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Comparison of Four Pump Systems on the Particle Size Distribution of Cabot SiLECT™ 6000 Slurry

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Introduction

Delivery systems are often used to supply the slurry used to planarize wafers during semiconductor chip manufacturing. These systems pressurize the slurry to deliver it to the tools and circulate it to help keep the particles in suspension. Pressurization and circulation are accomplished by various means including a variety of types of pumps 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 may be damaged due to the mechanical handling of the slurry. For example, particle agglomerates may form which can limit the life of filters or even reduce yield by causing wafer defects.

Previous experiments have shown significant increases in the large particle concentrations when certain types of pumps (diaphragm and bellows) were used to circulate slurry, while Levitronix magnetically levitated centrifugal pumps have not [2-5]. These tests were performed to determine the effect of circulating SiLECT™ 6000 with four different pumps (bellows, diaphragm, BPS-3, and BPS-4 pumps) on the health of the slurry. During these tests, a number of slurry properties were monitored including the size distribution of the particles in the slurry.

Large differences in the large particle tail of the slurry particle size distributions (PSD) were observed after circulation with the bellows and diaphragm pumps, while relatively small changes were observed with the centrifugal pumps. Minimal changes were observed in other slurry properties.

Experimental

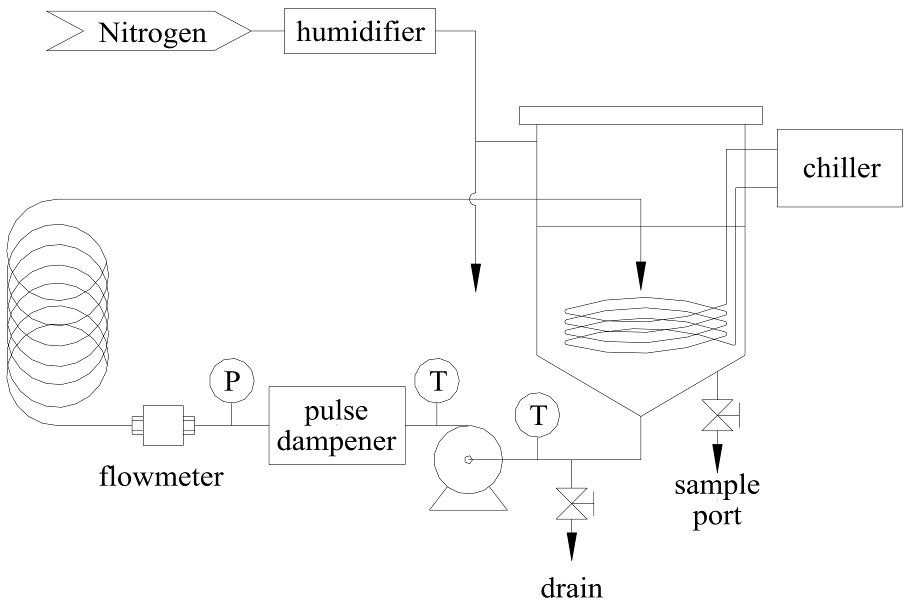
The pumps evaluated in this test were: an ASTI bellows pump, a Yamada diaphragm pump, and two Levitronix magnetically levitated centrifugal pumps. Table I shows the details of each pump. Manufacturer recommended pulse dampeners were installed downstream of both the ASTI and Yamada pumps to minimize pulsation. A schematic of the test system is shown in Figure 1.

Table I. Specifications of the four pump systems

Pump and Pulse Dampener Manufacturer	Type of Pump	Model # of Pump	Pulse Dampener Used?	Model # of Pulse Dampener
ASTI	Bellows	PFD3 322S	Yes	AMC3 222A
Yamada	Diaphragm	DP25F	Yes	AD-25TT
Levitronix	Centrifugal	BPS-3	No	NA
Levitronix	Centrifugal	BPS-4	No	NA

Each pump was used to circulate 12 liters of slurry at a flow rate of approximately 30 lpm (7.9 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 gas into the slurry. The return line was also positioned to minimize the formation of a large vortex in the tank that may entrain gas into the slurry. No valves or orifices 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 Yamada and ASTI pumps was adjusted to achieve a slurry flow rate of 30 lpm with the desired outlet pressure. Meanwhile, the speeds of the BPS-3 and BPS-4 pumps were varied to achieve the desired flow rate and pressure. The BPS-3 and BPS-4 pumps were operated at speeds of 7350 and 5830 rpm, respectively. In each test, the slurry was circulated until more than 3,000 tank turnovers were achieved. The test system was constructed of PFA, except for the conical bottom tank that was constructed of polyethylene. The slurry used in each test was taken from the same lot.

Figure 1: Test system schematic



The tank holding the slurry 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 dehydration of the slurry. Shifts in the pH and dehydration can both result in particle agglomeration in the slurry. The relative humidity in the tank was > 90% throughout the test. A chiller and stainless steel coil were used to maintain the slurry at 21 ± 2°C during the test. Platinum RTD sensors were used to measure the temperature at the inlet and the outlet of each pump being evaluated.

Samples were drawn from the system at selected times for analysis. The particle size distribution (PSD) was measured using 2 techniques. The size of the working particles was measured using a 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 optical particle counter.

The AccuSizer uses a combination of light scattering and light extinction to measure the size distribution of particles ≥ 0.56 μm. Since the PSD was so steep, each sample was measured at two dilution ratios. This allowed more accurate measurement of the particle concentrations over a wider size range. Measurement of particle sizes from 0.56 to 1.5 μm was performed by diluting the slurry sample by a factor of 1,750:1 and using the summation mode. In the summation mode, a combination of light scattering and light extinction is used to size particles up to 1.5 μm in size. Measurement of particle sizes ≥ 1.5 μm were performed by diluting the slurry sample by a factor of 32,000:1 and using the extinction mode. In the extinction mode, light extinction is used to size the particles. Between samples, the entire system was thoroughly flushed with deionized water. Data from selected particle size channels were analyzed.

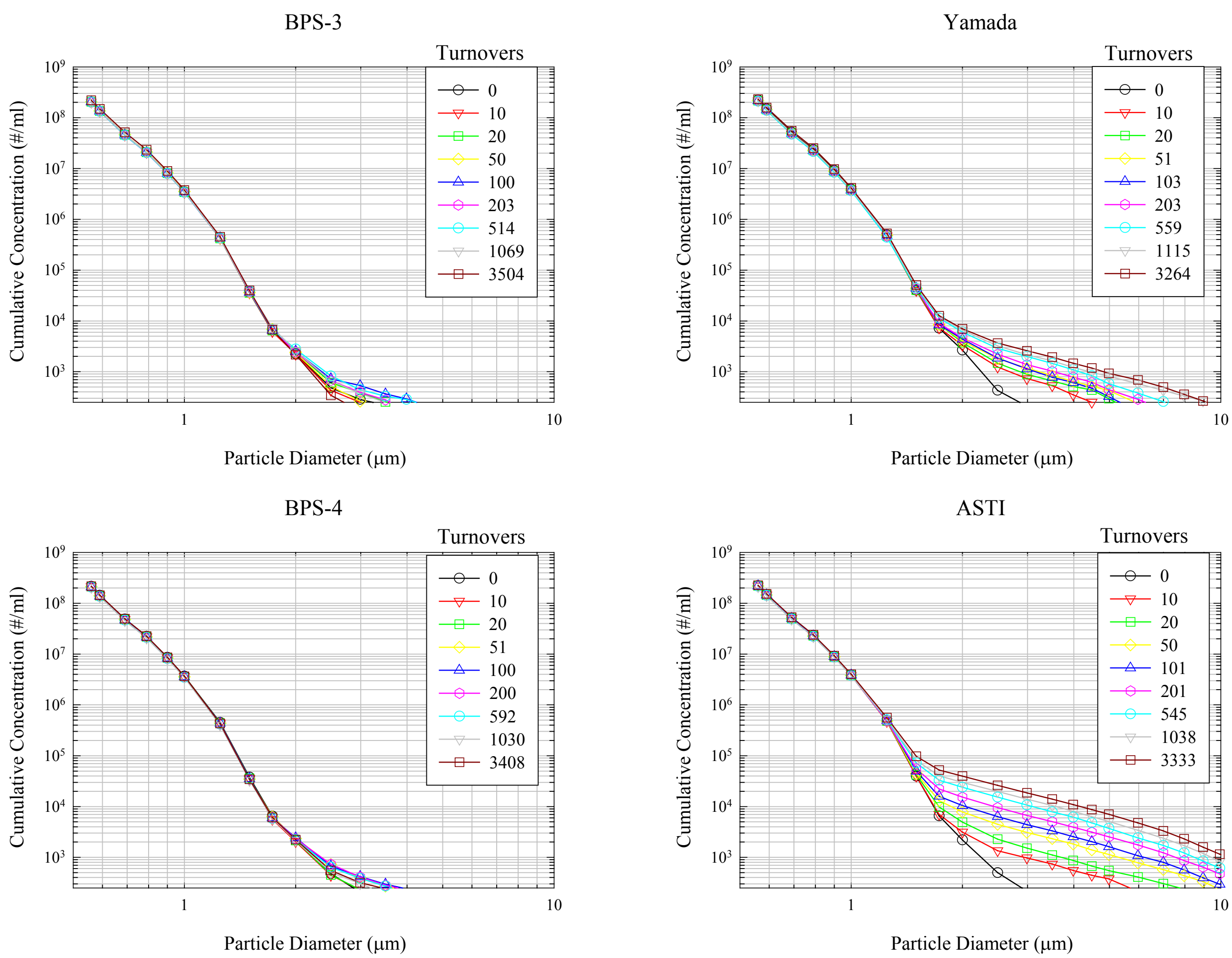
The zeta potential and working PSD measurements were made using the NICOMP 380ZLS. The samples were diluted approximately 40:1 into deionized water and analyzed at 23°C. Each PSD measurement was made over 10 minutes while each zeta potential measurement was made over 2 minutes. The PSD and zeta potential measurements of each sample were performed in triplicate and quintuplicate, respectively. The size measurement data were analyzed using the instruments 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 four graphs in Figure 2 show the cumulative PSDs of the slurry large particle tail during each pump test. Each graph presents the results from a different pump. The initial PSD, measured prior to the start of each test, is presented in each graph as well as PSDs after selected numbers of turnovers.

Figure 2: Cumulative PSDs of the large particle tail measured during pump tests



The background particle levels for this slurry was on the order of 250 particles/ml or lower for particle sizes larger than 3 µm. The initial particle concentration for sizes larger than 3 µm was indistinguishable from the background of the measurement system. Particle concentrations ≥ 3 µm, remained below this level during both centrifugal pump tests.

Minimal change in the large particle tail of the slurry PSD was observed during either centrifugal pump test, even after more than 3,000 turnovers. Meanwhile, large increases in large particle concentrations were observed during the Yamada and ASTI pump tests for particle sizes larger than 2 µm.

Figures 3 presents the ratio of particle concentrations at each test point to the initial particle concentration. Each graph presents concentration ratios for selected size channels as a function of tank turnovers for each pump test.

Figure 3: Particle concentrations relative to the initial concentration for selected size channels

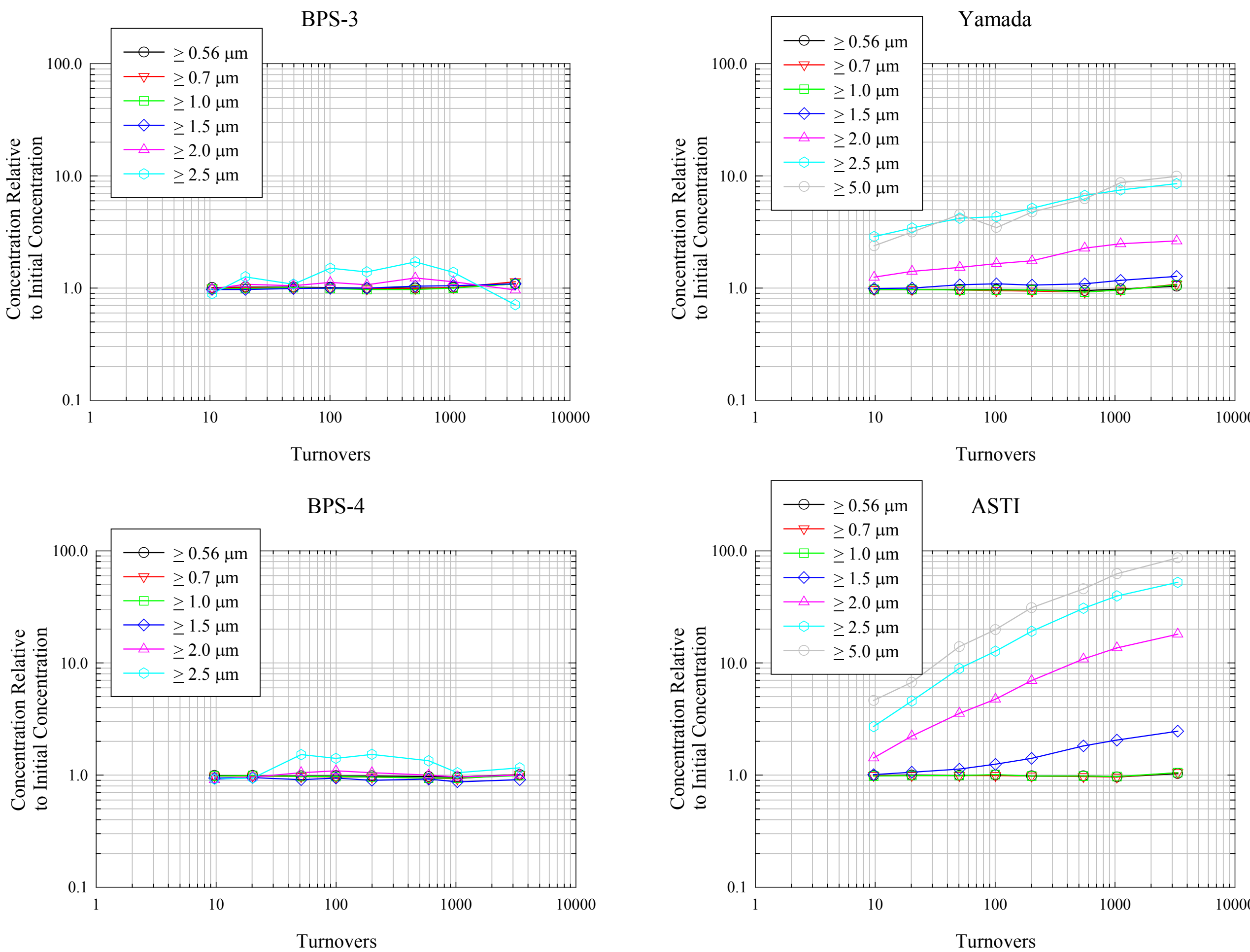


Figure 4 presents the particle concentration ratios during each pump test as a function of particle size after approximately 100 and 1,000 turnovers. These points were chosen since slurry is typically turned over on the order of 100 times prior to use while 1,000 turnovers is probably a conservative upper estimate in most slurry delivery systems.

Minimal changes were observed in the PSD during the BPS-3 and BPS-4 pump tests relative to the other two pumps. Meanwhile, large increases in the large particle tail were observed for particles ≥ 2 µm for both the Yamada and ASTI pumps. Growth in the large particle tail probably occurred at smaller size channels as well, but since the concentrations of particles were higher at these smaller sizes, no increase was observed. After 100 turnovers, particle concentrations ≥ 2 µm increased by about a factor of 1.7 during the Yamada pump test and by a factor of about 5 during the ASTI pump test. After 1,000 turnovers, particle concentrations ≥ 2 µm increased by factors of about 2.5 and 14 during the Yamada and ASTI pump tests, respectively. This equates to about a 0.25% and 1.4% increase in the particle concentrations ≥ 2 µm during each pass through the pump for the Yamada and ASTI pumps, respectively. For the Yamada and ASTI pump tests, the actual concentration ratios are likely even higher than indicated for particles ≥ 3 µm, since the initial particle concentration for these large sizes was indistinguishable from the background of the test system.

Figure 4: Concentration increases measured during all tests after 100 and 1,000 turnovers

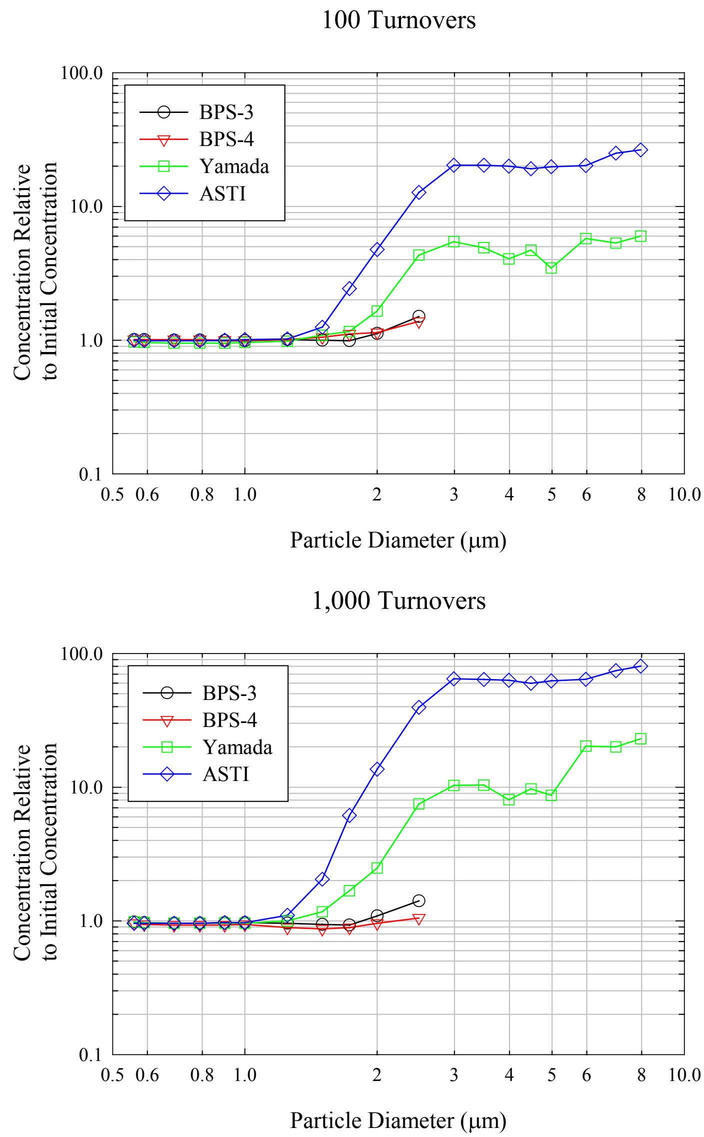


Table II shows a summary of the relative particle concentration changes during each test after approximately 100 and 1,000 turnovers for selected particle sizes.

Figure 5 presents the volume-weighted mean and 99th percentile particle diameters (99% of the particles have diameters less than this size) of the working PSD as a function of tank turnovers. Error bars are included and represent ± 3 standard deviations. The volume-weighted mean and 99th percentile particle diameters at the start of each test were 99 and 229 nm, respectively. According to the Cabot Microelectronics certificate of analysis, the mean particle size for this slurry is 127 nm, as measured by a different measurement technique. Also included in Figure 5 were the zeta potential measurements taken during each test. The zeta potential of this slurry was +21 mV. No significant change in the working PSD or zeta potential was observed during any of the tests.

Table II. Summary of the relative particle concentrations changes for selected times and particle sizes

Particle Concentrations Relative to Initial Particle Concentrations										
Particle Size	100 Turnovers					1000 Turnovers				
	Yamada	BPS-3	BPS-4	BPS-4 HP	ASTI	Yamada	BPS-3	BPS-4	BPS-4 HP	ASTI
≥ 0.56 µm	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
≥ 1.0 µm	1.0	1.0	1.0	1.0	1.0	1.0	1.0	0.9	1.0	1.0
≥ 1.5 µm	1.1	1.0	0.9	0.9	1.3	1.2	1.0	0.9	1.0	2.1
≥ 2.0 µm	1.7	1.1	1.1	1.2	4.8	2.5	1.1	1.0	1.0	13.6
≥ 5.0 µm	>2	-	-	-	>20	>5	-	-	-	>50
≥ 10 µm	>2	-	-	-	>20	>10	-	-	-	>50

Figure 5: Working particle size and zeta potential measurements as a function of turnovers

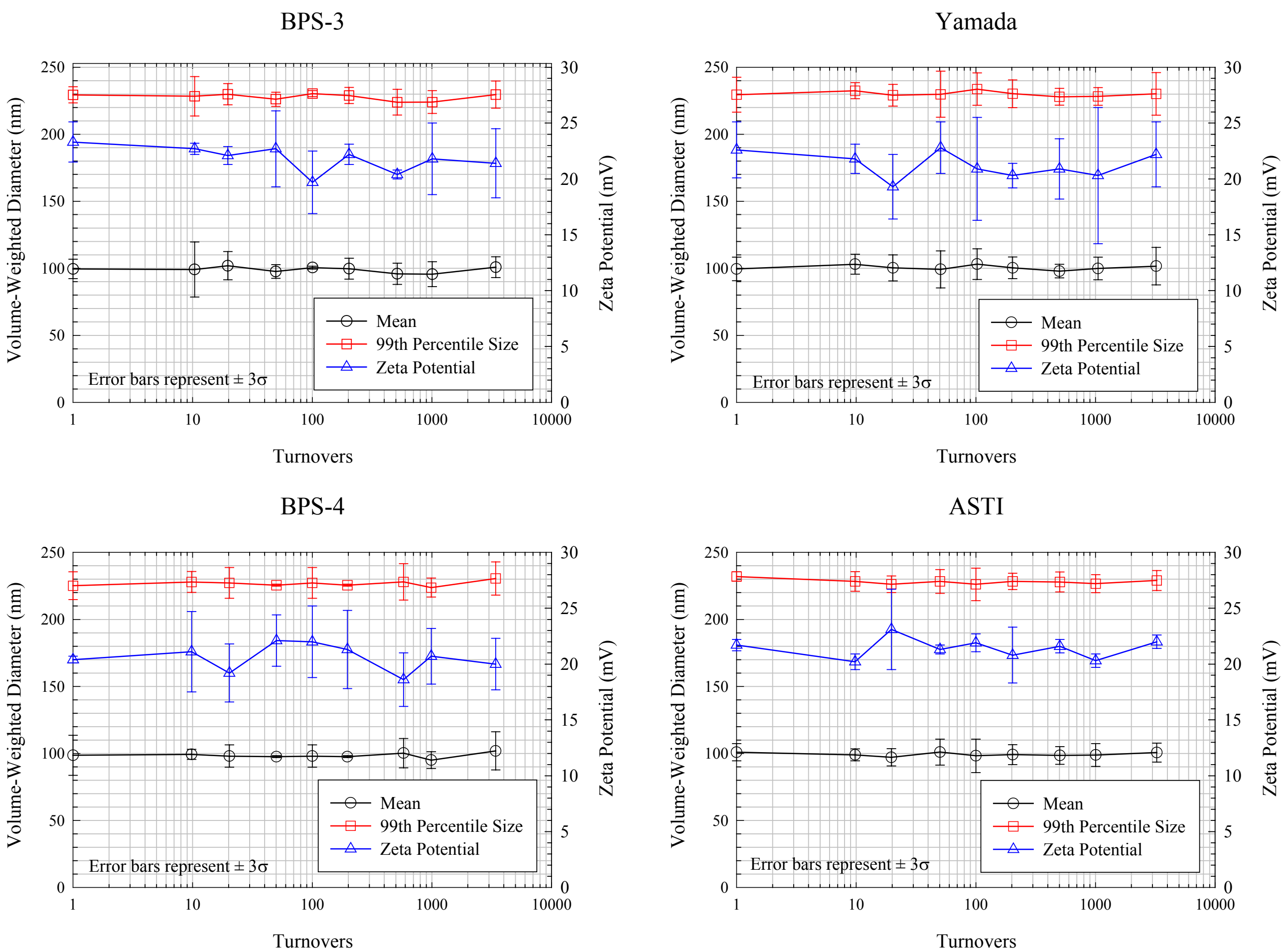


Figure 6 presents the total percent solids, specific gravity, and pH as a function of tank turnovers during each test. Error bars are included in each graph and represent ± 3 standard deviations. No changes in these slurry properties were observed during any of the tests.

Table III presents the average temperature difference across each pump during each test. The BPS-3 and BPS-4 pumps increased the temperature by 0.08°C and 0.12°C, respectively. No significant change in temperature was observed across the ASTI and Yamada pumps. The uncertainty in the results is approximately 0.02°C since the sensors were calibrated just prior to the tests.

Figure 6: Total % solids, specific gravity and pH measurements as a function of turnovers

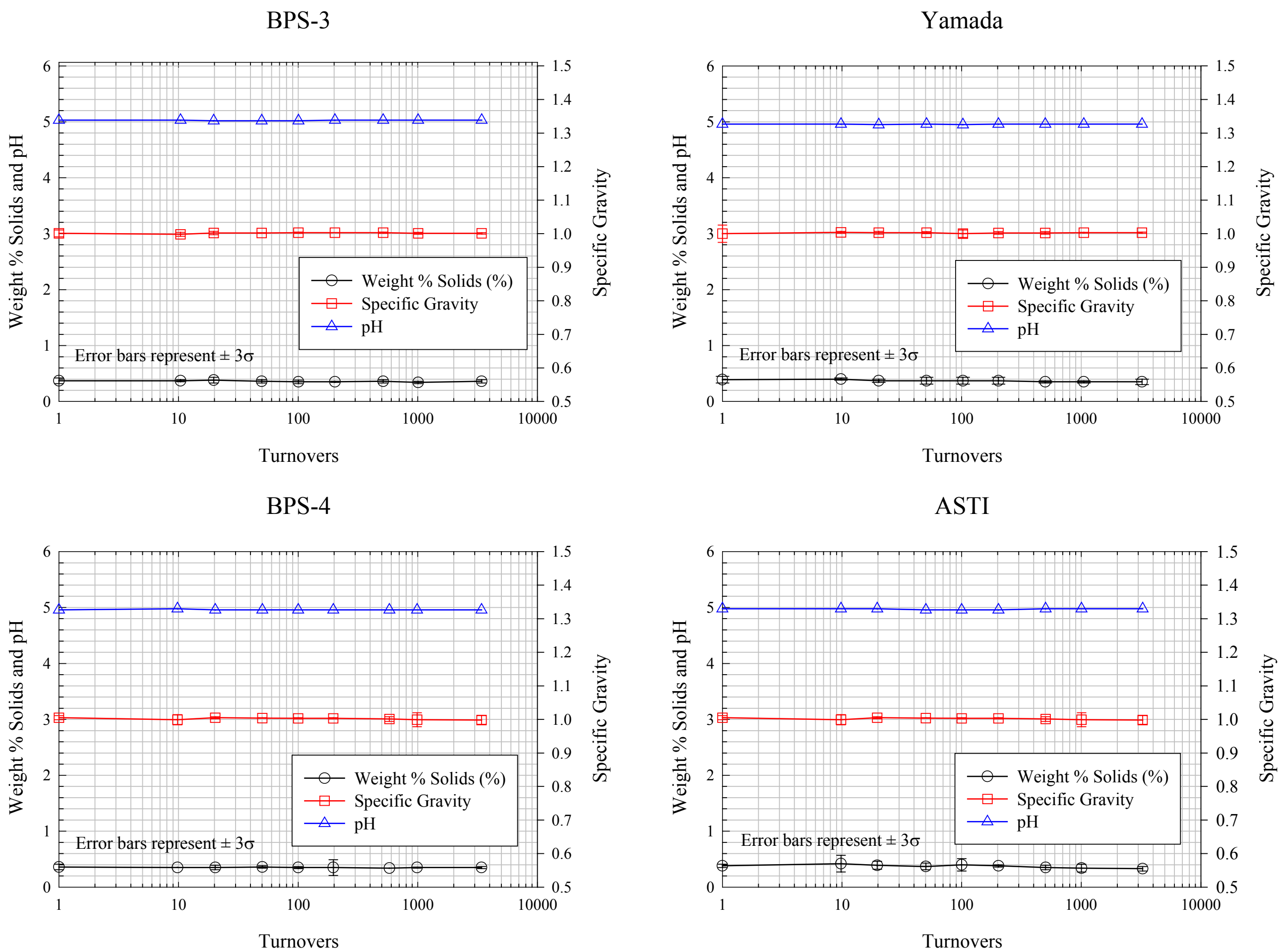


Table III. Average temperature difference across each pump during the tests

Pump	Speed	Flow rate (lpm)	Average Temperature Difference (outlet-inlet) °C
ASTI	NA	30	0.02
BPS-3	7350	30	0.08
BPS-4	5830	30	0.12
Yamada	NA	30	-0.01

Summary

A Yamada diaphragm pump, an ASTI bellows pump, and two Levitronix centrifugal pumps were tested to determine how their use affected the size distribution of particles in Cabot SiLECT™ 6000 slurry. The slurry was circulated until it had passed through each pump a minimum of 3,000 times.

Minimal change in the PSD was observed during the BPS-3 and BPS-4 tests regardless of particle size, while significant increases in the large particle concentrations, particularly for particles ≥ 2 μm, were observed during the Yamada and ASTI pump tests. For particles ≥ 2 μm, the concentration increases were substantial for the both the ASTI and Yamada pumps. After 100 turnovers, the concentration of particles ≥ 2 μm increased by factors of 5 and 1.7 for the ASTI and Yamada pumps, respectively. After 1,000 turnovers, the concentration of particles ≥ 2 μm increased by factors of at least 14 and 2.5 for the ASTI and Yamada pumps, respectively.

No significant changes were observed in the other slurry properties (working PSD, total percent solids, pH, density, and zeta potential) during any of the pump tests.

The average temperature increase across the BPS-3 and BPS-4 pumps was 0.08°C and 0.12°C, respectively. No significant temperature change was observed across the Yamada or ASTI pumps.

References

1. Personnel communication with J. Kvalheim, BOC Edwards Chemical Management Division, Chanhassen, MN, March, 2003.
2. CTA Report #: LTX 775 0935. M. Litchy (2004). "Comparison of Three Pump Systems on the Particle Size Distribution of Cabot Semi-Sperse® 12 Slurry".
3. M. Litchy and R. Schoeb, "Effect of shear stress and pump method on CMP slurry," *Semiconductor International* **27** (12), 87-90 (2004).
4. Litchy MR and R Schoeb (2005), "Effect of particle size distribution on filter lifetime in three slurry pump systems," *Materials Research Society Symposium Proceedings* Vol. 867, W2.8.1, (2005).
5. CTA Report #: LTX 979 1589. M. Litchy (2006). "Comparison of Four Pump Systems on the Particle Size Distribution of Cabot iCue® 600Y75 Slurry".