MICROCONTAMINATION CONTROL | APPLICATION NOTE

Effect of Flow Rate and Concentration on Filtration Efficiency of Colloidal Abrasives

Authors: Mia Wu, James Lee, Henry Wang, Steven Hsiao, Bob Shie, HJ Yang – Entegris

ABSTRACT

Sub 14 nm technology nodes require increased chemical mechanical planarization (CMP) steps compared to previous generation semiconductor devices. For both bulk slurry manufacturers and integrated device manufacturers (IDM), it's critical to reduce large particle counts (LPC), which are undesirable during the CMP process and can create micro-scratches that lead to wafer defects. Slurry filters strive to remove LPC that are generated during the mixing process and formed as gels or agglomerates while maintaining the overall particle size distribution of working particles. Efficient filtration of large particles reduces the number of micro-scratches during final wafer polishing and enables higher wafer yields.

To achieve a very low LPC level, filters are used in multiple locations. These locations are categorized as bulk, point-of-tool (POT), and point-of-dispense (POD). The right choice of filtration product at each point of the liquid slurry delivery system, which have different slurry concentrations and flow rate requirements, will affect the outcome. Many studies in slurry filtration have shown the importance of media structure and material characteristics to improve filtration efficiency. However, none addresses the importance of flow rate and concentration conditions together. This study focuses on understanding the effect of these critical factors on filtration efficiency by evaluating two nano-meltblown (NMB) Entegris CMP filters (NMB01 100 nm and NMBA5 50 nm) with colloidal ceria (CeO_2) and silica (SiO_2) abrasives. The importance of optimization of abrasive concentration, flow rate, and filtration media is demonstrated.

INTRODUCTION

Silica- and ceria-based slurries are widely used in CMP processes. Advanced filtration is necessary in most CMP processes to reduce unwanted large particles to decrease micro-scratch-related wafer defects. Depending on the filtration point, either at the chemical manufacturer (bulk filtration), IDM facilities, or at point of use (POT or POD), both concentration of slurry and flow rate will be different (see Figure 1).

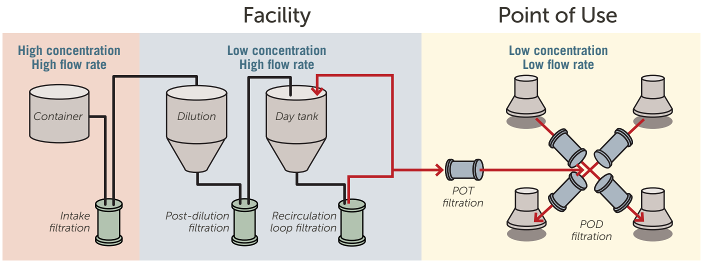

Figure 1. Concentration and flow rate conditions in the slurry delivery system.

The high concentration/high flow-rate-condition is typically used by the bulk chemical manufacturers as well by IDMs in their facilities systems. Post dilution to the desired concentration, the slurry is typically recirculated through a filter at a low concentration and high flow rate. At the point of dispense filtration location, the slurry is dispensed on wafer at the low concentration/low flow rate condition, though in rare cases a high flow rate may be chosen by the end user.

EXPERIMENTAL

Experimental Conditions

In this study, we measured large particle count pre- and post-filtration with various abrasive concentrations and two different flow rates. Colloidal silica and ceria slurries were tested with two Entegris nanofiber CMP filters (NMB01 100 nm and NMBA5 50 nm). Particle counts were measured using an AccuSizer® Fx Nano. As shown in Figure 2, we focused on the experimental understanding of the filters performance in the three most commonly encountered CMP slurry filtration system conditions.

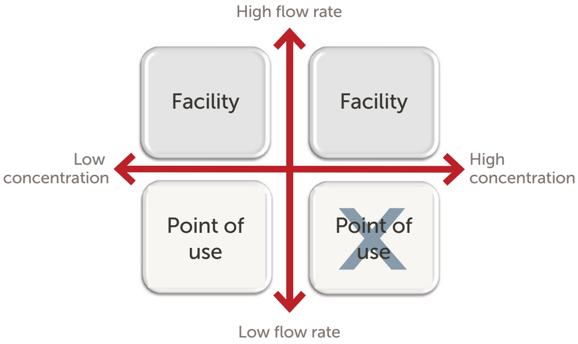

Figure 2. CMP slurry filtration conditions evaluated.

Slurries and Filtration Media

Utilizing Entegris' advanced membrane technology, the study finds that NMB-based filters provide very fine fibers from 50 nm to 1 µm and offer lower shear force than traditional microfiber devices to minimize abrasive agglomeration.[1] Two retention rated NMB media were selected: 100 nm and 50 nm.

Most commercial slurries contain abrasive particles (silica, ceria) and additive chemicals for optimal removal rate and selectivity. In this study, two concentrations of pure abrasive silica (20% and 4%) and ceria (1% and 0.1%) with no additive chemical, were used as the challenge particles (see Table 1). Two flow rates were selected: 250 mL/min to simulate the low flow rate condition and 5 L/min for the high flow rate condition. Large particle count (LPC) or particles greater than 0.5 µm and 0.8 µm were recorded after up to 50 bath turnovers (T50).

Experimental Setup

A CMP test stand shown in Figure 3 and described in Lu et al.[2] was used for this study.

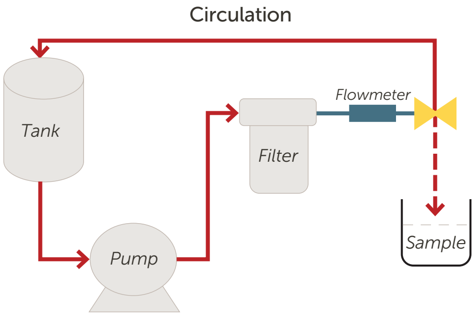

Figure 3. CMP filtration test stand configuration.

Table 1. Experimental conditions

Filter	Abrasive type	Mean particle size	Concentration	FLOW RATE	
				High	**Low**
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

RESULTS AND DISCUSSION

Colloidal Silica Abrasive Results

Figure 4 describes the retention results of the two filters (NMB01 and NMBA5) under three different conditions:

a. High concentration, high flow rate

b. Low concentration, high flow rate

c. Low concentration, low flow rate

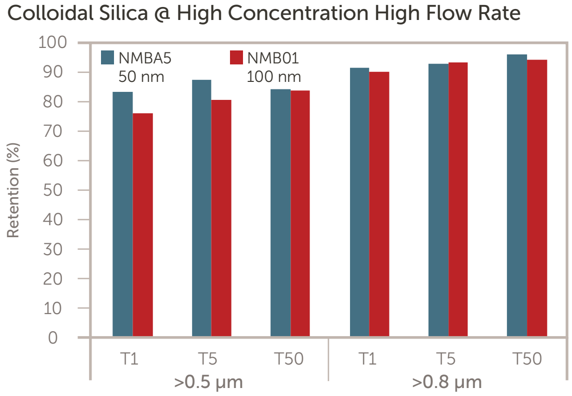

Figure 4a. Retention results of colloidal silica abrasive in a high concentration/high flow rate.

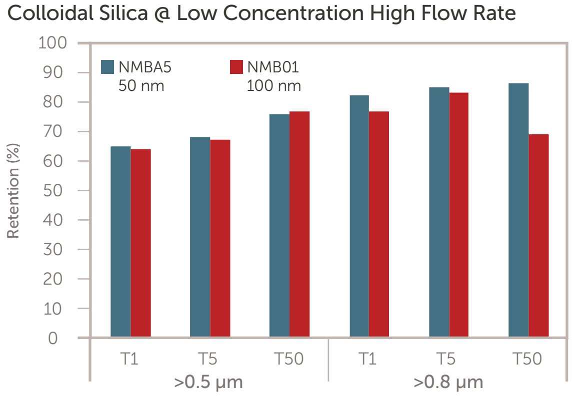

Figure 4b Retention results of colloidal silica abrasive in a low concentration/high flow rate.

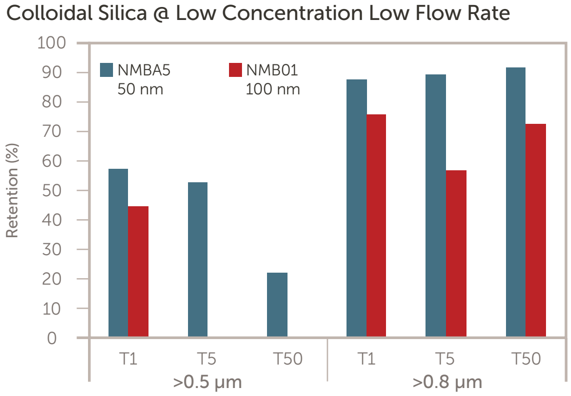

Figure 4c Retention results of colloidal silica abrasive in a low concentration/low flow rate.

High concentration with high flow rate: For both NMB01 and NMBA5, the retention results of particles greater than 0.5 and 0.8 µm are similar and high at T1 and T50 (Figure 4a). The high retention can be explained by the relatively large zeta potential gap between the filter material (polypropylene) and the colloidal silica abrasive, described in Figure 5, which promotes attraction of the particles to the media and results in high retention performance.

Low concentration with high flow rate: The retention results are slightly lower (Figure 4b) compared to the high concentration/high flow conditions. From Figure 5, the zeta potential of the colloidal silica approaches the isoelectric point at lower pH (i.e., low silica concentration). We suspect the large colloidal silica particles to be unstable at their isoelectric point and potentially agglomerate. Future testing is planned to verify: 1) no change in size distribution of the working particles and 2) potential agglomeration of large particles.

Low concentration with low flow rate: The retention of colloidal silica under those conditions (Figure 4c) can be explained by analyzing the agglomeration behavior of smaller (>0.5 µm) and larger size (>0.8 µm) particles. Ideally, one would expect that a lower flow rate would increase the chance of capture in the filter media compared to the high flow rate because of the lower flux/higher residence of the colloidal silica at all particle sizes. Though the larger size particles follow the expected trend, our results consistently show erratic retention behavior at the lower bin size (>0.5 µm) after multiple turnovers (T5 and T50). Our current hypotheses for the observed low retention measurements are as follows: (a) Higher agglomeration rates due to low bath turnover rates with the retention dropping at higher turnover counts; (b) Agglomeration after the collection of post-filtration samples in addition to (a) which shifts the measured particle size distribution to larger particle sizes.

We are currently in the process of conducting additional experiments to verify the hypotheses.

Regardless, for particles larger than 0.8 µm, we see that NMB filters provide stable, high retention values which would ultimately minimize micro-scratching during "on-wafer" CMP processes.

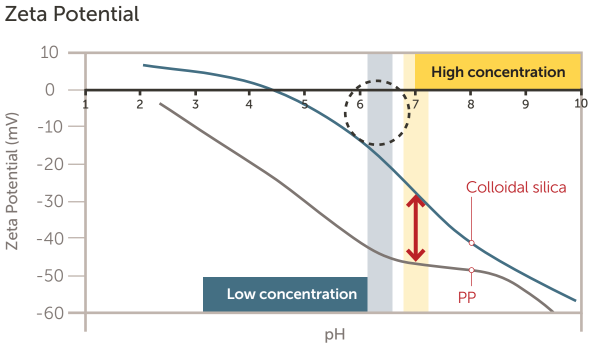

Figure 5. Zeta potential curves of colloidal silica and polypropylene (PP) across the pH range. At high concentration of slurry particles, the pH is alkaline as opposed to lower concentrations where it is more acidic/neutral.

Colloidal ceria abrasive results

Retention performances of NMB01 and NMBA5 were evaluated for colloidal ceria abrasive under three conditions (See Figure 6): (a) High concentration, high flow rate; (b) Low concentration, high flow rate; (c) Low concentration, low flow rate.

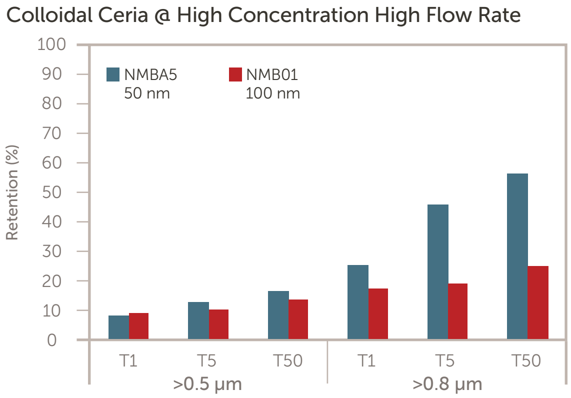

Figure 6a. Retention results of colloidal ceria abrasive in a high concentration/high flow rate.

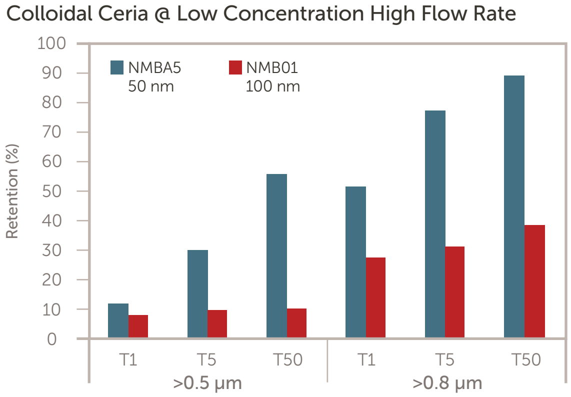

Figure 6b. Retention results of colloidal ceria abrasive in a low concentration/high flow rate.

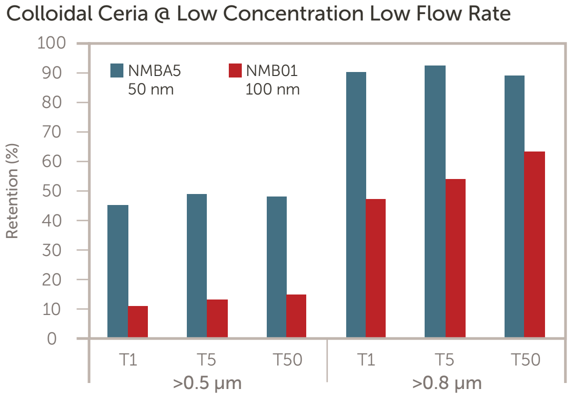

Figure 6c. Retention results of colloidal ceria abrasive in a low concentration/low flow rate.

High concentration with high flow rate: The lower retention performance for colloidal ceria abrasive (Figure 6a) can be attributed to low electrostatic interactions between particles and the polypropylene media. This is illustrated in Figure 7, comparing the zeta potential between ceria abrasive and PP fibers.

Low concentration with high flow rate: The retention behavior is improved compared to the high concentration and high flow rate conditions. We attribute these results to a higher zeta potential gap at low concentration of ceria particles which enhances the nonsieving effect (Figure 7). At higher turnover counts, the retention of particles, especially larger than 0.8 µm, gradually increases via cake filtration with the effect more pronounced in the tighter pore size NMBA5 filter.[2]

Low concentration with low flow rate: Under those conditions, the retention performance of both NMB01 and NMBA5 is constant with the turnover count in contrast to the low concentration high flow rate case where the retention increases with increasing turnover count. At lower flow rates, the lower flux/higher residence time through the filtration media increases the possibility of particle capture. As expected, from Figure 6c, it's clear that a lower flow rate at the same ceria concentration allows both the filtration media to reach the same retention efficiency as observed in the high flow rate case (Figure 6b) at much lower turnover counts (T5 as compared to T50). Overall, the improved retention seen with the tighter pore size NMBA5 50 nm filter illustrates the importance of sieving retention in next generation filtration solutions.

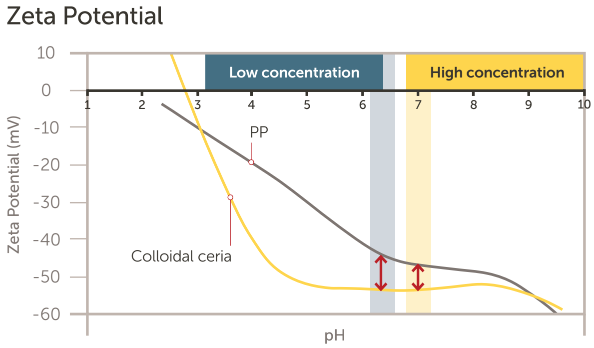

Figure 7. Zeta potential curve of colloidal ceria at different pH.

CONCLUSIONS

The filtration efficiency testing was performed on pure abrasive slurries (silica and ceria) to eliminate possible interaction with slurry chemical additives. Two products were evaluated: Entegris NMB01 and NMBA5 CMP filters. It was found that depending on the nature of the slurry, the flow rate and the concentration, which are representative of the filtration point in the slurry delivery system, the filtration performance can be different. The importance of both a tighter filtration media (sieving) and the zeta potential gap between the particle and filter media (PP) was demonstrated in the cases considered. In the case of silica abrasive, the zeta potential gap plays an important role in enhancing retention, especially at higher concentration. At lower concentration, agglomeration of colloidal silica is a challenge we are currently trying to characterize and explain – the agglomeration effect is severe at low flow rates. In contrast, the ceria abrasive follows the expected trends at all combinations of concentration and flow rate. Overall, the multiple case studies in this paper aimed at replicating the conditions observed during slurry filtration and underscoring the importance of optimization of abrasive concentrations and flow rates. In the future, three way collaborations between bulk chemical manufacturers, IDMs and Entegris can help create optimal solutions geared to overcome specific challenges in CMP slurry filtration.

REFERENCES

1. Y.W Lu, Bob Shie, Steven Hsiao, HJ Yang and Sherly Lee., *"CMP Filter Characterization with Leading Slurry Particles",* ICPT 2013, Taiwan (2013)
2. Y.W Lu, Bob Shie, Dean Tsou, Steven Hsiao and Henry Wang, *'Reducing Slurry Agglomeration with Low Shear Filtration',* ICPT 2014

LIMITED WARRANTY

Entegris' products are subject to the Entegris, Inc. General Limited Warranty. To view and print this information, visit entegris.com and select the Legal & Trademark Notices link in the footer. Entegris does not warranty any failure in the case of customers using unapproved foreign components.

FOR MORE INFORMATION

Please call your Regional Customer Service Center today to learn what Entegris can do for you.
Visit entegris.com and select the Contact Us link to find the customer service center nearest you.

TERMS AND CONDITIONS OF SALE

All purchases are subject to Entegris' Terms and Conditions of Sale. To view and print this information, visit entegris.com and select the Terms & Conditions link in the footer.

Entegris
PURE ADVANTAGE

Corporate Headquarters
129 Concord Road
Billerica, MA 01821
USA

Customer Service
Tel +1 952 556 4181
Fax +1 952 556 8022
Toll Free 800 394 4083