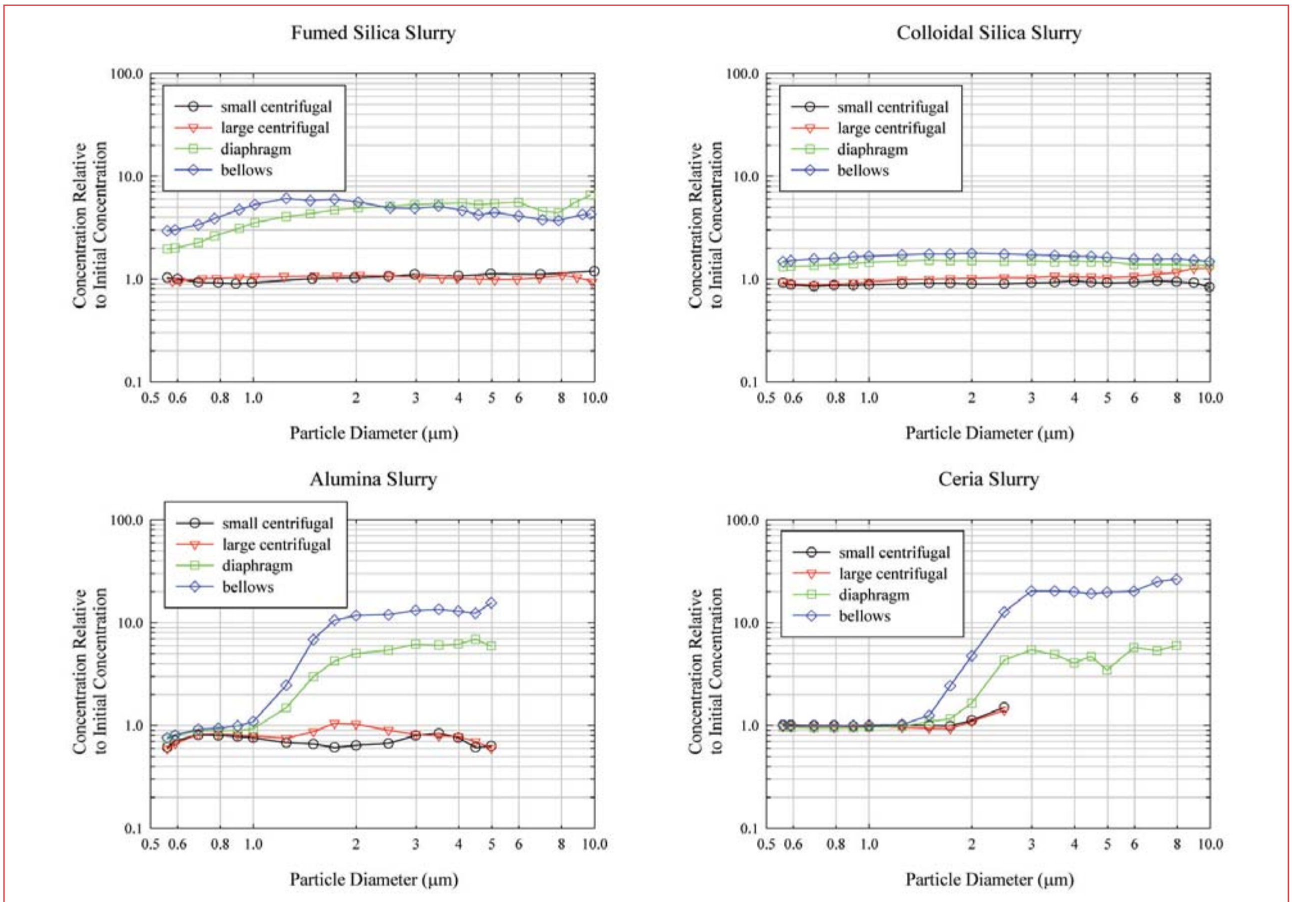

Figure 8. Concentration increases measured during all tests after 100 turnovers.

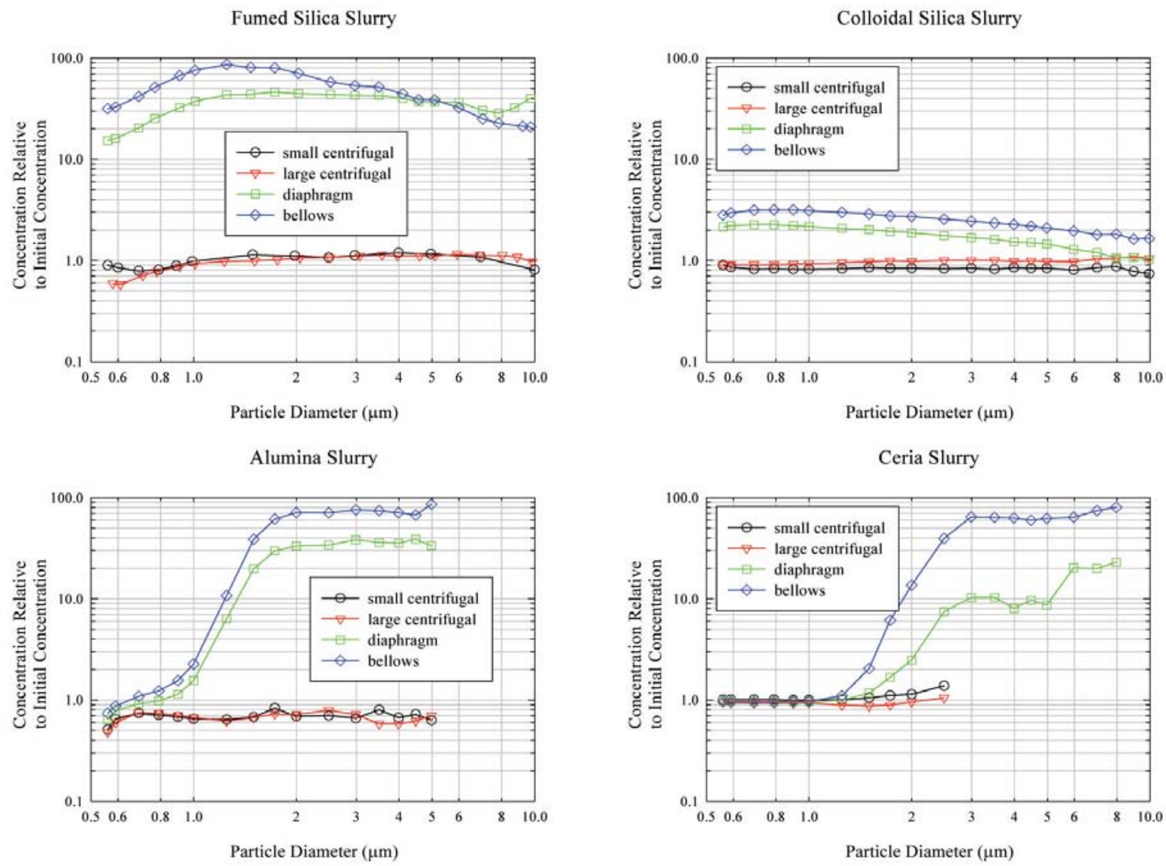

Figure 9. Concentration increases measured during all tests after 1,000 turnovers.

linearly with increasing turnovers. After 1,000 turnovers, the concentration of particles ≥ 2μm increased approximately 35- and 70- fold during the diaphragm and bellows pump tests, respectively. This equates to about a 3.5% and 7% increase in particle concentrations each time the slurry passes through these pumps, respectively. Meanwhile, concentrations measured during both centrifugal pump tests tended to decrease with increasing turnovers.

Figures 8 and 9 present a subset of the data collected in this study. The data are organized by slurry type rather than by pump type to more easily compare the effects of the pumps on each of the slurries. For each slurry, the concentration ratios are presented as a function of particle size for each pump after approximately 100 and 1,000 turnovers. These points in time were chosen since slurry is typically turned over on the order of 100 times prior to use, with 1,000 probably being a conservative upper limit in most delivery systems.

There were no observed significant increases in the particle concentrations with either centrifugal pump at any particle size in any of the slurries. In fact, a decrease in the particle concentrations was observed in some of the slurries during the centrifugal pump tests. For example, in the alumina slurry centrifugal pump tests, the particle concentrations decreased on the order of 30-50% for particle sizes ranging from 0.56μm to more than 5μm. Ratios were not plotted for some of the large particle sizes in the alumina and ceria centrifugal pump tests since the particle concentrations were so low, but no increases in particle concentrations were apparent.

Meanwhile, the concentrations of particles dramatically increased during the diaphragm and bellows pump tests in three of the four slurries tested. In the fumed and colloidal silica slurries, the concentration increases occurred over a wide range of particle sizes, from 0.56μm to larger than 10μm. However, concentration increases were very large during the diaphragm and bellows pump tests in fumed silica slurry, while only small increases were observed in the colloidal silica slurry. Meanwhile, in the alumina and ceria slurries, little or no particle concentration increase was observed for particles < 1.0μm in size, but very large concentration increases were observed with both the diaphragm and bellows pumps for particles

TABLE I: SUMMARY OF CONCENTRATION CHANGES IN SUBMICRON AND SUPERMICRON PARTICLE SIZES DURING ALL TESTS

Slurry	Submicron particles (0.5-1.0 μm)				Supermicron particles (> 3.0 μm)			
	Small Centrifugal	Large Centrifugal	Diaphragm	Bellows	Small Centrifugal	Large Centrifugal	Diaphragm	Bellows
Fumed Silica Slurry	0	0	++	++	0	0	++	++
Alumina Slurry	0	0	0	0	0	0	++	++
Colloidal Silica Slurry	0	0	+	+	0	0	0	+
Ceria Slurry	0	0	0	0	0	0	++	++

Key:

Symbol	Concentration change	Concentration ratio after 1,000 turnovers
0	None	0.5 - 2.0
+	Increase	2-10
++	Large Increase	> 10

≥ 2.0μm in size. (For the ceria slurry, the actual concentration ratios are likely to be even higher than indicated for particles ≥ 3μm, since the initial particle concentration for these large sizes was near the background of the test system.)

The particle sizes at which concentration increases occurred during the diaphragm and bellows pump tests were remarkably similar in all slurries. In general, the concentration increases observed during the bellows pump tests were larger than those seen during the diaphragm pump tests. The concentration increases observed with the bellows pump were typically about twice the level of increase with the diaphragm pump, except during the ceria slurry tests in which the increases appeared to be even higher.

Table I shows a summary of the concentration changes relative to the initial concentration during each test for both submicron (0.56 to 1.0μm) and supermicron (≥ 3μm) particle sizes. Minimal changes in particle concentrations were observed during the 1,000 turnover test for both centrifugal pumps. For submicron particles sizes, minimal changes were also observed with the diaphragm and centrifugal pumps in both the alumina and ceria slurries. However, concentration increases greater than an order of magnitude were observed for supermicron particles in three of the four slurries tested (fumed silica, alumina, and ceria) during both the diaphragm and bellows pump tests. For the fumed silica slurry, the concentration increases were much greater than an order of magnitude for both submicron and supermicron particle sizes.

Summary

Previous work has shown that silica-based CMP slurries are sensitive to agglomeration induced by extensive slurry handling. This study has shown that non-silica-based CMP slurries such as alumina and ceria are also sensitive. Both slurry type and pump type are key factors influencing the magnitude of agglomeration during slurry handling. Minimal changes in the large particle tail of the slurry PSD were observed during tests with magnetically levitated centrifugal pumps, while large increases in the large particle tail were observed in silica, alumina, and ceria-based slurries during circulation tests with either diaphragm or bellows pumps. The bellows pump caused more agglomeration than the diaphragm pump during most of the slurry tests.

No significant changes were observed in the other slurry health parameters (working PSD, zeta potential, total percent solids, pH, and specific gravity) for any of the pumps during any of the slurry tests.

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