

## Effect of Particle Size Distribution on Filter Lifetime in Three Slurry Pump Systems

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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 pumps and pressure-vacuum technology. Typically, slurry passes through a distribution system approximately 100 times before it is used to polish wafers[1].

Many slurries are easily damaged by mechanical handling. Damage often takes the form of changes in the size distribution of the slurry particles. Large particles that can scratch wafer surfaces are often removed by filtration. The large particles tend to occlude the filter, causing reduced flow rates, increased pressure drops, and frequent filter changes. The frequency of filter replacement depends on many factors including slurry type, filter type, filter pore size rating, etc. The increase in pressure drop across a filter determines its life.

Experiments were performed to determine the effect of circulating Semi-Sperse<sup>®</sup> 12 (Cabot Microelectronics Corporation) with three different types of pumps (bellows, diaphragm, and centrifugal) on the size distribution of the particles in the slurry in the absence of filtration[2]. Significant differences in the large particle tail of the slurry particle size distribution (PSD) were observed after circulation with the different types of pumps.

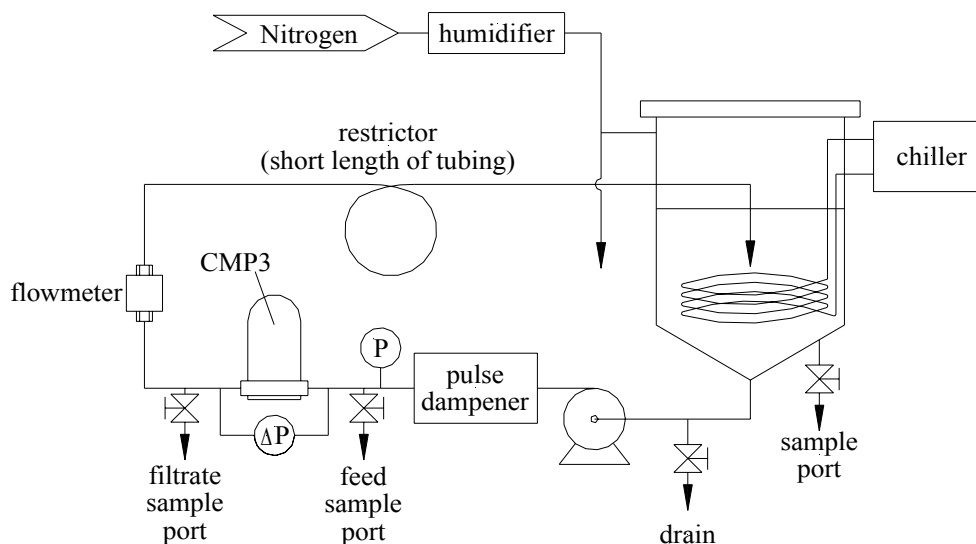
In addition, experiments were performed to determine if the changes observed in the large particle tail of the PSD resulting from pumping correlated to changes in the lifetime of filters used to remove large particles from the slurry. The increase in pressure drop across a 10” Mykrolis Planargard<sup>™</sup> CMP3 filter as a function of delivered slurry was characterized for three types of pumps. The expected results were observed: the higher the concentrations of large particles, the faster the filters clogged.

### EXPERIMENTAL METHODS

Testing was performed using the test system shown in Figure 1. Experiments were performed with and without filtration to measure the effect of large particle generation by the pumps and the effect of the particle generation on filter lifetime. Bellows, diaphragm, and centrifugal pumps were evaluated. Manufacturer recommended pulse dampeners were installed downstream of both the bellows and diaphragm pumps to minimize pulsation.

Each filter life test was initiated by installing a new 10” Mykrolis Planargard<sup>™</sup> CMP3 filter downstream of the pump or pump/pulse dampener combination. The pressure drop ( $\Delta P$ ) across the filter was monitored with a differential pressure transducer. Pressure ports were plumbed directly into the filter housing on the upstream and downstream sides of the filter. The slurry was circulated until a  $\Delta P$  increase of at least 10 psi was achieved or more than 3,000 system

turnovers (passes through the pump) were performed. These types of filters are often considered to have reached the end of their life when the  $\Delta P$  increases by 10 psi[3]. Slurry sample ports were located on the upstream and downstream sides of the filter.



**Figure 1.** Test system schematic

Each pump was used to circulate approximately 28.5 liters of slurry at a flow rate of  $29.5 \pm 1.0$  lpm. Settling of the slurry in the conical bottom tank was minimized by drawing from the bottom of the tank and by turning the volume of slurry in the tank over in less than one minute. 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 prevent the formation of a vortex in the tank that may entrain air into the slurry. 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.

No devices that generate a rapid pressure drop (valves, orifices, etc.) were used to generate backpressure at the outlet of the pump. Instead, in the absence of filtration, a long length of PFA tubing was used to gradually reduce the pressure from 30 psig at the pump outlet to ambient pressure at the tank. With filtration, a combination of the  $\Delta P$  across the filter and a length of tubing downstream of the filter were sufficient to provide pump outlet pressures ranging from 22-37 psig (depending on the  $\Delta P$  across the filter). The air pressure supplied to the bellows and diaphragm pumps and the rotational speed of the centrifugal pump were adjusted to maintain the slurry flow rate as the pressure drop across the filter increased.

The tank holding the slurry was blanketed with nitrogen to prevent absorption of carbon dioxide from the air that can change the pH and chemical composition 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  $20 \pm 2^\circ\text{C}$  during the test. The relative humidity in the tank was  $> 90\%$  throughout the test.

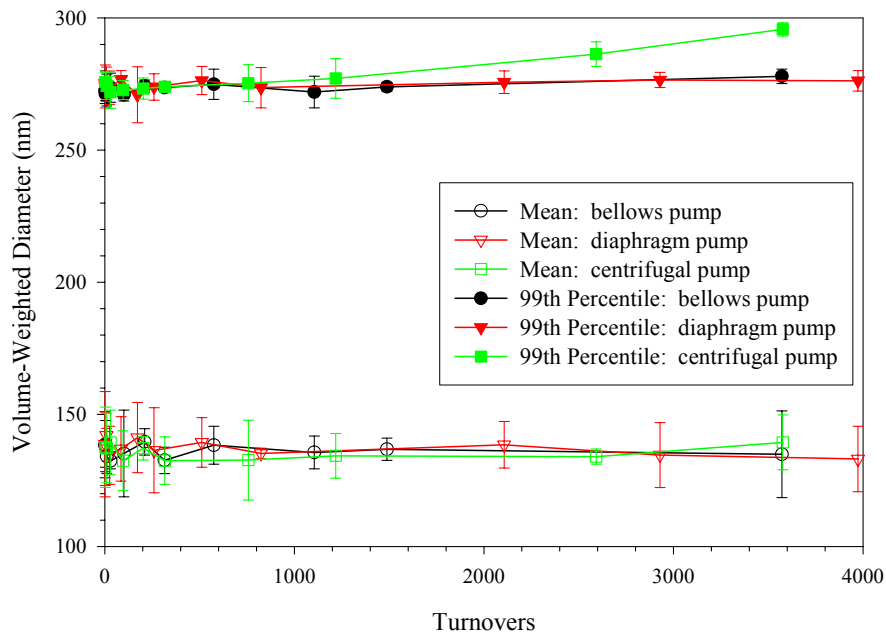
In all tests, samples were drawn from the system at selected times for analysis. The PSD was measured using 2 techniques. The size of the working particles was measured using a NICOMP 380ZLS (Particle Sizing Systems, Santa Barbara, CA) that determines particle size by dynamic

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).

## RESULTS

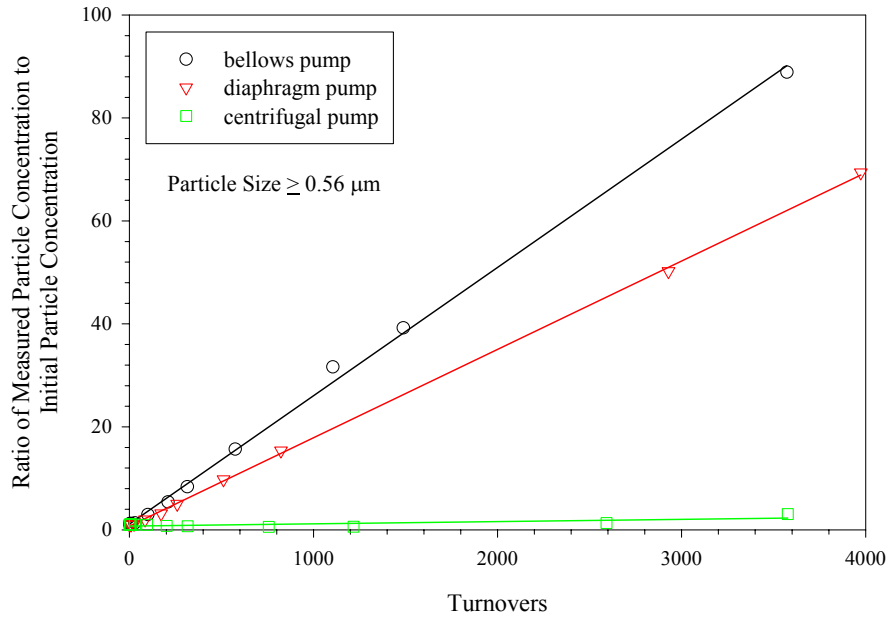
### Particle size distribution results without filtration

Figure 2 shows the volume-weighted mean and 99<sup>th</sup> percentile particle diameters of the working PSD as a function of tank turnovers. Error bars are included in the figure and represent  $\pm 3$  standard deviations. No significant change in the working PSD was observed for any of the pumps within 1,000 turnovers. No increase was observed in the mean diameter in any of the pump systems after 3,500 turnovers. However in the centrifugal pump test, the 99<sup>th</sup> percentile particle diameters appeared to increase slightly after about 2,000 turnovers.



**Figure 2.** Effect of pumps on working particle size distribution

Figure 3 presents an example of the effect of the pumps on the large particle tail in the absence of filtration. The ratios of the cumulative particle concentrations downstream of each pump relative to the initial particle concentrations for particles  $\geq 0.56 \mu\text{m}$  as a function of turnovers are shown. The particle concentration increase was essentially linear with turnovers for both the bellows and diaphragm pumps. Similar concentration changes were observed at larger particle sizes. Concentration ratios for different sizes at 100 and 1,000 turnovers are summarized in Table I. Typical initial concentrations are included for reference. These results suggest that the bellows and diaphragm pumps generate a constant number of large particles per pump stroke. Within 100 turnovers, the concentrations of large particles increased roughly 4 fold for these types of pumps. After 1,000 turnovers, the concentrations were 20-70 times higher than the initial concentrations (depending on particle size). Meanwhile, the large particle concentrations were essentially unchanged in the centrifugal pump system.



**Figure 3.** Effect of pumps on large particle concentrations

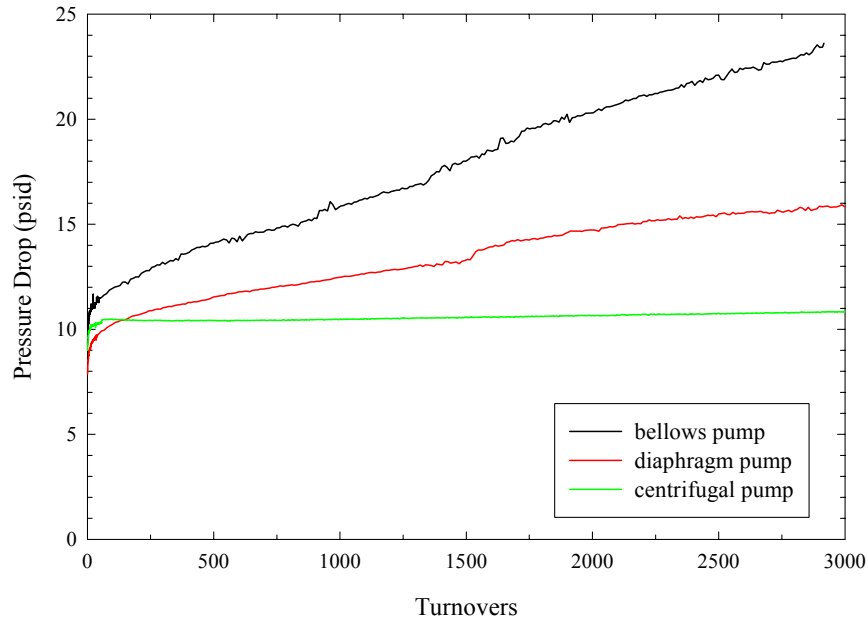
**Table I.** Summary of the effect of pumps on slurry large particle concentrations

Particle Size (μm)	Typical Initial Concentrations (#/ml)	Particle Concentrations Relative to the Initial Particle Concentrations					
		100 Turnovers			1,000 Turnovers		
		Bellows	Diaphragm	Centrifugal	Bellows	Diaphragm	Centrifugal
≥ 0.56	450,000	2.9	2.3	1.0	29	19	0.6
≥ 1.0	40,000	5.3	4.1	1.0	69	45	0.9
≥ 2.0	9,000	5.6	5.7	1.1	64	54	1.0
≥ 5.0	2,700	4.4	6.3	1.0	35	45	1.1
≥ 10	600	4.3	7.7	0.9	19	49	1.1

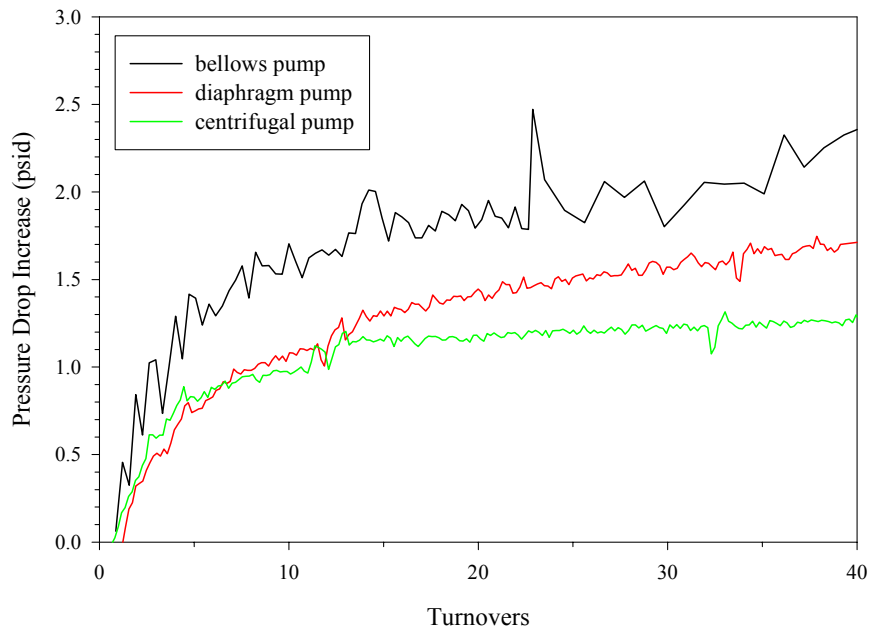
### **Pressure drop results across filters**

The  $\Delta P$ s measured across each CMP3 filter as a function of tank turnovers are presented in Figure 4 for the three pumps. In all of the tests, there was a relatively rapid increase in  $\Delta P$  that occurred during the initial 10 turnovers followed by a more gradual rate of increase thereafter. The ‘initial’  $\Delta P$ s were about 9.2, 8.0, and 9.0 psid for the bellows, diaphragm, and centrifugal pumps, respectively. The variation in the ‘initial’  $\Delta P$  is most likely attributed to variation in the filter media.

Figure 5 presents the  $\Delta P$  increase across the filter during the beginning of each test. The  $\Delta P$  increase was calculated by subtracting the ‘initial’  $\Delta P$  values from subsequent  $\Delta P$  measurements. This graph shows the rapid  $\Delta P$  increase across each filter (~1.0-1.5 psid) during the initial 5-10 turnovers of each test. This initial  $\Delta P$  increase is thought to be due to removal of large particles from the as-received slurry rather than from particles generated by pumping the slurry. As the large particles were removed from the slurry during the first few passes through the filter, the rate of change in  $\Delta P$  decreased. Measurements of large particle concentrations in the slurry support this observation.



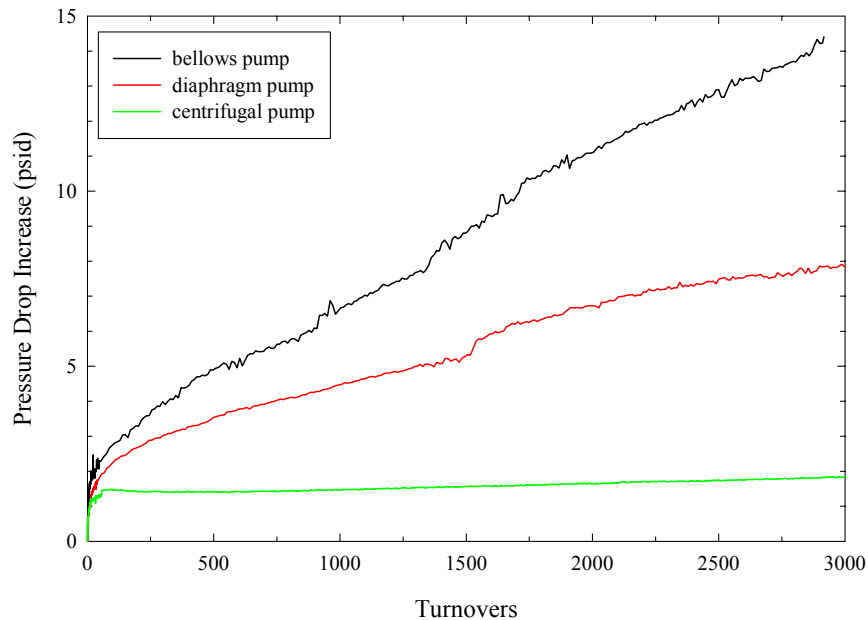
**Figure 4.**  $\Delta P$  across each filter for all 3 pump tests



**Figure 5.** Increase in  $\Delta P$  across each filter during the beginning of each test

Figure 6 presents the increase in  $\Delta P$  across the filter throughout each test. It shows the initial rapid increase in  $\Delta P$  followed by a slow and fairly linear increase in  $\Delta P$  during the remainder of the test. The increase in  $\Delta P$  for the centrifugal pump was substantially less than the increase for the other pump systems. The slopes of linear regressions of each curve (not shown) were used to approximate the  $\Delta P$  increase per turnover as shown in Table II. Based on these slopes the rates

of  $\Delta P$  increase (relative to the centrifugal pump) were about a factor 9 and 23 fold higher for the diaphragm and bellows pumps, respectively.



**Figure 6.** Increase in  $\Delta P$  across each filter for the three pump tests

**Table II.** Rate of increase in  $\Delta P$  for each pump

Type of Pump	$\Delta P$ Increase per 1,000 Turnovers (psi/1,000 turnovers)	Rate of $\Delta P$ Increase (relative to centrifugal pump)
Bellows	4.1	23
Diaphragm	1.7	9.3
Centrifugal	0.18	-

## CONCLUSIONS

A diaphragm pump, bellows pump, and centrifugal pump were tested to determine how their use affected the lifetime of CMP3 filters in Semi-Sperse<sup>®</sup> 12 slurry. The tests were continued until the  $\Delta P$  across the filter increased by at least 10 psig or more than 3,000 tank turnovers in the system were achieved.

The rates of  $\Delta P$  increase measured using the diaphragm and bellows pumps were approximately 9 and 23 times higher than those measured when using the centrifugal pump. These results are consistent with particle size distribution measurements that showed the centrifugal pump generates fewer particles than the diaphragm or bellows pumps.

## REFERENCES

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2. M. Litchy and R. Schoeb, Semiconductor International **27** (12), 87-90 (2004).
3. Z. Lin, G. Vasilopoulos, K. Devriendt, and M. Meuris, presented at SEMICON West 99 Technical Program, July, 1999.