Laboratory AccuSizer® Systems for CMP Slurries

AccuSizer® SPOS System

Chemical mechanical polishing/planarization (CMP) is a process widely used in the microelectronic industries to smooth surfaces with the combination of chemical and mechanical forces. This process uses an abrasive and corrosive slurry to help planarize the surface of a wafer. The CMP slurry is a complex mix of nanosized abrasive particles and other chemicals including surfactants, pH adjusters, oxidizers, organic acids, and complexing agents. The particle size distribution of the abrasive is a critical parameter affecting the overall process in several ways. The abrasive mean size and distribution width impacts the material removal rate (MRR). The presence of large particle counts (LPCs) can have a deleterious effect on yield by causing scratches and defects[1,2]. LPCs are essentially the right side (larger) tail of the distribution and are often described as the particle concentration in counts/mL > 1 µm, although the exact size range varies by application. Different analytical techniques are used for measuring mean size and tails. No single instrument can provide all desired data across an entire dynamic range. Dynamic light scattering (DLS) is often employed to measure the mean size and single particle optical sizing (SPOS) is used to measure the LPC tails.

Mean Size vs. Tails

Figure 1 shows the mean size as measured by dynamic light scattering of four abrasives used in CMP slurries: silica, ceria, alumina, and colloidal silica. Notice the variation in both mean size and distribution width.

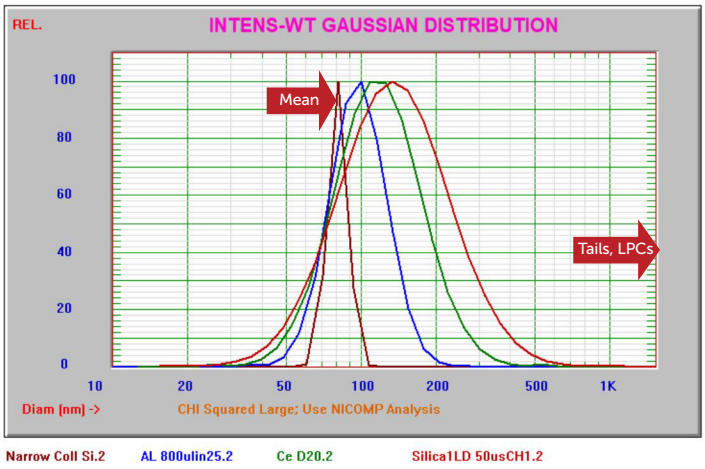

Figure 1. Mean size vs. tails of distribution

The mean size data shown was collected using the Nicomp® DLS system. A separate Entegris application note focuses on measuring mean size and zeta potential of CMP slurries.[3]

The tails[4] are typically measured using the technique of single particle optical sizing. The AccuSizer® lab and online systems are used by slurry manufacturers, researchers, and in fabs around the world. The focus of this application note is laboratory measurements of CMP slurries using the range of AccuSizer instruments. Models used in the laboratory to measure CMP slurry LPCs include the AccuSizer AD, AccuSizer APS, and AccuSizer FX Nano systems.

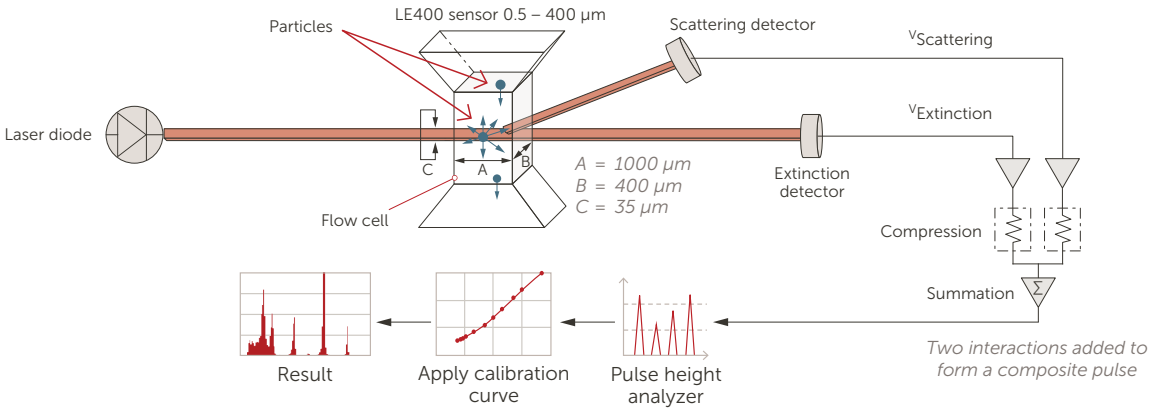

Figure 2. LE400 sensor and operation

AccuSizer System Components

All AccuSizer systems include a sensor, pulse height analyzer (counter), and fluidics for diluting and transporting the sample. The same 1024 channel counter is used for all systems and performs the task of converting pulses from the sensor into particle size and count using a calibration curve. Two sensor models, the LE400 and the FX Nano, are chosen depending on the sample requirements.

The LE400 sensor, Figure 2, measures from 0.5 – 400 µm and counts every particle passing through the system, providing a 100% counting efficiency. This sensor uses a collimated laser beam and both light obscuration and light scattering detectors to provide an extremely wide dynamic range. The concentration limit is approximately 10,000 particles/mL.

The FX Nano sensor, Figure 3, uses a focused laser beam to measure only at the very center of the flow cell. This sensor requires a proprietary algorithm to convert particle pulses into sizes because of the focused beam and non-unique pulse-to-particle size relationship. The FX Nano sensor measures down to 0.15 µm and can measure at much higher concentrations (~10^6 particles/mL). A combination of FX Nano and LE400 sensors delivers a dynamic range of 0.15 – 400 µm.

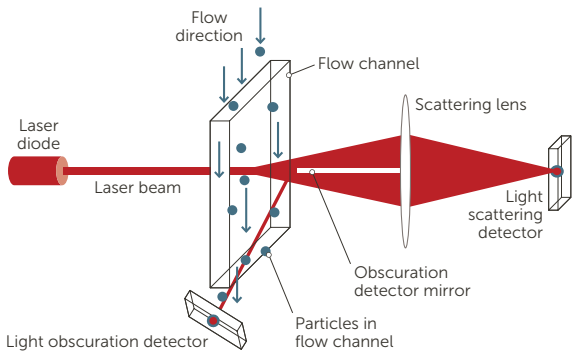

Figure 3. FX Nano sensor

Two dilution fluidics options are available to dilute the sample, control flow rate through the sensor, and flush to clean the system after every measurement. The software then calculates the actual concentration in the original non-diluted sample across as many size channels the user selects.

AccuSizer AD System

The AccuSizer AD system is a single-stage exponential dilution system that incorporates the LE 400 sensor. The AccuSizer AD dilution fluidics diagram (not to scale) is shown in Figure 4.

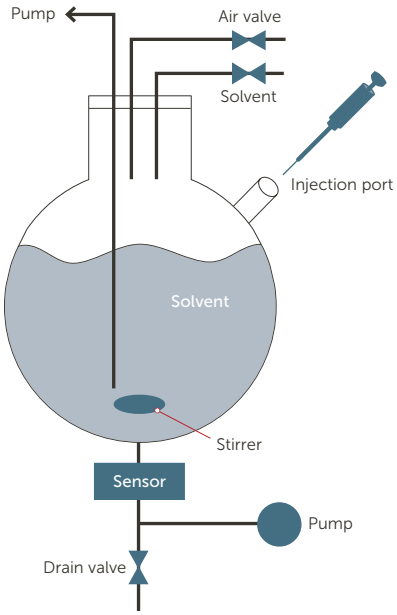

Figure 4. AccuSizer AD system and dilution fluidics

A sample is injected into the dilution chamber and dilution begins immediately. Solvent (DI water) is added to the chamber at the same flow rate sample is drawn through the sensor. The concentration is monitored, and the measurement begins when the concentration is below the coincidence limit of the sensor. The system is automatically flushed to an acceptable background level after the measurement is completed.

AccuSizer APS System

The AccuSizer APS system incorporates both single and two-stage dilution fluidics in a single system. The AccuSizer APS two-stage dilution fluidics diagram is shown in Figure 5.

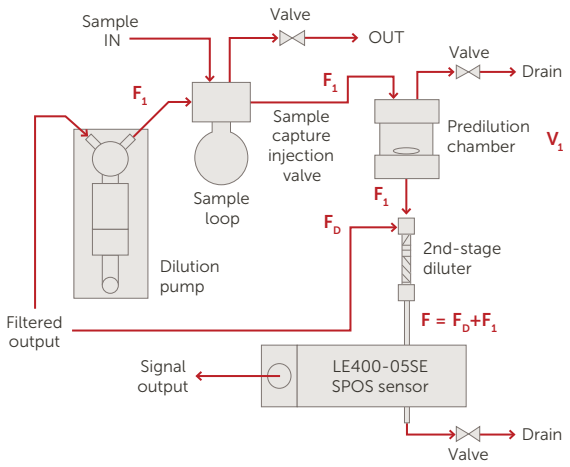

Figure 5. AccuSizer APS two-stage dilution fluidics

The AccuSizer APS system can operate in several modes. When operating in two-stage dilution sample is drawn into sample loop with volume V_L. The first stage of dilution occurs when the sample loop is injected into the dilution chamber with volume V_1. The first stage of dilution is V_1/V_L. The second stage of dilution occurs when the diluted sample flow F_1 mixes with filtered water F_D. The total flow F flows through sensor for analysis. This approach allows for a stable concentration over the course of the measurement.

To better visualize the difference between these two approaches to autodilution, AccuSizer AD vs. APS systems, a comparison of counts vs. time during data collection is shown in Figures 6a and 6b.

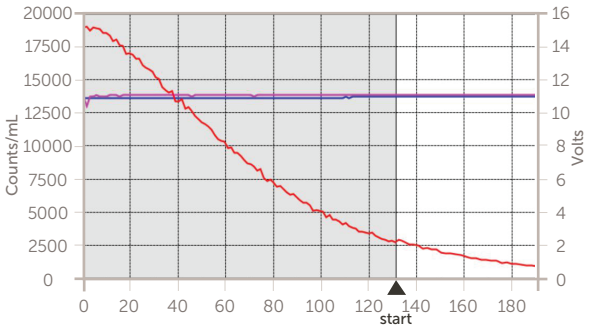

Figure 6a. AD system dilution

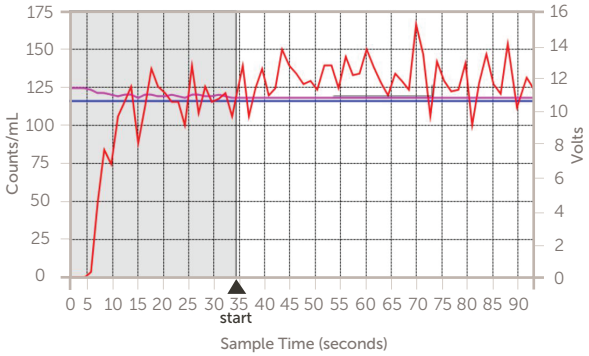

Figure 6b. APS system dilution

Plot interpretation:

X axis	*Sample Time in seconds*
Left Y axis	*Particle Counts/mL*
Red line	*Counts/mL vs. time*
Right Y axis	*Sensor voltages*
Pink line	*Scattering voltage*
Blue line	*Extinction voltage*
Grey area	*Dilution time*
White area	*Measurement time*

The time history graph in Figure 6a, is from a concentrated ceria slurry measured on the AccuSizer AD system. The concentration (counts/mL) starts extremely high and it takes over 120 seconds of dilution time before the measurement begins. The measurement then occurs while the sample is still be diluted. The time history graph in Figure 6b, is from a silica CMP slurry where the concentration is acceptable at the beginning of the sequence, but the measurement does not begin until after a 30 second equilibration time. The measurement then proceeds at a stable rate of counts/mL. This approach is preferred for samples with lower count rates in the LPC range so that the total number of counts collected is statistically valid for accurate, repeatable results.

AccuSizer FX Nano System

The AccuSizer FX Nano system is a single-stage exponential dilution system that incorporates both the LE 400 and FX Nano sensors. The dilution fluidics are similar to what is shown in Figure 4 and dilution occurs similar to the AccuSizer AD system. The sample measurement is made in three ranges. The system first collects data with the FX Nano sensor in low gain (.fyl) plus the LE400 range (.led). Then the system switches and measures the FX Nano sensor in high gain (.fyh). After all three ranges are collected all three result files are combined into a single file (.fyc) covering the entire dynamic range.

The AccuSizer FX Nano system is ideal for providing sensitivity down to 0.15 µm across a broad dynamic range. Some very clean colloidal silica slurries are best analyzed using the FX Nano system where the sensitivity to 0.15 µm is required to collect enough counts to be statistically valid. This system is also used internally within Entegris for testing filter retention of CMP slurries.[5]

Example Results

KLEBOSOL™ 1501 colloidal silica CMP slurry was measured using the AccuSizer AD system before and after filtration through an Entegris Planarcap® NMB point-of-dispense CMP slurry filter. The upstream vs. downstream comparison results are shown in Figure 7.

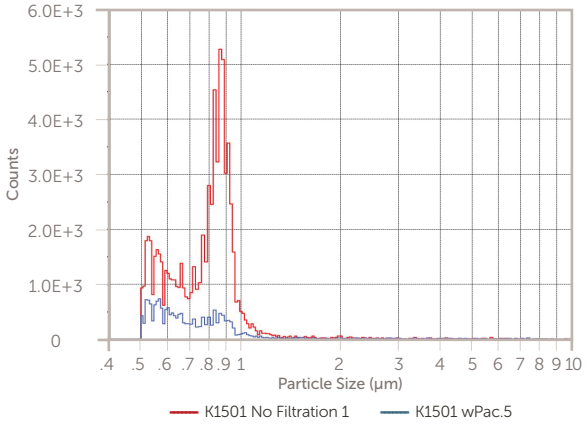

Figure 7. AccuSizer AD system results before (red) and after (blue) filtration

This graph clearly shows a spike of LPCs centered near 0.85 µm being removed by the filtration process. The AccuSizer AD lab system can be used to perform a QC check of incoming CMP slurry and as a tool for filtration studies to determine the optimum filter for a given slurry and process requirements.

A silica CMP slurry was analyzed using the AccuSizer APS system operating in two-stage dilution mode. A typical result is shown in Figure 8, which is the result from the run time data shown in Figure 6b.

The table in Figure 8 shows particle concentration at five user selected size ranges. This report can be customized to display and focus results at ranges considered important for tracking LPCs. The concentration shown is the actual concentration in the original sample prior to dilution. A complete table of up to 1024 size channels is available, as well as many report formats.

Caption: 2 stage
Sensor model: LE400
Calibration file: 1806927_S070618.SNS
Volume Sampled: 1.875 mL
Flow rate: 61.9 mL/min
Number sized(≥ 0.5 µm): 253330
Report Date/Time: 16:57 04/01/2020

Collection time: 16:12 10/18/2018
Sensor S/N: 1806927
Calibration date: 07-06-18
Sensor mode: Summation
Measurement time: 60
DF: 18.324
Pre DF: 1.00
Number of channels: 256

	Diameter µm	Region µm	Concentration #/mL	Mean Diameter µm
1	0.600	0.6-500	197794	1.126
2	0.700	0.7-500	113609	1.485
3	0.900	0.9-500	61949	2.053
4	1.000	1-500	53844	2.215
5	2.000	2-500	15963	4.301

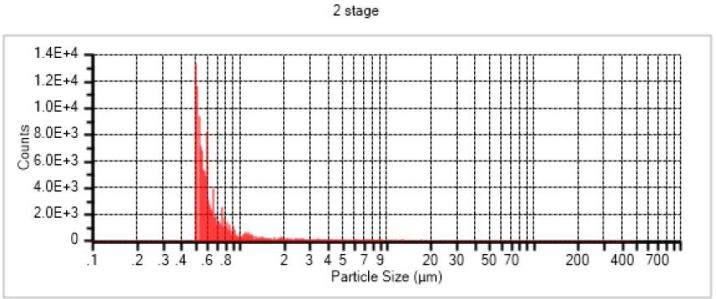

Figure 8. CMP AccuSizer system APS results

An alumina CMP slurry was analyzed on a dual sensor AccuSizer FX Nano system during an internal Entegris training class. This sample was pre-diluted 1000:1, then analyzed using a 0.5 mL sample loop dispersed into an 11.8 mL vessel volume. A 60 second sample time for each measurement range generated an average of approximately 200,000 total particles counted for each analysis. The sample was analyzed by four different Entegris field application engineers and the results from four independent measurements are shown in Figure 9 with the Y axis (concentration) shown on a logarithmic scale.

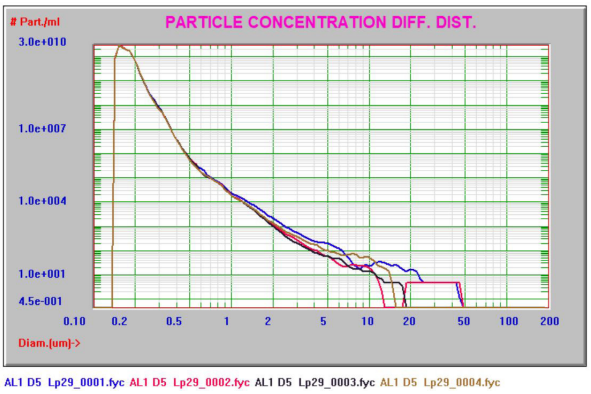

Figure 9. Alumina CMP slurry AccuSizer FX Nano system results

These independent results display excellent reproducibility. The four overlays are essentially indistinguishable below 1 µm. The variation seen above 10 µm is due to the poorer statistics at larger sizes. Since these are actual sample concentration values calculated after a 1000:1 pre-dilution and exponential dilution by the fluidics, the variations above 10 µm could be from single particles.

CONCLUSIONS

The range of AccuSizer laboratory particle size and count analyzers are ideally suited for testing for LPCs in CMP slurries. The advantages of the AccuSizer system include the widest dynamic range (0.15 – 400 µm), sophisticated autodilution fluidics, advanced reporting options, and broad acceptance by both CMP slurry manufacturers and end users in the fabs.

Entegris recommends sending slurry samples to our application labs for analysis and review so we can recommend the optimum system configuration for specific slurries and customer requirements.

References

[1] Remsen, E. et al., *Analysis of Large Particle Count in Fumed Silica Slurries and Its Correlation with Scratch Defects Generated by CMP,* Journal of The Electrochemical Society, 153 (5) G453-G461 (2006)

[2] Kim S.-K. et al, *Effect of calcination time on the physical properties of synthesized ceria particles for the shallow trench isolation chemical mechanical planarization process,* Journal of Ceramic Processing Research, Vol. 7, No. 1, pp. 53-57 (2006)

[3] Entegris Application Note – *Mean Size and Zeta Potential of CMP Slurries*

[4] Entegris Application Note – *Detecting Tails in CMP Slurries*

[5] Entegris Application Note – *CMP Slurry Filter Testing*

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

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