Detecting Tails in CMP Slurries

AccuSizer® FX

OVERVIEW

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. The particle size distribution of the slurry is a critical parameter controlling the success of the planarization process. A few large particles can scratch the surface of wafers, or disc drives, reducing yields and profits. The AccuSizer® particle size and concentration analyzer is uniquely capable of detecting the few large particles at the tail of the distribution that can be harmful during the CMP process.

INTRODUCTION

The CMP process and CMP slurries are widely used for polishing during microcircuit manufacturing. The health of the CMP slurry is crucial to maximize device yields, requiring regular measurement of the particle size distribution (PSD) of the slurries. In addition to the mean size of the PSD, the monitoring technique should ideally be sensitive to the presence of tails (i.e.; the small concentration of larger particles) away from the main peak of the distribution. These tails can come from contamination, aggregation due to changes in chemistry, CMP delivery systems, or from applied shear forces. The relationship between the large particle count (LPC) >1 µm and defect, or scratch count, has been well established and the ideal characterization system should provide an accurate LPC value.

PARTICLE SIZE/COUNTING TECHNIQUES

There are many particle characterization techniques being used to measure the size and concentration of particles in CMP slurries. Light scattering techniques, including dynamic light scattering (DLS) and laser diffraction, can measure the size and breadth of the distribution but do not provide any useful concentration information. Single particle optical sizing (SPOS) measures the particles one at a time as they pass through a narrow measurement chamber, providing both an accurate size and concentration (particles/mL) result. Since the particles are measured individually, this technique is inherently very high resolution and can detect even very few individual particles removed from the main distribution. It is therefore the ideal technique for detecting the LPCs that causes the most trouble with CMP slurries.

Some instruments are best used in the laboratory, while others, such as SPOS, can be used both in the lab and at point-of-use (POU).

THE ACCUSIZER RANGE OF SYSTEMS

AccuSizer's particle sizing systems has been used for years by CMP slurry manufacturers and end users to detect the presence of large particles in tails. Depending on the slurry, the measurement can be made at full concentration or with automatic dilution to optimize conditions for the analysis.

The Entegris AccuSizer Mini FX system (pictured above) is designed to work with smaller particle sizes and higher concentrations. The AccuSizer FX sensor uses a focused beam to reduce the total volume inspected, thus increasing the concentration limit of the sensor and often allowing for measurement without dilution. The FX sensor measures particles from 0.65 – 20 µm at concentrations 200 times greater than standard light extinction or scattering sensors. The results can be displayed in up to 512 size channels.

The system can be configured for standard laboratory analysis including automatic dilution, for point-of-use (pictured above). Both of these configurations provide the sensitive and accurate LPC data critical for CMP slurries.

LPC DETECTION

The AccuSizer has proven in the past to be ideal for the detection of LPC >1 µm in CMP slurry. Figure 1 shows a comparison of techniques used to detect a known concentration of 1 µm SiO_2 particles added to a base of silica oxide CMP slurry. The concentration of the "spike" particles ranged from 0.175 – 17,500 mg/L.

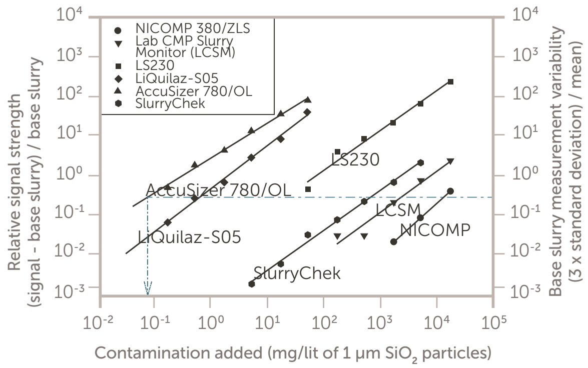

Figure 1. Perturbation detection analysis.
From Nichols, K., et. al., Perturbation Detection Analysis: A method for comparing instruments that can measure the presence of large particles in CMP Slurry, report published by BOC Edwards, Chaska, MN.

In this study the particle concentration for the >0.56 µm size channel was used as the sensor "signal." For the AccuSizer (older model 780) the perturbation detection limit was reported to be at 0.07 mg/L.

EXPERIMENTAL

A new experiment was designed to confirm the ability to detect 1 µm particles in the presence of a silica CMP slurry. Several common silica CMP slurries were spiked with 1 µm polystyrene latex (PSL) spheres. The spike particles were confirmed for both size and concentration and then diluted for the various studies. All samples were analyzed on the AccuSizer Mini FX system equipped with an FX sensor, range 0.65 – 20 µm.

RESULT 1, SLURRY A

Slurry A was first diluted 250:1 to assure measurements were outside of the coincidence region. 1.44 mL of 1000 × diluted PSL stock (1.74x107/mL) was added to 250 mL of sample suspension (containing 1 mL of original slurry). The intended spike concentration was 100,000/mL of 250:1 diluted sample suspension. The results are shown in Figure 2 and Table 1 and Table 2.

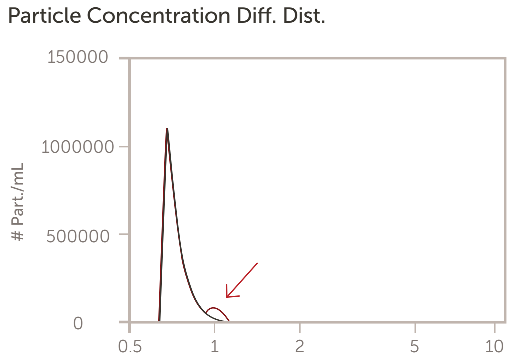

Figure 2. Slurry A before (blue) and after 1 µm spike (red)

Diameter range	#Particles sized	Cumulative number ≥ diameter
0.70 – 0.72 µm	1107410 mL	2637543 mL
0.72 – 0.77 µm	738493 mL	1530133 mL
0.77 – 0.82 µm	372885 mL	791640 mL
0.82 – 0.87 µm	203005 mL	418755 mL
0.87 – 0.92 µm	100429 mL	**215749 mL**
0.92 – 0.98 µm	54625 mL	115320 mL
0.98 – 1.04 µm	32665 mL	60695 mL
1.04 – 1.11 µm	13392 mL	28030 mL
1.11 – 1.18 µm	7375 mL	14639 mL
1.18 – 1.25 µm	4147 mL	7264 mL
1.25 – 1.33 µm	1762 mL	3117 mL
1.33 – 1.41 µm	709 mL	1355 mL
1.41 – 1.50 µm	213 mL	645 mL
1.50 – 1.60 µm	60 mL	432 mL
1.60 – 1.70 µm	88 mL	372 mL

Table 1. Slurry A result before spike

Diameter range	#Particles sized	Cumulative number ≥ diameter
0.70 – 0.72 µm	1005006 mL	2484223 mL
0.72 – 0.77 µm	633343 mL	1479216 mL
0.77 – 0.82 µm	343434 mL	845874 mL
0.82 – 0.87 µm	183631 mL	502439 mL
0.87 – 0.92 µm	96589 mL	**318808 mL**
0.92 – 0.98 µm	53409 mL	222219 mL
0.98 – 1.04 µm	82906 mL	168810 mL
1.04 – 1.11 µm	64733 mL	85904 mL
1.11 – 1.18 µm	11916 mL	21172 mL
1.18 – 1.25 µm	4192 mL	9256 mL
1.25 – 1.33 µm	2477 mL	5064 mL
1.33 – 1.41 µm	939 mL	2587 mL
1.41 – 1.50 µm	371 mL	1647 mL
1.50 – 1.60 µm	171 mL	1276 mL
1.60 – 1.70 µm	0 mL	1105 mL

Table 2: Slurry A result after spike.

Note the increase of ~100,000 particles/mL in the expected channel.

RESULT 2, SLURRY B

Silica Slurry B was measured without dilution on the FX POU system. Figure 3 shows the results of the undiluted results before the spike was added. The reported concentration is ~20,000 particles/mL.

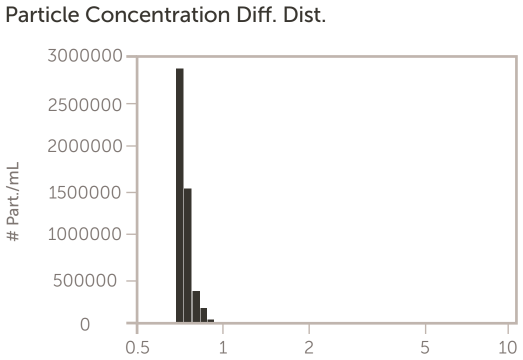

Figure 2. Slurry B, no dilution, no spike

This sample was then spiked by adding 57 µL of the 1000 × diluted 1 µm PSL standard (concentration = 1.74×10^{7} particles/mL) to 200 mL of original slurry. The estimated spike concentration = 5,000 particles/mL. The results before and after the spike are shown in Figure 3.

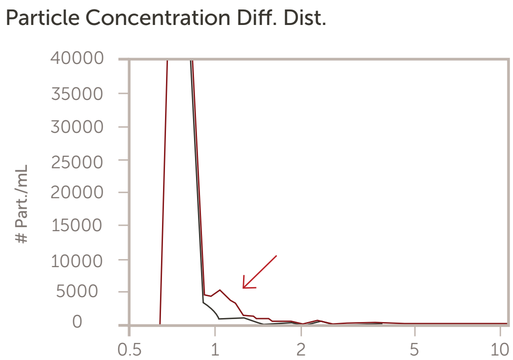

Figure 3. Slurry B before (blue) and after spike (red)

RESULT 3, SLURRY C

Slurry C is typically diluted one part slurry to two parts DI water when used in the fab, so this was used as the base sample. The base slurry was first measured without dilution using the FX POU system. The result before spiking is shown in Figure 4. Note that the LPC tail does not drop as sharply as seen in Slurry A or B. The concentration is ~30,000 particles/mL >0.7 µm, with only ~5,500 particles counted in 30 mL of slurry, a two minute analysis time.

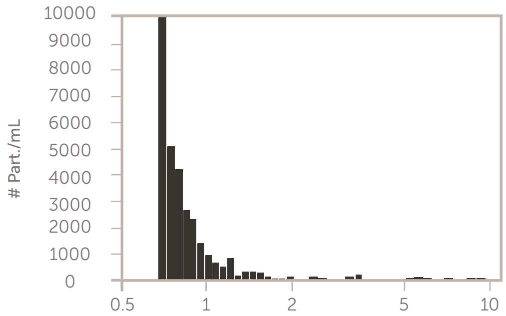

Figure 4. Slurry C, no dilution, no spike

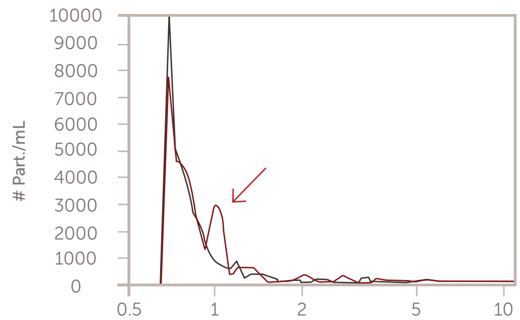

Figure 5. Slurry C before (blue) and after (red) spike

Slurry C was then spiked by adding 35 µL of the 1000 x diluted 1 µm PSL standard (concentration = 1.74×10^7 particles/mL) to 250 mL of base slurry. The estimated spike concentration = 2,500 particles/mL. Figure 5 shows the results for Slurry C before and after the spike.

CONCLUSIONS

These results confirm that the AccuSizer Mini FX is an accurate, easy-to-use analytical tool to detect the presence of low concentration LPC particles in CMP slurries. This system can be used in the lab or in the fab.

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