Influence of Components in the Slurry Delivery Chain on Slurry Health and CMP Defects

Reto Schoeb[a], Mark R. Litchy[b], Donald C. Grant[b]

[a] Levitronix GmbH, Zurich 8005, Switzerland
[b] CT Associates, Inc., Eden Prairie 55344, USA

The impact of many components and handling conditions on slurry health has been investigated extensively in previous studies. Numerous studies have shown that changes in the particle size distributions in CMP slurries can have a strong impact on scratch densities and surface roughness after CMP. This paper summarizes and quantitatively compares the impact of various components and conditions on slurry particle size distributions. The paper further summarizes oversize particle and micro scratch data from five different studies with 14 different slurry & substrate combinations. The goals of this paper are to identify the key elements that influence slurry health in the slurry delivery chain and establish a correlation between particle size distributions and micro scratch defect densities.

Introduction

Particles in some CMP slurries tend to agglomerate when the slurry is exposed to chemicals and gases, foreign materials and mechanical forces. These agglomerates may adversely impact surface roughness and increase micro scratch defects during CMP [1 - 5]. The impact of many components and handling conditions on slurry health has been investigated extensively in previous studies [6 - 16]. The slurry handling elements considered in this comparison are shipping containers, tanks, valves, different kinds of recirculation pumps, loop filters, point of use filters and variations in loop design. One goal of this paper is to quantitatively compare the impacts of these components and conditions on large particle counts (LPC) and to identify the key elements that influence slurry health. Another goal is to correlate changes in LPCs to defect densities for various CMP processes.

Overview of the Slurry Delivery Chain

CMP slurries are usually shipped to chip fabs in drums or totes in the form of formulated slurry for direct usage or in the form of concentrated raw slurry which is diluted and mixed with pH stabilizers, oxidizers and other constituents in the subfab. The formulated slurry is then transferred into a day tank, and from there delivered to the CMP tools in the fab. A schematic of a typical slurry mixing and distribution system is shown in figure 1. In order to prevent particle settling and to achieve stable pressure independent of slurry demand, the slurry is usually continuously recirculated through a global loop at flow rates ranging from approximately 10 to 30 lpm. Recirculation is achieved by either pumps or a

CTA publication 114: China Semiconductor Technology International Conference (CSTIC 2013), Shanghai, China, March 2013.

vacuum-pressure-dispense system. On its way through the loop, the slurry passes loop-filters, several hundred feet of piping, different valves including a back-pressure regulator, and a day tank. Only a small portion of the recirculated slurry leaves the global loop and is fed to and consumed by the CMP tools. Singh, Conner and Roberts [2] estimate a typical consumption rate of 200 liters / day which results in an average of approximately 100 turnovers before consumption at a slurry flow rate of 14 lpm. This figure can vary considerably depending on the number of CMP tools on a loop and capacity utilization. In any case, it is important to understand that the components in the global loop see the same slurry many times and are therefore the most critical ones concerning their impact on slurry health.

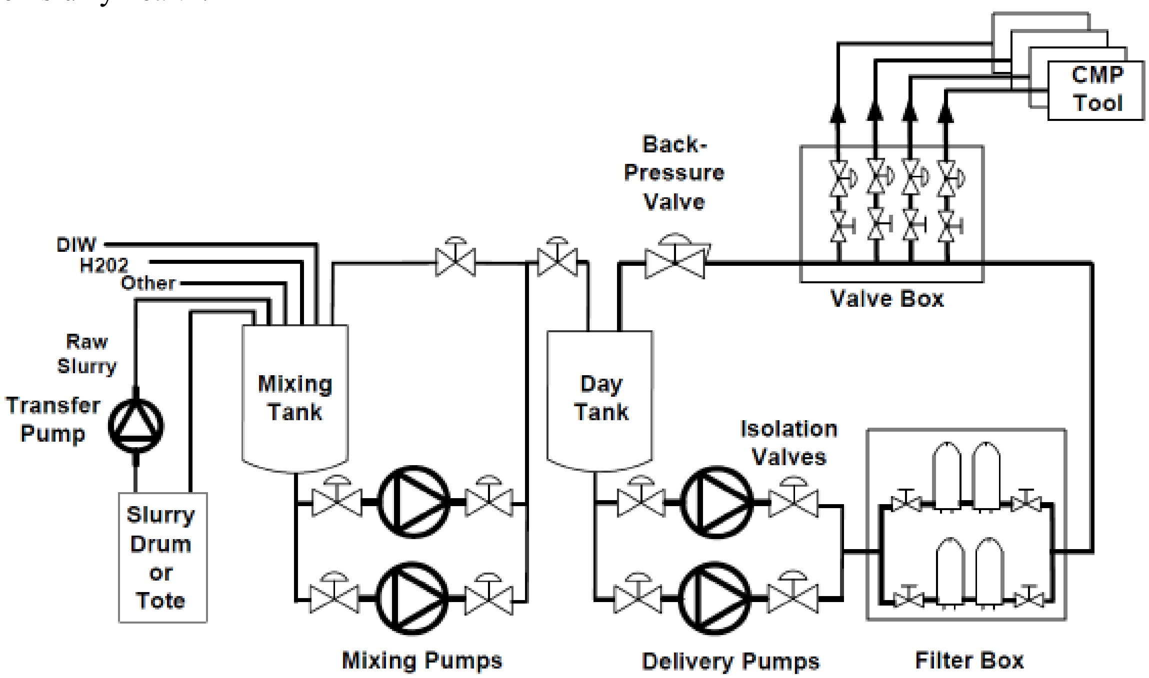

Figure 1. Schematic of typical slurry distribution system

On its way from the global loop to the platen, the slurry passes through several valves, long lengths of tubing and sometimes a point-of-use or point-of-tool filter. The slurry is then dispensed onto the pad by a peristaltic pump or a flow controller. The main concern in this section of the slurry delivery chain is precise control of the slurry flow and prevention of particle settling or even clogging of components since continuous slurry flow is unlikely.

The Impact of Various Components on Slurry PSDs

Shipping Containers and Tanks

Shipping and storage containers can have a strong impact on slurry health. Interaction of the gas/slurry interface and drying of slurry exposed to air are the main concerns. In a study involving 17 shipping containers it was demonstrated that the concentration of large particles (> 2µm) was approximately 10 times higher in drums containing headspace than in drums without heasdspace (figure 2) [20]. While it may be possible to ship slurry in containers without headspace, the slurry levels in mixing and day tanks changes over time resulting in particle agglomeration at the moving interface. To prevent dehydration of slurry, the tanks should be blanketed with humidified nitrogen. A study about surface mediated particle-particle agglomeration demonstrated that the choice of materials can also impact slurry large particle concentrations [10]. In the study, the LPC in a Glass container increased nearly five times faster than in a container with a polypropylene surface (figure 3).

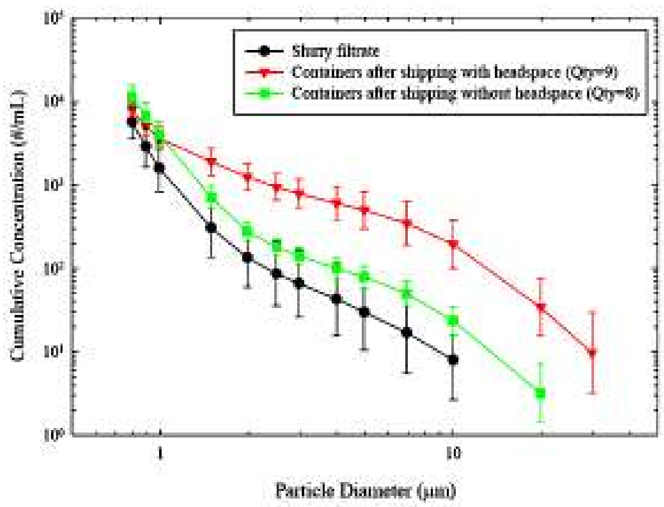

Figure 2: Effect of Headspace in Shipping Containers on Slurry PSD (Source: Ref. 20)

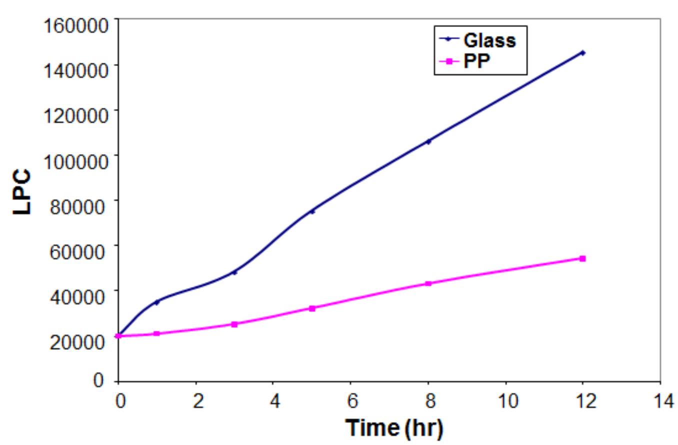

Figure 3: Influence of Container Material (Glass vs. Polypropylene) on Slurry LPC (Source: Ref. 10)

Pumps

The potentially high impact of various pumps on slurry PSD has been reported in many studies [2-10, 13, 16-19]. Figure 5 shows the influence of various pump types on PSD in a fumed silica slurry. After 1000 turnovers, LPCs > 2μm increased 53 and 63 fold with diaphragm and bellows pumps, respectively. After 100 turnovers, a more realistic figure for a global loop, the LPCs increased more than 5 times for both volumetric pump types.

In contrast, the PSD changed only minimally when the slurry was circulated with maglev centrifugal pumps and the LPCs may have actually decreased slightly.

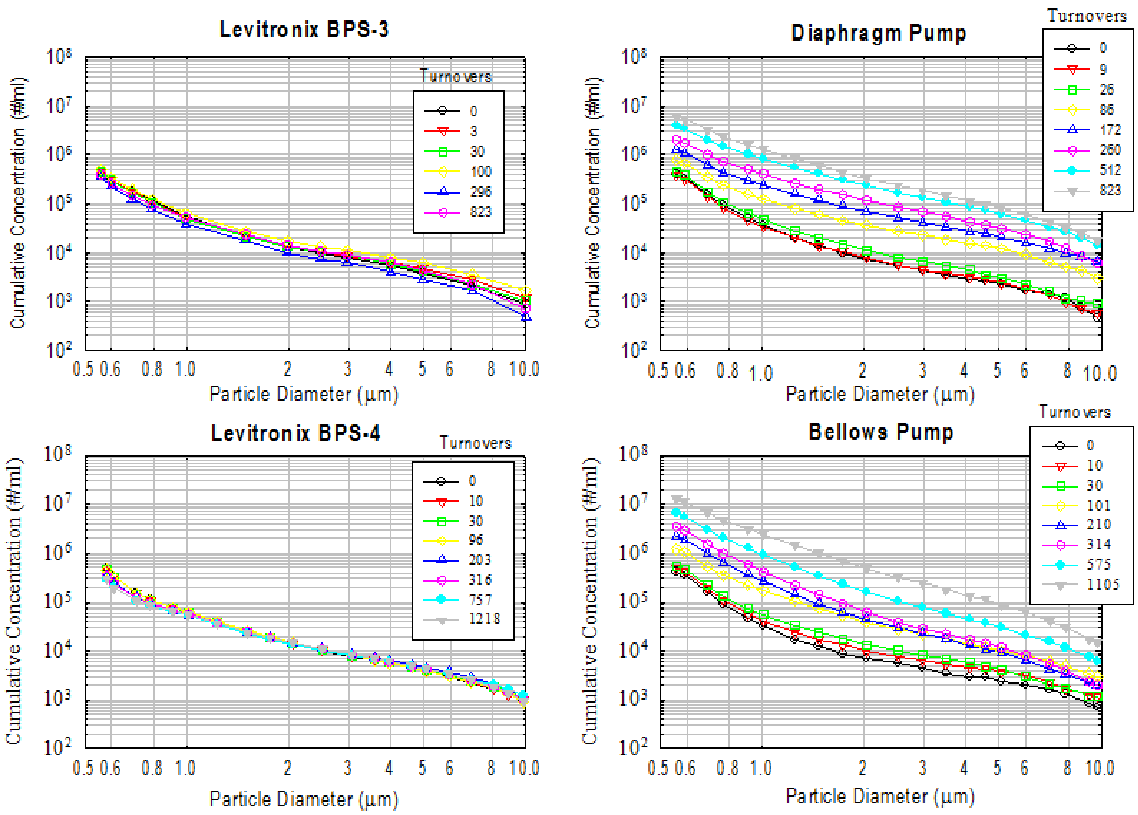

Figure 4: Influence of pump type on PSD in fumed sillica slurry (Source: Ref. 13)

The impact of pumps (and other components) on changes in PSD is dependent on the type of abrasives and on the chemical composition and properties of slurries (i.e. pH, buffer content, oxidizers, surfactants and chelating agents). Reference [19] reports a strong influence of pH and salt content on the tendency of slurries to agglomerate and demonstrates a clear relationship between pH and LPC after slurry circulation. LPCs after 1000 turnovers with positive displacement pumps increased much more in acidic slurries compared to alkaline slurries. Reference [18] describes the influence of a "removal rate enhancer" on LPCs after circulation with different pump types. Reference [16] discusses the susceptibility of 9 different slurries including 4 abrasive types to particle agglomeration. When the slurries were circulated 100 times with volumentric pumps, a significant LPC increase (> 2x) could be measured in 6 out of 9 slurries. When the slurries were circulated with a maglev centrifugal pump, only one slurry showed a significant increase in LPCs. Two slurries turned out to be relatively robust and LPC changes remained in a window of -50% to + 100%. With one slurry, a significant decrease of LPCs was measured after circulation with any pump type.

It seems that particle agglomeration is not limited to any one abrasive type; rather agglomeration has been observed with 4 different abrasive types. In one alumina slurry, the LPC increased more than 10 times after 100 turnovers with a bellows pump, while in another alumina slurry, the LPC decreased significantly. Similarily, one of the ceria slurries tested turned out to be highly susceptible to agglomeration while a second ceria slurry was the most robust slurry in the testfield. Nevertheless, all tested silica slurries were relatively susceptible to agglomeration during volumetric pumping.

TABLE I. Summary of changes in large particle concentrations after 100 and 1,000 turnovers with different pumps in various slurry types (Source: Ref. 16)

Abrasive	After 100 Turnovers			After 1,000 Turnovers		
	Centrifugal	Diaphragm	Bellows	Centrifugal	Diaphragm	Bellows
fumed silica	0	+	+	0	++	++
colloidal silica 1	0	0	0	0	+	+
colloidal silica 2	+	+	+	+	+	++
colloidal silica 3	0	+	+	0	+	++
colloidal silica 4	0	+	++	0	++	++
alumina 1	-	-	-	-	-	-
alumina 2	0	+	++	0	++	++
ceria 1	0	++	++	0	++	++
ceria 2	0	0	0	0	0	0
Totals with Increase	1	6	6	1	7	7
Totals with Decrease	1	1	1	1	1	1

Key		
Symbol	Concentration change	Concentration ratio
-	Decrease	< 0.5
0	None	0.5 - 2.0
+	Increase	2-10
++	Large Increase	> 10

Valves

Many valves are used in CMP slurry delivery systems. On average, slurry passes through about a dozen different valves before it is dispensed on the pad. In the global loop, the slurry passes through the same valves about a hundred times. Reference [20] describes the effect of diaphragm valves on the particle size distribution of a fumed silica slurry. During steady-flow through the open valves, LPCs remained constant even after 2100 turnovers; however, during valve cycling, LPCs increased approximately linearly with increasing valve cycles. Figure 4 shows the effect of valve cycling on the particle size distribution in a relatively small simulated distribution loop. A significant impact on the LPCs is only seen after more than 10,000 cycles. This effect will be even smaller in a distribution loop that contains a larger volume of slurry. As a result, the effect of valves on LPCs is miminal compared to the effect of volumetric delivery pumps.

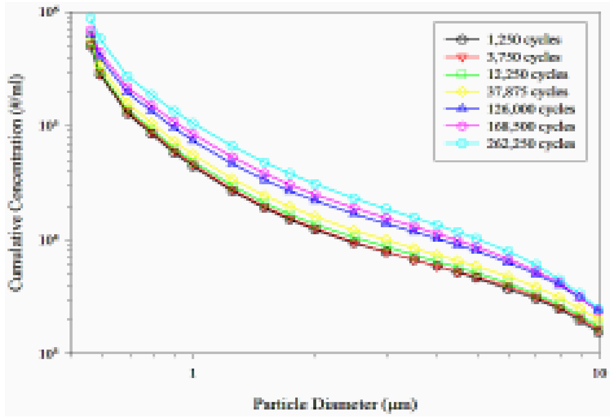

Figure 5: Influence of valve cycling on fumed sillica slurry PSD (Source: Ref. 20)

<u>Loop Design</u>

In a study with fumed silica slurry, it was shown that cavitation can lead to significant particle agglomeration [11]. Cavitation occurs when the liquid pressure temporarily falls below the liquid vapor pressure. This can happen in high speed gradients at edges, bends, orifices, blades or valves. With high static liquid pressure, the risk of cavitation is low. The risk of cavitation increases if the static liquid pressure falls significantly below atmospheric pressure or if the liquid temperature rises close to the boiling point. In CMP slurries, the later risk is small, since the CMP slurries are normally processed at room temperature. Conditions of high negative pressure can occur when slurries are sucked with a pump or with vacuum out of a drum or a day tank. In order to avoid cavitation, the suction pressure at the pump inlet should be controlled carefully. If feasible, the pump should be mounted below the tank level and the distance between pump and tank should be kept short. The pipe diameter between the tank and the pump sould be reasonably large and the connection should contain as few bends as possible. Valves, other than isolation valves which are normally open when the pump runs, should be avoided on the inlet side of the pumps. If the slurry is sucked out of tanks by vacuum, the vacuum pressure should be controlled and not fall below -0.3 Bar in order to maintain some safety margin.

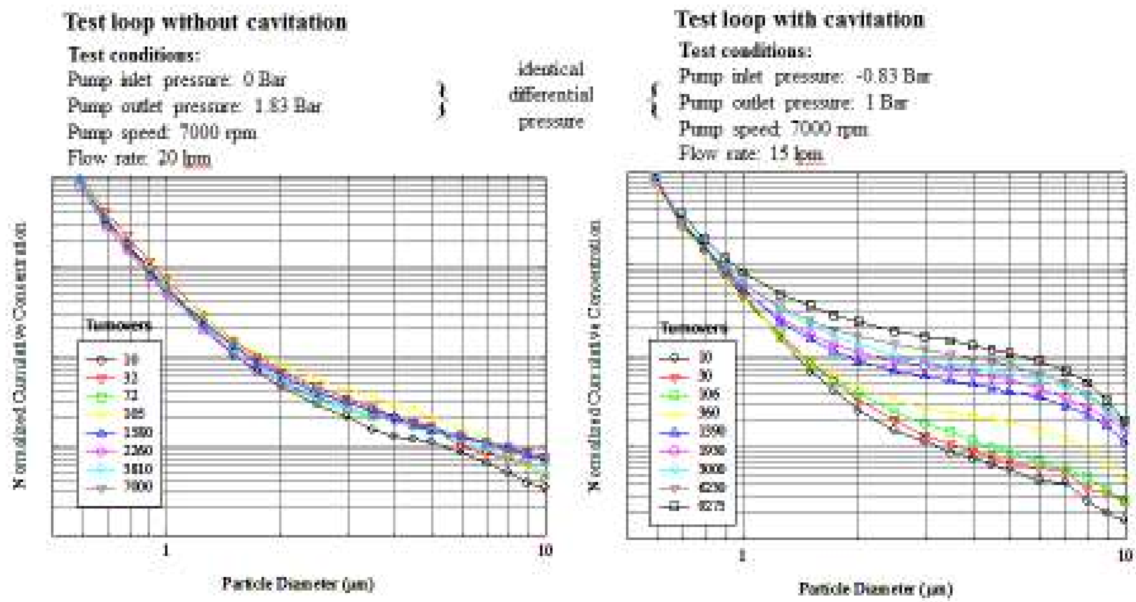

Figure 6: Influence of slurry loop design on PSD in fumed silica slurry (Source: Ref. 11)

<u>CMP Filters</u>

Filters are the only known way to actively remove oversize particles from CMP slurries. The effectiveness of slurry filtration has been demonstrated in various studies [2, 6, 15, 17, 22]. In a test with a silica slurry, a loop filter rated at 3μm removed approximately 60% of particles >0.56 μm and 90% of particles >2 μm. A 0.5 μm rated point of use filter removed 70% of particles >0.56 μm and 95% of particles >2 μm [22]. Despite the effectiveness of slurry filtration, other components in the slurry loop can still make a big difference. Figure 7 shows the PSD of a fumed silica slurry after 24 hours of circulation with either a maglev or a bellows pump, with either a 3 micron or a 5 micron filter installed in the test loop. While the two filters effectively reduced the high number of oversize particles created by the diaphragm pump, the resulting LPC remained significantly higher in the test loop with the diaphragm pump than in the loop with the maglev pump. The removal of high concentrations of oversize particles also has a direct impact on filter life. In a test with fumed silica slurry and three different pumps, the filter life with the maglev pump was 8 and 23 times longer than with diaphragm and bellows pumps, respectively [12].

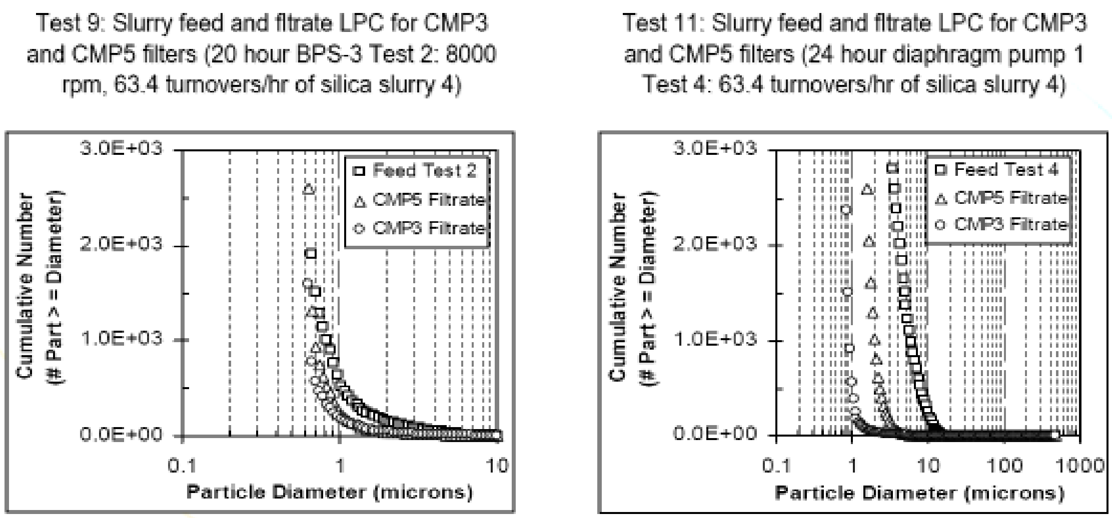

Figure 7: Influence of filter type and pump type on PSD in fumed silica slurry (Source: Ref. 6)

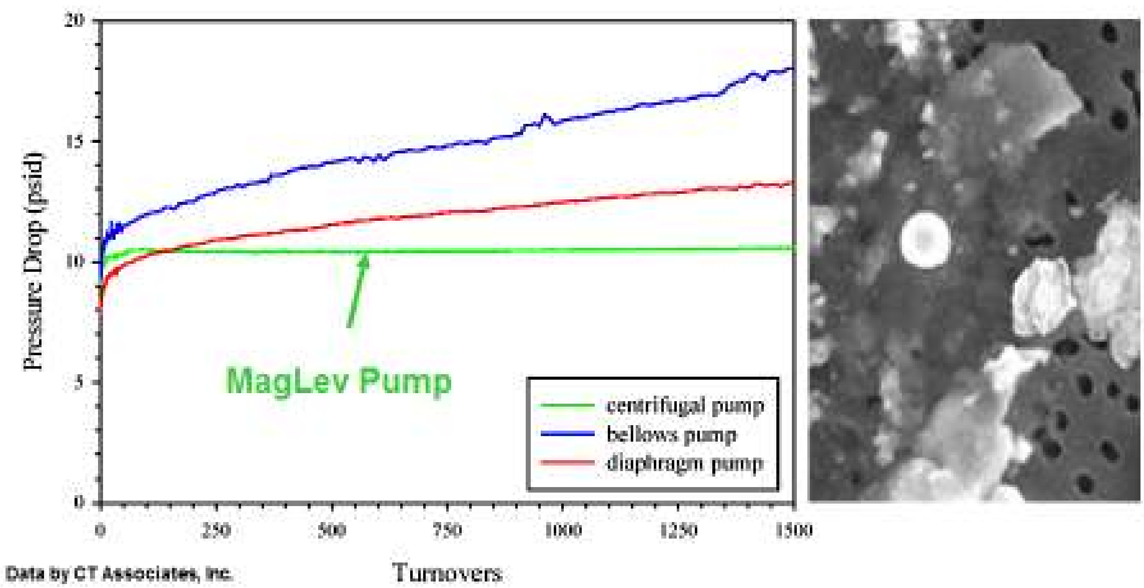

Figure 8: Pressure drop across an Entegris® Plangard™ CMP3 filter in a fumed silica slurry circulated with a bellows pump, a diaphragm pump and a maglev centrifugal pump at a constant flow rate of 30 lpm (Ref. 12)

Comparison of Impacts of Various Elements

Table II summarizes the relative impact of various elements on LPCs in silica slurry. Shipping containers and tanks, circulation pumps and loop design can have a high negative impact with reported LPC increases between 500% and 6300% for particles > 2 µm and up to 2800% for particles >0.56 µm. The impact of these elements can be reduced to a medium to low level by avoiding headspace and dry air in shipping containers and tanks, installing maglev pumps and avoiding negative suction head in the connection of the tanks and the pumps. Loop filters and point of use filters can have a high positive impact with reported LPC decreases on the order of 90-95% for particles > 2 µm and up to 70% for particles >0.56 µm. Other components like valves and dispense pumps demonstrated relatively low impact on LPCs. There are several other factors besides LPCs such as tendency of clogging, service intervals and dispensing precision that influence the selection of these components.

TABLE II. Relative Impact of Different Elements on LPCs in Sillica Slurry

Element		**Potential Impact on LPC**		
		Particles >0.56 µm	**Particles >2 µm**	**Impact Rating**
Shipping Containers & Tanks	No headspace	**+80%**	**+100%**	**medium**
	with headspace	**+100%**	**+1'000%**	**high**
Valves (10'000 cycles on volume of 25L)		**+3%**	**+5%**	**low**
Recirculation Pumps (1000 turnovers)	Bellows	**+2'800%**	**+6'300%**	**high**
	Diaphragm	**+1'800%**	**+5'300%**	**high**
	MagLev	**- 40%**	**-**	**low**
Loop Filters (3 µm, steady state, continuous recirculation)		**-60%**	**-90%**	**high**
POU Filters (0.5 µm, 1 pass)		**-70%**	**- 95%**	**high**
Dispense Pumps (1 pass)	Peristaltic	**+6%**	**+7%**	**low**
	MagLev	**-**	**-**	**low**
Loop Design (impact of extreme negative suction head of -0.83 Bar)		**+10%**	**+500%**	**high**

The Impact of LPC Levels on Scratch Rates

A key question is: Do increased LPC levels (due to slurry handling elements) correlate with higher CMP defectivity and reduced surface quality? Several independent studies involving different slurries and test substrates have attempted to address this question [3,4,5,18,19]. In all studies, CMP slurries were extensively handled with different types of pumps before polishing substrates. A relatively good correlation between LPC levels and scratch density was reported in a CMP study involving low-k wafers and silica slurry [19] (figure 9). In the same study, an even stronger correlation was reported between LPC levels and surface roughness (figure 10). Other studies reported similar results for other slurries and substrates [4,5,18]. Since the test conditions for these studies were quite different, the comparability of the data is limited. There were significant differences in measurement methods, measurement instruments and even in the definition of LPC levels and scratch densities.

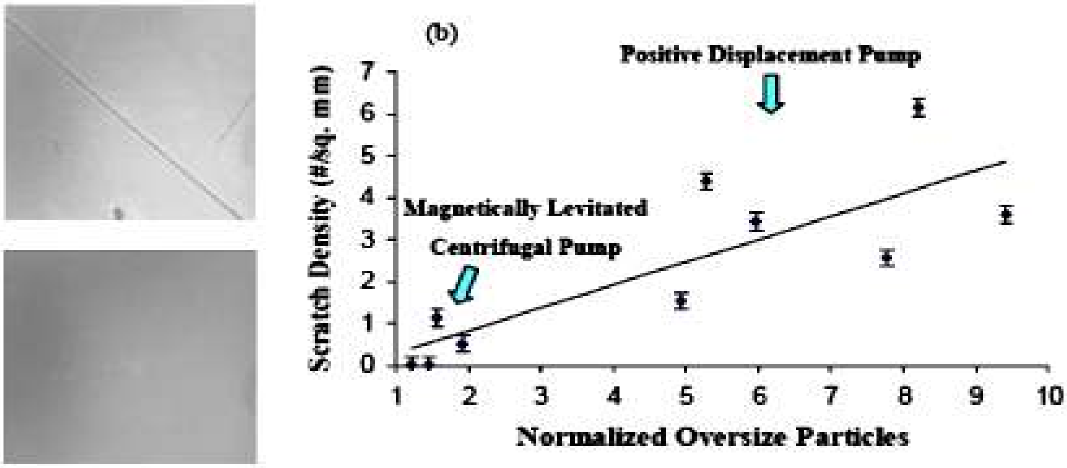

Figure 9: Correlation between scratch density on low-k wafers and LPC in silica slurry after slurry handling with different pumps (Source: Ref. 19)

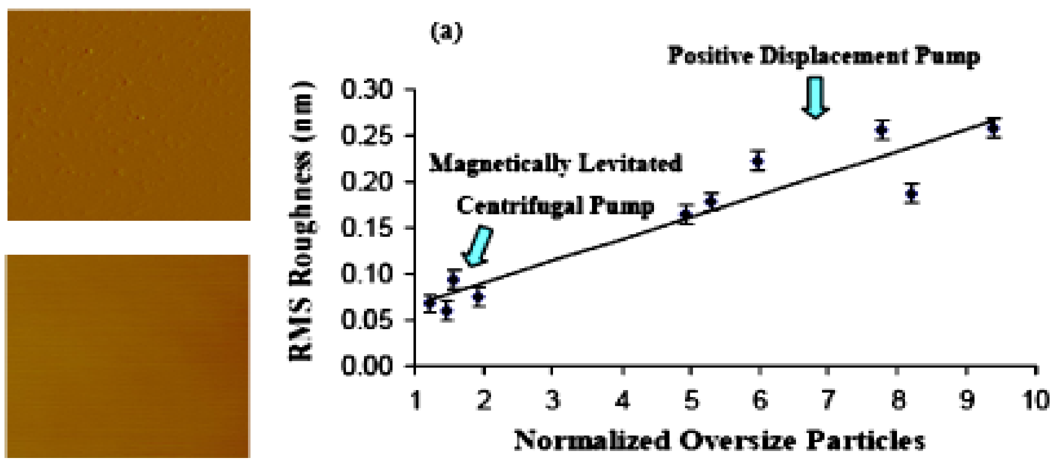

Figure 10: Correlation between surface roughness on low-k wafers and LPC in silica slurry after slurry handling with different pumps (Source: Ref. 19)

In order to make a better comparison, the LPC and scratch density increases resulting from pneumatic pumps seen in these studies were normalized to the increases seen with maglev pumps. By analyzing relative increases of LPC levels and scratch densities rather than absolute increases, the effect of variations in measurement methods and definitions should be diminished. The results of the normalization are summarized in Table III.

In 13 of the 14 tests summarized in Table III, a significant increase of LPCs >0.56 µm was reported when the slurries were circulated with a pneumatic pump compared to a maglev pump. In only one test, involving ball milled ceria slurry, no significant difference of LPCs resulted from the different circulation pumps. For some of the tests, LPCs > 1 µm and > 2 µm were also reported. In all 7 experiments (using 3 different types of slurries) in which supermicron data were available, the relative concentration increases of particles > 1 µm significantly exceeded those > 0.56 µm. For the alumina slurry used in four CMP tests, the relative concentration of particles > 2 µm exceeded the relative concentration of particles > 1µm by more than 20 times and the relative concentrations of particles > 0.56 µm by more than 100 times.

In 12 of the 14 tests summarized in Table III, significantly higher (70% to 1200%) scratch densities were observed in tests with the pneumatic pump relative to the maglev pump (column 8 in Table III). No significant impact of pump type on scratch density was found in the remaining two polishing tests. To compensate for the differences in the number of turnovers performed in each experiment, the relative increase in scratch densities were normalized to 100 turnovers. (It was assumed that the increase in scratch density increased linearly with increasing turnovers). Even at 100 turnovers, 11 of the 14 tests had significant (meaning > 10% per 100 turnovers) increases in relative scratch density, varying from 12% to 65% per 100 turnovers. In all 12 tests in which a relative increase in scratch density after circulation with pneumatic pump compared to maglev pump was observed, a significant relative increase in LPCs > 0.56 µm was also measured. There was only one case in which a significant increase of LPCs > 0.56 µm was seen with no measured difference in scratch density.

TABLE III. Relative increase in LPCs and scratch densities after circulation of slurries with pneumatic pump vs. maglev pump for various slurry and substrate types.

Ref. #	Slurry type	# of turn overs	Relative increase in LPCs after circulation with pneumatic pump compared to maglev pump			Substrate	Relative increase in scratch density after circulation with pneumatic pump compared to maglev pump	Normalized relative increase in scratch density (per 100 turnovers)	Ratio of relative increase in LPCs to the relative increase of scratch density		
			0.56 µm	1 µm	2 µm				0.56 µm	1 µm	2 µm
3	Colloidal silica	500	320%	920%	880%	BD1	190%	38%	1.7	4.8	4.6
		500	320%	920%	880%	ULK	75%	15%	4.4	12	11
22	Ceria	500	60%	1600%	n.a.	TEOS	140%	28%	0.43	11	n.a.
5	Alumina	2000	60%	370%	8200%	TEOS	>600%	>30%	<0.4	<0.6	<13
		2000	60%	370%	8200%	Cu	1300%	65%	0.05	0.28	6.2
		2000	60%	370%	8200%	BD1	600%	30%	0.10	0.62	14
		2000	60%	370%	8200%	Patterned	850%	43%	0.07	0.43	10
4	Colloidal silica	2900	280%	n.a.	n.a.	NiP	840%	29%	0.33	n.a.	n.a.
18	Colloidal silica	2900	700%	n.a.	n.a.	Glass	360%	12%	2.0	n.a.	n.a.
	Colloidal silica	2900	600%	n.a.	n.a.	Glass	680%	23%	0.88	n.a.	n.a.
	Colloidal ceria	2900	380%	n.a.	n.a.	Glass	350%	12%	1.1	n.a.	n.a.
	Fumed silica	2900	110%	n.a.	n.a.	Glass	0%	0%	0	n.a.	n.a.
	Ball milled ceria I	2900	15%	n.a.	n.a.	Glass	0%	0%	0	n.a.	n.a.
	Ball milled ceria II	2900	470%	n.a.	n.a.	Glass	70%	2%	6.7	n.a.	n.a.

In order to directly compare the relative increases in LPCs to the relative increases of scratch densities in each experiment, the ratio of relative increase in LPCs to the increase of scratch densities was calculated (columns 10-12 in Table III). For the 12 tests which showed an effect on scratch density, the ratios of relative increase of LPCs > 0.56 µm to increase in scratch densities varied by two orders of magnitude (0.05 to 6.7). Therefore it seems to be impossible to predict scratch densities quantitatively just from LPCs > 0.56 µm for different slurries and substrates. For the tests in which supermicron data were available, a dramatic change in large particle concentration increase ratios (LPCs > 1 µm and 2 µm) was observed, compared to a more moderate difference in increase ratios for LPCs > 0.56 µm. For such slurries, increase ratios for LPCs > 2 µm seem to be a better indicator for scratch densities. The ratios of relative increase of LPCs > 2 µm to increase in scratch densities varied only by a factor of 3 (4.6 to 14). It seems to be necessary to select the right indicator (in terms of large particle size) for a given slurry and substrate combination in order to predict scratch densities halfway accurately.

Summary

Recirculation pumps, shipping containers and tanks can have a significant negative impact on slurry health by increasing LPCs. High negative suction pressure can also have a profound impact on large particle generation and should be avoided. CMP slurry filters can partly remove large particles and have a positive effect on slurry health. Despite the effectiveness of slurry filtration, other components in the slurry loop can still significantly degrade slurry health. Proper selection of pumps, minimizing gas headspace in containers, blanketing mixing and day tanks with humidified nitrogen and proper loop design are key measures to retain slurry health. Increased LPC levels (due to suboptimal slurry handling elements) was found to correlate with higher scratch densities in 12 out of 14 CMP tests involving different slurries and substrates. Prediction of scratch densities from LPC results remains difficult since it depends on a number of factors including slurry type, substrate type and LPC particle size.

Acknowledgments

The authors would like to thank Rajiv Sing, Feng-Chi Chang, Siddharth Tanawade, Gary Scheiffele, Yuzhuo Li, Craig Burkhard, Yongquin Lan, Shyam Venkataraman, Rakesh Singh, Rob Donis, Matthew Fisher, S. Hamid and Leland Bauck for providing data and graphs for this paper.

References

1. K Nicholes, R K Singh, D Grant, and M R Litchy, “Measuring particles in CMP slurries”, *Semicond. Int.*, vol. 24, pp. 201-206, 2001
2. R K Singh, G Conner, and B R Roberts, “Handling and filtration evaluation of a colloidal silica CMP slurry“, *Solid State Technology*, vol. 47, pp. 61-66, 2004.
3. FC Chang, S Tanawade, R Singh: “How pump-induced particles affect low-k CMP defectivity,” *Semiconductor International*, September 2008, pp46-50.
4. YQ Lan and Y Li: “Effect of pump-induced particle agglomeration on CMP” *SolidState Technology*, August 2008, pp40-48.

5. R Donis, M Fisher, L Bauck: "Effects of slurry distribution using diaphragm and centrifugal pumps on the defectivity in a Cu CMP process" *International Conference on Planarization/CMP Technology,* Fukuoka, J (2009)
6. R K Singh, "CMP pump effects on filter life" *Levitronix CMP Users' Conference*, Santa Clara, CA, 2005
7. MR Litchy and R Schoeb: "Effect of shear stress and pump method on CMP slurry," *Semiconductor International*, December 2004, pp87-90.
8. L Bauck and R Donis: "Slurry pump affects on CMP" *International Conference on Planarization/CMP Technology,* Seoul (2005)
9. MR Litchy, DC Grant and G Van Schooneveld: "Effects of fluid handling components on slurry health," *Transaction on Electrical and Electronic Materials* of the Korean Institute of Electrical and Electronic Material Engineers (2006).
10. YQ Lan, C Burkhard and Y Li: "Surface mediated particle-particle aggregation during CMP slurry delivery and handling", 2007 Levitronix CMP Users Conference.
11. DC Grant MR Litchy and R Schoeb: "The effect of cavitation on particle agglomeration in CMP slurries," *Proceedings of the 26th Annual Semiconductor Pure Water and Chemicals Conference*, Sunnyvale, CA., (2007) pp119-134.
12. MR Litchy and R Schoeb: "Effect of particle size distribution on filter lifetime in three slurry pump systems," *Materials Research Society Symposium Proceedings*, 867 W2.8.1 (2005).
13. MR Litchy, DC Grant, and R Schoeb: "The effect of pump type on various CMP slurries," *Proceedings of the 26th Annual Semiconductor Pure Water and Chemicals Conference*, Sunnyvale, CA., (2007) pp119-134.
14. MR Litchy and DC Grant: "Effect of Pump Type on the health of various CMP slurries", *Semiconductor Fabtech*, 33rd Edition, (2007), pp53-59.
15. RK Singh, K Anderson B Bjorneberg and J Hahn:"Precision Flow Control and Enhanced Filter Lifetime in Magnetically Levitated Pump Based CMP Slurry Delivery Systems", CMP-MIC Conference Fremont, CA (2007)
16. MR Litchy, DC Grant, and R Schoeb: "Susceptibility of different slurry types to agglomeration", presented at the 2009 CMP Users Conference, sponsored by Levitronix.
17. P Levy: "Issues associated with testing the consistency of CMP filtration products", *Levitronix CMP Users Conference*, Santa Clara, CA, 2009
18. S Venkataraman and Y Li: "Chemical mechanical polishing of hard disk drive substrates",*Levitronix CMP Users Conference*, Santa Clara, CA, 2009
19. FC Chang: "Externally induced agglomeration during chemical mechanical polishing of Metals and Dielectrics", Dissertation, University of Florida, 2008.
20. MR Litchy and DC Grant: "Effects of slurry handling components on slurry health", *Semiconductor Pure Water and Chemicals Conference*, Sunnyvale, CA, 2005
21. R K Singh, C R Wargo, B Mullee and B Milmore :" Handling, Filtration and Polishing Performance Characterization of Next Generation CMP Slurries", *Levitronix CMP Users Conference*, Santa Clara, CA, 2009
22. R Singh and FC Chang: "Why do Specific pumps increase over-size particle size distribution in CMP slurries",*Levitronix CMP Users Conference*, Santa Clara, CA, 2008

CTA publication 114: China Semiconductor Technology International Conference (CSTIC 2013), Shanghai, China, March 2013.