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Comparison of Three Pump Systems on the Particle Size Distribution of Cabot Semi-Sperse® 12 Slurry

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Introduction

Slurries, suspensions of fine particles dispersed in a liquid, are often used in semiconductor chip manufacturing to planarize wafer surfaces. The effectiveness of these slurries in achieving a flat surface free of scratches is highly dependent upon the physical properties of the slurry. Perhaps the most important physical properties of the slurry are the average size of the fine or "working" particles in the slurry and the presence of large particles, often referred to as the large particle tail, which can cause scratches.

Delivery systems are often used to supply the slurry to the planarization tools. 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 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].

Many slurries are easily damaged by mechanical handling. Damage often takes the form of changes in the size distribution of the slurry particles. This test was performed to determine the effect of circulating Semi-Sperse® 12 (Cabot Microelectronics Corporation, Aurora, IL), a suspension of fumed silica particles in dilute potassium hydroxide, with three different types of pumps (bellows, diaphragm, and centrifugal) on the size distribution of the particles in the slurry.

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 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.

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 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 Yamada and ASTI pumps was adjusted to achieve a slurry flow rate of 30 lpm with a 30 psig outlet pressure, while the Levitronix pump was operated at 5900 rpm. 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. The slurry used in each test was taken from the same drum of slurry.

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. A chiller and stainless steel coil were used to maintain the slurry at 20 ± 2°C during the test. The relative humidity in the tank was > 90% throughout 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 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 an AccuSizer 780 optical particle counter (Particle Sizing Systems, Santa Barbara, CA).

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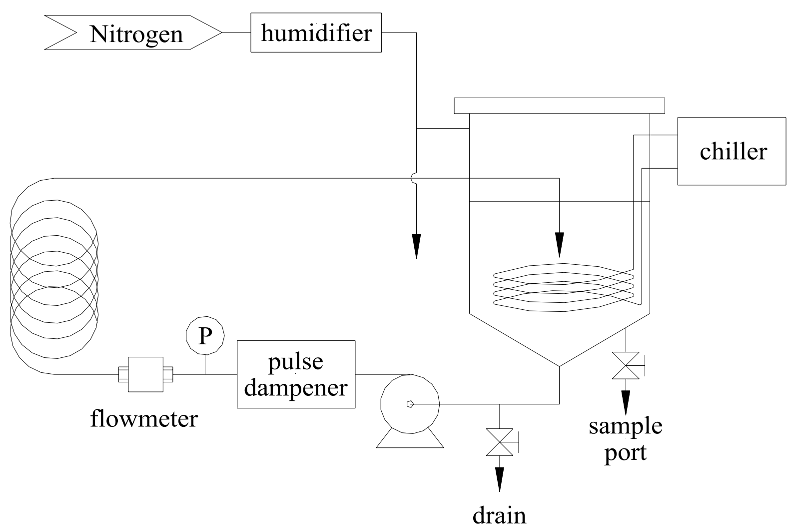
Table I. Specifications of the three pump systems

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	DP20F-FT	Yes	AD-25TT
Levitronix	Centrifugal	BPS-4	No	NA

Figure 1: Test system schematic



The measurements made using the NICOMP 380ZLS 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 instruments gaussian distribution assumption.

The size distribution of the large particle tail of the slurry was measured with an AccuSizer 780A. This instrument uses a combination of light scattering and light extinction to measure the size distribution of particles $\geq$ 0.56 µm. The size measurements were performed by diluting the slurry sample by a factor of about 500:1 into deionized water. Between samples, the entire system was thoroughly flushed with deionized water. Data from selected particle size channels were analyzed.

Results and Discussion

Figures 2 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 2. 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 3 shows the geometric mean PSD at the beginning of each pump test. The error bars in the figure represent ± one geometric standard deviation. Since there was some variation in the initial concentrations, particularly for the large particle tail, the ratios of the cumulative particle concentrations relative to the initial particle concentrations (C_I) will be presented in the following analysis (Figures 5-10) rather than actual particle concentrations.

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Figure 2. Initial working particle size distribution

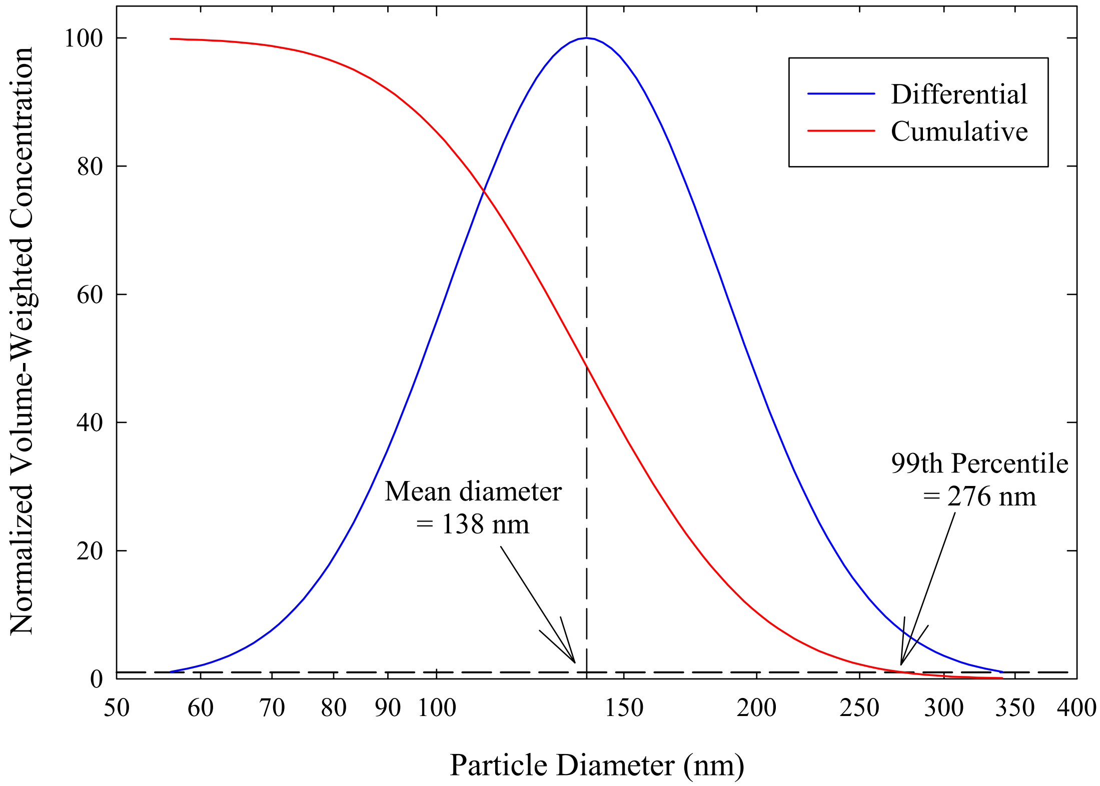

Figure 3. Initial size distribution of the large particle tail

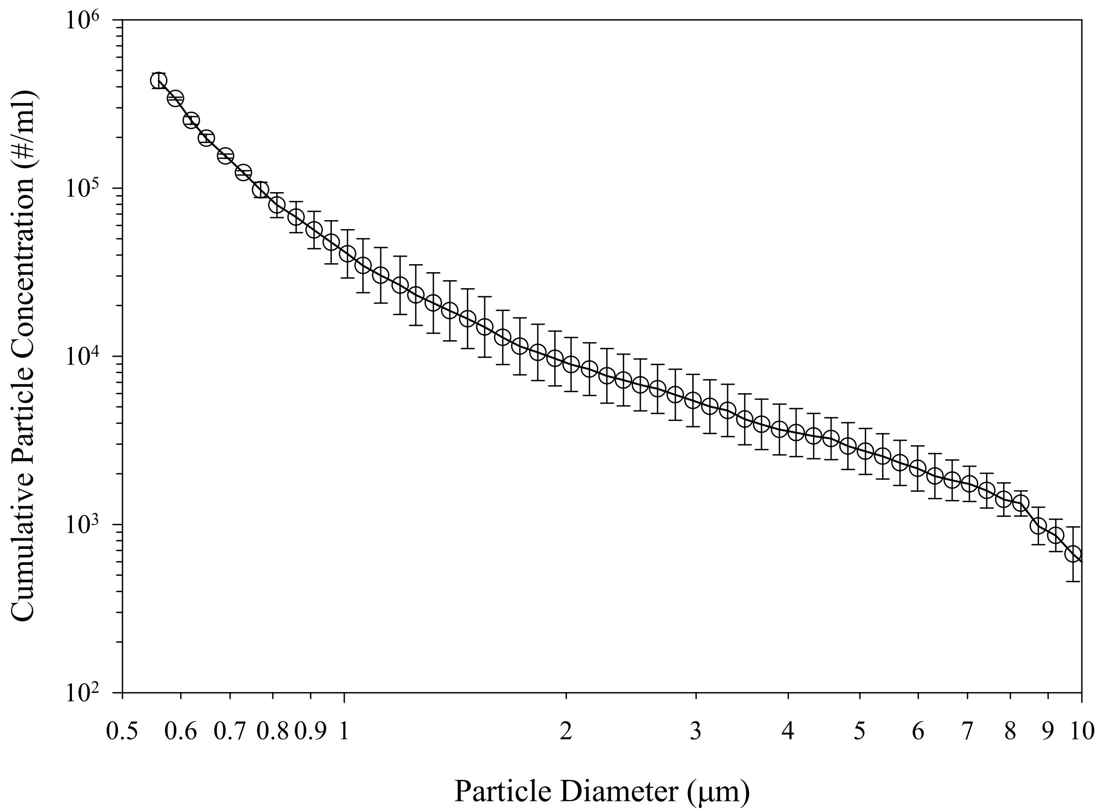

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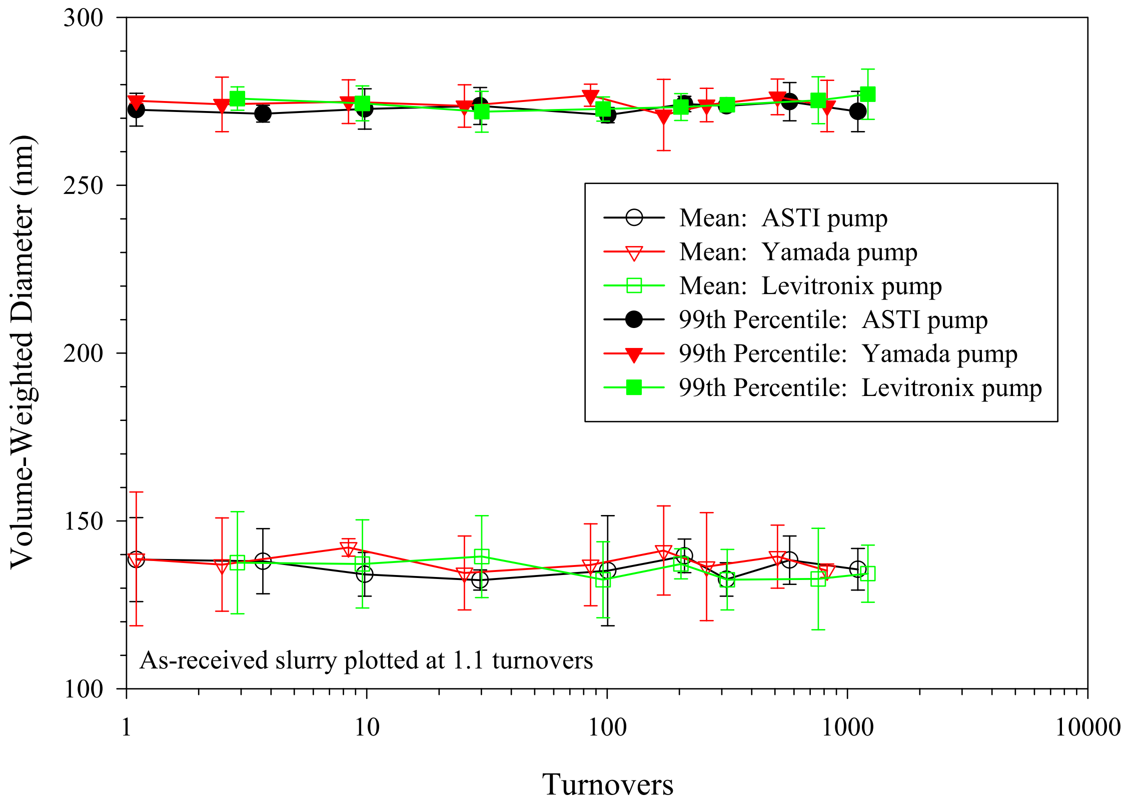
Figure 4 shows the volume-weighted mean and 99[th] percentile particle diameters of the working PSD as a function of tank turnovers. Error bars are included in the figure and represent ± 3 standard deviations. No significant change in the working PSD was observed for any of the pumps after 1,000 turnovers.

Figure 4. Effect of pumps on the working particle size distribution



Figures 5-9 present the ratios of the cumulative particle concentrations at different tank turnovers (C_T) relative to the initial particle concentrations (C_I) for 5 different size channels in the large particle tail as measured by the AccuSizer optical particle counter. Each plot presents the results from a different size channel as a function of tank turnovers. Each plot contains the test results from the three pumps. A summary of these results is presented in Table II.

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Figure 5. Effect of pumps on particles ≥ 0.56 μm

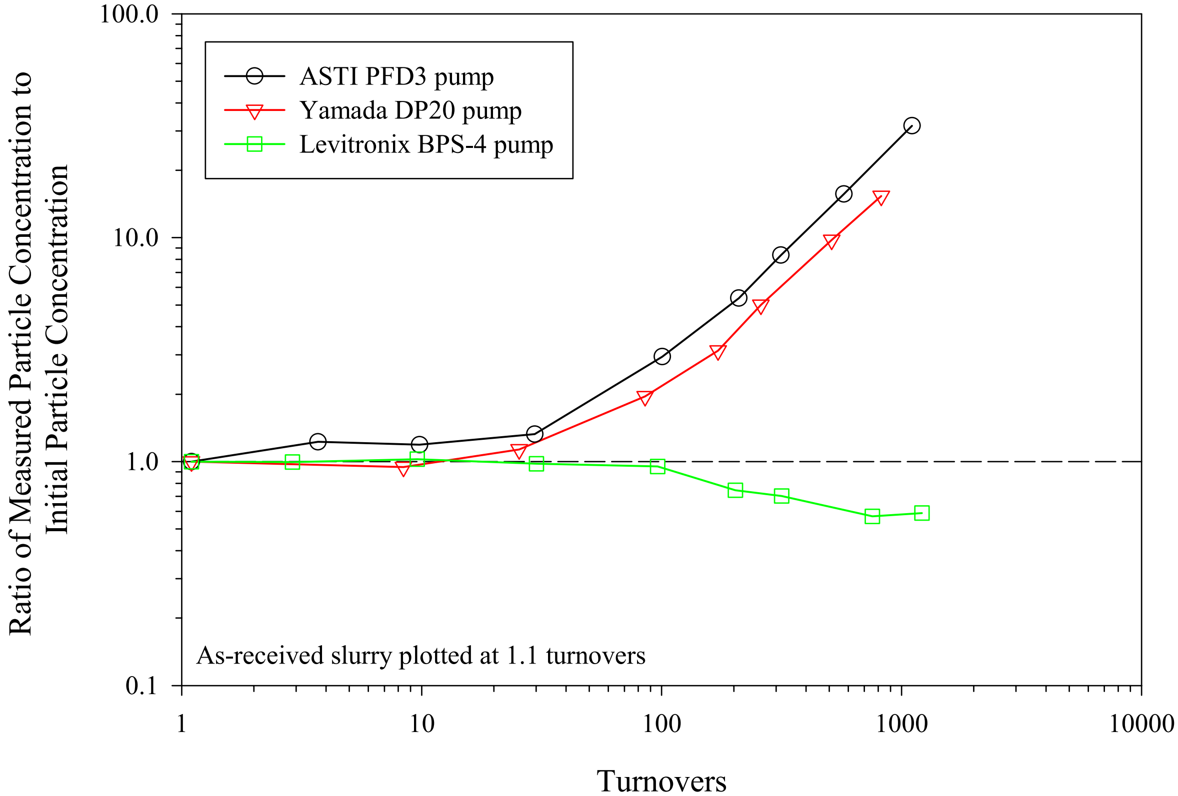

Figure 6. Effect of pumps on particles ≥ 1.0 μm

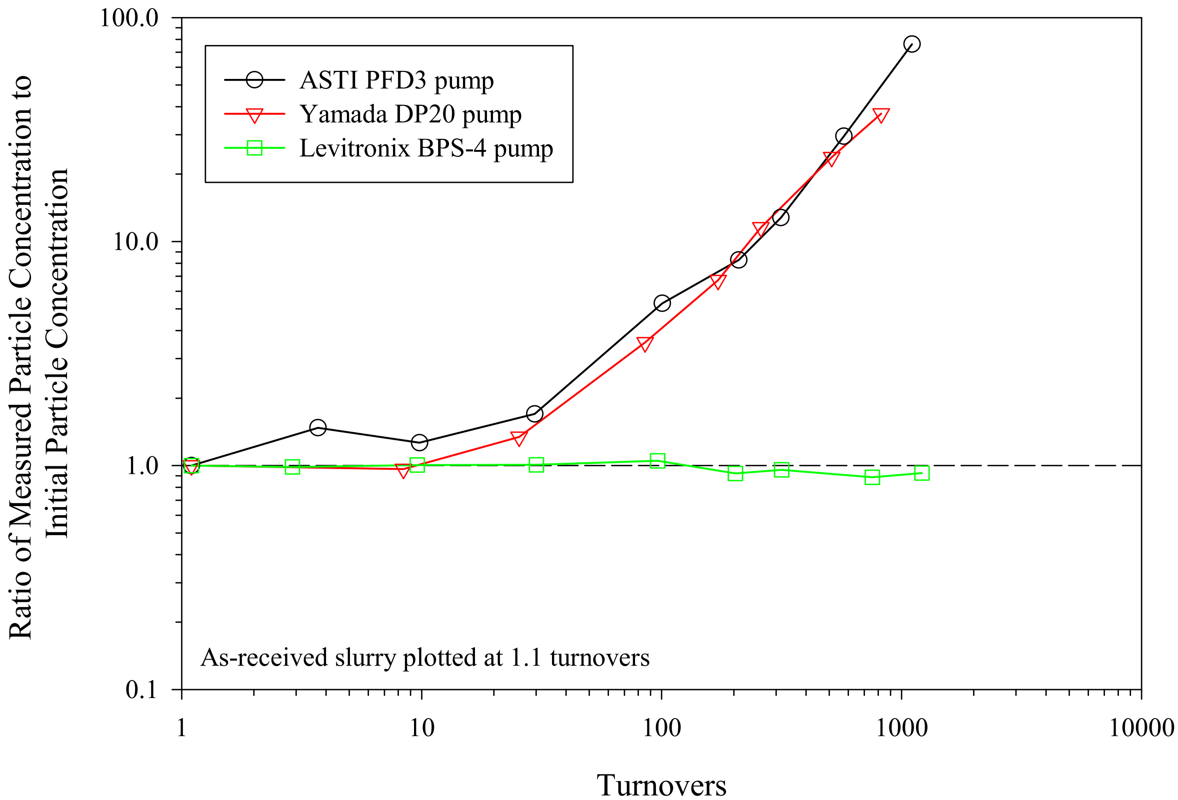

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Figure 7. Effect of pumps on particles ≥ 2.0 µm

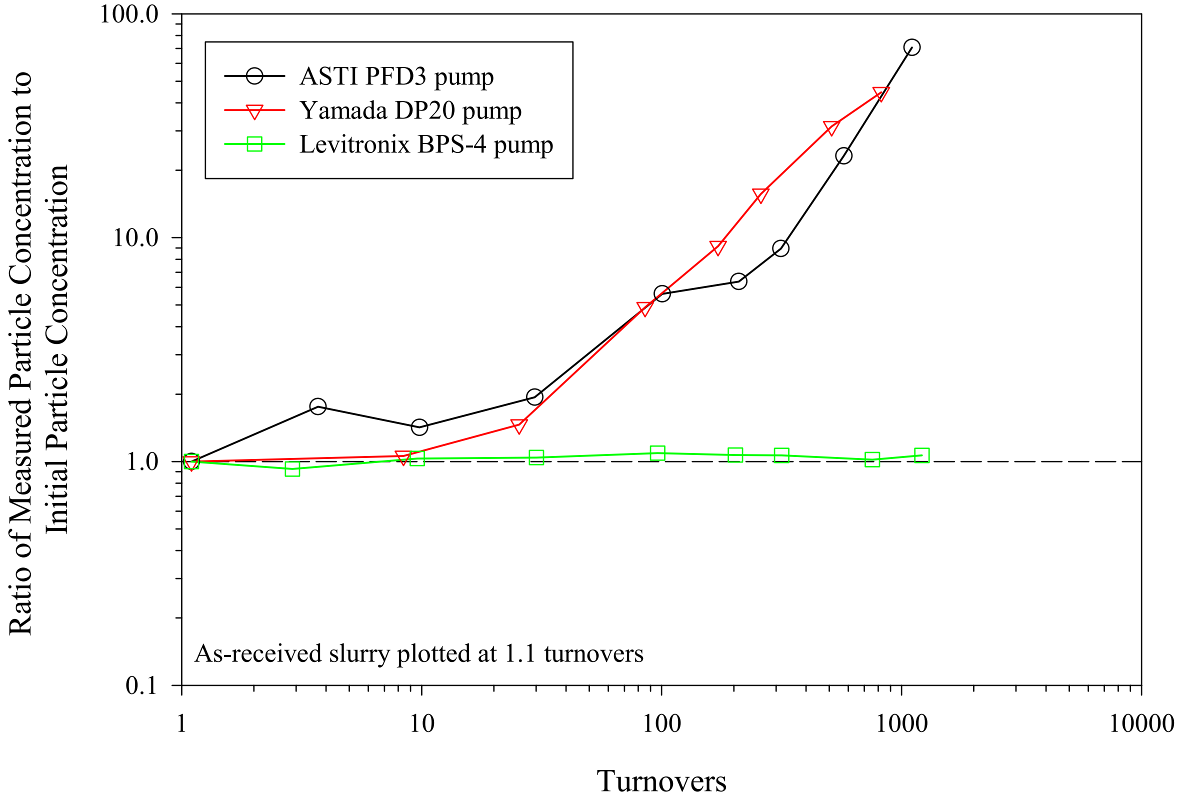

Figure 8. Effect of pumps on particles ≥ 5.0 µm

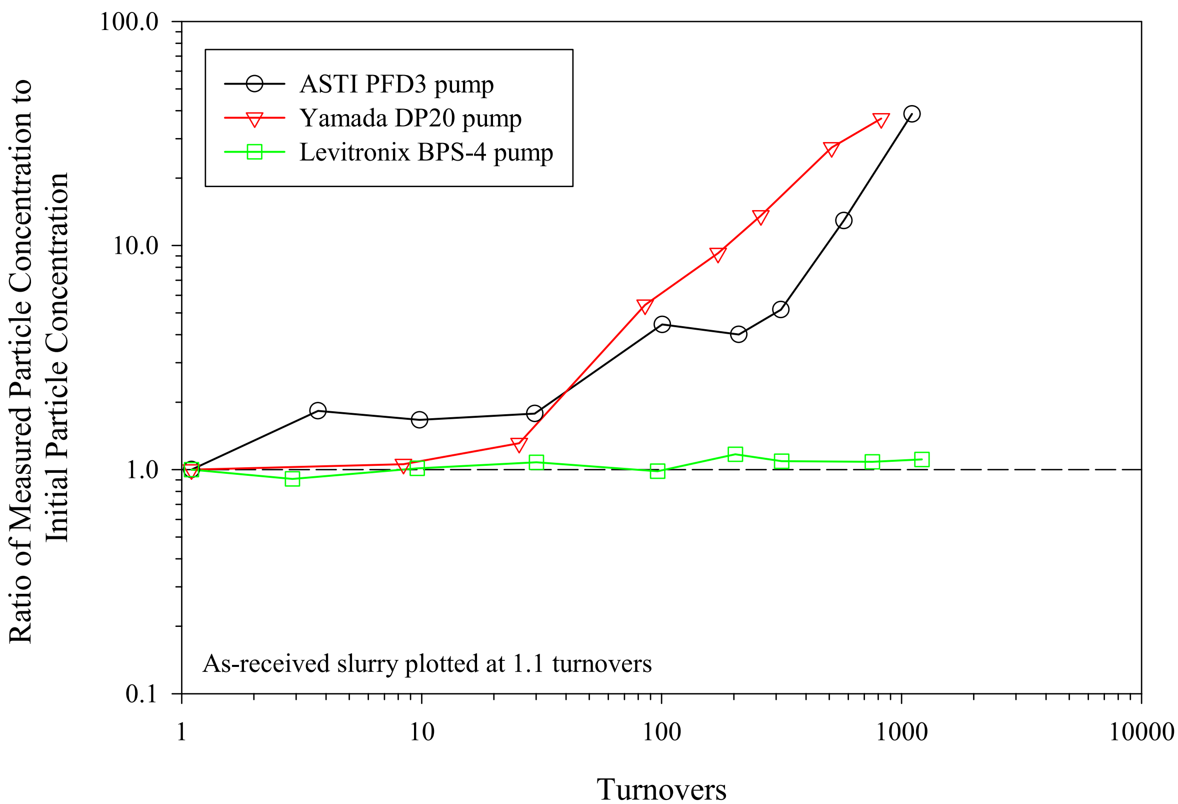

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Figure 9. Effect of pumps on particles ≥ 10 µm

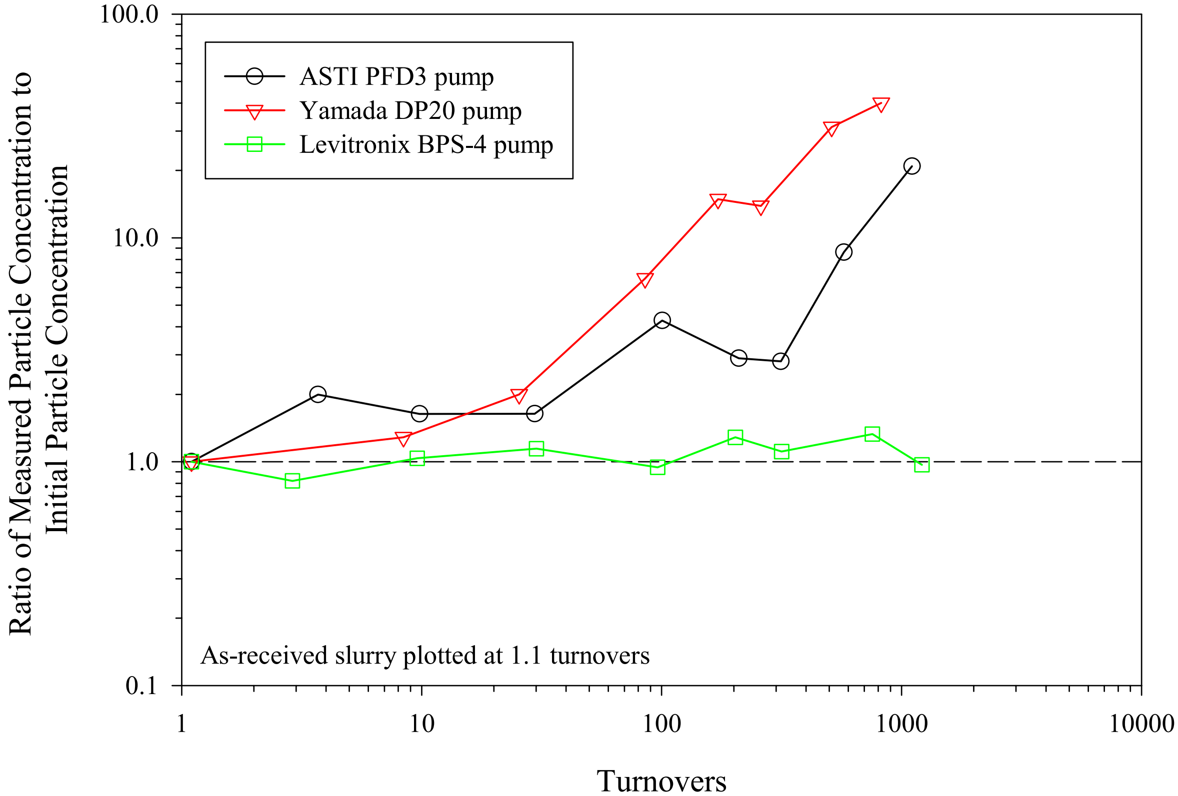

Table II. Summary of the effect of the pumps on the slurry PSD

Particle Concentrations Relative to the Initial Particle Concentrations (C_T/C_I)						
Particle Size	100 Turnovers			1,000 Turnovers		
	ASTI	Yamada	Levitronix	ASTI	Yamada	Levitronix
≥ 0.56 µm	2.9	2.3	1.0	29	19	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	19	49	1.1

Figure 5 presents the particle concentration ratios (C_T/C_I) for particles ≥ 0.56 µm. The particle concentration increase was essentially linear with tank turnovers for both the Yamada and ASTI pumps as shown in Figure 10. (This graph presents the same data as in Figure 5, except the data are plotted on linear rather than logarithmic scales.) These results suggest that the Yamada and ASTI pumps generated a constant number of large particles per pump stroke, approximately 1.1 and 2.4 million particles ≥ 0.56 µm per pump stroke, respectively. Although this appears to be a rather large amount of particle agglomeration, the particle generation must be put into perspective relative to the initial particle concentration in the slurry (approximately 400,000 particles/mL ≥ 0.56 µm).

Within 100 turnovers, the particle concentrations measured in the Yamada and ASTI pump systems had doubled and tripled, respectively, relative to the initial particle concentration in the slurry. After about 1,000 turnovers, the particle concentrations measured in the Yamada and ASTI pump systems increased by factors of about 20 and 30, respectively. Meanwhile, the particle concentrations in the Levitronix pump system

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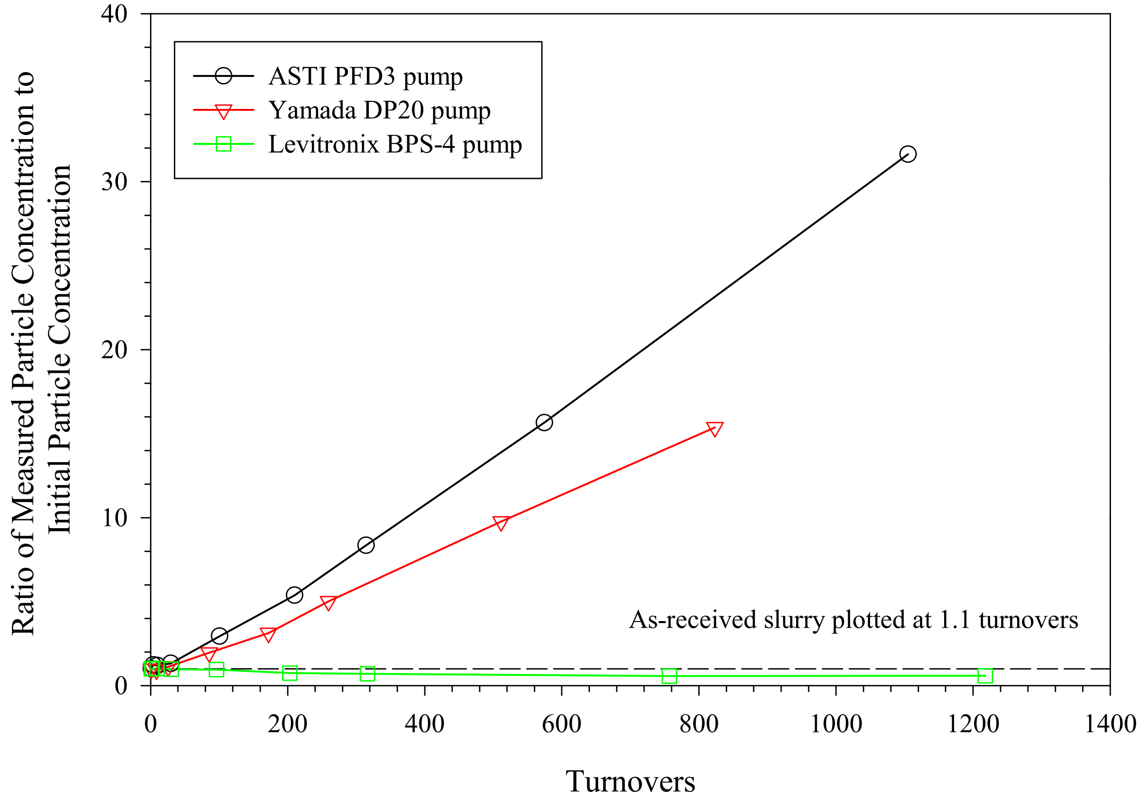
remained unchanged until after more than 100 turnovers. After 1,000 turnovers, the cumulative particle concentrations had decreased by more than 40%.

Figure 10. Effect of pumps on particles ≥ 0.56 µm (linear scales)



Figures 6-9 show the particle concentration ratios for particles ≥ 1.0 µm, ≥ 2.0 µm, ≥ 5.0 µm, and ≥ 10 µm, respectively. For these large particle sizes, the particle concentrations increased 4-8 fold relative to the initial particle concentration in the slurry for both the Yamada and ASTI pumps after 100 turnovers. Meanwhile, over the same particle size range, there was essentially no change in the particle concentrations for the Levitronix pump system.

Particle agglomeration has been shown to occur in Semi-Sperse® 12 when it is subjected to handling. In previous evaluations, large particle agglomeration of Semi-Sperse® 12 circulated by diaphragm pumps has been observed [2,3]. In these tests, the concentrations of particles in the large particle tail were found to increase significantly after less than 100 turnovers, while changes in the working particle size distribution were not observed until 2,000-3,000 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 Semi-Sperse® 12 slurry. The slurry was circulated until it passed through each pump approximately 1,000 times.

After 1,000 turnovers, no change in the working particle size distribution was observed with any of the pumps. However, the large particle concentrations increased by a factor of 2-8 within 100 turnovers and 20-70 within 1,000 turnovers for the ASTI and Yamada pump systems. Meanwhile, the particle concentrations in the Levitronix pump system remained unchanged after 100 turnovers. After 1,000 turnovers, the

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cumulative particle concentrations ≥ 0.56 μm decreased by more than 40% while the particles ≥ 1.0 μm remained unchanged.

References

1. Personnel communication with J. Kvalheim, BOC Edwards Chemical Management Division, Chanhassen, MN, March, 2003.
2. Nicholes K, R Singh, D Grant, and M Litchy (2001). "Measuring particles in CMP Slurries," Semiconductor International, 24(8): 201-206.
3. Litchy MR, K Nicholes, DC Grant, and RK Singh (2001). "Perturbation Detection Analysis: A method for comparing instruments that measure CMP slurry health," Proceedings of the 19th Annual Semiconductor Pure Water and Chemicals Conference, Sponsored by Balazs Analytical Laboratory, Sunnyvale, pp 153-171.

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