Effects of Fluid Handling Components on Slurry Health

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This study investigated the effects of pumping systems and diaphragm valves on the physical properties, or "health," of a CMP slurry. Health was determined by measuring changes in the slurry particle size distribution. Three types of pumping systems were tested: bellows, diaphragm and magnetically-levitated centrifugal. The bellows and diaphragm systems caused significant increases in concentrations of large particles in the slurry while the centrifugal pump had little effect. The increased concentrations resulted in shorter lifetimes of filters used to remove large particles from the slurry. Valves had little effect on the properties of slurry flowing through them, but increased large particle concentrations when the valves were cycled. The rate of increase was substantially less than that seen with the bellows and diaphragm pumping systems. Overall, this paper shows that how a slurry is handled following its manufacture can have a significant effect on the concentration of large particles in the slurry. Some handling techniques substantially increase large particle concentrations and can be expected to decrease slurry polishing effectiveness. Other techniques that are available cause significantly less change in slurry properties.

Keywords : CMP, slurry, particle, pump, valve.

1. INTRODUCTION

The physical properties of slurries used in chemical mechanical polishing (CMP) can strongly influence the effectiveness of the slurries in polishing wafer surfaces [1]. Perhaps the most important physical property of the slurry is the size distribution of the particles in the slurry [2-4]. Two attributes of the particle size distribution (PSD) are important. The PSD of the smaller "working" particles, typically < 0.2 µm, is important in determining the polishing rate and uniformity. The presence of numerous particles in the large particle tail (particles > 0.5 µm) can cause imperfections (scratches, pits, etc.) in the polished wafer surface that lead to electrical defects and reduce manufacturing yield [5-7].

The PSD of slurries used in CMP is influenced by how the slurry is handled [8-15]. This study examined the effects of handling on the PSD of the silica particles in Cabot Semi-Sperse®-12 CMP slurry. The effects of pumping systems and diaphragm valves were investigated. Both the working particle PSD and the large particle tail PSD were examined. Three types of pumping systems were compared: a bellows pump with a pulse dampener, a diaphragm pump with a pulse dampener and a magnetically-levitated centrifugal pump. The effects of the pumping systems on the slurry PSD were determined by circulating the slurry through a simulated distribution loop at a fixed flow rate and delivery pressure. The effects of circulating the slurry using the same pumping systems on the lifetime of filters used to remove large particles from the slurry were also investigated. The effect of diaphragm valves on slurry PSD was tested under both steady-flow and cycling conditions.

2. EXPERIMENTAL PROCEDURES

2.1. Size distribution (PSD) measurement

The particle size distribution (PSD) was measured using two techniques. The size of the working particles was measured using a NICOMP 380ZLS (Particle Sizing Systems, Santa Barbara, CA) that determines particle size by dynamic light scattering. The size distribution of the large particle tail was measured using either an AccuSizer 780 optical particle counter (Particle Sizing Systems, Santa Barbara, CA) or a LiQuilaz-SO5 optical particle counter (Particle Measuring Systems, Boulder, CO).

The measurements made using the NICOMP 380ZLS counter were performed at 23°C on samples of slurry that were diluted approximately 40:1 into deionized water. Each sample was measured for 10 minutes. Triplicate measurements of each sample were made. The size measurement data were analyzed using the instrument's gaussian distribution assumption.

The large particle tail measurements were performed by first diluting the slurry sample using the system shown in Figure 1, then flowing the diluted slurry through the optical particle counter. In this system, the slurry sample was injected into a flowing stream of essentially particle-free deionized water at a fixed rate. The injection rate was selected to yield a particle concentration in the diluted stream that was just below the concentration where scattering from the working particles interfered with the analysis. The dilution factor was typically about 500:1. Between samples, the entire system was thoroughly flushed with deionized water.

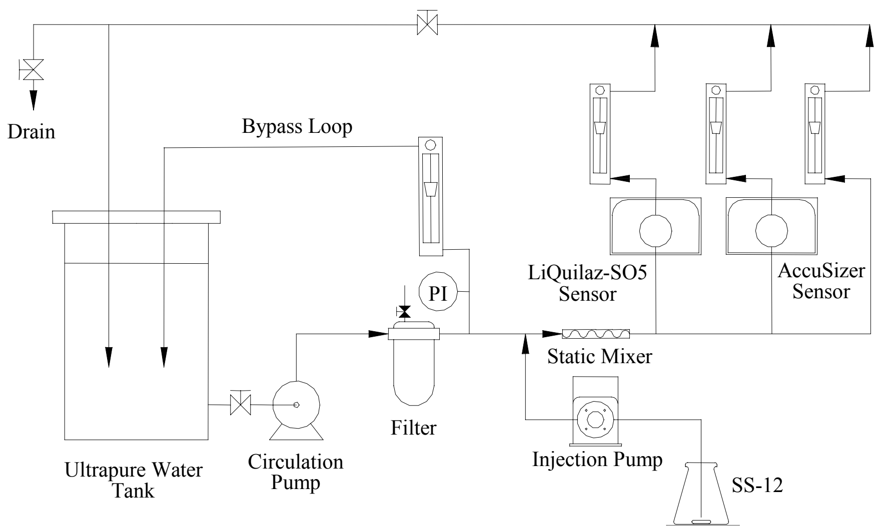

Fig. 1. In-line dilution system for large particle tail measurement

2.2. The effect of pumping systems on slurry PSD

A schematic of the test system used for testing the effect of pumping systems on slurry PSD is shown in Figure 2. Each pump was used to circulate 12 liters of slurry at a flow rate of approximately 30 lpm (8.0 gpm) and outlet pressure of 30 psig (2.1 bar). 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 air into the slurry. The return line was also positioned to minimize the formation of a large vortex in the tank that may entrain air into the slurry. No metering valves were used to generate back pressure at the outlet of the pump. Instead, a long length of ½" PFA tubing was used to gradually reduce the pressure from 30 psig at the pump outlet to ambient pressure at the end of the return line to the tank. The air pressure supplied to the bellows and diaphragm pumps was adjusted to achieve the desired flow rate and pressure, while the rotational speed was adjusted on the centrifugal pump. The bellows and diaphragm pumps were fitted with manufacturer recommended pulse dampeners.

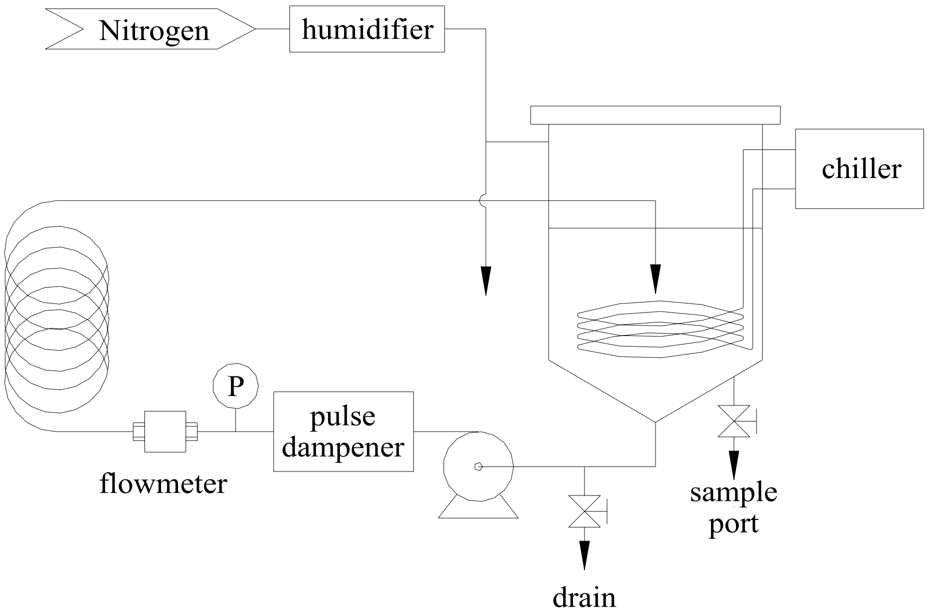

Fig. 2. Test system used to determine the effect of pumping systems on slurry PSD

The test system was constructed of PFA, except for the conical bottom tank which was constructed of polyethylene. The tank was blanketed with nitrogen to prevent absorption of carbon dioxide from the air that can change the pH of the slurry. The nitrogen was humidified to prevent slurry dehydration. The relative humidity in the tank was > 90% throughout the test. Shifts in the pH and dehydration can both cause particle agglomeration in the slurry. A chiller and stainless steel coil were used to maintain the slurry at 20 ± 2°C during the test. Fresh slurry from the same drum was used in each test and the slurry was circulated until more than 1,000 tank turnovers were achieved. Samples were drawn from the tank at selected times for analysis.

2.3. The effect of pumping system on filter lifetime

The test system used to evaluate the effect of pumping system on filter lifetime is shown in Figure 3. The system was similar to the one described in the previous section, with three modifications. A 10” PFA filter housing was installed immediately downstream of the pump or pump/pulse dampener combination and a differential pressure transducer was installed to measure the pressure drop across a filter in the housing. Also the tubing restrictor was modified such that the pump outlet pressure was 22 psig when a new filter was installed in the system.

Tests were conducted using 10” Mykrolis Planargard™ CMP3 filters. (cat. no. CMP301E06, lot no. CB4DNP3M2). A new filter was installed at the beginning of each test. The test protocol was similar to the tests designed to measure the effect of pumping system on slurry PSD. Each pumping system was used to circulate 28.5 liters of fresh slurry at a flow rate of 29.5 lpm (7.8 gpm). As the experiment progressed, the filter resistance to flow increased as it removed large particles from the slurry. Therefore, the air pressure supplied to the bellows and diaphragm pumps and the rotational speed of the centrifugal pump were increased during the course of the experiments to maintain a constant flow rate through the filter and allow the pressure drop across the filter to increase. The average pump outlet pressure during the tests was approximately 30 psig, the same as the outlet pressure during the tests to determine the effect of the pumping systems on the slurry PSD.

A filter was considered to reach the end of its life when the ΔP increased by 10 psid [16]. Therefore, the slurry was circulated until a ΔP increase of at least 10 psi was achieved or more than 10,000 tank turnovers (or passes through the pump) were performed. Samples were drawn from the system at selected times for PSD analysis.

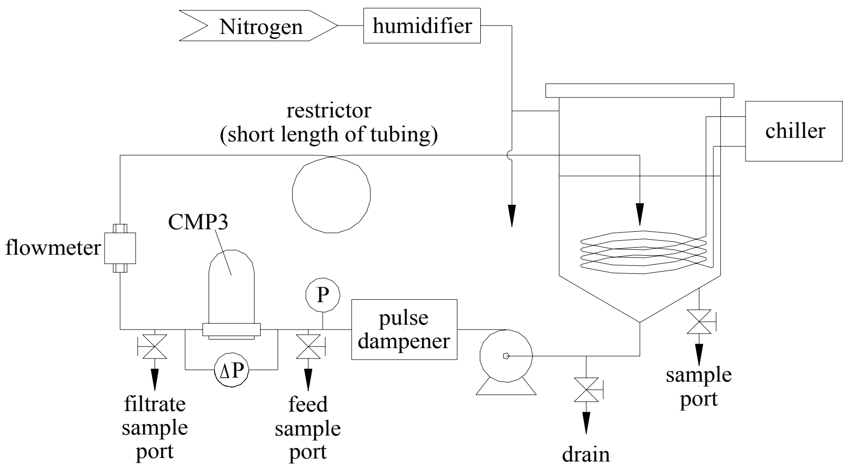

Fig 3. Test system used to determine the effect of pumping systems on filter lifetime

2.4. The effect of valves on slurry PSD

The test system used to measure the effect of valves on slurry PSD is shown in Figure 4. Eight valves were tested in the system at the same time. Each test included two steps: steady-flow through each valve for 48 hours followed by flow through the valves as they were cycled for 48 hours. For the steady-flow portion of the test, the valves were assembled in a manifold containing all eight valves, with 4 pairs (each pair of valves in series) of valves arranged in parallel. For the cycling portion of the test, the valves were assembled to allow parallel flow through two groups of 4 valves in parallel. The valves were configured in this manner to maintain the mean velocity of the slurry through each valve at 0.5-1.0 ft/sec in both tests. Figure 4 shows the valves in the cycling configuration. To measure changes in the slurry PSD caused by the circulating system in the absence of valves, control tests were performed in which spool pieces, which were simply unions, were tested in place of the valves.

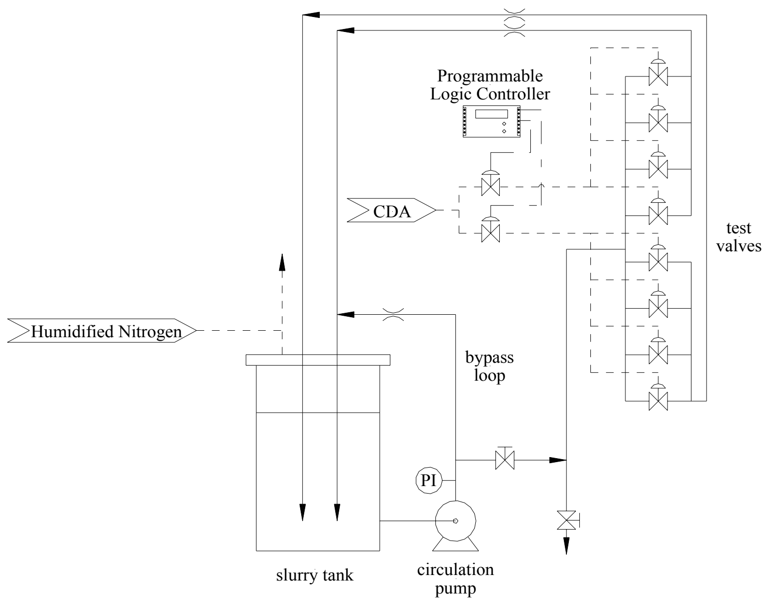

Fig. 4. Test system used to measure the effect of valves on slurry PSD during valve cycling

The slurry was circulated through the valves using a Levitronix BPS-3 centrifugal pump drawing from a 30 L conical bottom slurry reservoir. This pump was chosen because previous testing has shown that it causes minimal slurry damage [17]. The system was used to circulate 25 liters of slurry at a flow rate of 18.5 ± 2 lpm and outlet pressure of 22 ± 2 psig. Under these conditions, the slurry in the tank was turned over approximately every 80 seconds. Other test system details were similar to those described for the pumping system tests.

During the cycling portion of the test, the valves were cycled at a rate of 12 cycles/min (2.5 seconds open and 2.5 seconds closed). All valves in each group of four were actuated/deactuated at the same time. One group was actuated as the other group was deactuated, so that flow through one group was initiated as flow through the other group stopped. This sequencing schedule ensured that flow was constant throughout the test. Each group of 4 valves was connected to a separate outlet manifold, which allowed the outlet pressure on a group of valves to drop to ambient when the valves were closed.

3. RESULTS AND DISCUSSION

3.1. Initial slurry PSD

Figures 5 shows the initial size distributions of the working particles measured using the dynamic light scattering instrument. Both the cumulative and differential distributions are presented in Figure 5. The volume-weighted mean and 99th percentile particle diameters (99% of the particles have diameters less than this size) are included in the figure.

Figure 6 shows the geometric mean of the large particle tail PSD at the beginning of the pumping system tests measured using the Accusizer 780 sensor. The error bars in the figure represent ± one geometric standard deviation. Since there was some variation in the initial concentrations for each test, particularly for the large particle tail, the ratios of the cumulative particle concentrations relative to the initial particle concentrations will be presented in the following analysis rather than actual particle concentrations.

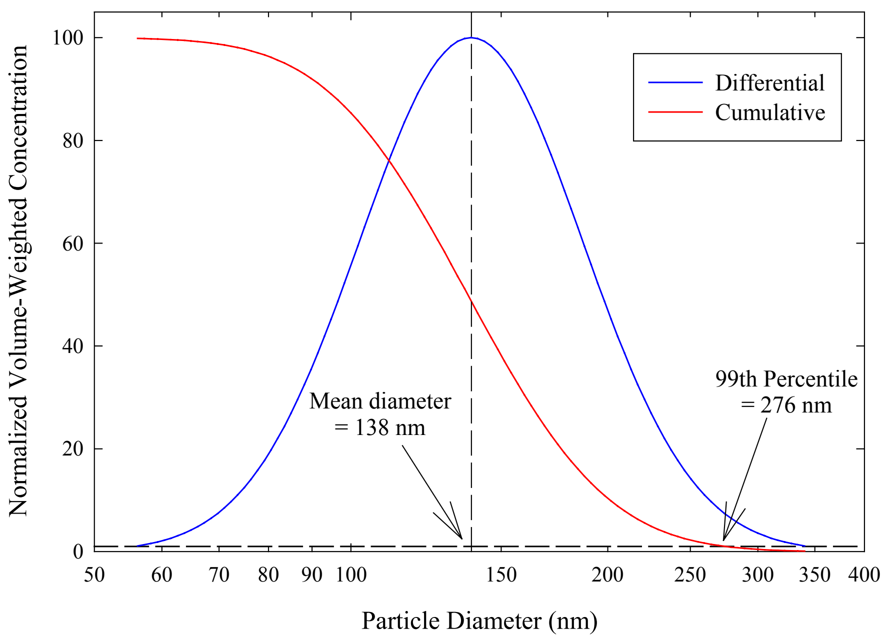

Fig. 5. Initial working particle size distribution

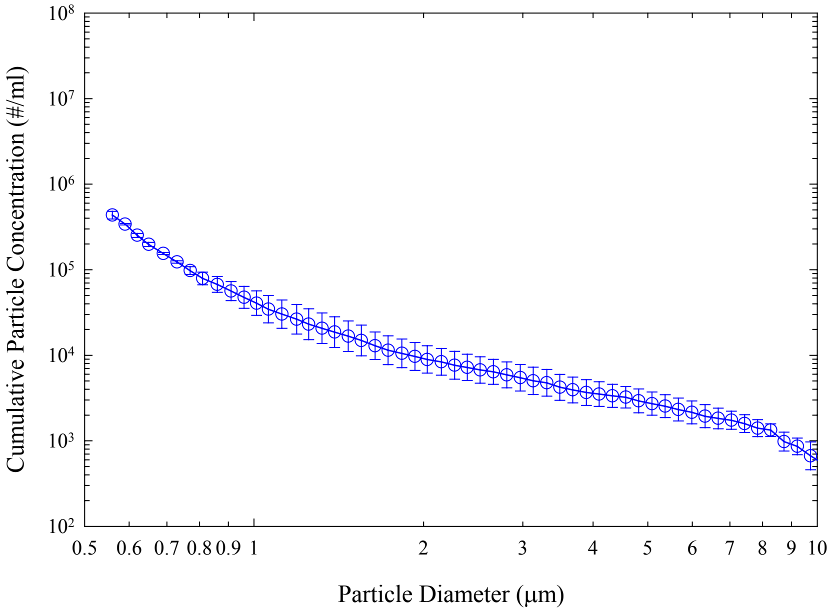

Fig. 6. Initial size distribution of the large particle tail

3.2. The effect of pumping systems on slurry PSD

Figure 7 shows the volume-weighted mean and 99th percentile particle diameters of the working PSD as a function of tank turnovers measured during the experiments to determine the effects of pumping systems on slurry PSD. Error bars are included in the figure and represent ± 3 standard deviations. No significant change in the working PSD was observed for any of the pumping systems during 1,000 turnovers.

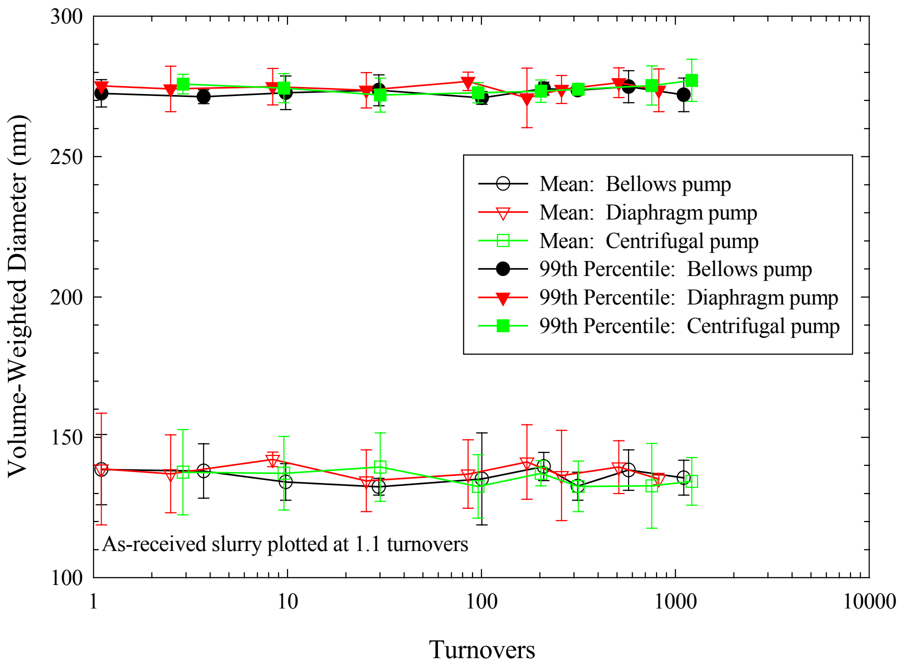

Fig. 7. Effect of pumping systems on the working particle size distribution

Figure 8 presents an example of measurements of the large particle tail PSD made during an experiment. The example is from the test with the bellows pump. Figure 8 shows PSDs measured after the tank of slurry had been turned over (passed through the pumping system) selected numbers of times. Particle concentrations increased throughout the size range measured as the number of turnovers increased.

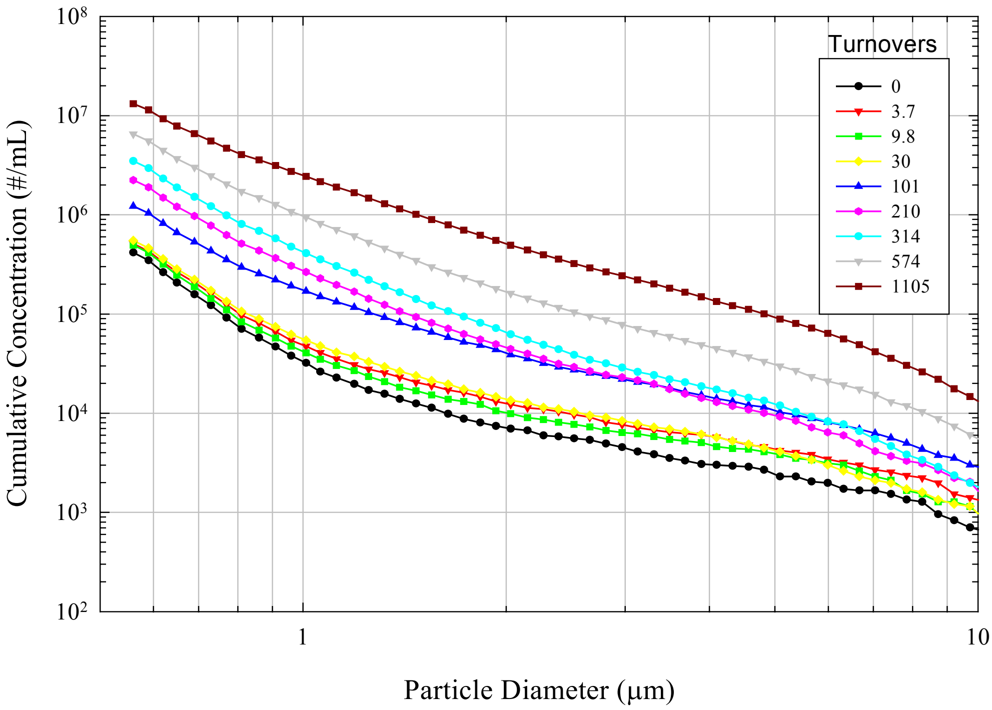

Fig. 8. Effect of bellows pumping system on the large particle tail PSD

Figure 9 and Table 1 compare the effects of the three pumping systems on the large particle tail PSD. Figure 9 contains 2 graphs that show the ratio of the particle concentration in the slurry at a given number of slurry turnovers to the initial concentration as a function of slurry turnovers. The graphs depict particles ≥ 0.56 µm and particles ≥ 10 µm. Concentrations of particles of both sizes increased linearly with turnovers in the bellows and diaphragm pumping systems and remain essentially constant in the centrifugal pumping system. Table 1 presents particle concentration ratios for various sized particles in the three pumping systems after 100 and 1000 turnovers. Table 1 indicates that concentrations in the bellows and diaphragm pumping systems had increased 2 to 8 fold after 100 turnovers and 20 to 70 fold after 1000 turnovers. Concentrations in the centrifugal pumping system remained the same or decreased slightly.

Particle concentrations relative to the initial particle concentrations (C_T/C_I)						
Particle	100 Turnovers			1,000 Turnovers		
size	Bellows	Diaphragm	Centrifugal	Bellows	Diaphragm	Centrifugal
≥ 0.56 µm	2.9	2.3	1.0	28	18	0.6
≥ 1.0 µm	5.3	4.1	1.0	69	45	0.9
≥ 2.0 µm	5.6	5.7	1.1	64	54	1.0
> 5.0 µm	4.4	6.3	1.0	35	45	1.1
≥ 10 µm	4.3	7.7	0.9	17	52	1.1

Table 1. The effect of pumping systems on large particle concentrations

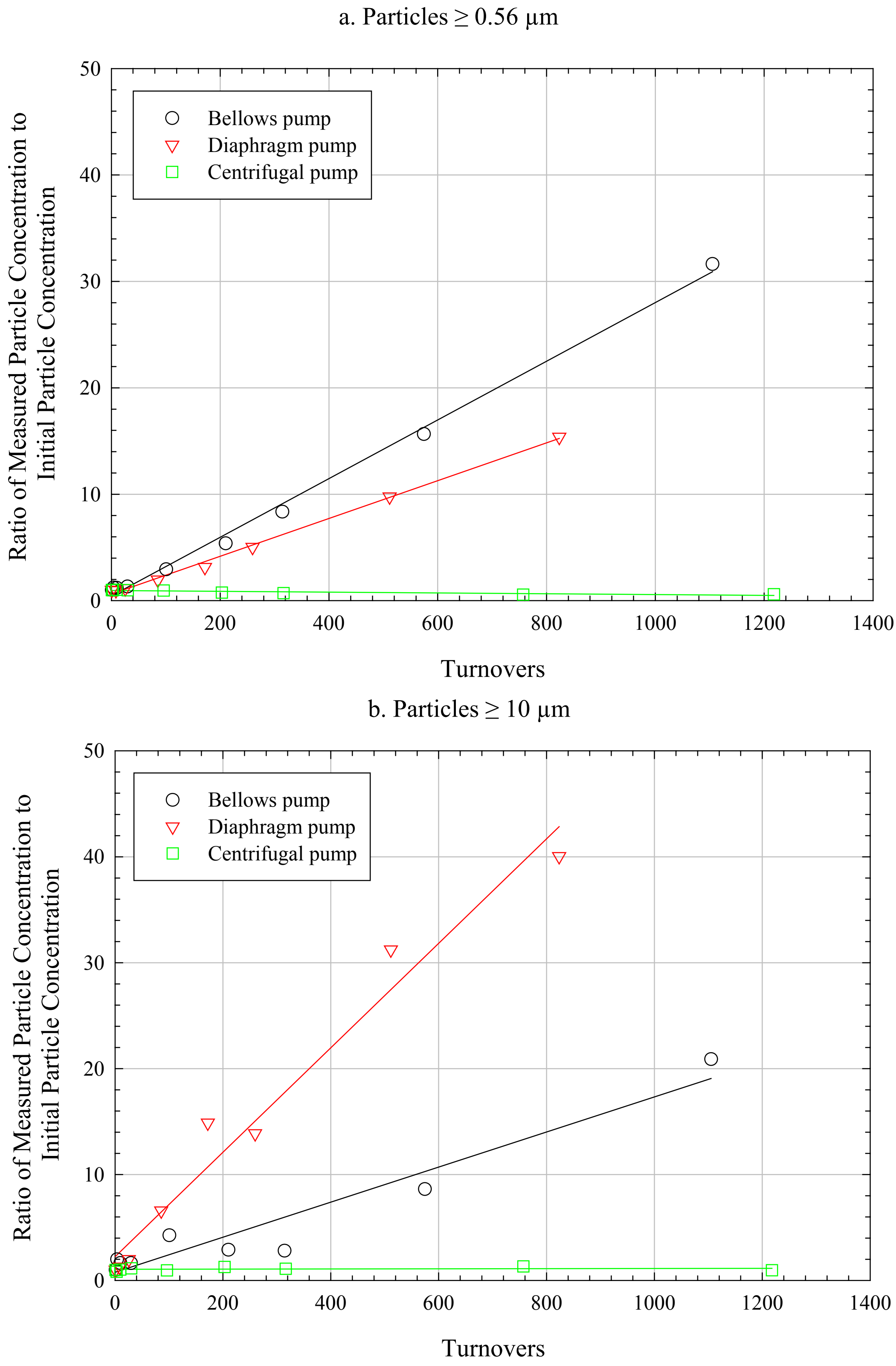

Fig. 9. The effect of pumping systems on large particle concentrations

3.3. The effect of pumping systems on filter lifetime

Figure 10 compares the increase in pressure drop across a CMP3 filter as a function of turnovers in the three pumping systems. The curves in Figure 10 are characterized by a fairly rapid initial rate of increase followed by a slower constant rate of increase. The initial rapid increase rate is attributed to removal of large particles in the fresh slurry, while the slower constant rate resulted from removal of particles generated by the pumping system. The rate of pressure drop increase was greatest for the bellows pumping system, followed by the diaphragm pumping system. The pressure drop across the filter in the centrifugal pumping system was essentially constant after the initial pressure rise. The rates of pressure increase from the linear portion of each curve are compared in Table 2. The pressure drops across the filters in the bellows and diaphragm pumping systems increased 23 and 9.3 times faster than pressure drop across the filter in the centrifugal pumping system. These results indicate that the bellows and diaphragm pumping systems generate considerably more large particles than the centrifugal pumping system. Hence they are consistent with the results described in section 3.2.

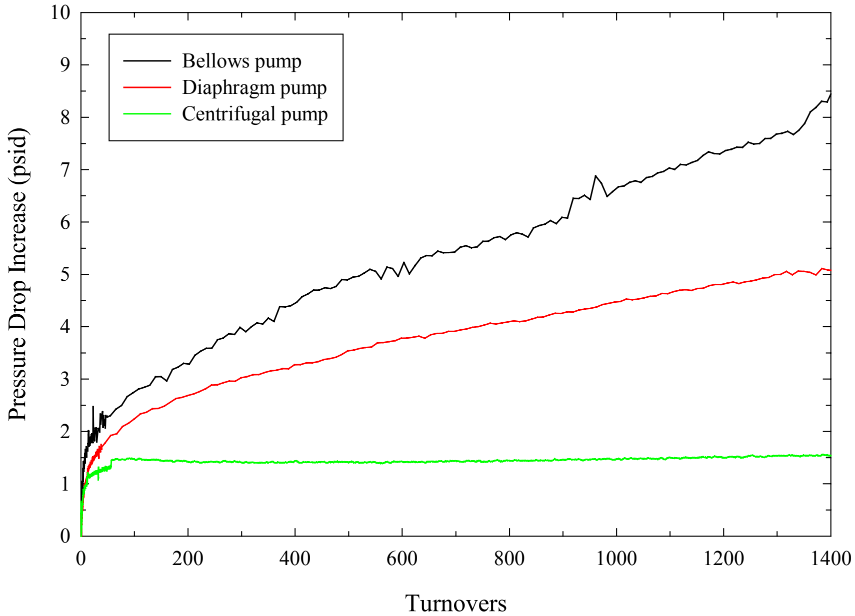

Fig. 10. The effect of pumping systems on filter pressure drop

Type of Pump	ΔP Increase per Turnover (psi/turnover)	ΔP Increase per 1000 L (psi/1000 L)	Rate of ΔP Increase (relative to centrifugal pump)
Bellows	0.0041	0.14	23
Diaphragm	0.0017	0.058	9.3
Centrifugal	0.00018	0.006	-

Table 2. The effect of pumping systems on filter pressure drop rate of increase

3.4. The effect of diaphragm valves on slurry PSD

The effect of valves on slurry large particle tail PSD is shown in Figure 11. Six curves of particle concentrations ≥ 0.60 µm versus turnovers are shown; three for spool pieces and three for test valves. There was no discernable change in particle concentration when spool pieces were tested up to 4500 turnovers. During the steady-flow portion of the valve test (up to 2100 turnovers) concentrations remained constant. However, during valve cycling, particle concentrations increased approximately linearly with turnovers. Since the valves were subjected to a constant number of valve cycles per turnover, this corresponds to a linear increase with valve cycles. Particle concentrations increased throughout the size range measured as shown in Figure 12. No change in the working PSD was observed.

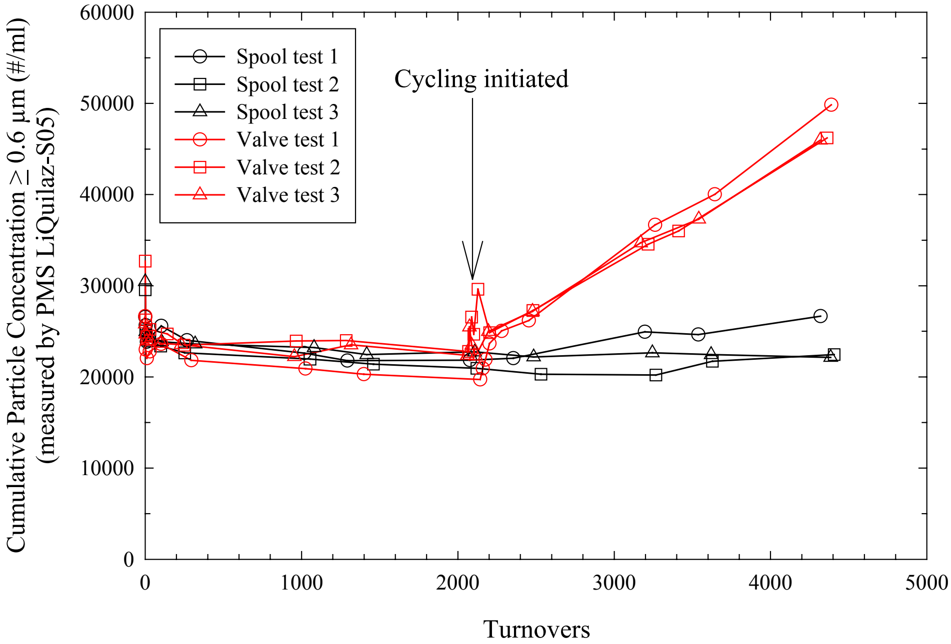

Fig. 11. The effect of diaphragm valves on slurry PSD

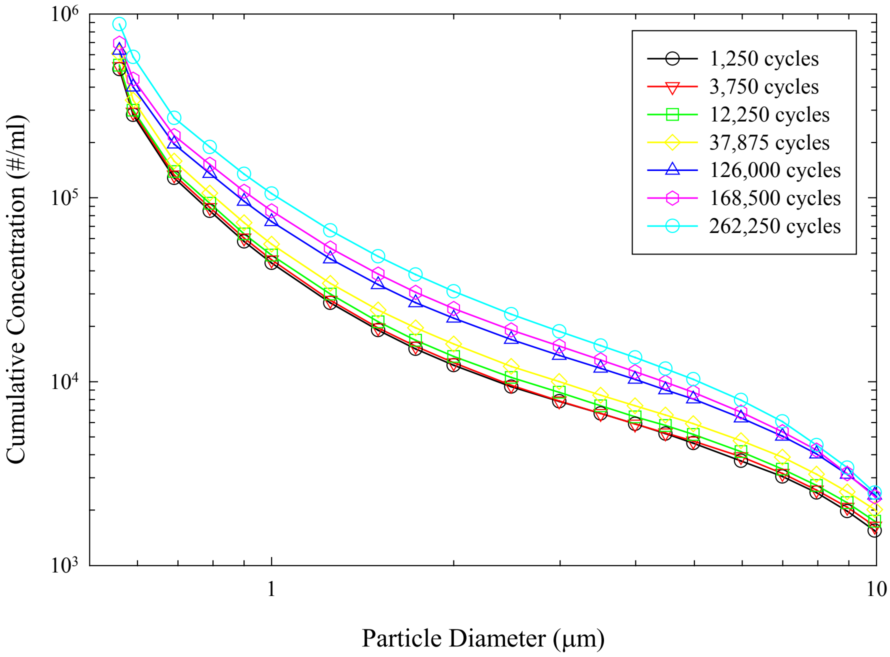

Fig 12. The effect of valve cycling on slurry large particle tail PSD

3.5. Comparison between the effect of pumping systems and diaphragm valves on slurry PSD

The data presented in previous sections indicate that concentrations of large particles increased linearly with handling in bellows and diaphragm pumping systems and when subjected to valve cycling. Therefore, the data can be converted to particles generated per pump stroke or per valve cycle as shown in Figure 13. Figure 13 indicates that these pumping systems generate 10 to 100 times more particles per stroke then are generated by the valve per cycle. The ratio decreases with increasing particle size. Data for the centrifugal pumping system are not shown in the figure as it did not appear to contribute particles in the range measured.

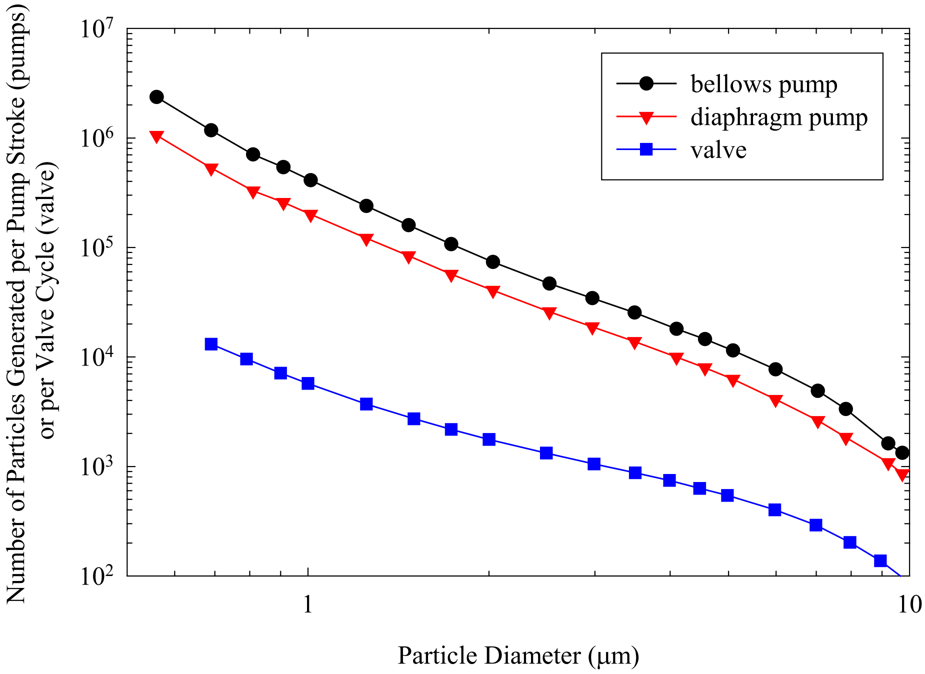

Fig 13. Particle generation by pumps and valves

The data in Figure 13 can be used to predict the effect of pumping systems and valves on the PSD of handled slurry. For example, if it is assumed that the slurry in a delivery system with a bellows pumping system and no filtration is subjected to an average of 100 turnovers and 100 valve cycles per liter of slurry delivered, the particle concentration in the delivered slurry can be estimated. Table 3 shows the predicted effect of these assumptions on the slurry used in this study. Under these assumptions, the bellows pumping system is the major contributor to particles in the large particle tail.

Particle diameter (μm)	Concentration (#/ml)			
	Incoming slurry	Increase due to pumping system	Increase due to valves	Delivered slurry
0.7	158,000	586,000	130	744,130
1.0	32,000	206,000	57	238,057
2.0	7,000	36,700	18	43,718
5.0	2,300	5,800	5	8,105
10.0	700	670	1	1,371

Table 3. The effect of slurry handling components in a hypothetical delivery system

4. SUMMARY AND CONCLUSIONS

This study examined the effects of handling on the particle size distribution (PSD) of the silica particles in Cabot Semi-Sperse®-12 CMP slurry. The effects of pumping systems and diaphragm valves were investigated. Both the working particle PSD and the large particle tail PSD were examined.

Three types of pumping systems were compared: a bellows pump with a pulse dampener, a diaphragm pump with a pulse dampener and a magnetically-levitated centrifugal pump. The pumps were found to have little effect on the working particle PSD. However, both the bellows and diaphragm pumps caused significant increases in the concentrations of large particles. No significant change in large particle concentrations was observed after more than 1000 passes through the centrifugal pumping system. The effect of circulating the slurry using the same pumping systems on the lifetime of filters used to remove large particles from the slurry was also investigated. As expected, filters in the bellows and diaphragm pumping systems clogged more quickly than those in the centrifugal pumping system since the bellows and diaphragm pumps generate more large particles.

The diaphragm valves were tested under both steady-flow (no cycling) and cycling conditions. No change in either the working particle PSD or the large particle tail PSD was seen after 2000 passes through the valves under steady-flow conditions. When the valves were cycled, the working particle PSD was not affected, although large particle concentrations increased. Concentrations doubled when the slurry was subjected to approximately 10,000 actuation cycles per liter of slurry. The damage caused by valves should be substantially less than that caused by the bellows and diaphragm pumps used in a slurry delivery system.

Overall, this paper shows that the way slurry is handled following its manufacture can have substantial effects on the concentration of large particles in the slurry. Some handling techniques substantially increase large particle concentrations and can be expected to decrease slurry polishing effectiveness and increase cost of ownership due to increased filter change frequency. Other techniques are available that cause significantly less change in slurry properties.

ACKNOWLEDGMENTS

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