



CMP SLURRY MEASUREMENT USING LASER DIFFRACTION

Chemical mechanical polishing (or planarization) is the most popular technique for removing the surface irregularities of silicon wafers. Typical CMP slurries consist of a nano-sized abrasive dispersed in acidic or basic solution. A chemical reaction softens the material during mechanical abrasion. The abrasive particles have a size distribution which directly affects critical metrics including rate of removal and wafer defects. Particle size analysis is therefore a key indicator of CMP slurry performance. The popularity and utility of several particle sizing techniques will be discussed within.

Introduction

The CMP process is an essential part of modern multiple-layer semiconductor manufacturing. The ability to provide nano-scale smoothness makes the technology indispensable as both die size and lithographic techniques continue to shrink. Given the high costs associated with state of the art semiconductor device manufacture and the crucial role of CMP, there's little surprise that the abrasive slurries used undergo thorough characterization. A sub-standard batch of slurry may consist of particles too fine or too coarse thus affecting rate of removal, or just a few oversize aggregates causing micro scratches to the wafer surface. Naturally, CMP slurry manufacturers and users possess a keen interest in particle size information. This measurement can prevent the loss of millions of dollars worth of wafers simply by identifying good slurry from bad. Good slurry removes

material at a specific rate, evenly across the wafer. Good slurry features predictable performance stemming from a uniform particle size distribution with excellent stability. Low stability slurry, on the other hand, may quickly form large particle aggregates which then cause irreparable damage to the wafer. The importance of particle characterization for CMP slurries is obvious.

A Unique Challenge Narrows Options

Choosing to monitor CMP slurry particle size is easier done than choosing how to measure it because of the technical challenge posed. The typical size range of CMP abrasive particles is 50-250 nanometers and several particle sizing techniques are capable of measuring in this range with varying accuracy and precision. The typical oversize aggregate in CMP slurry is 1-10 microns and appears on a parts per million scale. Again, several techniques can

Technique	Nano-Particle Measurement	Oversize Measurement	Analysis Speed	Dilution Required?
Laser Diffraction	Very good	Excellent	Excellent	Yes
Dynamic Light Scattering	Excellent	Very poor	Very good	Yes
Acoustic Spectroscopy	Excellent	Excellent	Good	No
Light Obscuration	Good	Good	Poor	Yes
Disc Centrifuge	Very good	Excellent	Very poor	Yes
Microscopy (SEM, TEM)	Excellent	Very poor (statistics)	Very poor	No

Table 1: Several mainstream particle size analysis techniques and their suitability for CMP slurry measurement



perform this analysis with degrees of accuracy and precision. The challenge comes from the combination of accurately sizing the nano-scale particles while also identifying a relative few micron-scale aggregates. Only a few techniques are capable of meeting both requirements in addition to usability and reliability criteria. Table 1 lists several CMP sizing techniques and their relative strengths and weaknesses.

CMP Particle Characterization Techniques

Laser diffraction is the most popular technique for sizing CMP slurries owing to its fast, accurate, and precise measurements of both the nano- and micro-scale particles. As laser diffraction tools evolve, smaller and smaller particles can be measured and the detection limit for oversize particles decreases. The excellent measurement performance of the laser diffraction technique is enhanced by unmatched usability and reliability. If CMP test labs can only choose one particle size analysis tool, they pick the current state of the art laser diffraction instrument.

Tools based on dynamic light scattering are only suitable for the nano-scale component – information about oversize particles is unreliable due to the technique's physical limitations. DLS has one advantage over laser diffraction: the ability to measure sub-100 nanometer particles at very low sample loading (particle concentration). After the presence of any destructive large particles has been verified, CMP test labs may add a DLS system to complement existing technologies.

Acoustic spectroscopy has also found popularity by measuring both particle size and zeta potential without dilution. CMP slurries are applied to silicon wafers at a particular concentration and test labs often choose to conduct material characterization while holding this and other variables constant. The idea is that the presence of oversize particles may be exaggerated or hidden when concentration is changed. Perhaps a less well known measurement, zeta potential can predict batch stability. Put simply and roughly, this value is the potential difference between

particle and liquid medium. If the magnitude of the potential difference decreases below a certain threshold, then particle-particle interactions including aggregation become much more likely. In this way zeta potential has joined particle size analysis as a critical quality control test of CMP slurries. Zeta potential measurements using the electroacoustic method show better accuracy for concentrated slurries than other methods.

Particle size instruments built upon light obscuration methods have historically found acceptance for CMP measurement. Light obscuration is capable of single particle resolution which improves the detection of oversize aggregates. The technique is exceedingly slow in comparison to laser diffraction, acoustic spectroscopy, and dynamic light scattering. The lengthy analysis time stems from diluting CMP slurry with a solids content of several percent so that only one particle at a time passes in front of a light source. The diluent must also need to be ultra pure as any water-borne particulates will be measured given the technique's unforgiving resolution. These instruments tend towards difficult maintenance and operation and are being replaced by other techniques for the CMP application.

Spinning disc centrifuge instruments have found a foothold in the CMP industry also because of high resolution. The 'density gradient stabilized' version of this technology allows for improved accuracy of sub-micron particles. Analysis times tend to run very long (15-60 minutes) in comparison to laser diffraction (1-2), dynamic light scattering (1-5), and acoustic spectroscopy (3-5). Disc centrifuges require calibration to achieve sufficient accuracy – a limitation not seen with laser diffraction, DLS, or acoustics as they are first principle measurements.

Ultramicroscopes suffer for the CMP application because of poor statistics, comparatively long sample preparation and measurement time, and very high cost. Microscopy will provide extremely high particle size resolution and accuracy, but remains unpopular because competing techniques offer



acceptable accuracy with none of the same drawbacks.

Testing Laser Diffraction Performance: Experimental

An experiment was designed for the current state of the art in laser diffraction, the [HORIBA LA-950](#), to test that instrument's suitability for both nano-particle and oversize aggregate detection. Two well-characterized silica products of different size were chosen to mimic the CMP application of a nano-scale primary distribution with micro-scale aggregates/impurities. The experiment consisted of absolute accuracy tests for each silica product, the lower limit of detection for oversize particles, and checking the accuracy of each component for the mixed silica result. The first material chosen was LUDOX TM colloidal silica from Grace Davison. The particle size of this silica has been characterized (1) by numerous techniques and reliably shows a D50 (volume median) value between 30-35 nanometers. The second material chosen was Geltech 1.5 powdered silica. The nominal particle size is 1-2 microns making it a suitable choice for testing oversize detection capability.

Experimental setup involved the creation of a 0.1wt% solution of Geltech 1.5 in 0.1M KCl and a 0.5wt% solution of Geltech 1.5 in LUDOX TM-50 (a.k.a. 50wt% colloidal silica). All measurements were conducted with a refractive index value of 1.47 and zero imaginary component; circulation pump activated, and de-ionized water as the dispersant. For individual accuracy tests, aliquots were drawn directly from the LUDOX TM-50 and 0.1wt% Geltech 1.5 and added (independently) to the LA-950. The mixed silica test consisted of adding the 0.5wt% Geltech 1.5 in LUDOX TM solution to the LA-950, acquiring a measurement, evaluating the results for both accuracy and oversize detection, diluting the mixture, and repeating the previous steps. The amount of sample added to the analyzer is inconsequential, only the concentration of oversize particles in the sample informs the performance and suitability of laser diffraction.

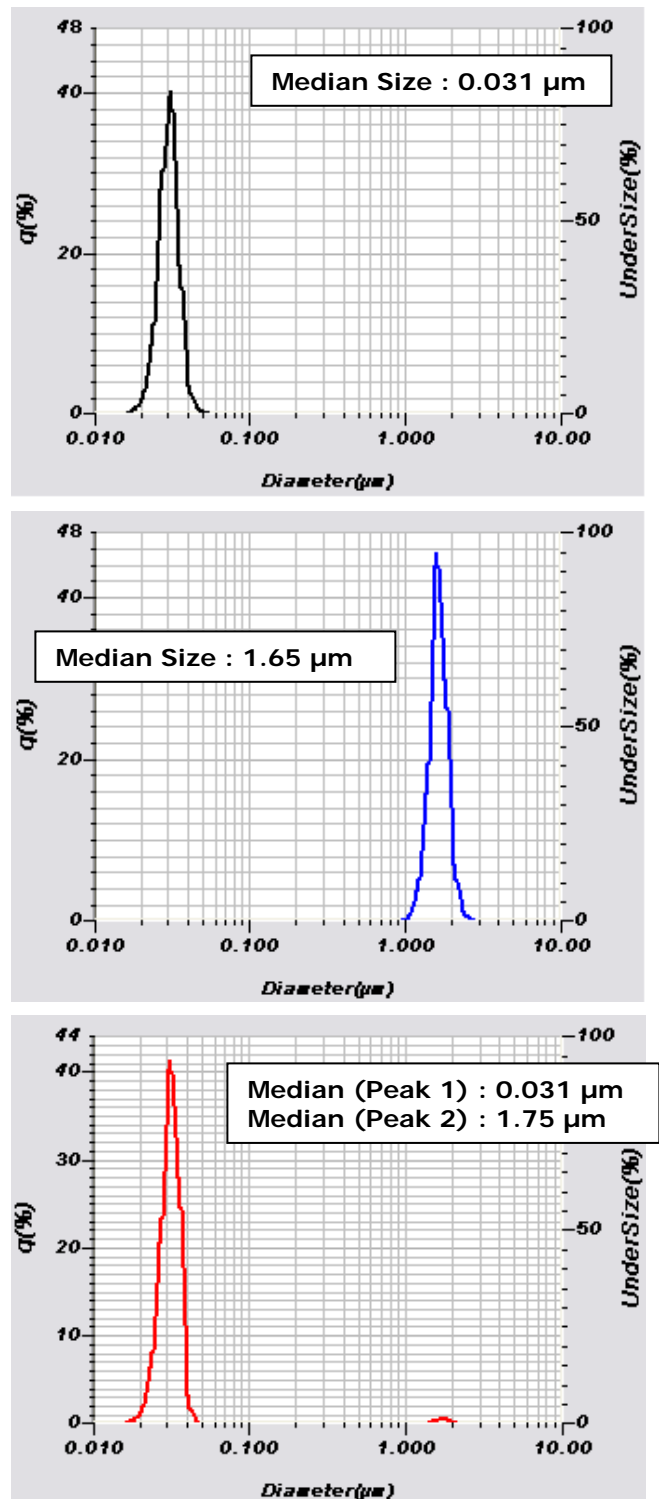


Figure 1: Top: LUDOX TM-50 particle size distribution, Middle: 0.1wt% Geltech 1.5 particle size distribution, Bottom: 0.05wt% Geltech 1.5 in LUDOX TM result shows both oversize particle detection and very good accuracy for both components.

**Testing Laser Diffraction Performance: Results**

The individual LUDOX TM-50 accuracy test produced a median size value of 31 nm which is consistent with product specification (Figure 1). The measurement of 0.1wt% Geltech 1.5 silica slurry produced a median size value of 1.65 μm which is also consistent with product specification. Measurement results for the 0.5wt% Geltech 1.5 in LUDOX TM solution showed correct location of the oversize particles and very good accuracy of the colloidal silica. Further dilutions of the Geltech 1.5 produced similar results with the only difference being the decreasing volume percentage of oversize particles in the particle size distribution. Finally, the LA-950 proved incapable of adequately measuring a solution of 0.025wt% Geltech 1.5 in LUDOX TM. The lowest acceptable dilution in terms of both accuracy and detection was 0.05wt%. Assuming equivalent density this corresponds to roughly 3 parts per billion Geltech 1.5 in LUDOX TM.

Conclusions

Given the demanding challenges of measuring CMP slurries no one particle sizing technique may be considered perfect. Laser diffraction, however, presents the best available option for measuring the primary nano-scale component and detecting a very small amount of damaging oversize particles. The combination of measurement accuracy and precision (results are highly reproducible across multiple instruments and operators) with best available speed and ease of use create a compelling case for using a state of the art laser diffraction particle size analyzer for CMP slurries.

References

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