Measuring Particles in CMP Slurries

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At a Glance

Slurry is a primary factor in the etch rate and quality of CMP processing. Although a slurry is chosen during process development, in the production environment its characteristics, or "health," can change during shipping, handling and distribution. Reliable CMP slurry analysis can be key to consistent, quality CMP processing.

With CMP becoming increasingly important in IC manufacturing, slurry characteristics play a major role in determining process conditions. Slurries are complex suspensions that can be comprised of shear sensitive solids and reactive chemicals. Slurry particles are optimized to enhance process performance, but handling can cause shifts in size distribution or create agglomerations that form larger particles that can cause scratching on the wafer surface. By monitoring slurry "health," engineers can proactively evaluate slurry quality to ensure the most reliable CMP processing.

Control of CMP processes has been hindered by the inability to detect slurry damage before it adversely affects product yield. For years, slurry pH, conductivity and total solids have been monitored during CMP processes.[1] More recently, CMP slurry analyzers have been used to characterize slurry particles in terms of concentration and size distribution.[2] This study was undertaken to determine which slurry characteristics change when slurry is circulated and which instruments can most sensitively detect the onset of change. Effective slurry monitoring depends greatly on the type of analytical instrument, its sensitivity, repeatability and resolution.

Materials and methods

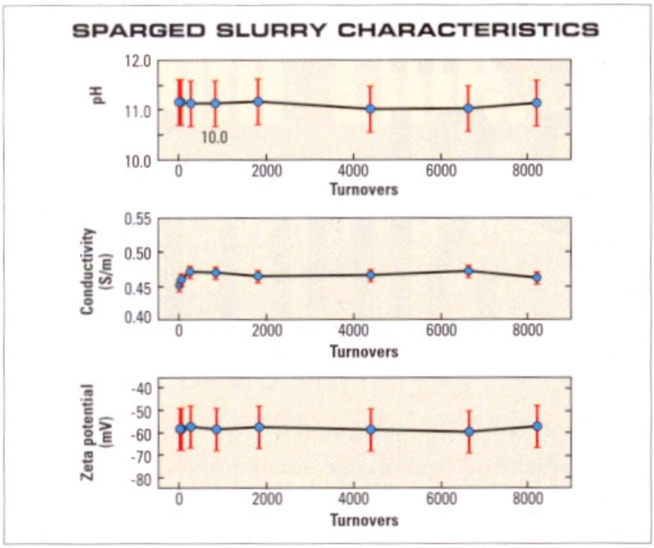
Six slurry analyzers were evaluated (Table 1), along with a description of the slurry characteristics they report and the principles used to measure them. All were tested off-line, although all (except the LS230 and NICOMP 380/ZLS) are designed for on-line use. The slurry used was Cabot Semi-Sperse 12, an oxide CMP slurry containing silica particles.

The first step was to determine situations that lead to changes in slurry characteristics or negatively affect slurry "health." To assess the damage caused by circulation and drying, parallel tests were conducted in three systems. In two parallel systems, a centrifugal and a diaphragm pump were used to circulate slurry from a humidified tank through a distribution loop. In the third system, circulated with a diaphragm pump, compressed dry air (CDA) was passed through the slurry in the tank using a sparger to accelerate change caused by drying.

The slurry was circulated for seven to 10 days to force measurable changes in characteristics. Typically, eight samples

Table 1. Slurry Health Analyzers Evaluated

Analyzer (manufacturer)	Parameters analyzed	Particle measuring principle
LiQuilaz-S05 (Particle Measuring Systems)	Cumulative particle number; particle concentration at 15 different sizes of ≥0.55-10 µm	Single-particle light scattering
AccuSizer 780/OL (Particle Sizing Systems)	Cumulative particle number; particle concentration at 15 different sizes of ≥0.56-10 µm	Single-particle light scattering and light extinction
LS230 (Beckman Coulter)	Cumulative particle volume fraction at sizes of 0.04-5 µm	Laser diffraction and light scattering
NICOMP 380/ZLS (Particle Sizing Systems)	Mean particle size; standard deviation of PSD; zeta potential	Dynamic light scattering
SlurryChek (Particle Measuring Systems)	Extinction coefficients at 17 different wavelengths of 200-1000 nm (wide cell only)	Multiwavelength light extinction
Lab CMP slurry monitor (Colloidal Dynamics)	Attenuation and dynamic mobility spectra at frequencies of 1-18 MGz, pH, conductivity, temperature, zeta potential, median diameter, and geometric standard deviation	Electroacoustics



1. Results obtained with the Lab CMP slurry monitor analyzer during measurement of the sparged slurry in the diaphragm pump system show statistically insignificant changes in pH, conductivity and zeta potential.

were withdrawn over the test period and analyzed within one hour. The measurements were repeated three to five times for each sample to determine sample variability.

Data analysis

Slurry analyzers use a variety of detection principles, so their outputs cannot be compared directly. Hence, all output data were converted to a uniform, dimensionless parameter: relative signal strength (RSS), defined as the ratio of the change in instrument response caused by circulation to the variability of the base slurry measurement. The RSS is calculated as:

$$RSS = \frac{\textit{Measurement after circulation} - \textit{base slurry measurement}}{\textit{Variability of the base slurry measurement}}$$

The variability of the base slurry measurement was defined as 3× the measurement standard deviation (3σ). A detailed explanation of RSS can be found elsewhere.[3] RSS analysis was performed for all of the parameters listed in Table 1. Parameters listed as a range were analyzed at multiple values. For example, cumulative particle concentrations were analyzed at 15 different particle sizes for the LiQuilaz-S05 analyzer.

Results

Several parameters indicated no measurable change even though the slurry was cycled for more than 8000 turnovers. Figure 1 presents some results obtained with the Lab CMP slurry monitor analyzer during measurement of the sparged slurry in the diaphragm pump system. The error bars represent ±3 standard deviations. Figure 1 demonstrates that, as a slurry is used, changes in pH, conductivity and zeta potential are statistically insignificant. These three parameters, then, make poor choices as slurry health indicators.

Table 2 presents the parameters for the sparged slurry in the diaphragm pump system that did not change with increasing turnovers of the slurry. The maximum RSS for each parameter is listed. The RSS must be greater than 1 for a change to be detected. An RSS of less than 1 indicates that the change in instrument response was less than the variability of the base slurry measurement. All of the RSSs in Table 2 were less than 1. Similar results were observed for the other distribution systems.

Only parameters related to particle characteristics — primarily particle size distribution — changed when circulating slurry. For each instrument, parameters and operating conditions that gave the overall best RSS in all circulation systems were used in the instrument comparison (Table 3).

RSS analysis allowed comparison of the instruments' abilities to detect change in circulating slurry (Fig. 2). The x-intercept of each linear regression (RSS=1) represents each instrument's detection sensitivity; that is, the fewest number of turnovers at which the instrument could detect change. The detection sensitivities of each instrument in each of the three systems are listed in Table 4.

The sensitivities of the instruments varied by more than four orders of magnitude. The LiQuilaz-S05 and AccuSizer 780/OL were the most sensitive instruments for the diaphragm pump systems, followed by the SlurryChek analyzer. For the centrifugal pump system, the LiQuilaz-S05 analyzer was the most sensitive followed by the SlurryChek and the AccuSizer 780/OL analyzers. However, the slope for the LiQuilaz-S05 data in the centrifugal system was very different from slopes for all other instruments in all systems. If the slope were similar to the others and weighted to the

Table 2. Parameters That Did Not Significantly Change in >8000 Turnovers

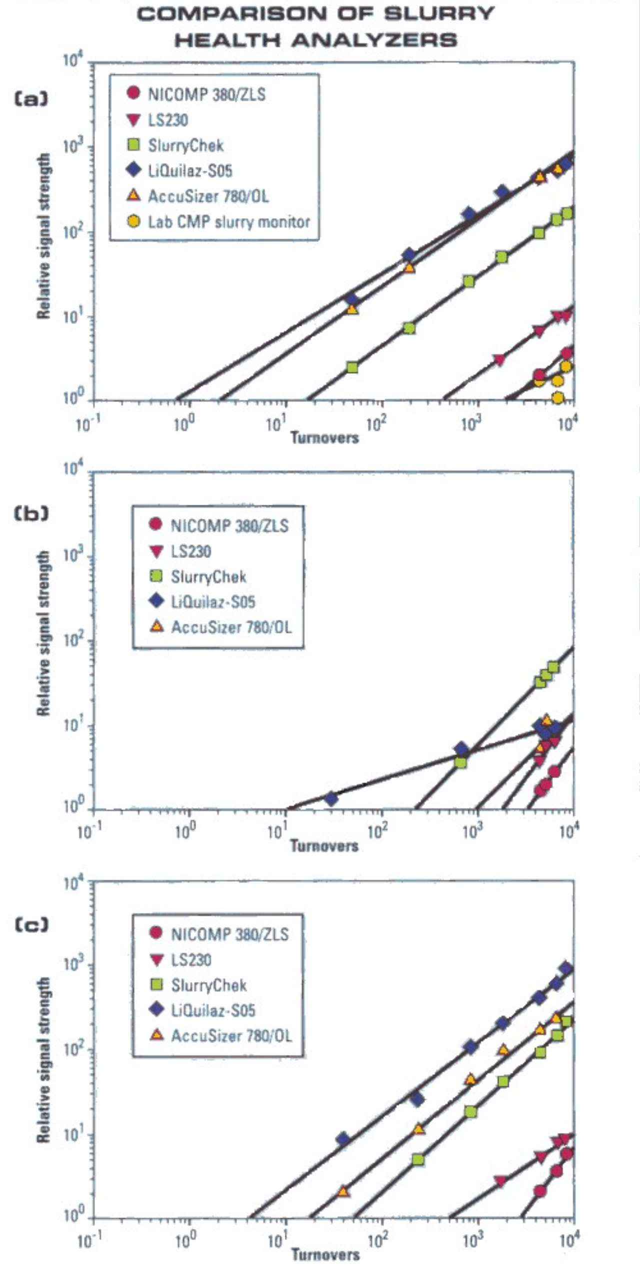
System	Instrument	Signal	Max. RSS
Diaphragm sparged	Lab slurry monitor	Conductivity	0.71
		Zeta potential	0.21
		pH	0.25
		Attenuation coefficient	0.81
	NICOMP 380/ZLS	Zeta potential	0.20

Table 3. Summary of Parameters Used in RSS Analysis

Instrument	Signal
LiQuilaz-S05	Cumulative particle concentration ≥0.77 μm
AccuSizer 780/OL	Cumulative particle concentration ≥0.56 μm
SlurryChek	Wavelength 750 nm
LS230	Cumulative volume fraction at 1 μm
Lab CMP slurry monitor	Frequency 5.4 MHz
NICOMP 380/ZLS	Mean diameter (nm)

RSS values at high turnovers, then the sensitivities of the LiQuilaz-S05, AccuSizer 780/OL, and SlurryChek instruments would all be similar.

In general, the two optical particle counters — the LiQuilaz-S05 and the AccuSizer 780/OL — were the most sensitive, with the LiQuilaz-S05 analyzer being somewhat more sensitive. The difference between the sensitivities of the two instruments is believed to be a result of the dilution systems used to supply sample to the sensors. The LiQuilaz-S05 sensor used a laboratory dilution system while the AccuSizer sensor used a patented autodilution system. The laboratory system achieved lower measurement variability than the autodilution system, as shown in Table 5. The AccuSizer 780/OL analyzer's higher variability means that a larger change is required before a signal can be detected by this analyzer. If the AccuSizer 780/OL sensor were to be used with the laboratory dilution system, similar sensitivities would be expected. In fact, because the AccuSizer 780/OL sensor uses light extinction rather than light scattering to size large particles, it is expected to provide better sensitivity than the LiQuilaz-S05 sensor for particles greater than ~1.5 µm.[4]



2. Using RSS analysis, slurry health analyzers are compared in systems with a diaphragm pump with humidified nitrogen blanket (a), centrifugal pump with humidified nitrogen blanket (b), and diaphragm pump with air-sparged slurry (c).

Table 4. Summary of Detection Sensitivities

Instrument	Detection sensitivities (turnovers to measurable signal)		
	Diaphragm sparged	Diaphragm humidified	Centrifugal humidified
LiQuilaz-S05	4.0	0.77	9.7
AccuSizer 780/OL	17	2.1	1000
SlurryChek	52	18	220
LS230	500	420	1800
NICOMP 380/ZLS	2800	2200	3200
Lab CMP slurry monitor	>8000	3200	>8000

Table 5. Comparison of Optical Particle Counter Measurement Variables

Instrument	Baseline measurement (#/mL)	Measurement variability 3σ (#/mL)	Relative variability (variability/baseline)
LiQuilaz-S05 (≥0.77 µm)	34,479	1310	3.8%
AccuSizer 780/OL (≥0.56 µm)	427,776	52,831	12.4%

Summary

Slurry parameters and analyzers were evaluated for their ability to detect change in CMP slurry circulated through three different pumping systems. Physical characteristics of the slurry — such as pH, conductivity and zeta potential — showed no change in more than 8000 turnovers. Measurements related to particle size characteristics — primarily particle size distribution — were the only indicators of slurry change. These measurements were converted to a relative signal strength to allow direct comparison of the analyzers.

The LiQuilaz-S05 optical particle counter was the most sensitive analyzer tested. The AccuSizer 780/OL was the second most sensitive analyzer in the diaphragm pump systems, and the SlurryChek analyzer had intermediate sensitivity in all three systems. The difference between the sensitivities of the LiQuilaz-S05 and AccuSizer 780/OL analyzers was attributed to the dilution systems used.

CMP particle size analyzers can provide a good basis for monitoring slurry health. With automated and in-line slurry sampling systems, handling variability can be reduced, yielding process-important data. It has been demonstrated in the production environment that changes in slurry particle size monitoring data correspond to changes in the slurry distribution system such as filter swaps. These correlations provide valuable information about the health of the slurry as it reaches the CMP tools for processing. •

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