



Online Monitoring of Particle Size in CMP Slurries by SPOS, Requiring Little or No Dilution

David Nicoli, Kerry Hasapidis, Paul Toumbas Particle Sizing Systems, Inc. Santa Barbara, CA and New Port Richey, FL

CMPUG Meeting, September 4, 2002 Sunnyvale, CA



♦ Features of AccuSizer_{FX}

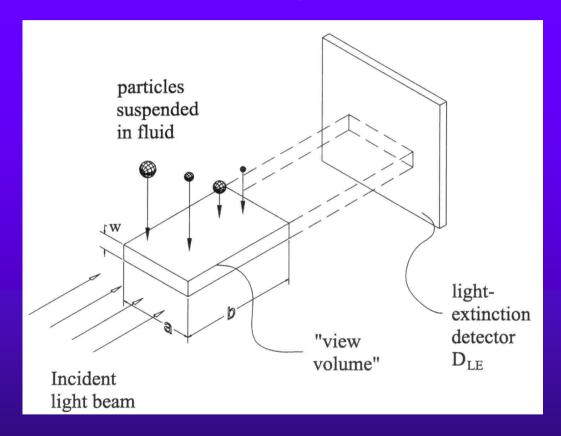
- High Concentration
 - Little or NO Dilution
- High Resolution: Singleparticle Optical Sensing
- High Accuracy
- High Reproducibility
- Sensitivity to outliers several standard deviations above the mean diameter
- Adjustable lower size threshold limit
- Fast, easy to use

Applications

- CMP Slurries
- Emulsions (oil/water)
- Dispersions
- Paints
- Inks / Pigments
- Ceramics
- Filtration media
- Homogenized products
- Abrasives
- Protein aggregates
- Macromolecules



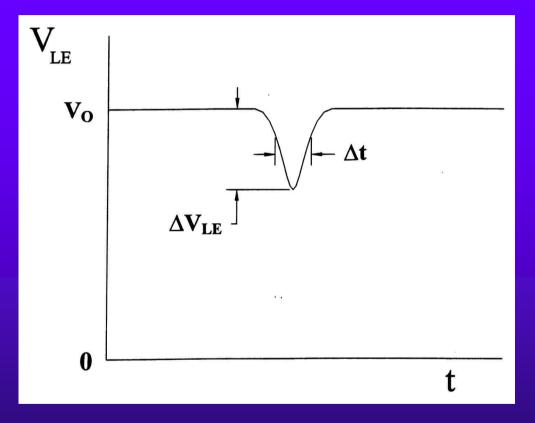
Single-Particle Optical Sensingtrue PSDs, of Highest Resolution



Light-Extinction (LE) -- Momentary decrease in light intensity transmitted across a flow channel, caused by the passage of a particle through a very small optical sensing zone (OSZ). Mechanisms: refraction and scattering



High Resolution ... using SPOS High Concentration ... AccuSizer $_{FX}$

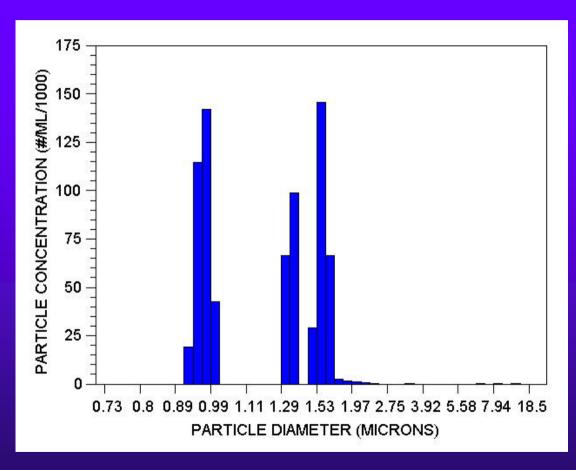


Light Extinction (LE) -- Stylized representation of signal pulse produced by a single particle passing through the OSZ.

Useful size range of AccuSizer_{FX}: ≈ 0.6 to 25 µm



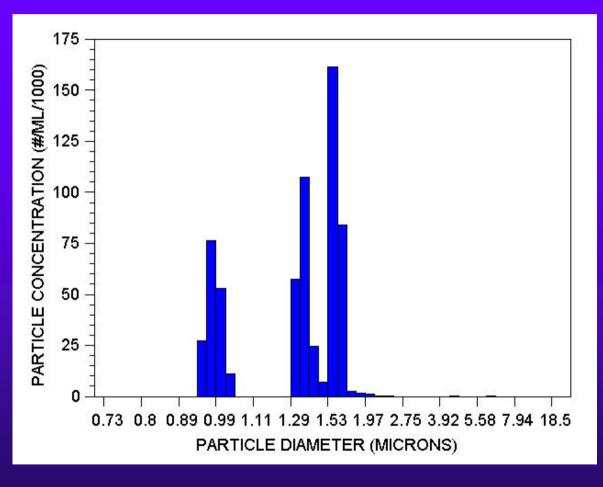
High Resolution (SPOS) and High Concentration (AccuSizer $_{FX}$)



TRI-MODAL #1: 0.5-uL 1-μm + 1-uL 1.36-μm + 2-uL 1.59-μm uniform latex particles (1% w/w), added to 40-ml water. Measured volume: 16-ml



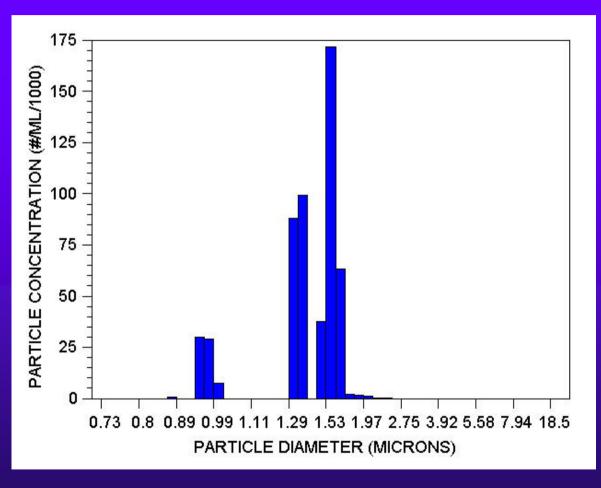
High Resolution (SPOS) and High Concentration (AccuSizer $_{FX}$)



TRI-MODAL #2: same as TRIMODAL #1, but only 1/2 the amount of 1-µm latex particles (0.25-µL added to 40-ml water).



High Resolution (SPOS) and High Concentration (AccuSizer $_{FX}$)



TRI-MODAL #3: same as TRI-MODAL #1, but only 1/4 the amount of 1-µm latex particles (0.125-µL added to 40-ml water)



High Resolution and Accuracy

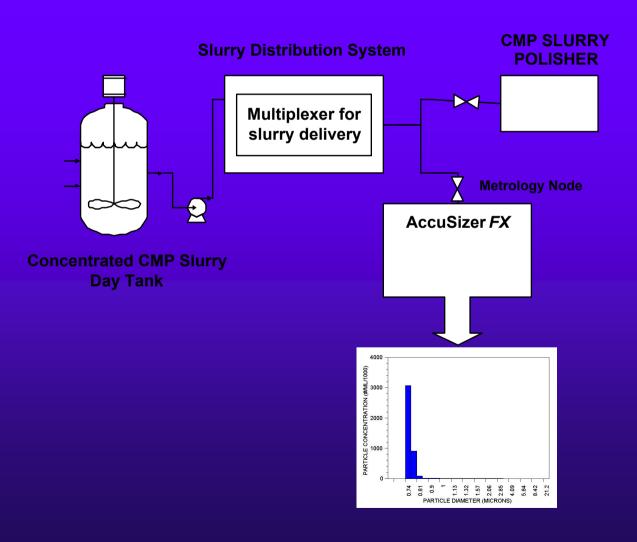
Summary of Latex Tri-modal Standard Results

Particles/ml/1000 -- Measured PSDs vs Expected Values

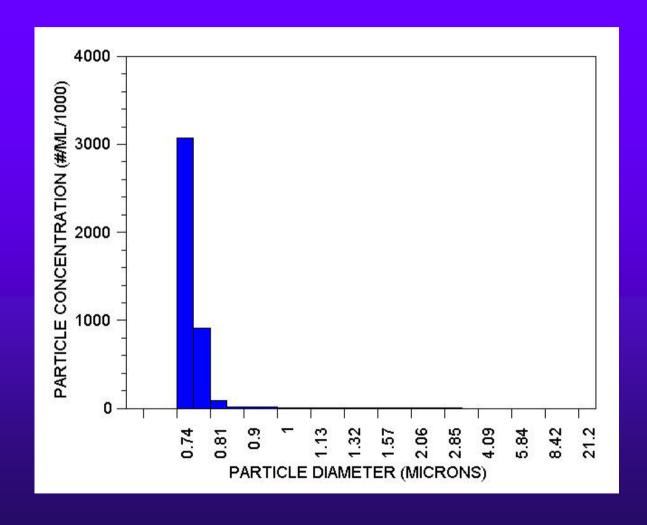
	1-μm Peak	1.36-µm Peak	1.59-µm Peak
Tri-Modal	PSD Expected	PSD Expected	PSD Expected
#1	318 325	165 177	242 272
#2	168 163	190 177	252 272
#3	67 81	187 177	272 272



Online Particle Size Monitoring of Concentrated CMP Slurries

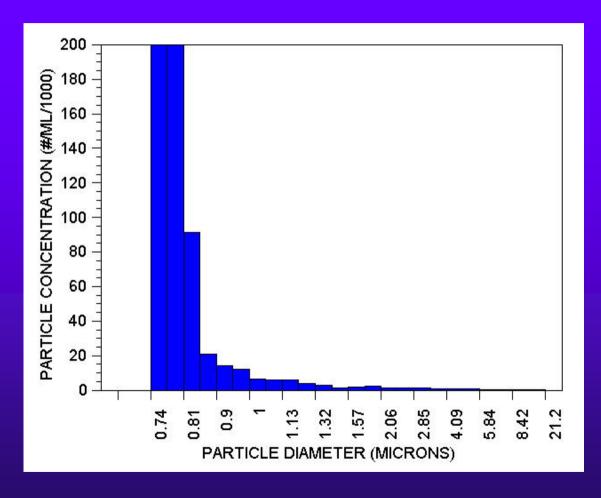






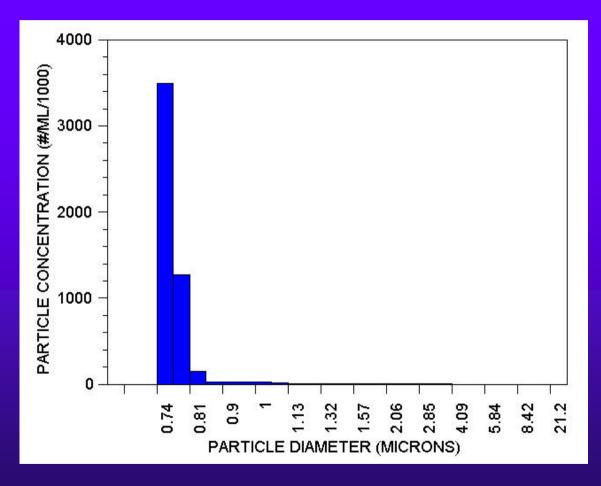
Silica CMP Slurry (SS25) -- full working concentration





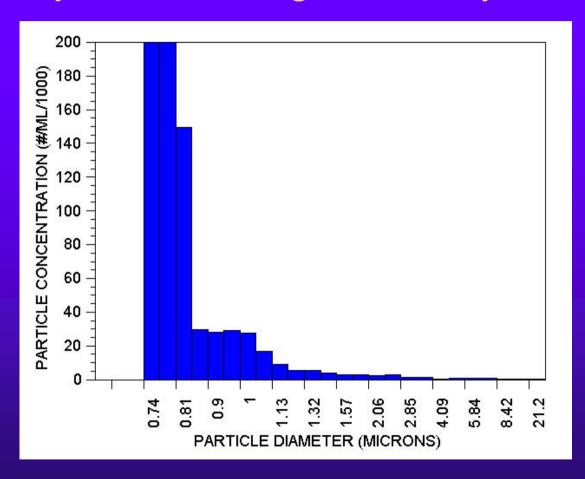
Silica CMP Slurry (SS25) -- *full working concentration* Y-Axis expanded 20× to emphasize large-particle outliers





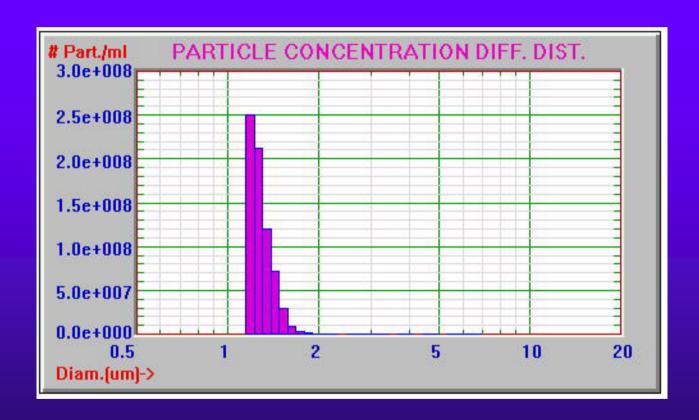
Silica CMP Slurry (SS25) -- *full working concentration* "Spike" of 1- μ m latex particles added to raw slurry ($\approx 1.3 \times 10^5$ /ml)





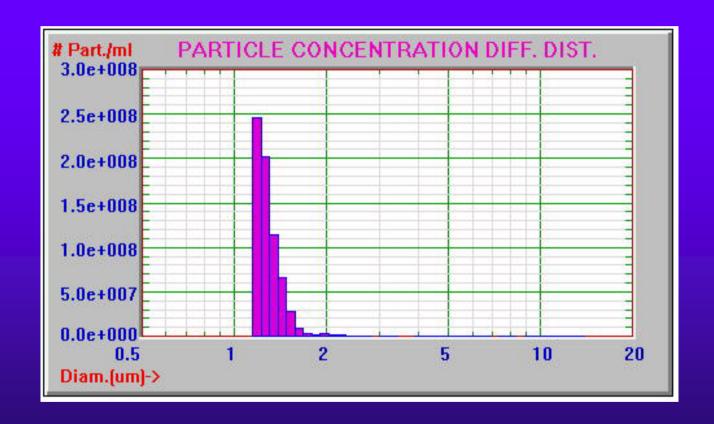
Silica CMP Slurry (SS25) -- *full working concentration* "Spike" of 1-μm latex particles (≈ 0.5 ppm) added to raw slurry Y-Axis expanded 20× to emphasize the added latex spike





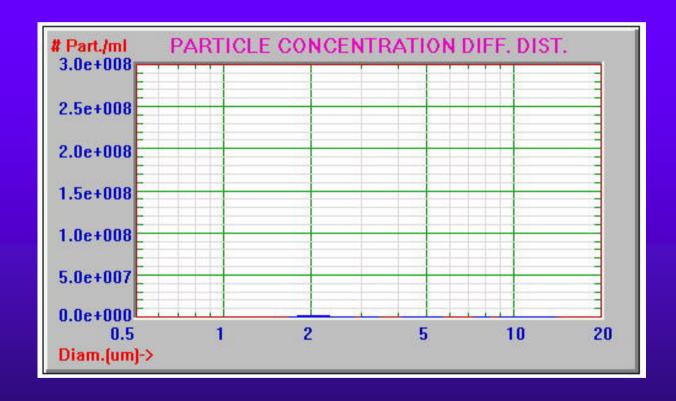
Cerium Oxide CMP Slurry (Hi8005) -- 160:1 dilution $\approx 7 \times 10^8$ particles/ml larger than 1.15-µm in starting slurry





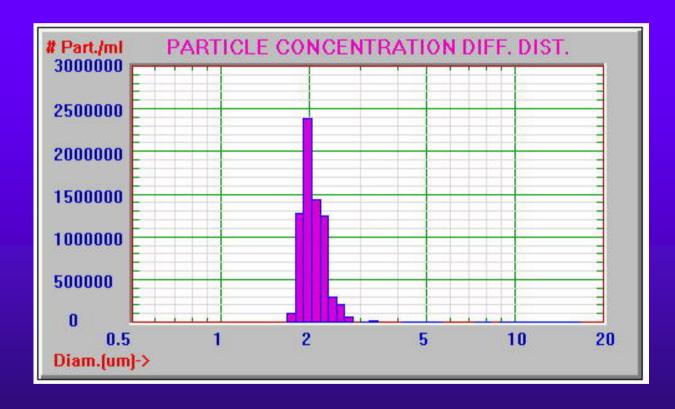
Cerium Oxide CMP Slurry (Hi8005) -- 160:1 dilution "Spike" of 2.013- μ m latex particles added to slurry ($\approx 9 \times 10^6$ /ml)





Cerium Oxide CMP Slurry (Hi8005) -- 160:1 dilution Result of subtracting plain-slurry PSD from spiked-slurry PSD



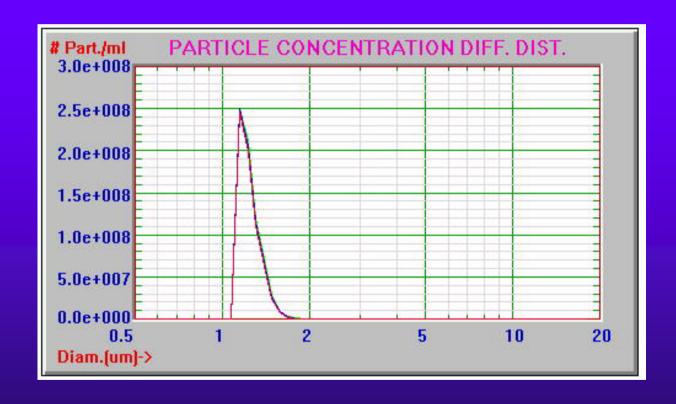


Cerium Oxide CMP Slurry (Hi8005) -- 160:1 dilution Result of subtracting plain-slurry PSD from spiked-slurry PSD Y-Axis expanded 100× Spike conc. ≈ 7 × 10⁶/ml



High Reproducibility ...

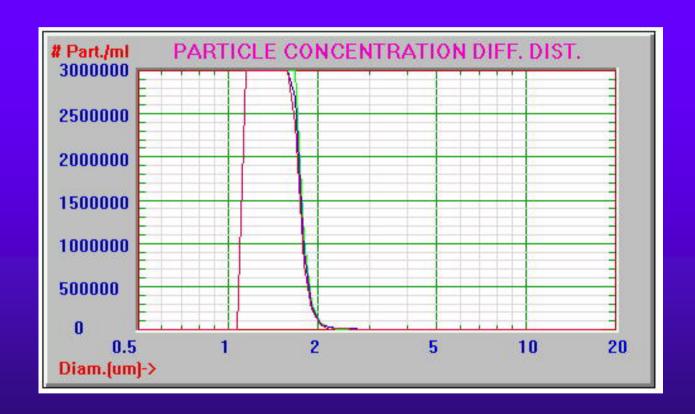
... the Key to Reliable AccuSizer _{FX} Measurements



Cerium Oxide CMP Slurry (Hi8005) -- 160:1 dilution Overlay of four consecutive measurements (120-sec each)



High Reproducibility the Key to Reliability of AccuSizer _{FX} Results

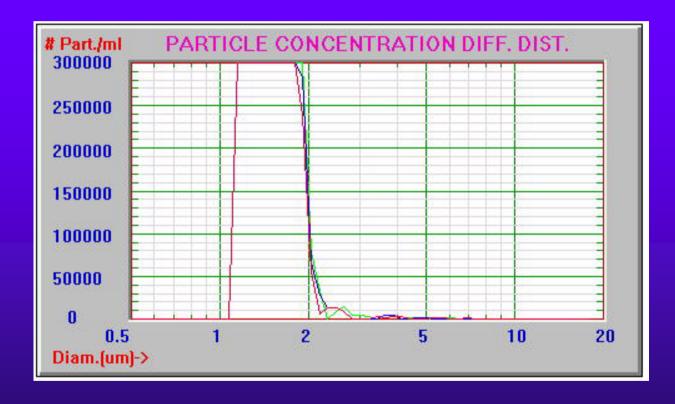


Cerium Oxide CMP Slurry (Hi8005) -- 160:1 dilution Overlay of four consecutive measurements (120-sec each) Y-Axis expanded 100×



High Reproducibility ...

... the Key to Reliability of AccuSizer _{FX} Results



Cerium Oxide CMP Slurry (Hi8005) -- 160:1 dilution Overlay of four consecutive measurements (120-sec each) Y-Axis expanded an additional 10× (total: 1000×)



AccuSizer $_{FX}$ High Sensitivity at High Concentration a powerful (and unique) combination!

- \bullet The AccuSizer_{FX}: The KEY to QUALITY
 - When particles several standard deviations away from the mean diameter of the distribution spell disaster (low yield) ... COUNT on the AccuSizer $_{FX}$ to detect and quantify them. The first step in solving a problem is identifying its root cause.
 - AccuSizerTM Trademark of Particle Sizing Systems
 - FX Technology Patents Pending