CMP Slurry Filter Testing

AccuSizer®

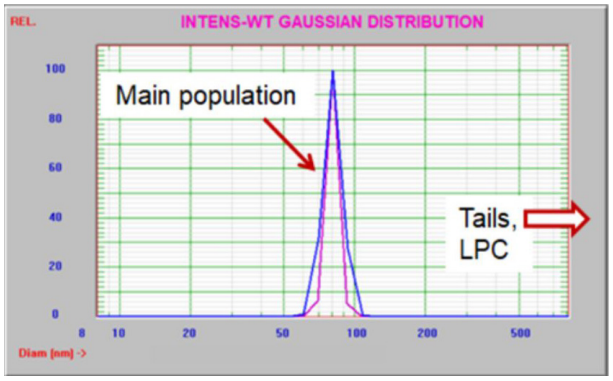

Figure 1. Main population vs. tails (LPC).

CMP SLURRY FILTER TESTING

Chemical mechanical planarization (CMP) slurries need to be controlled to reduce micro scratches on the wafer surface that reduce yield values. The goal is to reduce the large particle count (LPC) while leaving the main population unchanged. This can be accomplished through a combination of filtration and monitoring. Entegris now offers both the filters and particle size analyzers required to properly control CMP slurry particle size distribution to maximize yields.

INTRODUCTION

Maximizing yields requires tight control of the particle size distribution (PSD) of CMP slurries. This can be achieved through a combination of filtration and monitoring of the PSD. Removing the LPC component without altering the main population is achieved by proper filter selection and placement. Assuring that high LPC slurry never touches the wafer surface can be achieved through a combination of lab and/or in-process monitoring with a particle size counter/analyzer. In this study, the Entegris Planargard® NMB CMP slurry filters were challenged with two kinds of slurries. The particle size analyses were made using the AccuSizer® liquid particle counter system. Physical properties of the slurries are shown in Figure 2.

MATERIALS

Slurries

While many filtration studies use polystyrene latex (PSL) standard particles, this study used actual CMP slurries to better model actual fab conditions. This study used silica (mean 30 – 80 nm) and ceria (mean 50 – 100 nm) CMP slurries. Figure 1 shows the main distribution of a silica CMP slurry as measured on the Nicomp® dynamic light scattering (DLS) system. The AccuSizer is then used to measure the tails, or LPC concentration.

	COLLOIDAL SILICA (CS)	CERIA (CE)
Conc. %	20	30
Shape	Spherical	Irregular
pH	7.3	6.6
pH @1%	6.8	6.4

Figure 2. Slurry physical properties.

Filters

Filters used in this study were Entegris Planargard slurry filters NMB01 (0.1 µm pore size) and NMB03 (0.3 µm pore size). These filters are constructed of a new polypropylene membrane technology that produces nanofibers and multilayer continuous melt blown (CMB) media for improved flow path with high particle retention. Planargard filters (Figure 3) remove only those particles that are disruptive to the CMP process, not all the particles. The particle size distribution of the desired slurry particles does not change after filtration.

Figure 3. Entegris Planargard® NMB filter.

Instruments

The AccuSizer (Figure 4) uses the single particle optical sizing (SPOS) technique to count and size particles in liquid suspensions. Depending on the system configuration the AccuSizer can measure from 0.15 – 400 µm. The system used for this study was the AccuSizer FX Nano, but only utilizing the LE400 sensor range of 0.5 – 400 µm. This sensor was chosen because the focus of this study was large particle count (LPC), typically defined as particle concentration/mL >around 1 micron.

Note: The AccuSizer can also be placed in-process using the AccuSizer Mini also shown in Figure 4.

Figure 4. AccuSizer FX Nano (left) and AccuSizer Mini (right).

Filter Test Stand

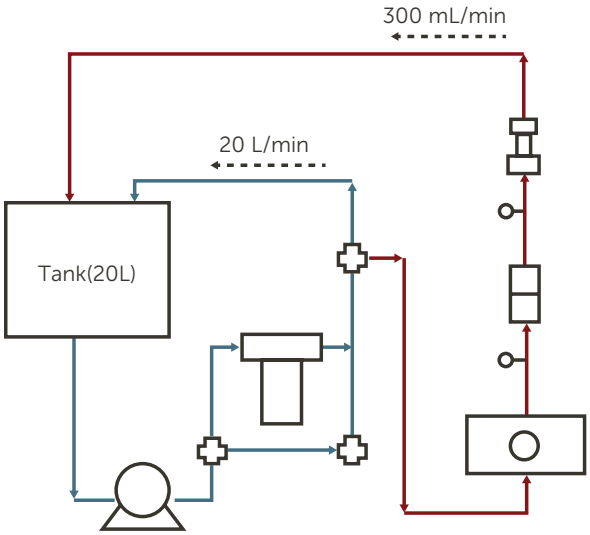

Figure 5. Filter test stand.

Experimental procedure:

- Dilute slurry to 1% concentration
- Install filter into CMP test stand
- Perform pressure activation operation*
- Flush filter and all system 5 min
- Collect downstream sample for LPC measurement
- Collect upstream sample for LPC measurement
- Continuously record pressure increase with time

**Nanofiber manufacturing processes can trap micro air bubbles in tiny spaces in the melt blown media structure. Sometimes the initial pressure will be higher than normal levels. To address this before usage, a liquid flush process will drive air away from media and activation filter to allow the filter to show better performance. Pressure activation uses DI water to flow through the filter at 20 psi and pulse several times. As a result, the initial pressure decreases significantly.*

LPC RESULTS

The two slurries show a different LPC curve model. As seen in Figure 6, the colloidal silica particles show a significant size decrease after filtration across the detected size distribution. This indicates capture of all large particle sizes.

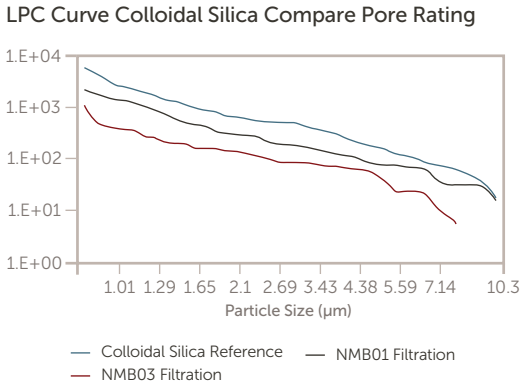

Figure 6. Silica LPC results, NMB01 vs. NMB03.

The ceria results shown in Figure 7 indicate a smaller, but discernable LPC difference up to 2 µm. After 2 µm essentially all the LPC counts have been removed.

LPC Curve Ceria Compare Pore Rating

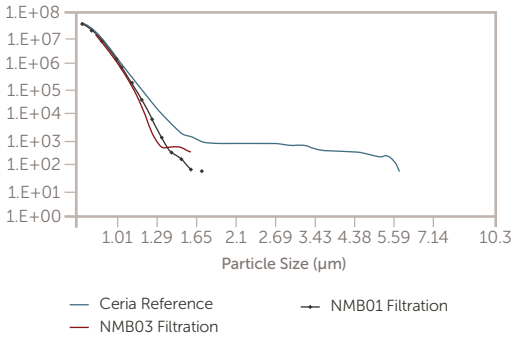

Figure 7. Ceria LPC results, NMB01 vs. NMB03.

SHEAR STRESS EFFECT

Slurry LPC counts typically increase as the sample is exposed to shear forces from pumping. In the next experiment silica and ceria slurries were pumped through the filter test stand without any filter in place. Shear was induced and tracked by changing the pump speed (rpm) and by the number of turnover the slurry was pumped through the test loop. T0 indicates the start of the test, T300 = 300 times through the loop, etc.

The low shear results were recirculated at pump rate 2500 rpm and the high shear results were collected at 5500 rpm. The results are shown in Figures 8 – 11.

Ceria Slurry - Low Shear Stress

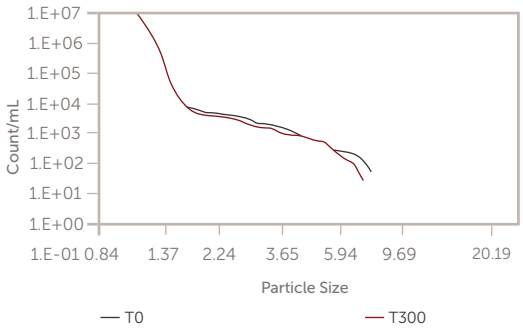

Figure 8. Ceria low shear stress.

Ceria Slurry - High Shear Stress

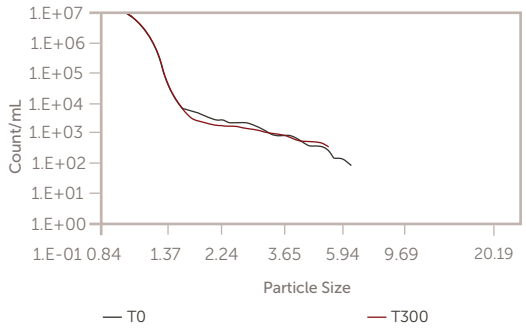

Figure 9. Ceria high shear stress.

These results indicate that a change in rpm and number of circulations through the test loop did not cause significant change in agglomeration for the ceria slurry.

Silica Slurry - Low Shear Stress

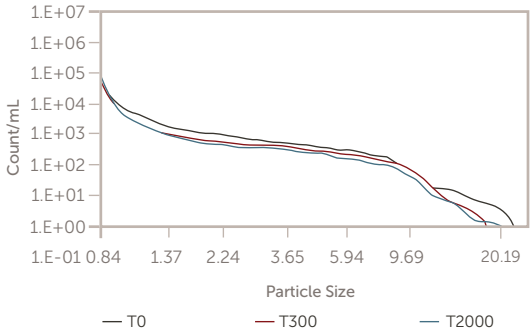

Figure 10. Silica low shear stress.

Silica Slurry - High Shear Stress

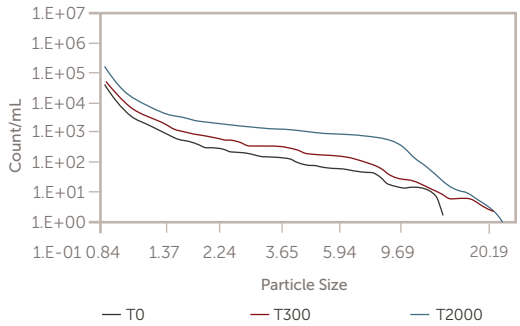

Figure 11. Silica high shear stress.

This data indicates that the silica slurry did exhibit agglomeration behavior at high shear rates.

ONLINE FILTER MONITORING

The AccuSizer Mini is used to continuously track LPC data downstream of filters in CMP delivery systems in many fabs. Results shared by a customer are shown in Figure 12.

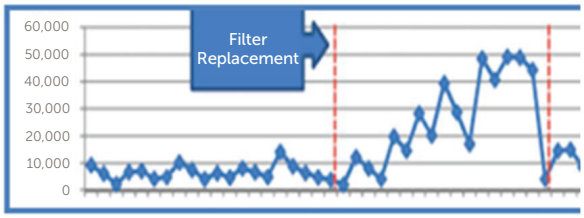

Figure 12. Online LPC downstream of filtration step.

CONCLUSIONS

The CMP slurries proved appropriate for evaluating filter performance using materials used in process. The AccuSizer can accurately count and size the LPC using the laboratory instrument. The AccuSizer Mini is used in fabs around the world for in-process LPC monitoring. Entegris now possesses both the filtration and particle size analyzer technologies, along with expertise to provide the most complete solutions for CMP slurry filtering and monitoring.

Acknowledgements: *Thanks to Yi-Wei Lu, Bob Hshie, Steven Hsiao, HJ Yang, Sherly Lee, and Dean Tsou for collecting most of the data shown in this application note.*

FOR MORE INFORMATION

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Entegris

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

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