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Comparison of Three Pump Systems on the Particle Size Distribution of Cabot WIN™ W7300-A18 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 pump types. 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]. These tests were performed to determine the effect of circulating Cabot Microelectronics WIN™ W7300-A18 with three different pumps (bellows, diaphragm, 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 pump. 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 a Levitronix magnetically levitated centrifugal pump. Table I provides pump details. 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.

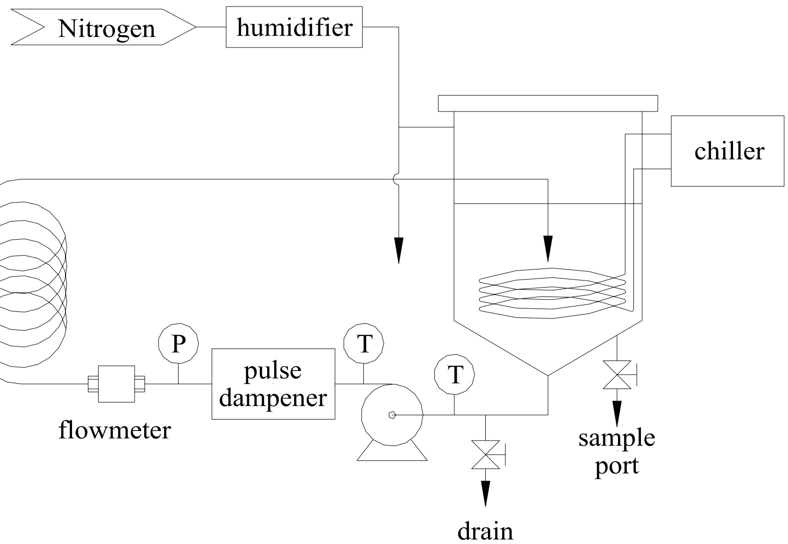
Table I. Specifications of the three pump systems

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-4	No	NA

Each pump was used to circulate 11 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 speed of the BPS-4 pump was adjusted to 5800 rpm to achieve the desired flow rate and pressure. In each test, the slurry was circulated until more than 1,000 tank turnovers were achieved. The test system was constructed of PFA, except for the conical bottom tank that was constructed of polyethylene. All of the tests were performed using slurry from the same lot.

Prior to the start of the test, the slurry was mixed using a 2.7" diameter Lightnin® A-100 impeller at speed of approximately 300 rpm for 15 minutes. The slurry was immediately blended with 30% hydrogen peroxide (J.T. Baker, CMOS grade) to obtain a 1.0% (by weight) H_2O_2 concentration. The amount of H_2O_2 added depends on the process, but typically ranges from 0.5-1.0 (wt%). Cabot Microelectronics provided these recommendations.

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 20 ± 2°C during the test.

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. Measurement of particle size distribution was performed by diluting the slurry sample by a factor of approximately 100:1 with filtered deionized water. Between samples, the entire system was thoroughly flushed with deionized water. Data from selected particle size channels were analyzed.

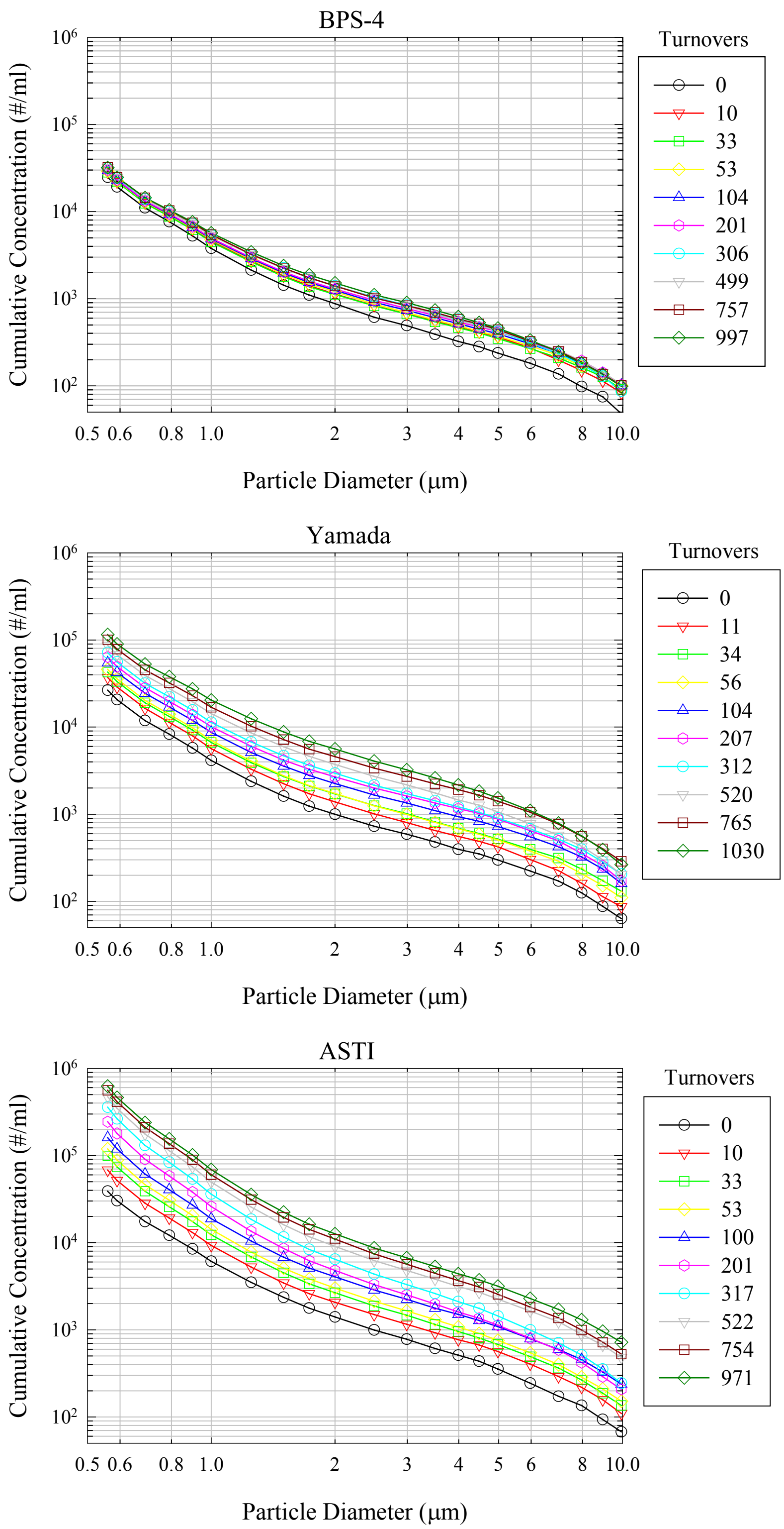
The zeta potential and working PSD measurements were made using the NICOMP 380ZLS. The zeta potential samples were diluted approximately 40:1 into deionized water while the size distribution samples were diluted by approximately 6:1 into deionized water. All measurements were performed at a temperature of 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 instrument's Gaussian distribution assumption.

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

Results and Discussion

The three 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



Small changes in the large particle tail of the slurry PSD were observed during the centrifugal pump test. Meanwhile, significant increases in large particle concentrations were observed during the Yamada and ASTI pump tests over a wide range of particle sizes.

Figure 3 presents the ratios of particle concentrations at each test point to the particle concentration at the start of the test. 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

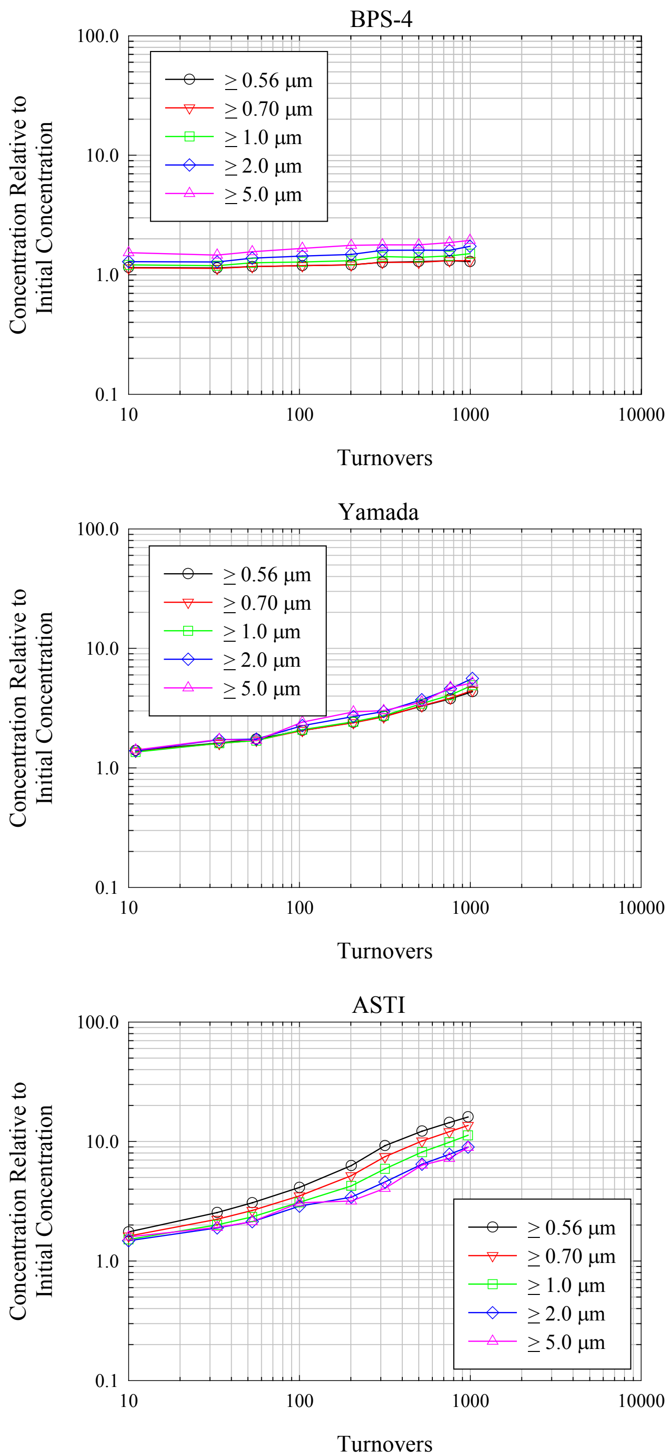
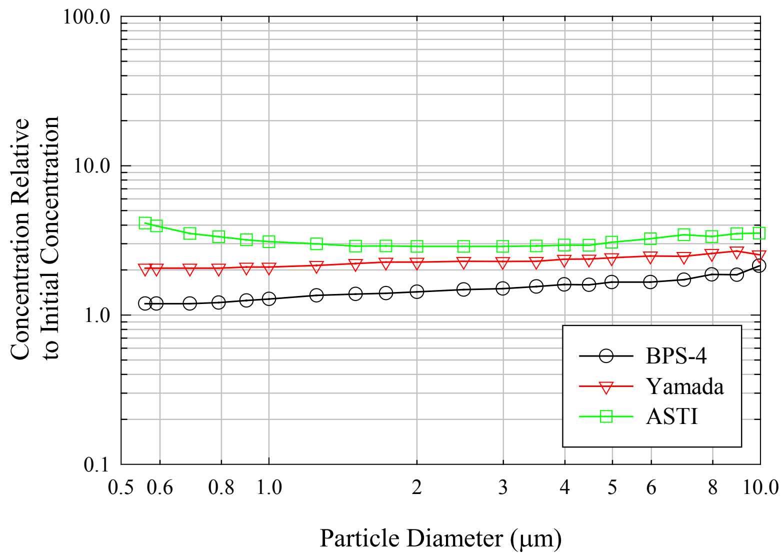


Figure 4 presents the particle concentration ratios during each pump test as a function of particle size after approximately 100 and 1,000 turnovers. These points were chosen since slurry is typically turned over on the order of 100 times prior to use, while 1,000 turnovers is believed to be a conservative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

For the BPS-4 pump, the concentration increases appeared to increase slightly as particle size increased, while the concentration increases appeared to go through a minimum as particle size approached 2 µm for the ASTI pump. Little dependence on particle size was observed during the Yamada pump test. Overall, the particle concentration increases appeared to be roughly independent of particle size.

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

100 Turnovers



1,000 Turnovers

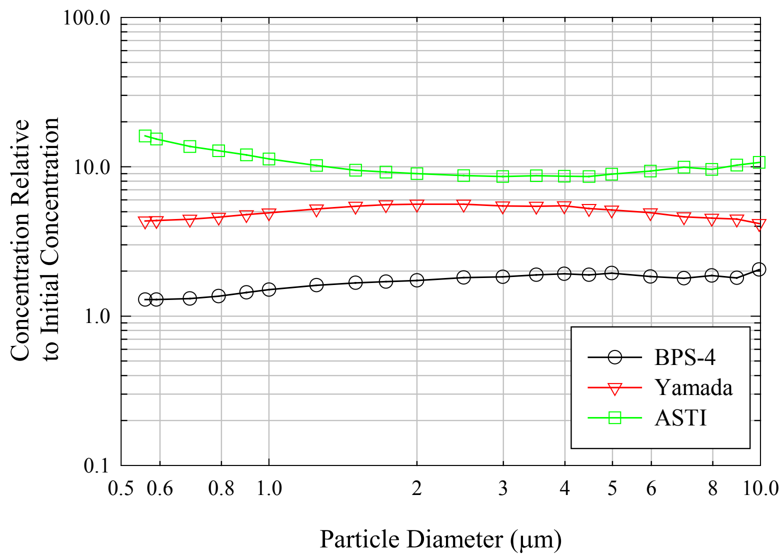


Table II shows a summary of the relative particle concentration changes during each test after approximately 100 and 1,000 turnovers for selected particle sizes.

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

Particle Concentrations Relative to the Initial Particle Concentrations						
Particle Size	100 Turnovers			1,000 Turnovers		
	BPS-4	Yamada	ASTI	BPS-4	Yamada	ASTI
≥ 0.56 µm	1.2	2.1	4.1	1.3	4.3	16.1
≥ 1.0 µm	1.3	2.1	3.1	1.5	4.9	11.3
≥ 2.0 µm	1.6	2.3	2.9	1.7	5.6	9.0
≥ 5.0 µm	2.1	2.4	3.1	1.9	5.1	8.9
≥ 10.0 µm	2.1	2.5	3.5	2.1	4.2	10.7

Relatively small changes were observed in the PSD during the BPS-4 pump test relative to the other two pump tests. Meanwhile, substantial increases in the large particle tail were observed over a wide range of particle sizes for both the Yamada and ASTI pump tests. After 100 turnovers, concentrations of particles ≥ 0.56 µm increased by 110% during the Yamada pump test and by more than 300% during the ASTI pump test. Meanwhile, the concentrations of particles ≥ 0.56 µm increased by only about 20% during the BPS-4 pump test. After 1,000 turnovers, concentrations of particles ≥ 0.56 µm increased by about 330% and 1500% during the Yamada and ASTI pump tests, respectively, while concentrations increased by only 30% during the BPS-4 pump test.

The increase in particle concentration appeared non-linear when plotted versus turnovers as shown in Figure 5. The rate of concentration increase decreased with increasing turnovers.

Figure 6 presents the volume-weighted mean and 99^{th} 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 initial values were included on the plot at 1 turnover.) The volume-weighted mean and 99^{th} percentile particle diameters at the start of each test were 42 and 79 nm, respectively. Also included in Figure 5 were the zeta potential measurements taken during each test. The zeta potential of this slurry was approximately 0 mV. No significant change in the working PSD or zeta potential were observed during any of the tests.

Figure 7 presents the total percent solids, specific gravity, pH, and hydrogen peroxide concentration as a function of tank turnovers during each test. Error bars are included in each graph and represent ± 3 standard deviations. (The initial values were included on the plot at 1 turnover.) No changes in these slurry properties were observed during any of the tests.

Figure 5. Particle concentrations as a function of turnovers for selected size channels on linear axes

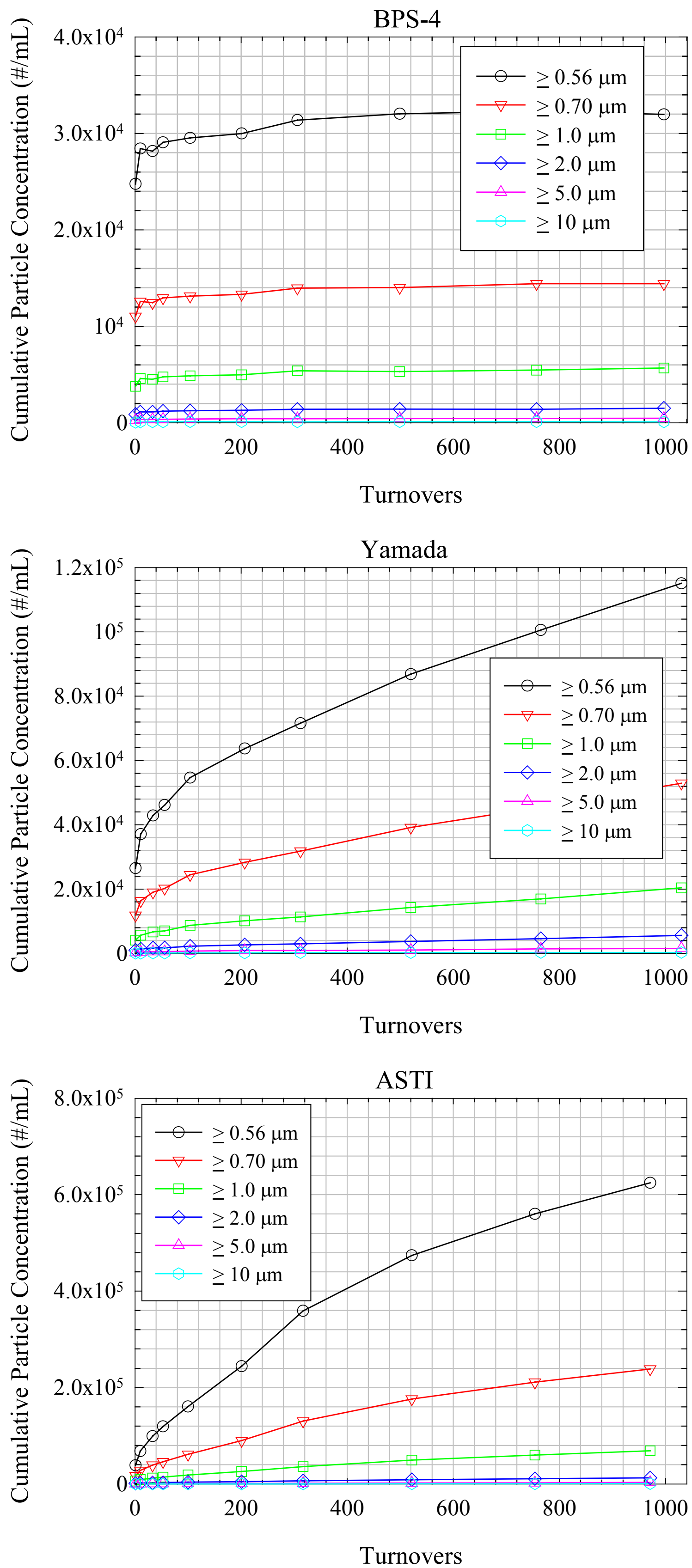


Figure 6. Working particle size and zeta potential measurements as a function of turnovers

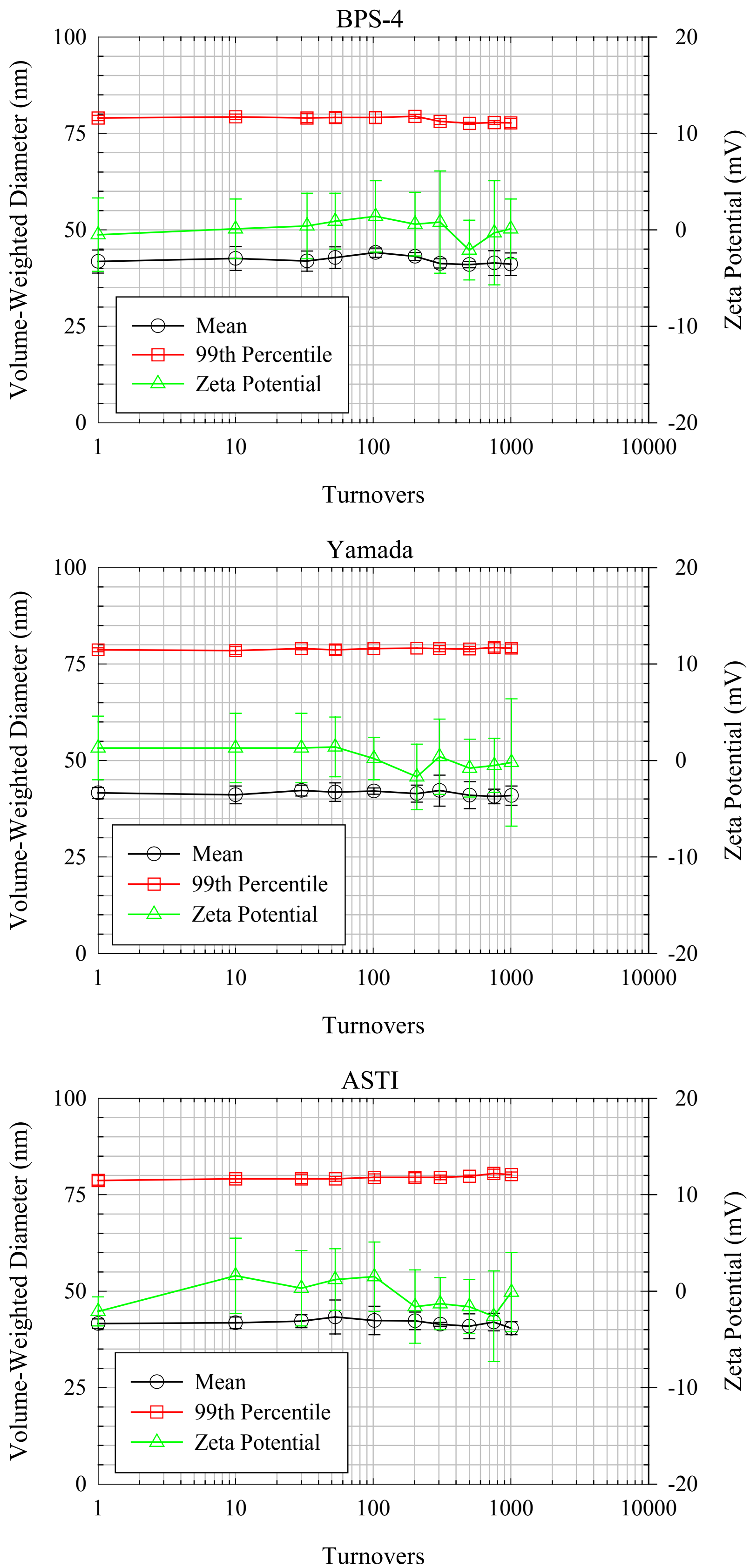
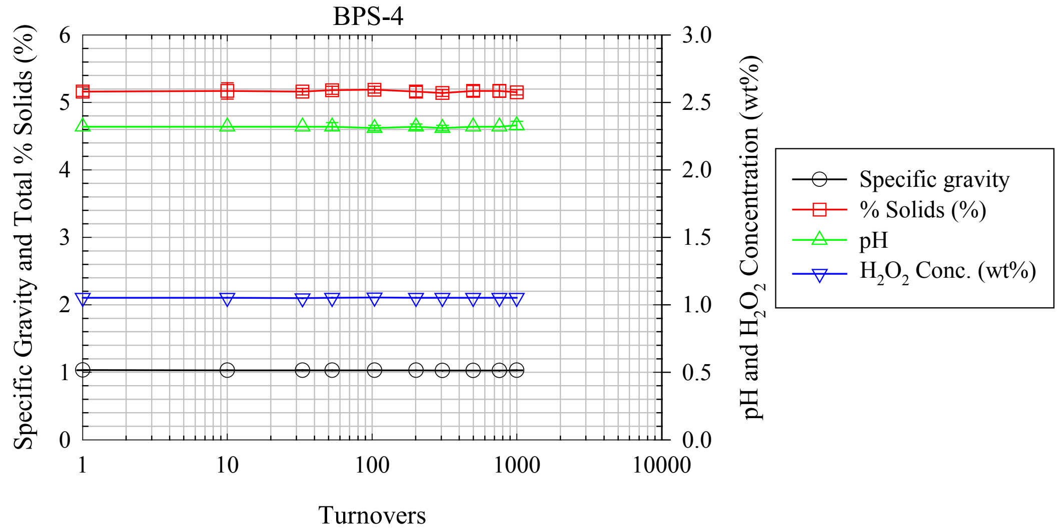
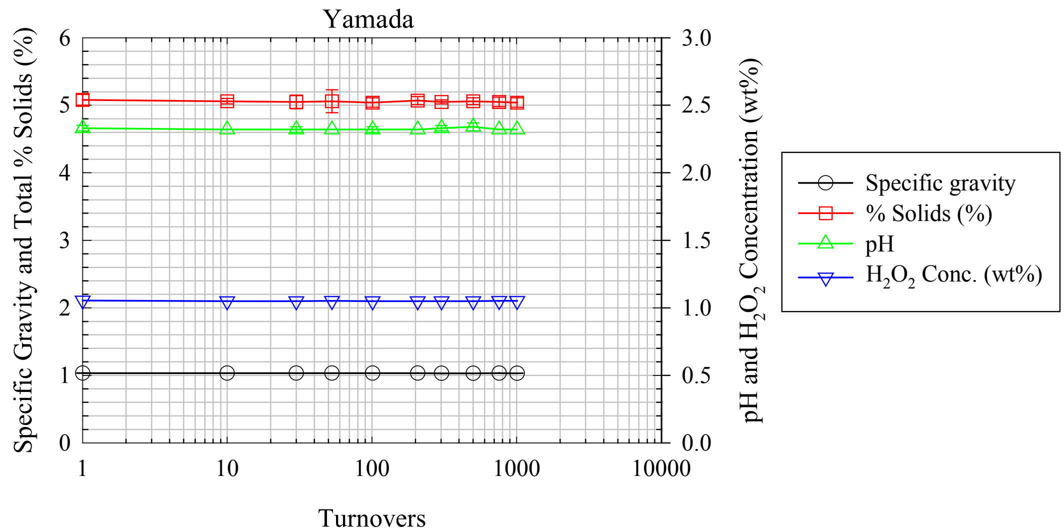
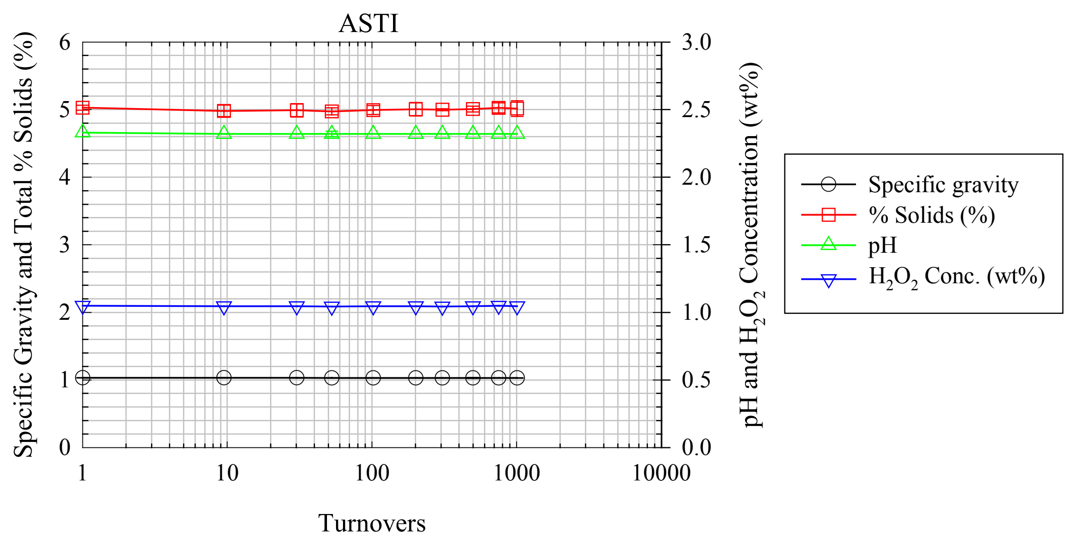


Figure 7. Total % solids, specific gravity, pH, and H_2O_2 concentration measurements as a function of turnovers







Summary

A Yamada diaphragm pump, an ASTI bellows pump, and a Levitronix centrifugal pump were tested to determine how their use affected the size distribution of particles in Cabot WIN™ W7300-A18 slurry. The slurry was circulated until it had passed through each pump approximately 1,000 times.

Relatively small changes in the PSD were observed during the BPS-4 pump test, while larger increases in the large particle concentrations were observed during the Yamada and ASTI pump tests. After 100 turnovers, the concentration of particles ≥ 0.56 μm increased by factors of 1.2, 2.1 and 4.1 for the BPS-4, Yamada, and ASTI pumps, respectively. After 1,000 turnovers, the concentration of particles ≥ 0.56 μm increased by factors of 1.3, 4.3, and 16.1 for the BPS-4, Yamada, and ASTI pumps, respectively. The particle concentration increases appeared to be roughly independent of particle size during each pump test. The rate of increase in particle concentration decreased with increasing turnovers during each test.

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

References

1. Personnel communication with J. Kvalheim, BOC Edwards Chemical Management Division, Chanhassen, MN, March, 2003.
2. Litchy MR and DC Grant (2007). "Effect of pump type on the health of various CMP slurries", *Semiconductor Fabtech*, 33rd Edition, pp 53-59.