A New Method for Determining the Size Distribution of Particles in CMP Slurries

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The particle size distribution (PSD) of the "working" particles in CMP slurries strongly influences the efficacy of the planarization process. Current techniques used to determine PSD typically only measure relative particle concentrations and often presume the shape of the distribution. This causes serious problems for CMP Engineers who require more precise information to optimize their processes. A new approach uniquely combining proven techniques used for counting and sizing aerosol PSDs has been developed to measure both particle size and number concentration in CMP slurries.

Keywords: Nanometer Particles, Particle Size Distribution, Chemical-Mechanical Planarization, Nebulizer, Differential Mobility Analyzer, Condensation Particle Counter,

1. Introduction

The use of CMP slurries, and CMP steps in Integrated Circuit processing is growing exponentially and becoming ever more critical to manufacturing vertically stacked semiconductors. While the International Roadmap for Devices and Systems (IRDS) does not specifically reference the sizing of CMP slurries, the need for measuring sub-10 nanometer particles in all materials that come in contact with a wafer is unmistakable. The killer particle size is rapidly getting smaller and smaller. By 2019 the killer size, defined as logic ¼ pitch, will be 7nm. The need for improved CMP particle size detection, particularly at sub-50nm, is urgent.

Current CMP sizing is done by using Dynamic Light Scattering (DLS), where temporal fluctuations are usually analyzed by means of the intensity or photon auto-correlation function. While this technique has the capability of measuring particles as small as 1nm under ideal conditions, a number of short-comings remain. The most significant short coming is DSL's inability to measure absolute particle concentration and its difficulty of measuring multi-model size distributions (most CMP slurries contain a mixture of particle sizes).

To address these problems Kanomax FMT, in collaboration with CT Associates (Eden Prairie, MN) has developed a non-optical particle sizing system based upon classifying a PSD using electrical mobility and subsequent counting with a condensation particle counter [1]. This sizing technique is used by both the National Institute for Standards and Technology (NIST in the USA) and by the National Institute of Advanced Industrial Science and Technology (AIST in Japan) as a fundamental particle sizing technique.

2. The Measurement Technique

The technique, illustrated in Figure 1, includes the use of an ultrafine nebulizer and a scanning mobility particle sizer. A colloidal suspension (such as CMP slurry) is diluted on-line using ultrapure water (UPW) and injected into the nebulizer. The nebulizer (NanoParticle Nebulizer) generates a droplet distribution in filtered air. The resultant droplet distribution is diluted and dried with additional filtered air to form an aerosol laden with the particles originally in the colloidal suspension. The aerosol PSD is then measured using a Differential Mobility Analyzer (also called an Annual Flow Ion Mobility Classifier) and a Condensation Particle Counter (Fast CPC). All three components are manufactured by Kanomax FMT and commercially available as the Liquid Nanoparticle Sizing (LNS) system.

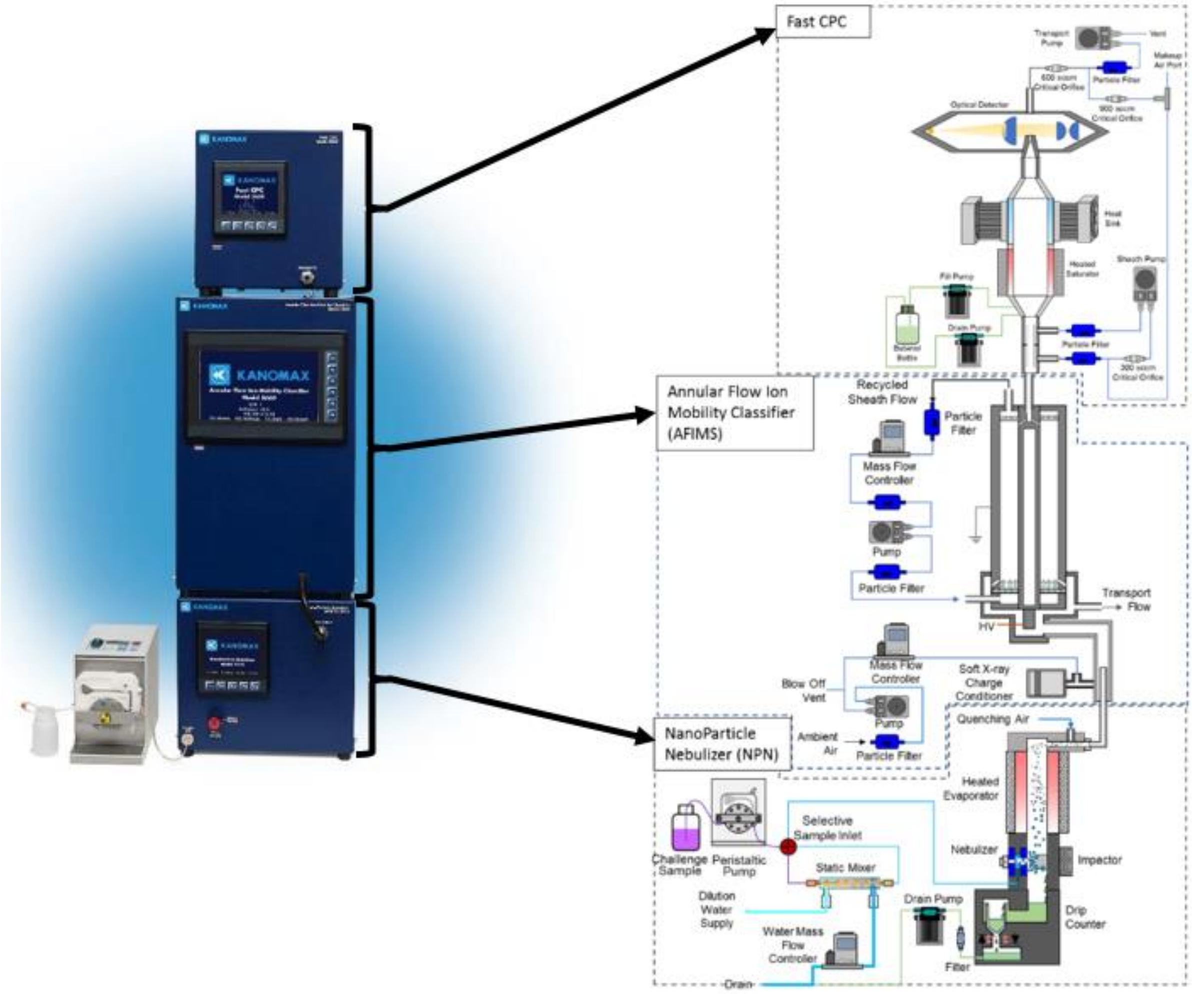


Figure 1. Photograph and schematic of the Kanomax FMT Liquid Nanoparticle Sizing system.

The key to the LNS measurement approach is the nebulizer [2]. Dissolved non-volatile residue in the suspension forms particles when the droplets from the nebulizer are dried. These residue particles can interfere with the particle analysis. The unique nebulizer used in the LNS system minimizes this effect by producing droplets that are sufficiently small and uniformly sized that particles formed from dissolved materials in the droplets are small enough as to not interfere with the particle size measurement. In addition, the particle suspension is sufficiently dilute that no more than one particle is present in each droplet. The LNS technique of measuring colloidal PSDs offers many interesting capabilities. This technique measures number concentrations, not relative particle concentrations (that is, one size relative to another) and makes no assumption regarding the shape of the PSD. The number-weighted PSD measured may easily be converted to alternative weightings such as diameter, surface area, or volume weighted distributions. Since this technique measures individual particles, it is highly sensitive to measuring very small changes in the PSD, and is not affected by the optical properties and density of the particles. Further, the technique is insensitive to air bubbles in the suspension, unlike other methods.

3. Sizing Accuracy

The accuracy of the LNS measurement technique is shown in Table 1. Three different sizes of gold colloids (supplied by BBI Research, Madison, WI) were measured individually. As seen in Table 1, the sizing measurements correlate well with the manufacturer's sizing claims. Measurements of polystyrene latex (PSL) and silica particles have also shown good agreement with manufacturer's claims [3].

Nominal Size (nm)	Manufacturer measured size		LNS measured size	
	Mean (nm)	CV (%)	Mean (nm)	CV (%)
10	9.3	< 15	8.4	13
20	20.3	< 8	20.8	7.4
30	30.3	< 8	30.5	7.3

Table 1. Sizing comparison between certified colloidal gold standards and LNS measurements

4. Measuring the PSD of CMP Slurries

The PSD of a tri-modal CMP polishing slurry measured by the LNS is presented in Figure 2. (Scanning Electron Microscopy of the slurry confirms the tri-modal PSD). As discussed previously, the LNS directly measures the sizes and numbers of individual particles in the colloidal suspension; variations in concentration of specific size particles amongst samples can readily be detected. Variations in particle concentration can affect process efficacy (such as affecting material removal rates and selectivity during polishing). In comparison data from a Dynamic Light Scattering (DLS) instrument used to measure the same tri-modal polishing slurry is shown in the right-hand graph in Figure 2. The DLS is an ensemble measurement technique that measures the properties of many particles simultaneously and then fits the data to an assumed lognormal PSD. In addition, only relative particle concentrations are provided.

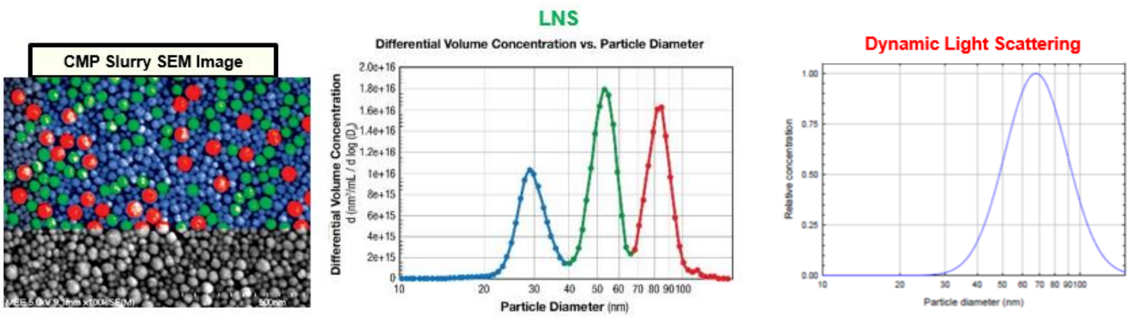


Figure 2. PSD measurement of the same tri-modal CPM slurry (identified by the colors blue, green and red) using LNS and DLS

Handling slurry (i.e. circulating through a distribution system) can result in changes in the particle size distribution and agglomeration of slurry particles [4]. The ability to measure the change in the "working particle size distribution" is beneficial to the CMP engineer seeking tighter process control and improved removal efficacy. Figure 3 shows the cumulative effect of circulating a batch of slurry containing colloidal silica particles. As the number of turn-overs (passes through the pumping system) increased, the concentration of large particles increased. This suggests that this slurry is susceptible to handling damage with small particles agglomerating to form larger particles.

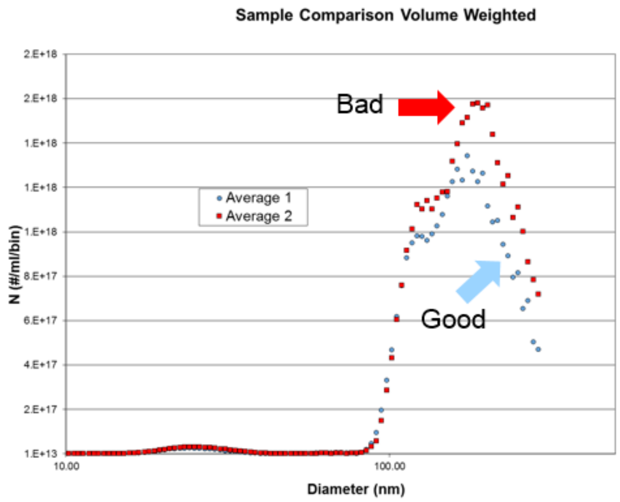
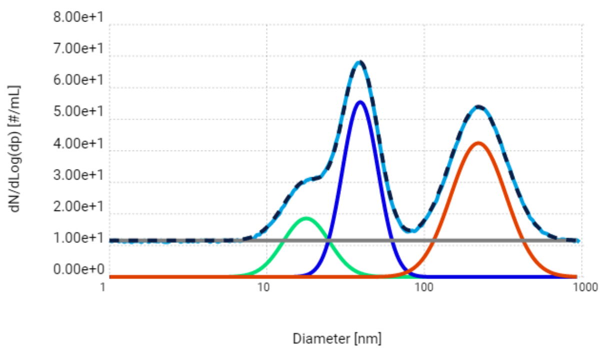


Figure 3. LNS differential volume data showing the subtle size difference between “good” and “bad” CMP slurry

5. Software Analysis

Kanomax FMT has developed a sophisticated curve fitting algorithm to analyze each individual component within a CMP slurry. Figure 4 shows a typical data analysis for a tri-model CMP slurry. Each individual mode is analyzed and size measurements reported for cumulative size distribution at D10, D50 and D90 (i.e. the size corresponding to 10%, 50% and 90%). Additional statistical information is also calculated and displayed.



Name	Total (#/mL)	d_{gm} (nm)	GSD	D_{10} (nm)	D_{50} (nm)	D_{90} (nm)
Mode 1	436	17.8	1.4	11.5	17.8	27.4
Mode 2	1017	39.2	1.3	28.4	39.2	54.2
Mode 3	1200	220.7	1.5	133.4	220.7	365.2
Background	2216	-	-	-	-	-
Sum	4.86e+3	-	-	4.5	47	339.8

Figure 4. LNS data output from a tri-model CMP slurry

Note: Patent numbers 8,272,253, 8,573,034, 9,086,350 and 9,513,198 have been issued to CTA and licensed by Kanomax FMT. Patent number 7,852,465 has been issued to Kanomax FMT. Kanomax FMT has applied for additional domestic and international patents for technology contained within the LNS.

6. Summary

A new method of characterizing CMP slurry using the Liquid Nanoparticle Sizer (LNS) was introduced. The method does not rely on optical methods and so can provide absolute concentration values and handle multi-modal particle distributions, unlike more traditional methods. The LNS System provides CMP engineers with a powerful analysis tool for optimizing their processes.

7. References

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