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Sample Dilution

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Traditional methods of measuring the large particle counts (LPC) in CMP slurry rely on dilution of a small sample to reduce the number of particles passing through an optical sensor at any one moment to a manageable level. To maintain reasonable productivity, a slurry sample < 1 ml is diluted between 10x and 10,000x and a portion of the diluted mixture is measured to represent to bulk slurry particle size distribution (PSD). Dilution with water can change the chemistry and the tendency of particles to agglomerate, possibly contributing to misleading albeit consistent results. In contrast, the SlurryScope measures 15 ml/min of undiluted slurry that can be returned to the supply stream after measurement.

One of the primary purposes of measuring PSD in CMP slurries is to monitor the number of particles > 1µm, which are not supposed to be present in chip production but almost always are. While sub-micron particles that perform the fine polishing number in the millions per milliliter, the larger particles number in the hundreds to tens of thousands per milliliter. This is a good thing for defect-free polishing, but can be a bad thing for reliable detection of these larger particles.

Over the course of a day, a slurry distribution system (SDS) in a chip fab may be tested every ten minutes using 1 ml slurry samples, for a total of 144 ml of slurry tested in 24 hours. If the slurry requires a high dilution factor so that the total volume of diluted slurry cannot be measured in 10 minutes, the total undiluted slurry volume tested over the course of the day is even less. This total sample volume provides an adequate representation of the PSD for small particles (≤ 1µm) where the particle counts are high, but not for the large particles where the particle counts are low. (See Applications Note: Perils of Low Particle Counts.)

In contrast, a SlurryScope operating on a SDS will monitor LPC continuously at a rate of 15 ml/min, (900 ml/hr or 21.6 liters/day). This is 150 times the total volume measured using traditional methods. Since the SlurryScope measures without dilution, the slurry can be returned to the distribution loop with no slurry consumption.

As discussed in the Applications Note: Perils of Low Particle Counts, a total particle count of 1,000 in any one particle size bin is required to provide < 10% deviation from one measurement data set to another. The SlurryScope can accomplish this over the full range of 1-12 µm in a single day. Traditional methods using the sampling parameters described above would require 150 days to achieve the same statistical repeatability for particles > 5 µm. In practice, slurry aging effects would confound any experimental efforts to demonstrate repeatability over this length of time.

The practice of diluting the slurry to the extent that only a single particle passes through the detector at a time contributes another layer of complexity to the data interpretation. If dilution is performed with a solution that preserves the pH and the ionic strength of the chemical environment that each particle sees, then the measured PSD is less subject to conjecture about spontaneous association and dissociation of soft particle agglomerates that may appear to the detector as large particles. More commonly, however, the slurry is diluted with deionized water as a practical convenience. This practice does change both the pH and the total ionic strength of the solution, and these properties have an effect on particle association and dissociation that is not well characterized. The saving grace is that if the dilution procedure is accurate and consistent, the measured results will likely be self-consistent over time. SlurryScope measurements are not subject to these uncertainties, since the slurry is measured without dilution.

