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Effect of Levitronix BPS-200 and BPS-600 Pump Systems on the Particle Size Distribution of Cabot Semi-Sperse® 12 Slurry

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Introduction

Delivery systems often 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, 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 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 Semi-Sperse® 12 (lot #: G080925010) silica slurry with two new Levitronix pumps (BPS-200 and BPS-600) 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.

Experimental

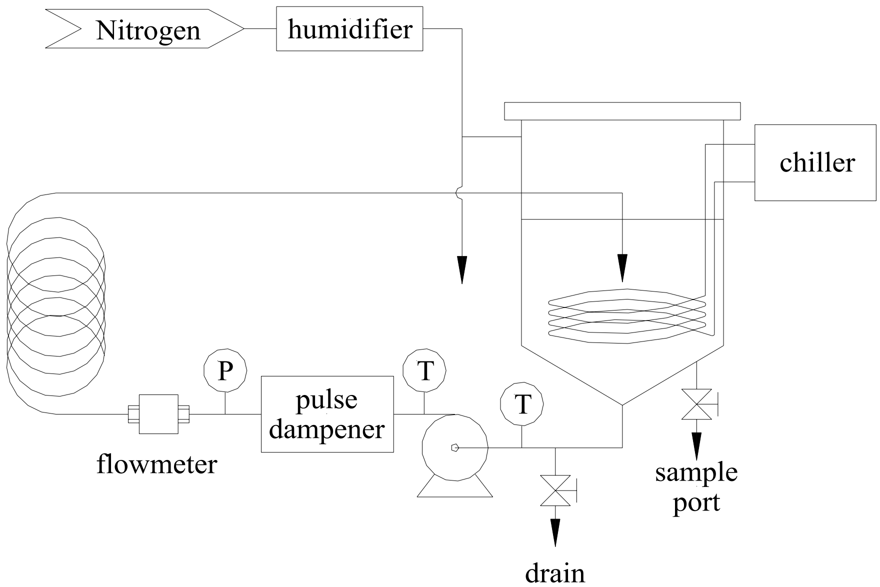
The BPS-200 pump was used to circulate 14.0 liters of slurry at a flow rate of approximately 4.8 lpm (1.3 gpm) and outlet pressure of 30 psig (2.1 bar), while the BPS-600 pump was used to circulate 12.5 liters of slurry at a flow rate of 30 lpm (7.9 gpm) and outlet pressure of 30 psig. Settling of the 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 3 minutes. 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, different lengths of PFA tubing were used to reduce the pressure at the pump outlet to ambient pressure at the end of the return line to the tank. The speed of the pumps was adjusted to achieve the desired flow rate and pressure. The BPS-200 pump was operated at a speed of 9,200 rpm, while the BPS-600 was operated at 7,700 rpm. 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.

Prior to the start of the test, the Semi-Sperse® 25E was slowly diluted with ultra pure water (approximately 1:1) while being agitated to form Semi-Sperse® 12.

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. Shifts in the pH and dehydration can both result in slurry particle agglomeration. 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. Both the large particle tail and the working particle size distributions (PSDs) were measured. The large particle tail PSD 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. The slurry samples were diluted approximately 1,700:1 with filtered deionized water prior to measurement of the PSD. Between samples, the entire system was thoroughly flushed with deionized water. Data from selected particle size channels were analyzed.

Figure 1. Test system schematic



The size of the working particles was measured using two techniques. In the first technique, the slurry was diluted 20,000:1 into filtered deionized water, the particles in the diluted slurry were dispersed into air using ultrafine atomization (UFA) and the size and concentration of the particles was measured using a scanning mobility particle sizer (SMPS) [3]. The working PSD was also measured using a Particle Sizing Systems NICOMP 380ZLS (Santa Barbara, CA) that determines particle size by dynamic light scattering. The particle zeta potential was also measured using this instrument. The zeta potential samples were diluted approximately 75:1 into deionized water while the size distribution samples were diluted approximately 38:1 into deionized water. These 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, and pH. Measurements of each sample were performed in triplicate.

Results and Discussion

Figure 2 shows the cumulative PSDs of the slurry large particle tail. The initial PSD, measured prior to the start of each test, is presented as well as PSDs after selected numbers of turnovers.

Figure 3 presents the ratios of particle concentrations at each test point to the particle concentration at the start of each test. The concentration ratios for selected size channels are presented as a function of tank turnovers.

Figures 2 and 3 indicate that there were relatively small changes in the large particle tail of the slurry PSD during both pump tests. During the BPS-200 pump test, the concentration of particles ≥ 2µm decreased with increasing turnovers. This effect increased as particle size increased. A possible explanation for the decrease in the large particle concentrations is that the pump may have been effective at breaking up some loosely held agglomerates. It appears less likely that the larger slurry agglomerates settled out during the test. Although there was less mixing in the tank since the flow rate during this test was only 5 lpm, compared to

30 lpm during the BPS-600 test, the time to turn the volume in the tank over was short (less than 3 minutes). Significant particle settling would not be expected under these conditions. Meanwhile, no measurable change in the concentration of large particles occurred during the BPS-600 pump test.

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

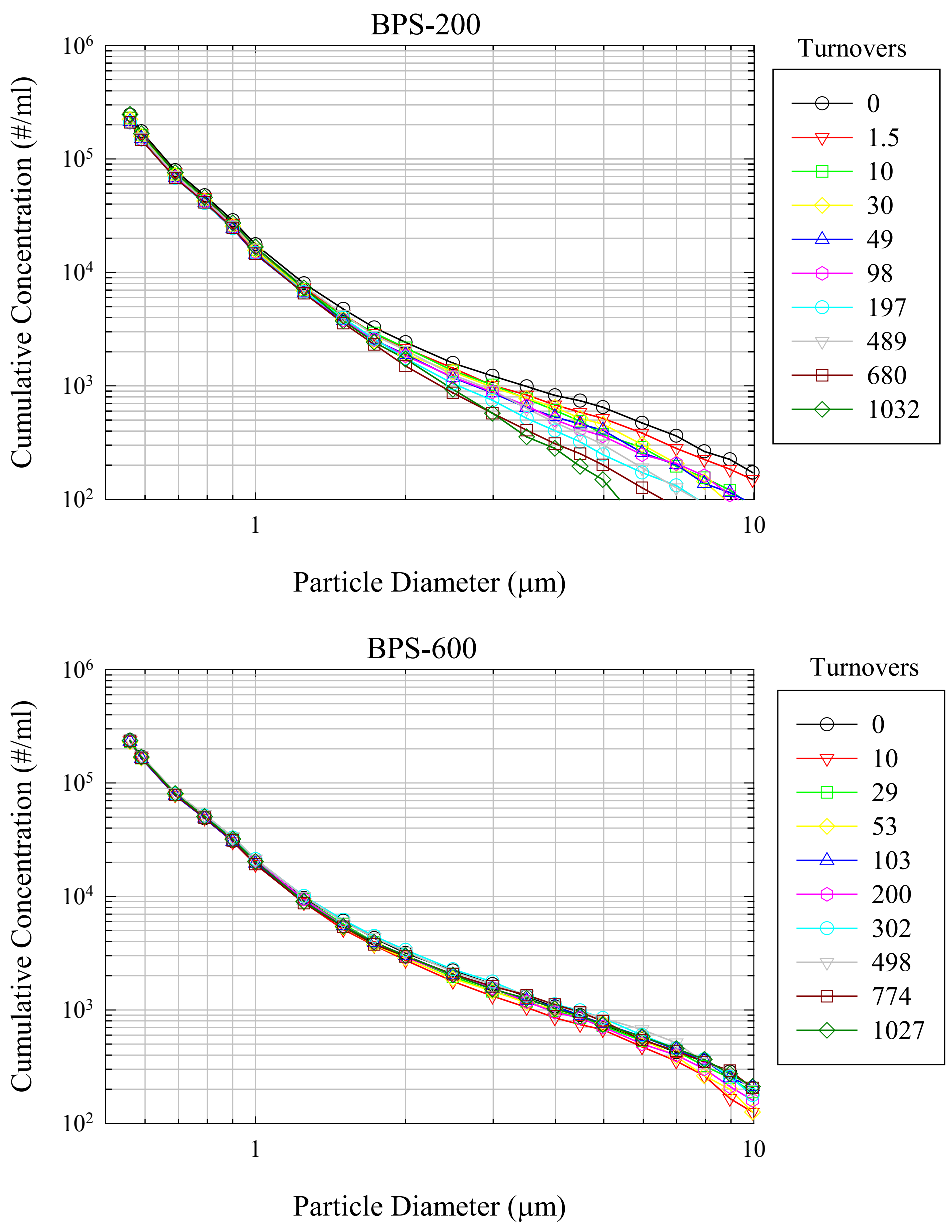
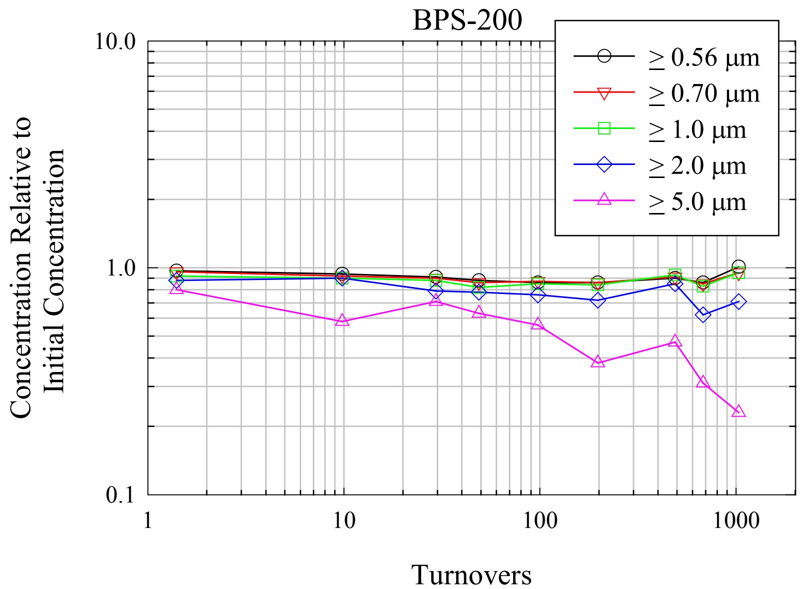


Table I shows a summary of the relative particle concentration changes during each test after approximately 100 and 1,000 turnovers for selected particle sizes. These points were chosen since slurry is typically turned over on the order of 100 times prior to use, while 1,000 turnovers is believed to be a conservative upper estimate in most slurry delivery systems.

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



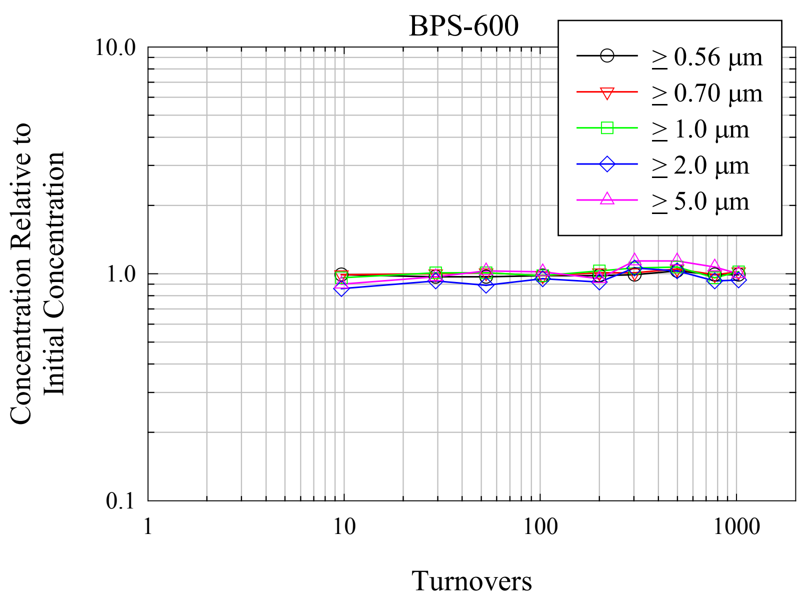


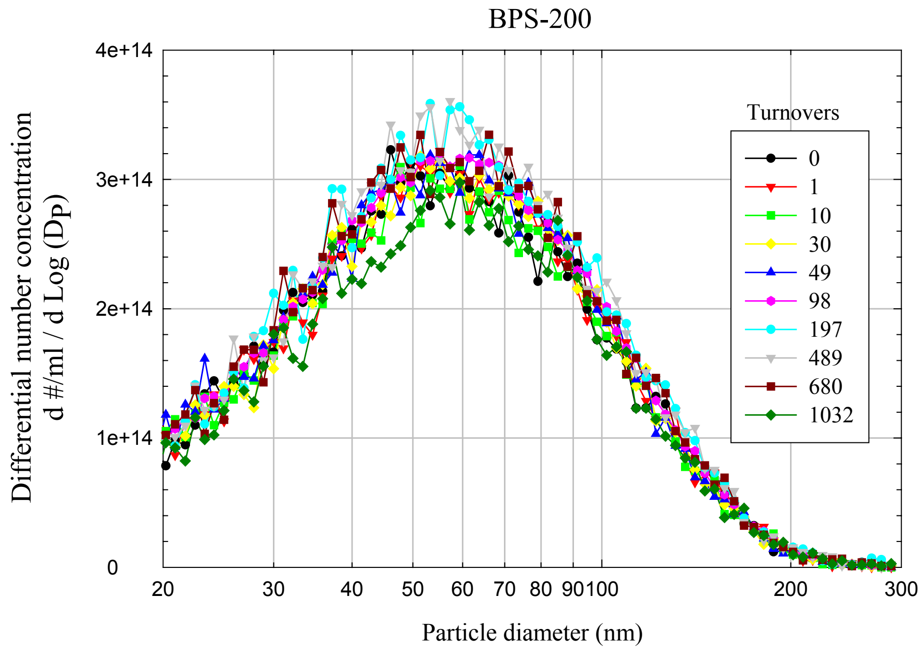
Table I. Summary of the relative particle concentrations changes for selected times and particle sizes

Particle Concentrations Relative to the Initial Particle Concentrations				
Particle Size	100 Turnovers		1,000 Turnovers	
	BPS-200	BPS-600	BPS-200	BPS-600
≥ 0.56 µm	0.9	1.0	1.0	1.0
≥ 1.0 µm	0.9	1.0	1.0	1.0
≥ 2.0 µm	0.8	1.0	0.7	0.9
≥ 5.0 µm	0.6	1.0	0.2	1.0
≥ 10.0 µm	<0.5	1.0	<0.5	1.0

Figures 4 and 5 present working particle size distributions measured using the UFA/SMPS technique. Figure 4 presents number-weighted distributions, while Figure 5 presents volume-weighted distributions. The graphs indicate that the distribution has a single mode with a number-weighted peak at approximately 55 nm and a volume-weighted peak at approximately 125 nm.

Figures 4 and 5 indicate that there was little change in either the number-weighted or the volume-weighted PSDs during either test. This is shown further in Figure 6, which presents cumulative number concentrations of selected particle sizes as a function of turnovers.

Figure 4. Differential number-weighted working particle PSDs



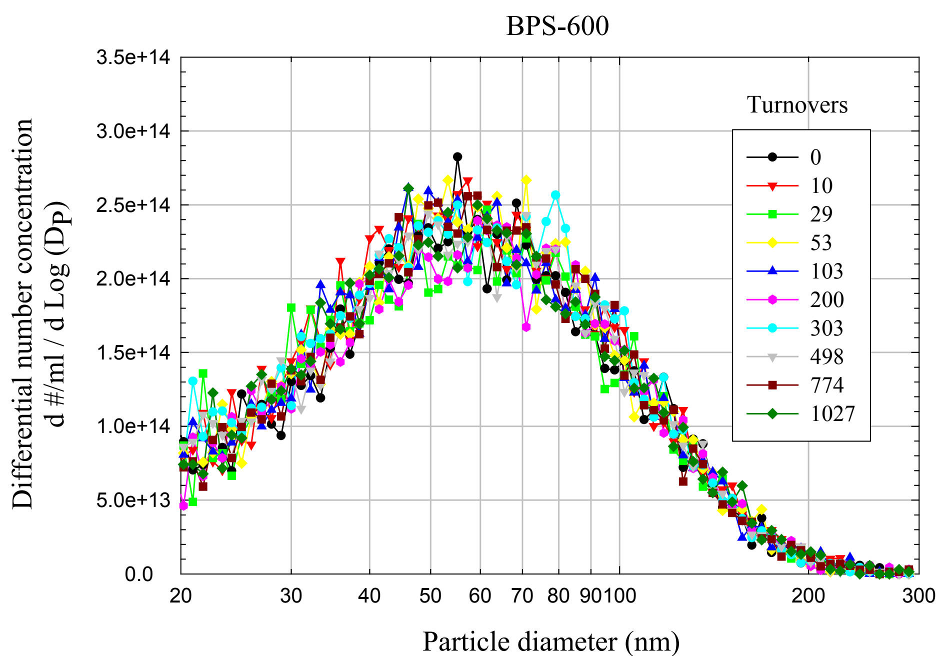
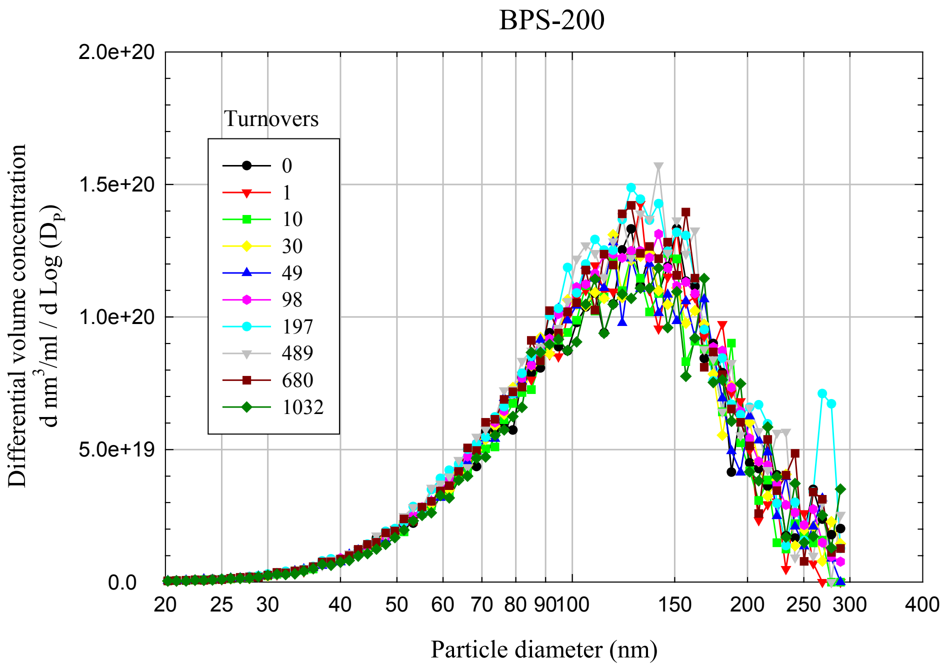


Figure 5. Differential volume-weighted working particle PSDs



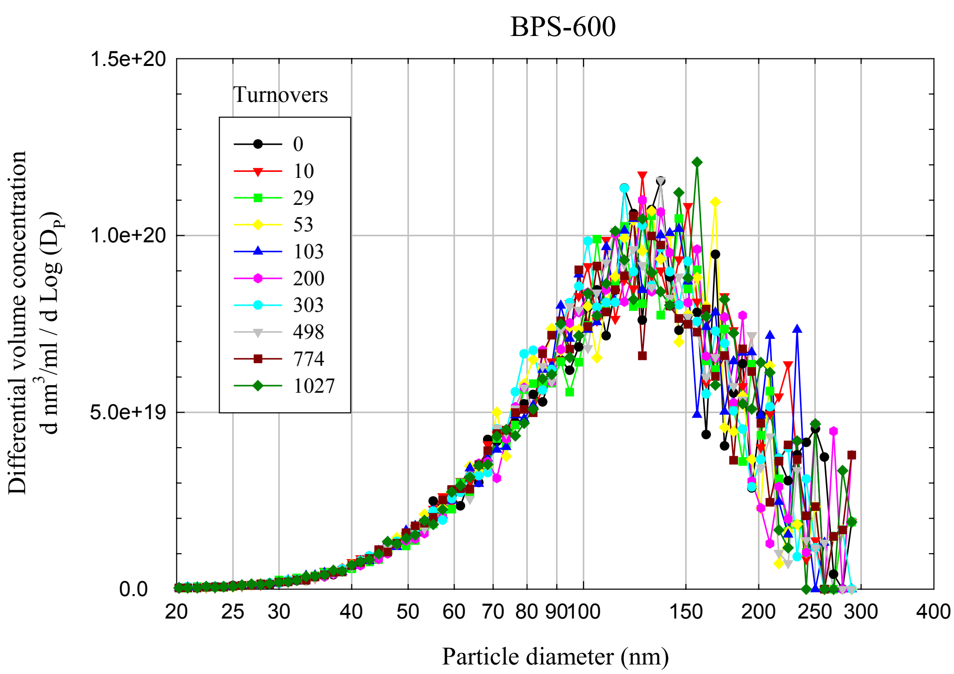
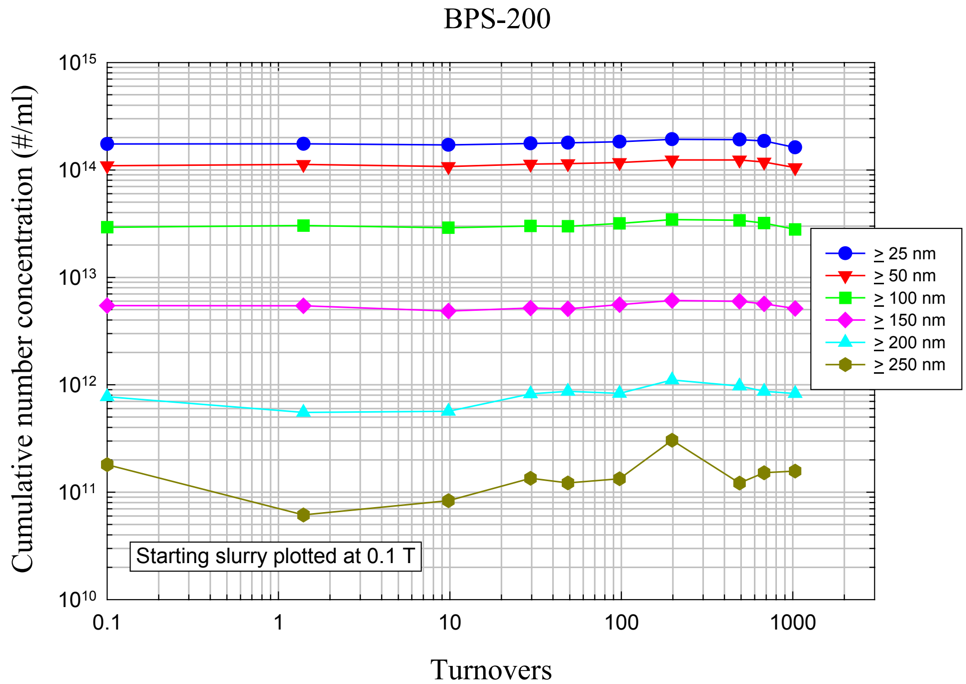
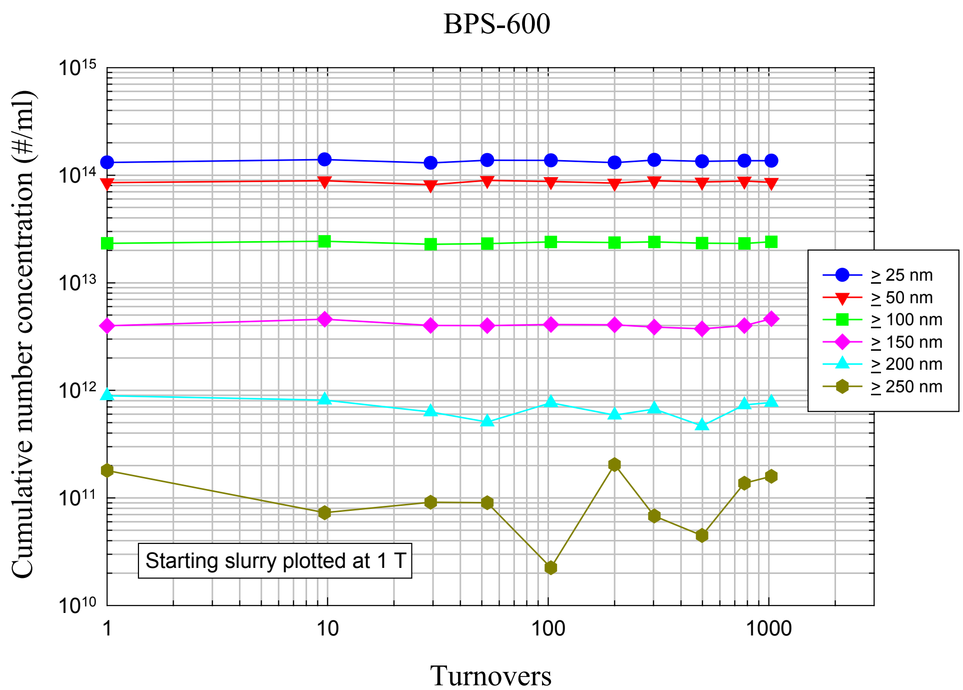


Figure 7 presents the volume-weighted mean and 99th percentile particle diameters (99% of the particles have diameters less than this size) of the working PSD measured by dynamic light scattering as a function of tank turnovers. Error bars are included and represent ± 3 standard deviations. (The initial values were included on the plot at 1 turnover.) The volume-weighted mean and 99th percentile particle diameters at the start of the test were 131 and 257 nm, respectively. The mean size of 131 nm agrees with the volume-weighted size distributions measured using UFA/SMPS (Figure 5). According to Cabot Microelectronics Certificate of Analysis, the mean particle size of this slurry is 141 nm, as measured by a different measurement technique.

Figure 6. Cumulative working particle concentrations as a function of turnovers

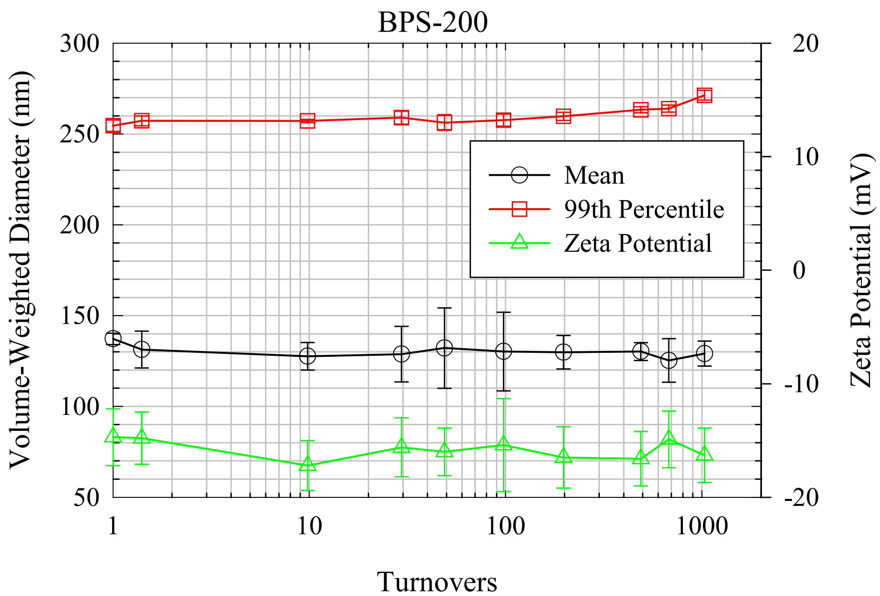




No change in the volume-weighted mean was observed in either pump test. At high turnovers, a slight broadening of the working PSD was observed during both pump tests. Within 200 turnovers, no measurable change in the 99th percentile particle size was observed in either test; however, the 99th percentile particle size increased about 5% and 2.5% after 1,000 turnovers during the BPS-200 and BPS-600 pumps, respectively.

Also included in Figure 7 were the zeta potential measurements taken during the test. The zeta potential of this slurry was approximately -16 mV. No significant changes in particle zeta potential were observed during these tests.

Figure 7. Working particle size and zeta potential measurements as a function of turnovers



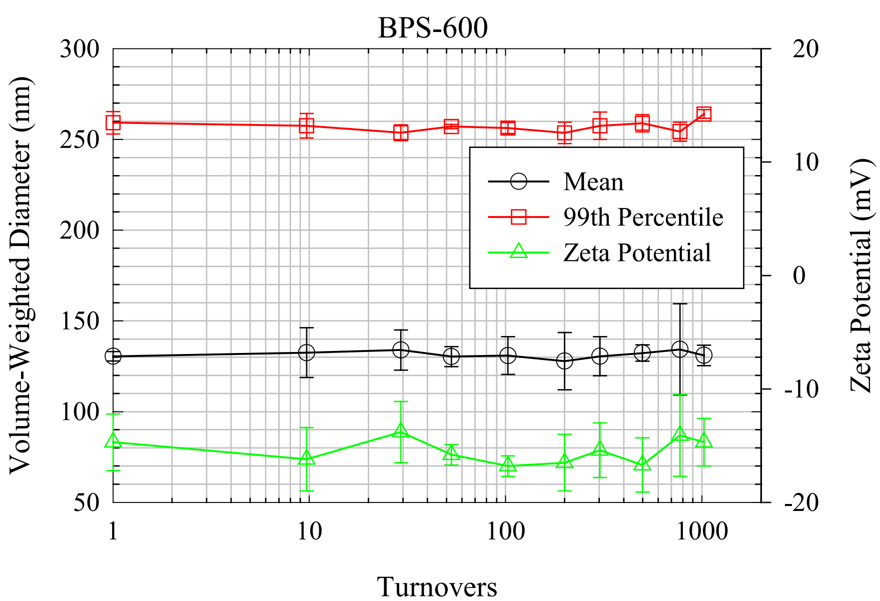
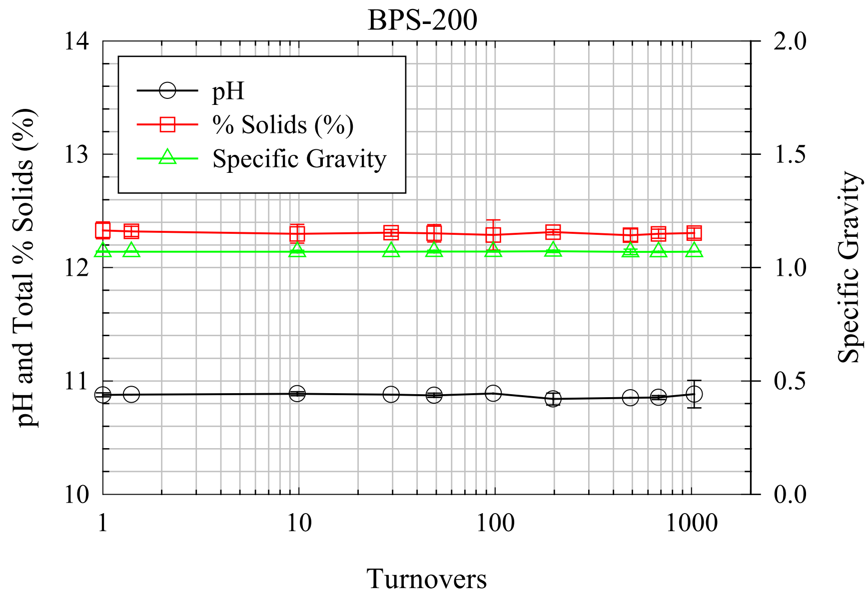
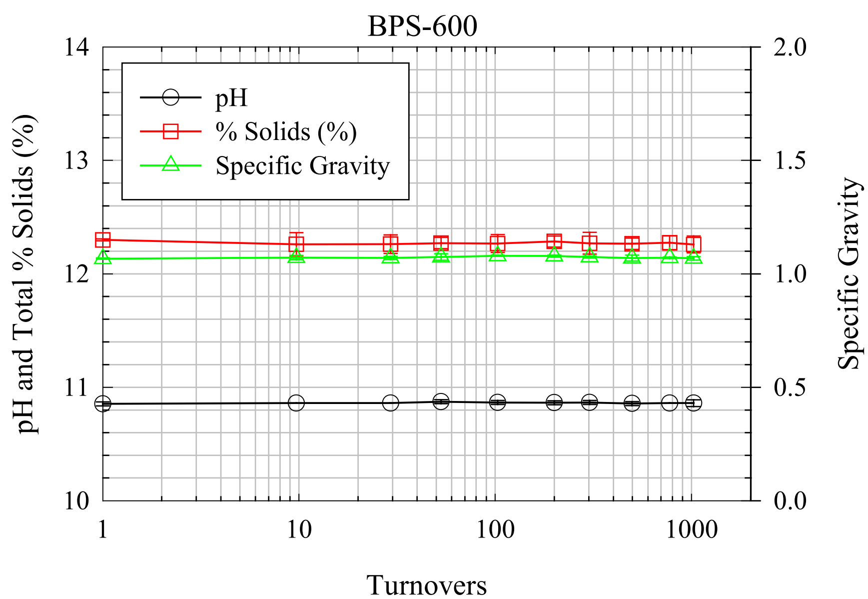


Figure 8 presents the total percent solids, specific gravity, and pH as a function of tank turnovers. Error bars are included in each graph and represent ± 3 standard deviations. (The initial values were included on the plot at 1 turnover.) No changes in these slurry properties were observed during the tests.

Figure 8. Total % solids, specific gravity, and pH measurements as a function of turnovers





Summary

BPS-200 and BPS-600 magnetically levitated centrifugal pumps were tested to determine how their use affected the size distribution of particles in Cabot Microelectronics Semi-Sperse® 12 slurry. In each test, the slurry was circulated until it had passed through each pump approximately 1,000 times.

No increase in the concentration of particles in the large particle tail was observed during either pump test. Rather, the concentration of large particles actually decreased during the BPS-200 pump test. This effect increased as particle size increased. Meanwhile, no measurable change in the concentration of large particles occurred during the BPS-600 pump test.

Little change in the working PSDs were observed by the NiCOMP and UFA/SMPS measurement techniques during the tests. A slight increase (≤ 5%) in the 99th percentile particle size (dynamic light scattering measurement technique) was observed after 1,000 turnovers during the BPS-200 and BPS-600 pump tests, respectively. No change in the volume-weighted mean was observed in either pump test.

No significant changes were observed in any of the other slurry properties measured (total percent solids, pH, density, or zeta potential) during either pump test.

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.
3. Grant DC (2008). "A New Method for Determining the Size Distribution of the Working Particles in CMP Slurries," presented at the 2008 CMP Users Conference, sponsored by Levitronix.