Examining the Relationship Between Silica Particle Behavior and CMP Defects

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ABSTRACT

CMP defects continue to challenge semiconductor manufacturers and require materials suppliers, equipment and process providers to deliver solutions that improve process yield and device reliability. An area of further study is the formation of particles and agglomerates during the mixing and dilution of slurries under excess shear stress. A key attribute of effective slurry performance is the particle distribution, and thus a low LPC (large particle count). We previously described the decrease of particle and agglomerate retention in Silica (SiO_2) slurry at a low concentration in a low flow rate system.[1] Three hypotheses were proposed: a) Shear stress from Point-of-use system (low concentration slurry at low flow rate filtration) leads to silica particle agglomeration; b) Higher agglomeration rates due to low bath turnover rates with the retention dropping at higher turnover counts; c) Agglomeration after the collection of post-filtration samples. This study focuses on the change in large particle size distribution during low turnover rates and the influence of large particles on filtration efficiency. Through this study we can suggest bulk and point-of-use (POU) filtration solutions for LPC improvement to reduce the number of defects in final wafer polishing to improve both wafer yields and device reliability.

INTRODUCTION

Micro-scratch defects can cause current leakage between gates in integrated circuit (IC) fabrication. Large and agglomerated slurry particles are known to be a critical micro-scratch defect source during chemical mechanical planarization (CMP).[2] For superior slurry large particle reduction, several common filtration points are set (Figure 1).

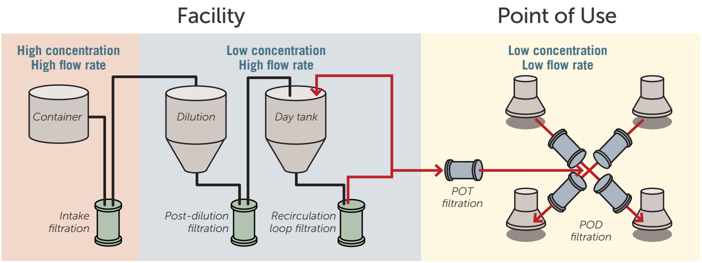


Figure 1. Common filtration points in a slurry delivery system.[1]

For Chemical Distribution System (CDS) loops, high concentration slurry from shipping containers is filtered at a high flow rate to dilution and mixing tanks. Then low concentration slurry is filtered at a post-dilution point and pumped to a recirculation loop in a day tank with a high flow rate. Point-of-use (POU) filtration, including point-of-tool (POT) and point-of-dispense (POD), are performed at low flow rates for optimal slurry dispense during polishing. In a previous study to investigate the filtration efficiency at different filtration points, various abrasive concentrations with different flow rates were compared (Table 1). Colloidal silica and ceria were selected for this case study. Entegris new polypropylene depth media filter with new Nano-Melt-Blown technology "NMB01" (pore rating 0.1 μm) and "NMBA5" (pore rating 0.05 μm) were tested.[1]

Table 1. Experimental condition to simulate facility and POU filtration in previous study[1]

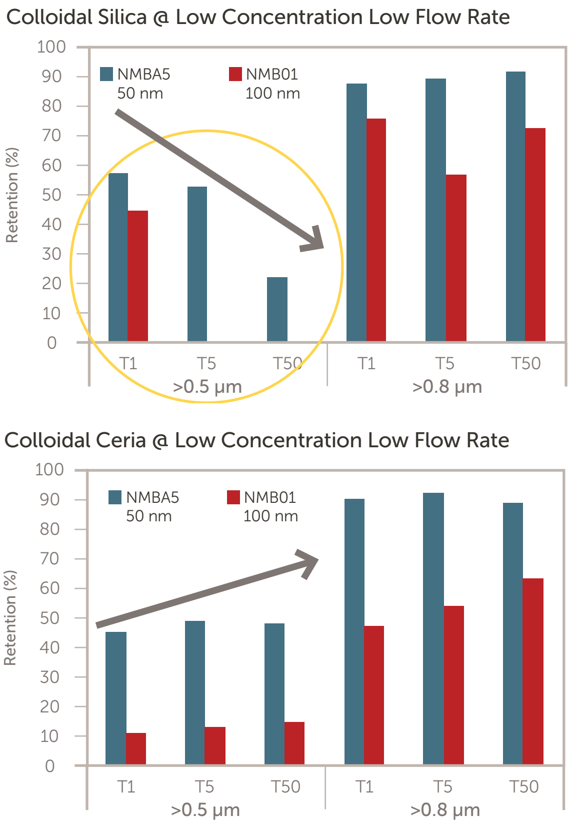
				FLOW RATE	
Filter	**Abrasive type**	**Mean particle size**	**Concentration**	**High (CDS filtration)**	**Low (POU filtration)**
NMBA5 and NMB01	Colloidal silica	55 nm	20%	5 L/min	—
			4%	5 L/min	250 mL/min
	Colloidal ceria	150 nm	1%	5 L/min	—
			0.1%	5 L/min	250 mL/min

As expected, a high flow rate system which simulated CDS filtration showed improving retention with repeated circulation. However, low concentration colloidal silica with a low flow rate (POU filtration system) showed decreasing particle retention with more circulation turnovers (Figure 2). Decreasing retention was attributed to increasing large particle counts during circulation. In the previous study, colloidal silica was found to be more sensitive to shear stress than ceria.[3,4] It is consistent to the result that only colloidal silica but not ceria suffered from particle agglomeration under the same condition.

Several hypotheses were proposed regarding particle agglomeration with turnovers:

1. Shear stress from the POU test system may lead to silica particle agglomeration. A peristaltic pump, which is commonly used for lab scale POU filtration, was tested at a low rate/low concentration condition in Figure 2. A magnetically levitated centrifugal (MLC) pump was used for the high flow rate system instead. Different shear stress generated from different pumps has been reported as a factor of silica agglomeration.[3]
2. Lower bath turnover rates would cause a higher agglomeration rate during the circulation.
3. Agglomeration happened after the collection of post-filtration samples which shifted the measured large particle size distribution to larger sizes.

In this paper, the proposed mechanisms of colloidal silica agglomeration were evaluated.


Colloidal Silica @ Low Concentration Low Flow Rate

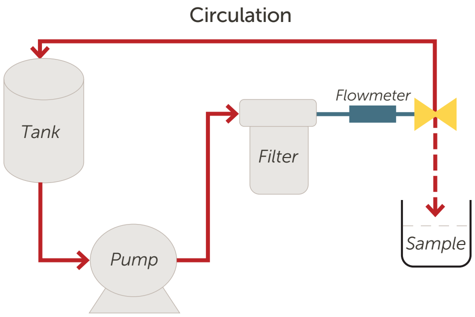
Colloidal Ceria @ Low Concentration Low Flow Rate

Figure 2. Retetion of colloidal silica and ceria POU filtration (Low concentration with low flow rate).[1] Retention (%) = [Feed LPC-filtrate LPC]/Feed LPC.

EXPERIMENTAL

Filtration

Ten liters of colloidal silica abrasive was filtered by NMB01 and NMBA5 5" filters. High-flow rate (5 L/min) filtration was performed by Levitronix® pump BSM-3.2. Low-flow rate (250 mL/min) filtration was set using peristaltic pump Masterflex® L/S® and MLC pump Levitronix® BPS-i30.2. At 5 L/min flow rate, one turnover (T1), five turnovers (T5), and 50 turnovers (T50) filtrate were collected after circulation of two min, 10 min, and 100 min. Likewise, T1, T5, and T50 samples were collected after circulation of 40 min, 200 min, and 2000 min at 250 mL/min flow rate (Figure 3). LPC was analyzed by the Entegris Particle Sizing System AccuSizer® FX-Nano. Accumulated particle counts at channels >0.5 µm and >0.8 µm were monitored. Particle size distribution (PSD) was measured by dynamic light scattering (DLS) using Particulate Systems NanoPlus-3.



Test volume	Flow rate	Turnovers	Circulation time
10 L	High-flow rate 5 L/min	T1	2 min
		T5	10 min
		T50	100 min
	Low-flow rate 250 mL/min	T1	40 min
		T5	200 min
		T50	2000 min

Figure 3. Configuration and test condition of CMP filtration test stand.

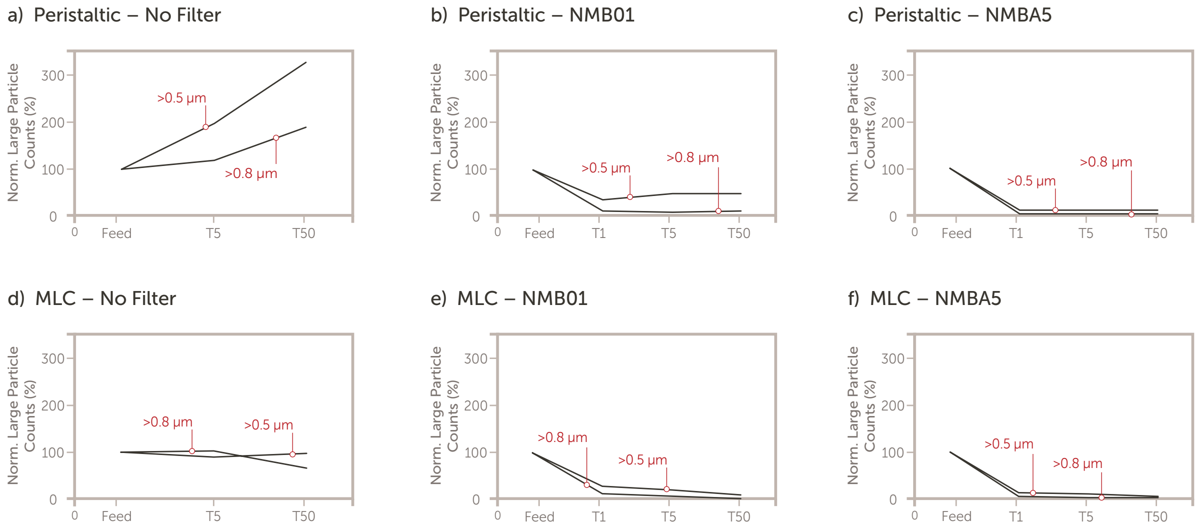
Polishing Setup and Wafer Defectivity

Commercial Cu slurry (colloidal silica) and oxide slurry (0.25 wt% ceria) were diluted to POU before filtration. POU slurry was filtered by NMBA5 with a flow rate of 250 mL/min. Eight inch Cu and PETEOS wafers were polished by Applied Materials Mirra® tool under down force pressure of 3 psi and platen speed of 90 rpm at a slurry flow rate of 175 mL/min for Cu slurry and 150 mL/min for Oxide slurry. Dow IC1010TM pad was used in-situ with conditioning disk 3MTM AP160. Post clean AG3300 after Cu polishing and SC1 after oxide slurry were used after polishing. Total defect counts were measured by KLA-Tencor Surfscan® SP1.

RESULTS AND DISCUSSION

Large Particle Counts Generation Rate Using Different Type of Pump

The MLC pump was compared to the peristaltic pump for low-flow rate filtration. In Figure 4a, LPC drastically increased to 328% (>0.5 um) and 190% (>0.8 um) after T50 using the peristaltic pump without filters. With filter NMB01, the LPC of monitoring particle size are 177% (>0.5 µm) and 278% (>0.8µm) reduced compared to the “without filter” condition after 50 turn-overs (Figure 4b). NMBA5 further reduced 188% (>0.5 µm) and 317% (>0.8 µm) (Figure 4c). By contrast, circulation using MLC pump without filter kept around 100% LPC compared with feed after T50 (Figure 4d). Therefore, LPC was reduced when using NMB01 and NMBA5 filtration using MLC pump (Figure 4e and 4f). Based on the result, the peristaltic pump would drive more shear stress than the MLC pump and significantly induce silica agglomeration. The peristaltic pump without a filter had the highest agglomeration ratio after T50 (Figure 4a), whereas the MLC pump with NMBA5 showed the lowest LPC after T50 (Figure 4f). However, their particle size distribution (PSD) remained the same (Figure 5). The agglomeration level was relatively trivial to influence the whole working particle PSD.



a) Peristaltic – No Filter

b) Peristaltic – NMB01

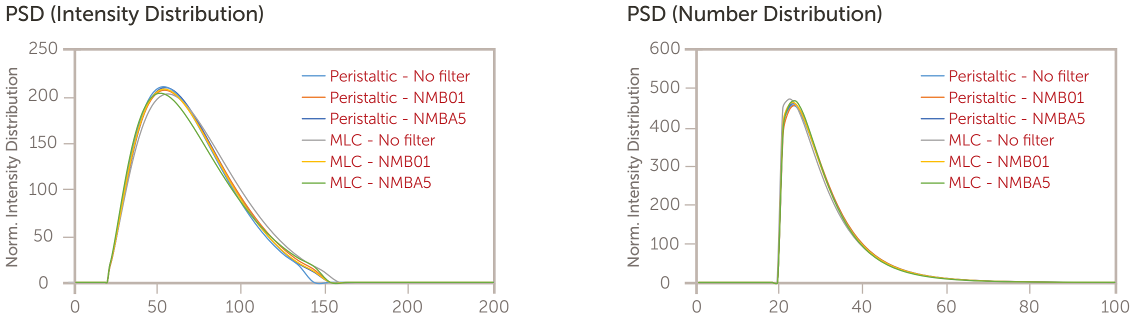
c) Peristaltic – NMBA5

d) MLC – No Filter

e) MLC – NMB01

f) MLC – NMBA5

Figure 4. Colloidal silica (1wt%) circulation by peristaltic pump: a) No filter, b) NMB01, c) NMBA5; MLC pump: d) No filter, e) NMB01, f) NMBA5. Particle agglomeration increased with turnovers using peristaltic pump without filters.



PSD (Intensity Distribution)

PSD (Number Distribution)

Figure 5. Particle size distribution of 1 wt% colloidal silica filtrate using two types of pump after T50.

Impact of Lower Bath Turnover Rates on Agglomeration Rate

Filtration at different flow rates was evaluated using MLC pump (Figure 6). Four wt% silica, which represented low concentration in the previous study, was tested. For the same concentration, low-flow rate showed better retention. This may be attributed to the low-shear flow, which caused less agglomeration and offered enough equilibrium time for silica filtration compared to the high-flow rate.

Agglomeration Level After the Collection of Post-filtration Samples

To investigate the agglomeration level after idle time of filtrate which was collected from low filtration rate (250 mL/min), the LPC was re-measured after three days (Figure 7a). Although the retention of T50 filtrate after three days showed a slight reduction due to the minor increase of LPC (Figure 7b), the LPC was still at the same level after three days idle. This result is consistent with our previous study.[4,5] Slurry agglomeration ratio remained at the same level after 15 hours idle time due to less shear stress caused by the nanofiber media (Figure 8). In contrast, the agglomeration ratio of a conventional non-nano-fiber filter showed three times higher at >1 µm particle size channel and 10 times higher at >8 µm. Agglomeration after the collection of low-flow rate filtrate would not be the main root cause for the retention drop with turnovers.

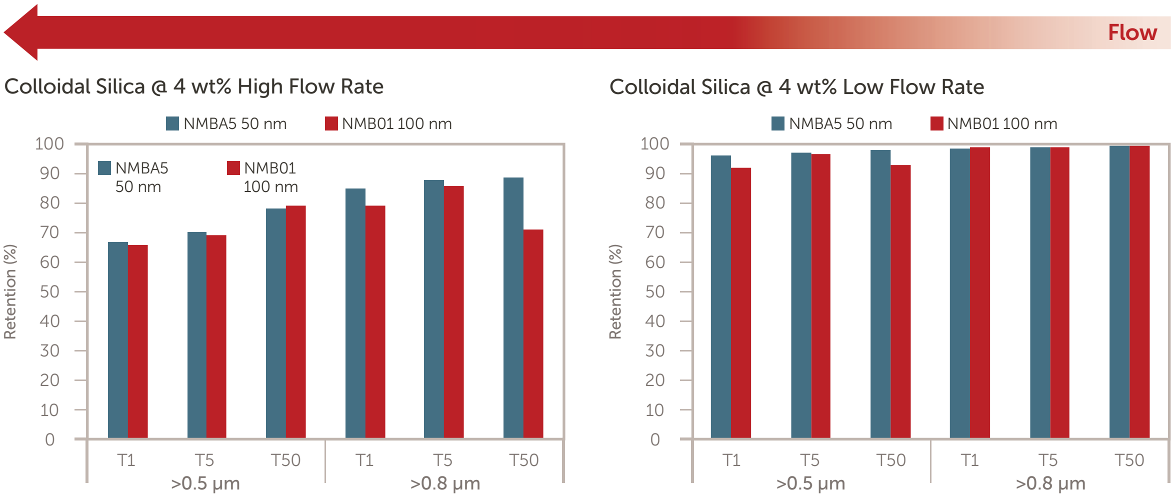
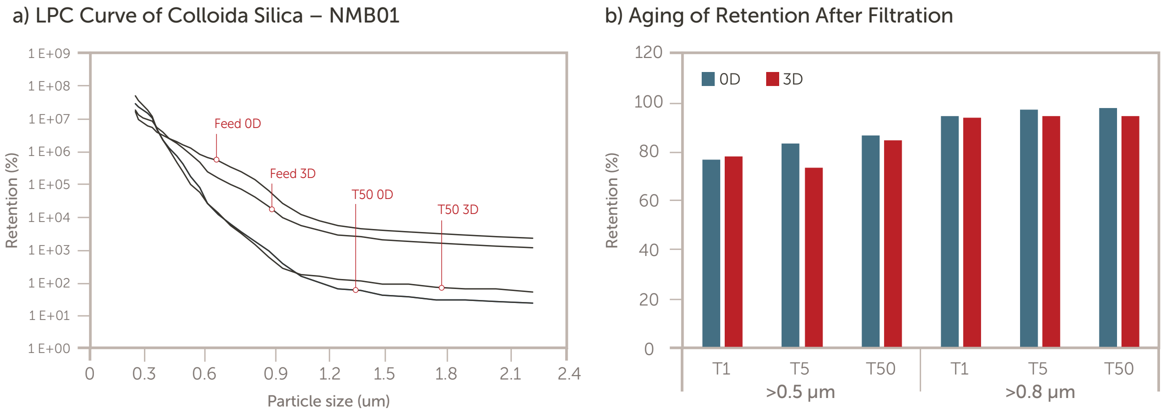


Figure 6. Colloidal silica filtration at high flow rate (5 L/min) or low flow rate (250 mL/min) using MLC pump.


a) LPC Curve of Colloida Silica – NMB01

b) Aging of Retention After Filtration

Figure 7. Aging of colloidal silica filtered by NMB01 at low flow rate (250 mL/min). a) The accumulative large particle distribution of feed and filtrate after 3 days idle time. b) Retention of freshly collected filtrate (0D) and filtrate after 3 days idle time.

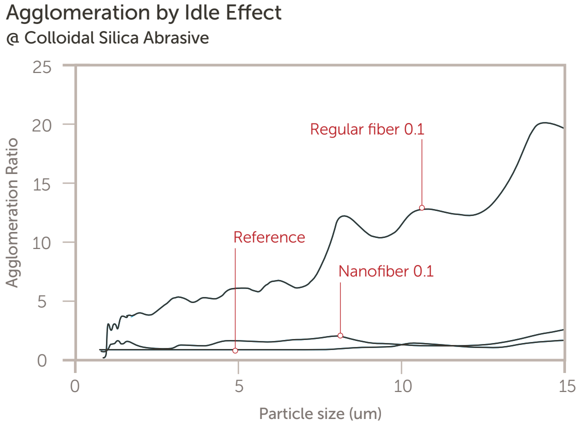
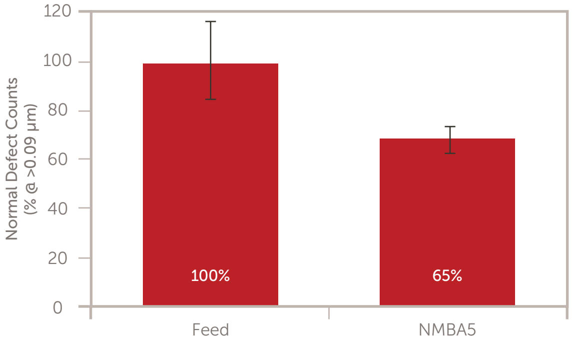


Figure 8. Agglomeration by idle effect.[4] Agglomeration ratio = (LPC after filtration)/(LPC without filtration). The agglomeration ratio of conventional filter is much higher than Nanofiber filter (NMB01).

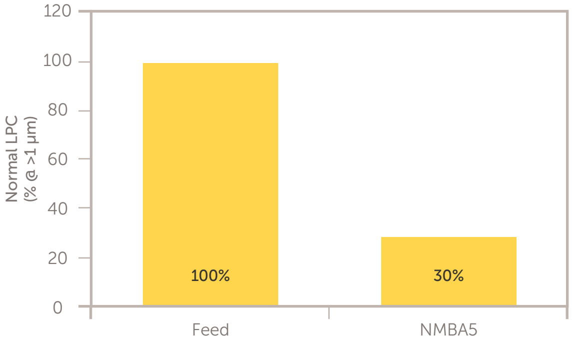
Impact of Filtration on Wafer Defectivity

To explore the NMBA5 filtration efficiency on defect count reduction, colloidal silica type Cu slurry and ceria type bulk oxide slurry filtrate were used for a polishing evaluation (Figure 9). Total defect counts on Cu could be reduced around 32% when the LPC reduced 70%. Total defects on an oxide wafer were 34% reduced when the LPC of oxide slurry decreased 74%. The result shows that filtration assists in defect improvement. While not all defects are removed, there are several other sources of on-wafer defectivity that cannot be addressed solely by filtration.

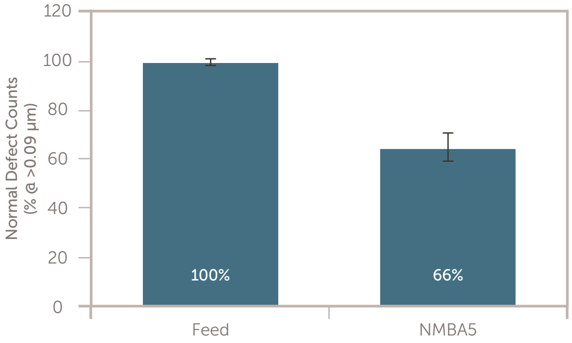
a) Defect of Cu Wafer



b) LPC of Cu Slurry



c) Defect of Ox Wafer



d) LPC of Ox Slurry

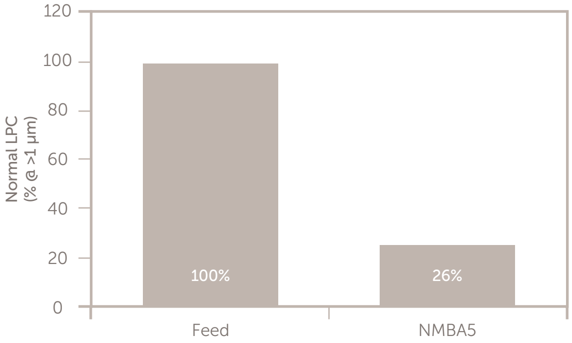


Figure 9. Normalized defect counts on polished wafers and LPC of polishing slurry. a) Cu defect counts and b) LPC of feed and NMBA5 filtered colloidal silica Cu slurry. c) Oxide defect counts and d) LPC of feed and NMBA5 filtered ceria slurry.

SUMMARY

The phenomena of particle agglomeration with turnovers under low flow rate conditions in a previous study was dominated by a peristaltic pump. This pump provided more shear stress on colloidal silica compared with a pulseless MLC pump. Under MLC pump filtration, low flow rate facilitated better filtration efficiency than a high flow rate. This may be attributed to the low shear flow which caused less agglomeration than a high flow rate. Low flow rate would also offer enough equilibrium time for filtration.

The results of the current study, showing that nanofiber filters NMB01 and NMBA5 significantly reduced large particles without changing the working particle distribution, and that low flow rates provide optimal filtration of colloidal silica, provide guidance to those seeking bulk and POU solutions to reduce defects in final wafer polishing that improve both wafer yields and device reliability.

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