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

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Introduction

• The particle size distribution (PSD) of the “working” particles in CMP slurries influences the efficacy of the planarization process.

• The techniques presently used to determine the PSD typically only measure relative particle concentrations and often presume the shape of the distribution.

• This presentation describes a new technique that allows measurement of both size and concentration of the working particle size distribution.
Outline

• Method description

• Measurement of “monodisperse” particles
  – Comparison to dynamic light scattering
  – Comparison of measured and claimed particle size uniformity
  – Comparison of measured concentrations

• Measurement of slurry particle size distributions
  – Comparison to dynamic light scattering

• Example of tracking slurry properties during handling
  – Comparison to dynamic light scattering
  – Comparison to large particle tail measurement

• Summary
Measurement method – Ultrafine atomization (UFA)

Patent pending.
Scanning mobility particle sizer (SMPS)

Polydisperse aerosol

Aerosol “neutralizer”

Differential mobility analyzer (DMA)

Monodisperse aerosol

Condensation Particle Counter
Differential mobility analyzer (DMA)
Condensation particle counter (CPC)


CTA Pub 74 Slide 7
Measurement method – Ultrafine atomization (UFA)

Pressure regulator

UPW

Slurry Sample

CDA

Ultrafine Atomizer

Particle Aerosol

Scanning Mobility Particle Sizer

Patent pending.
Key technical challenge

• Non-volatile dissolved residue in the UPW and slurry will form particles when the droplets from the atomizer are dried.
• These residue particles can interfere with the slurry particle analysis.
• The atomizer must produce small, uniformly-sized droplets to prevent interference with the particle analysis.
Droplet size distributions produced by different atomizers
Residue interference with Atomizer D

Particle diameter (nm)

Differential number concentration, $d \#/$ml / d log ($D_p$)

Limited to about 20 nm with this slurry.
Sizing of 20, 50, and 80 nm PSL particles

![Graph showing particle diameter vs. differential volume weighted concentration](image-url)
Sizing of 9 different sized PSL particles

Normalized size distributions at different coellicients of variation (CVs)

Measured CV of 20nm PSL

Measured CV of 50nm PSL

Measured CV of 80nm PSL
Comparison between claimed and measured PSL size uniformity

![Graph comparing claimed and measured PSL size uniformity](image-url)
Comparison to 50nm Optical Particle Counter

670 ppb of 30nm PSL

Cumulative concentration (#/ml)

Particle diameter (nm)
PSD in slurry A – Silica particles

UFA – Dynamic Light Scattering (DLS) Comparison
Silica particle slurry A

Particle diameter (nm)

Normalized differential volume weighted distribution

UFA
NICOMP 380ZLS
PSD in silica particle slurry B

Differential volume weighted concentrations.

Normalized concentration

Particle diameter (nm)

NICOMP
UFA
Measuring the effect of handling on Slurry A PSD

Increasing amount of handling.

Particle diameter (nm) vs. Differential number concentration $\frac{d\#}{d\ln(D_p)}$
Measuring the effect of handling on slurry PSD
Volume weighted concentrations


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Slide 21
Effect of handling – comparison to dynamic light scattering (NICOMP 380ZLS)*

* - Particle Sizing Systems, Santa Barbara, CA.


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Effect of handling – comparison to dynamic light scattering (NICOMPP 380ZLS) – NICOMPP normalized to UFA

UFA

NICOMPP

Increasing amount of handling.
Change in cumulative concentrations over time during handling

![Graph showing change in cumulative concentrations over time during handling](image_url)
Comparison between UFA and large particle tail measurements during slurry handling

Particle diameter (nm)

Cumulative number concentration, #/ml

UFA measurements

Accusizer 780* measurements

Increasing amount of handling.


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Summary

• A new method for the measurement of the working particle size distribution in CMP slurries has been described.
• The method allows measurement of particle concentrations as well as size.
• The method was shown to:
  – Accurately size and count PSL particles of known size
  – Allow measurement of the working particles in CMP slurries
  – Agree qualitatively with dynamic light scattering measurements of slurry working particle size distributions
  – Discern changes in slurry working particle size distributions induced by handling