Atomization of CMP Slurry and Its Characterization

Handol Lee, Seong Chan Kim, Shawn Chen and David Y. H. Pui

Particle Technology Laboratory
Mechanical Engineering
University of Minnesota

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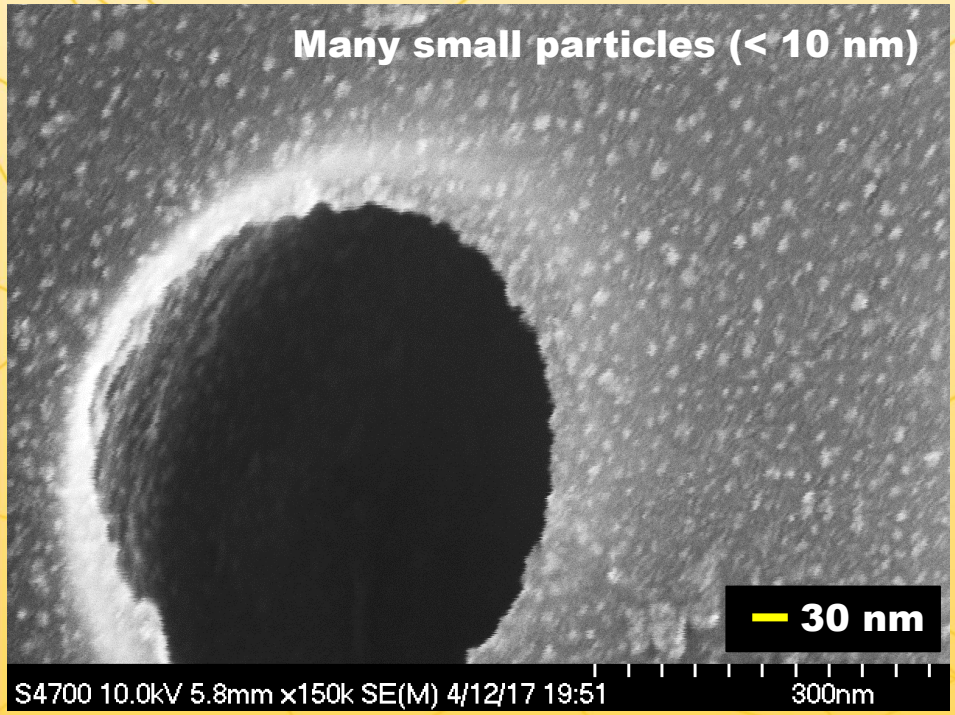
Introduction

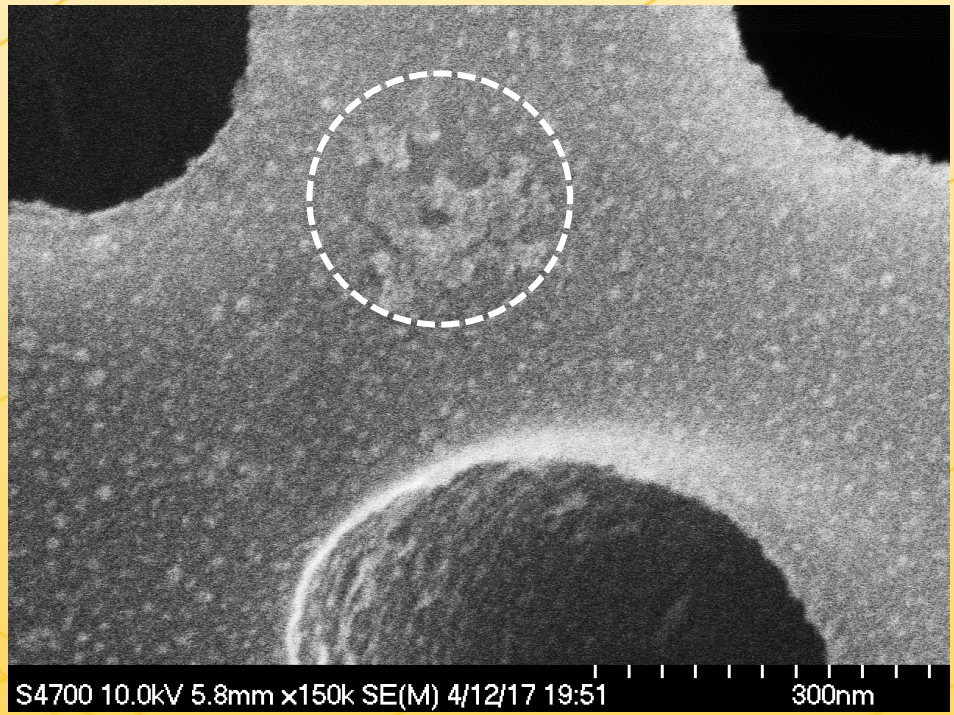
- Chemical mechanical planarization (CMP) is a material processing technique used to polish semiconductors.
- Abrasive particles in CMP slurry have a size distribution which directly affects critical metrics including rate of removal and wafer defects.
- Particle size analysis is therefore a key indicator of CMP slurry performance.

Objectives

- Characterization of CMP slurry, e.g., slurry particle size distribution

SEM Images of CMP



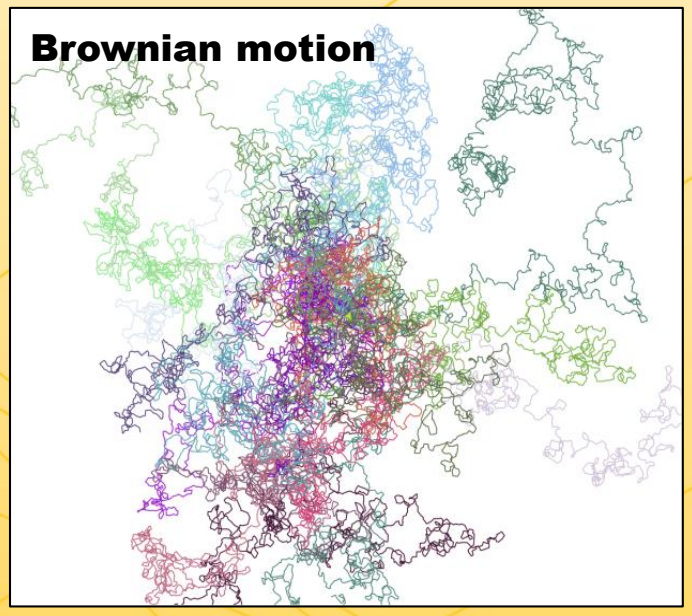


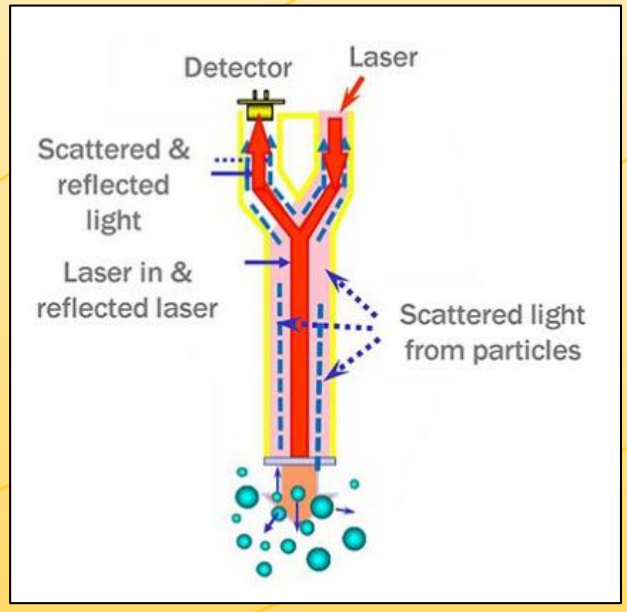
- **CMP slurry particles on a 400 nm rated Nuclepore filter**
- **Small particles (< 10 nm) and large aggregates are shown in SEM images.**

Size Measurement Methods

- **Dynamic light scattering (DLS)**
  - ✓ Measure the intensity of the scattered light fluctuates
  - ✓ Detection limit of particle size: > 1nm
- **Nanoparticle tracking analysis (NTA)**
  - ✓ Consist of laser and CCD camera to visualize particle behaviors
  - ✓ Measure particle size from diffusive Brownian motion of liquid-borne particles
  - ✓ Detection limit of particle size: > 20 ~ 30 nm
- **Aerosolization (scanning mobility particle sizer, SMPS)**
  - ✓ Dispersion by electrospray or atomizer
  - ✓ Measure airborne particle size distribution by SMPS
  - ✓ Detection limit of particle size: > 1 nm

DLS Measurement of CMP





- **Suspended particles exhibit Brownian motion.**
- **Particle velocity and size distributions are measured by light scattered from moving particles.**
- **Drawback**
  - ✓ **Low resolution (high concentration required)**
  - ✓ **Poor detection capability for particles with different sizes (mixtures)**
  - ✓ **Small particles hidden by larger particles**

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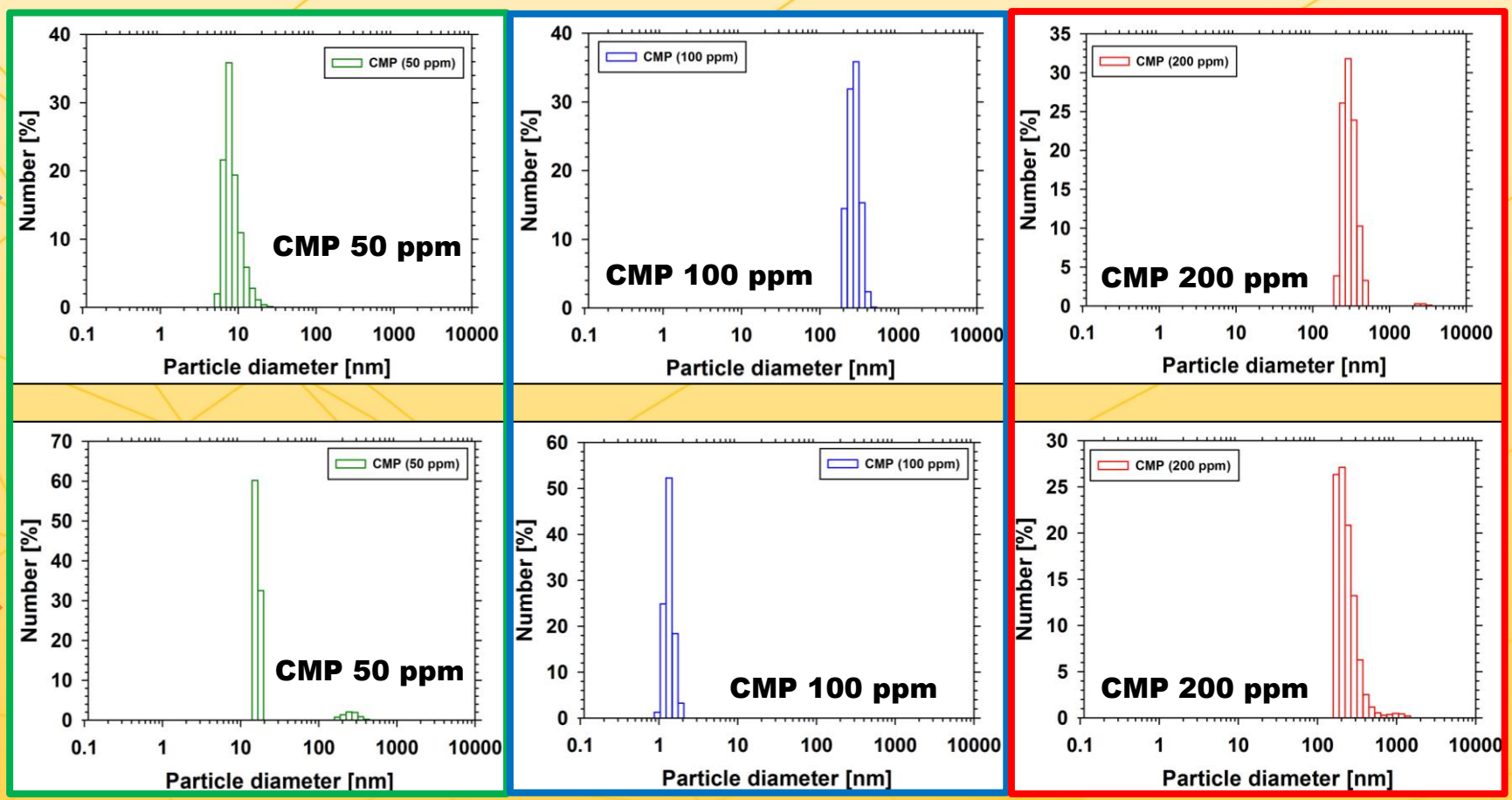
DLS Measurement of CMP with Different Concentrations

1st run

2nd run



- **Unstable (not consistent) size distribution, e.g., CMP 100 ppm**
- **Size distribution varies a lot with CMP concentration.**

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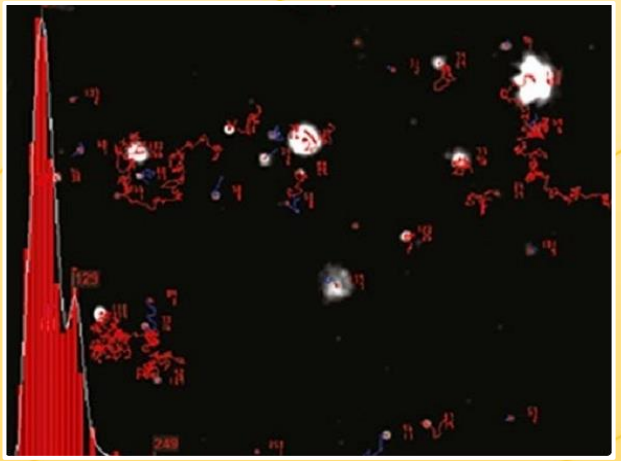
Nanoparticle Tracking Analysis (NTA)

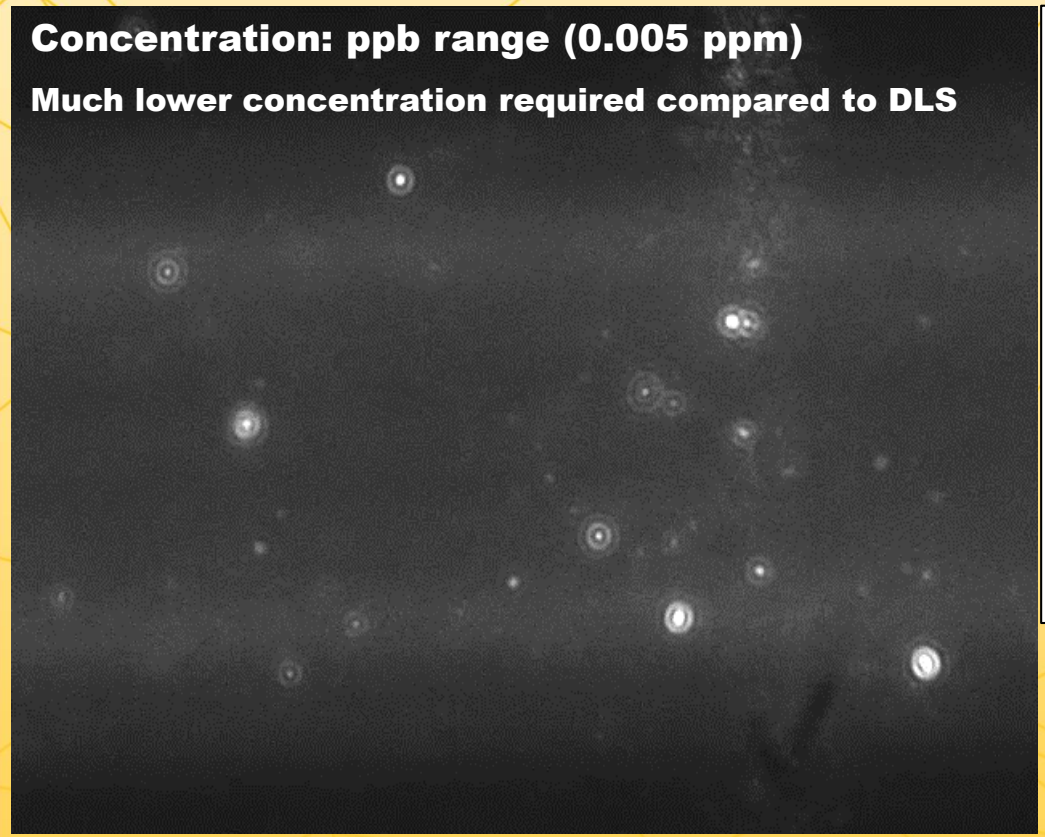
Recording

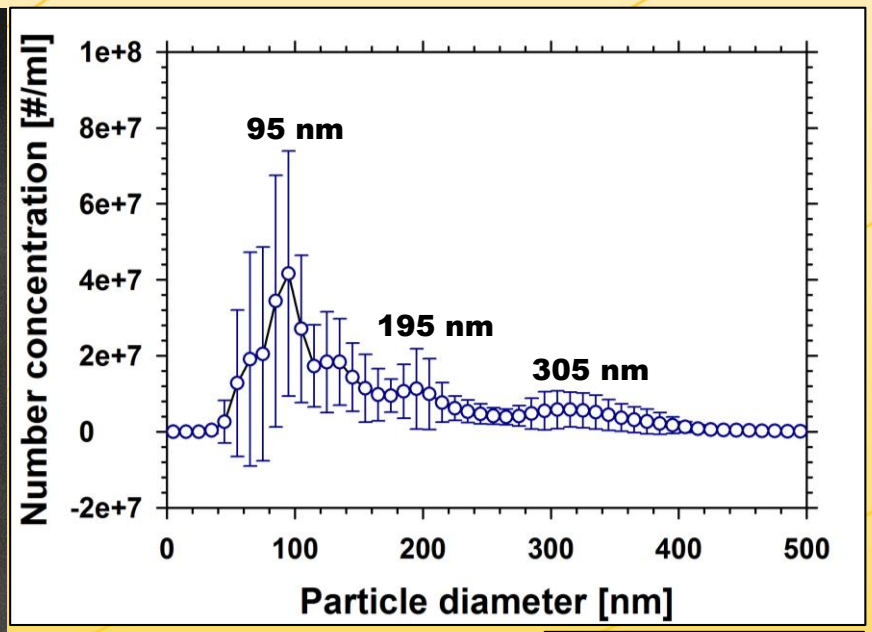


Tracking

- Composed of ultramicroscope, laser illumination and CCD camera
- Capturing light scattered by NPs using CCD camera
- Tracking Brownian motion of NPs from frame to frame
- Calculating particle size through the Stokes-Einstein equation
- Different setting required depending on types of samples (camera level and detection threshold)

NTA Measurement of CMP





- **Small particles (< 10 nm) cannot be seen by NTA.**
- **Visualized particles – larger than 20~30 nm (detection limit)**
- **Small particles (shown in the SEM image) are hidden by larger particles or cannot be seen (< 10 nm).**

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Aerosolization Methods

DISPERSION

Atomizer

Electrospray

MEASUREMENT

Scanning mobility particle sizer (SMPS)

- **Atomizer and electrospray are used for generating airborne particles.**
- **SMPS measures the size distribution of airborne particles (single particle counting).**
- **However, when using aerosolization method, residues contributed from water impurities and surfactant can interfere with the main particles.**

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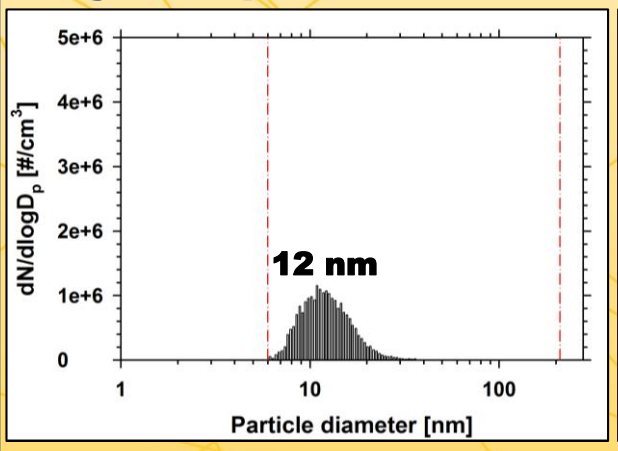
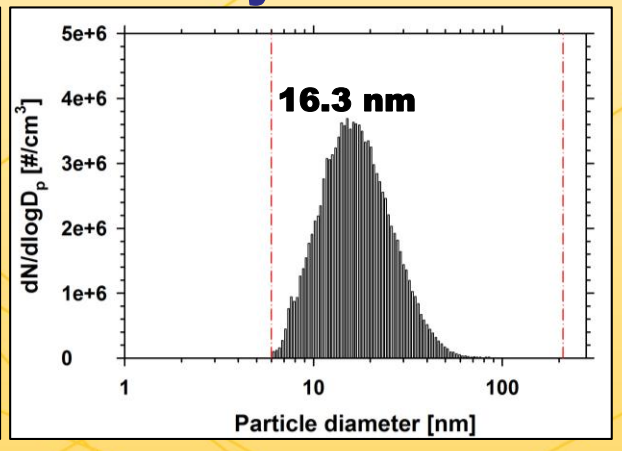
H. Lee, S.-C. Chen, C. Kim, E. Westenburg, S.I. Moon, D.Y.H. Pui, Sep. Purif. Technol. 184 (2017) 34–42.

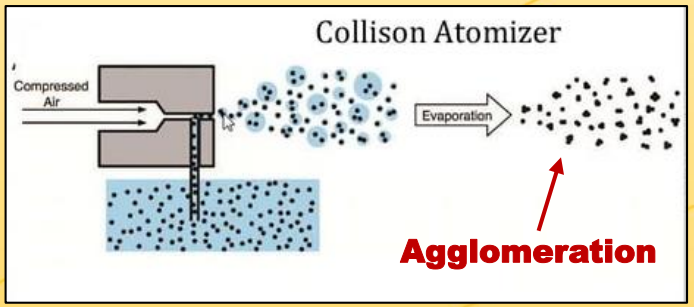
CMP Size Distribution Obtained by Atomizer - SMPS

Only ultrapure water



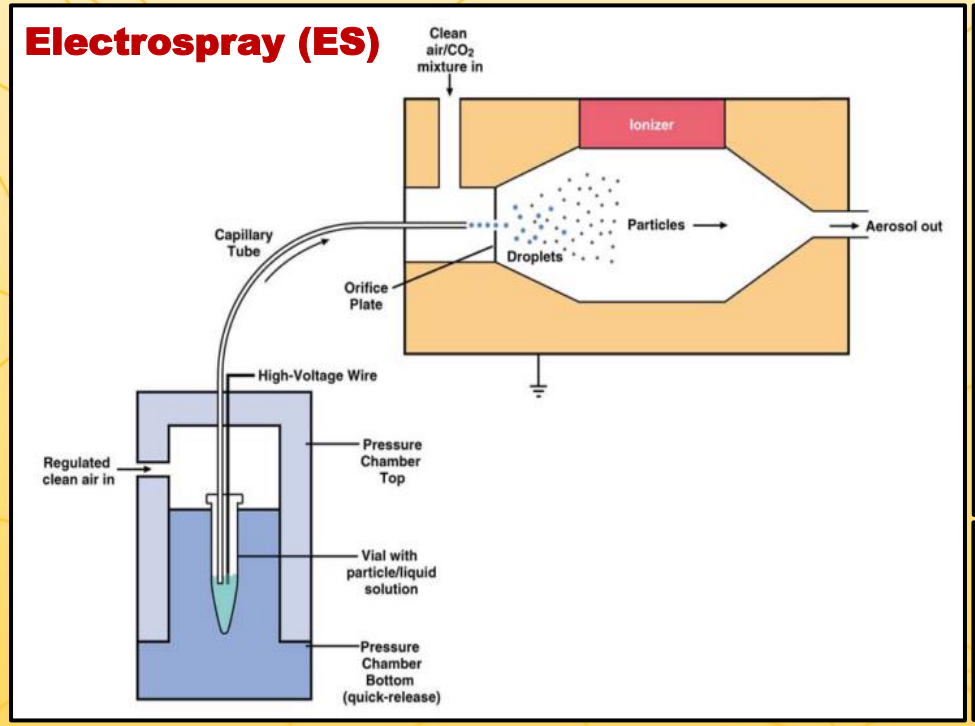
CMP slurry

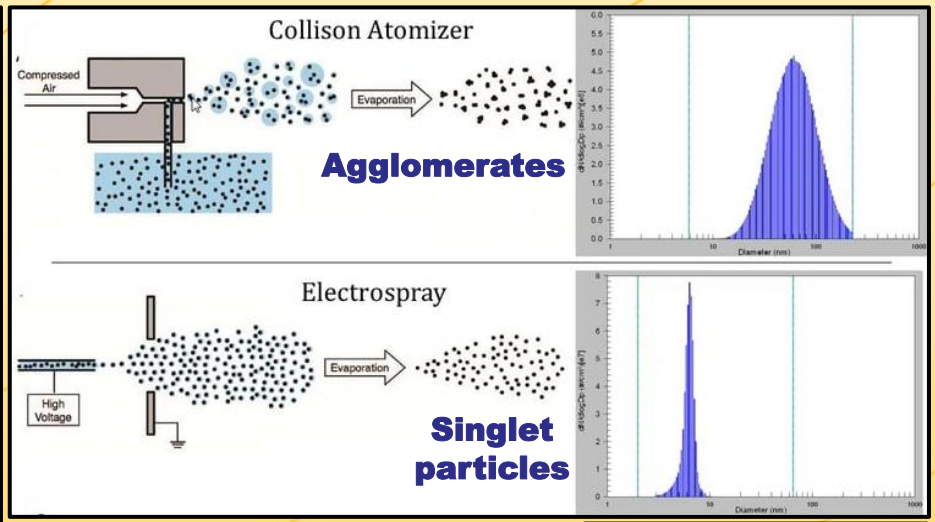


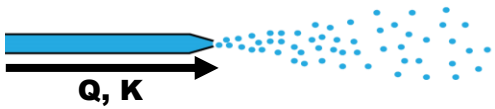


- **Residue particle size distribution from impurities of ultrapure water cannot be negligible (large droplets from atomizer).**
- **Due to the large droplets containing multiple particles, agglomeration occurs when the liquid is evaporated.**

Dispersion Methods







Q: flow rate in capillary tube

K: solution electrical conductivity

$D_d \sim (Q/K)^{1/3}$

Higher Q, Lower K

Larger droplet

Larger residue

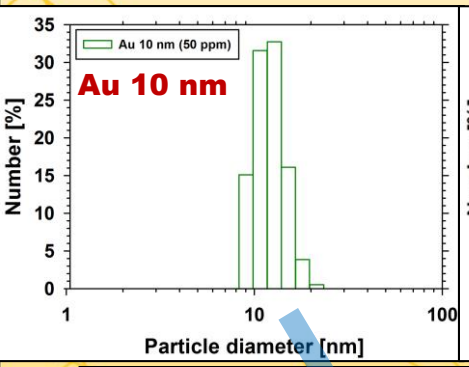
- ES generates very small monodisperse droplets (D_d ~ 200 nm).
- Due to the generation of small droplets, it can generate singlet particles (one particle in one droplet).

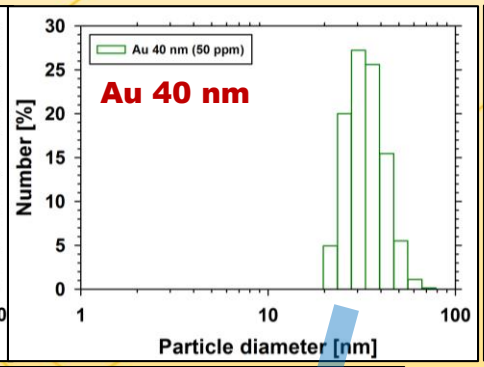
Mixtures of Different Particle Sizes

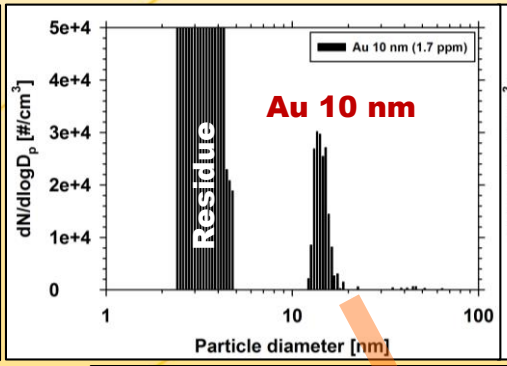
DLS vs ES-SMPS

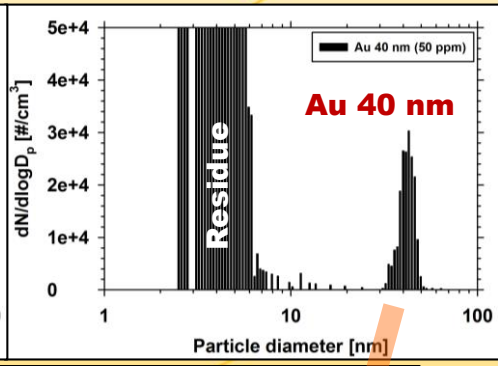
DLS

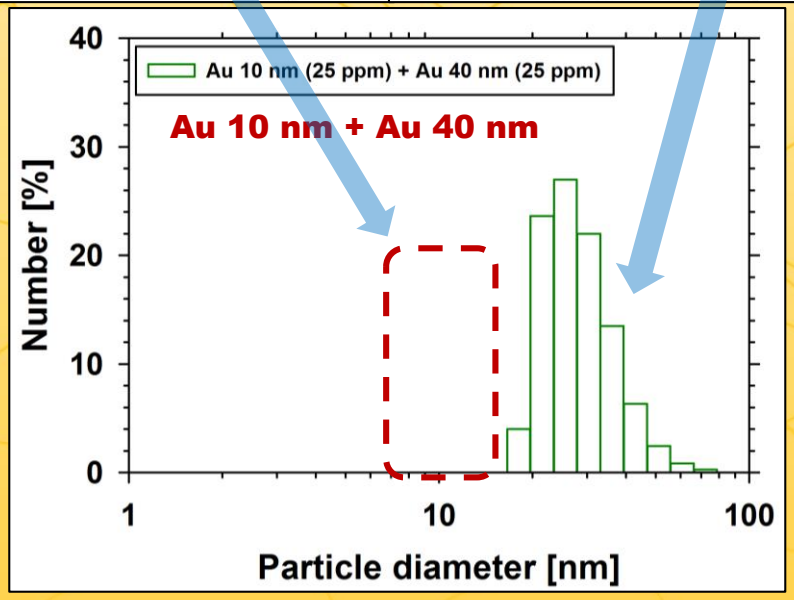
ES-SMPS

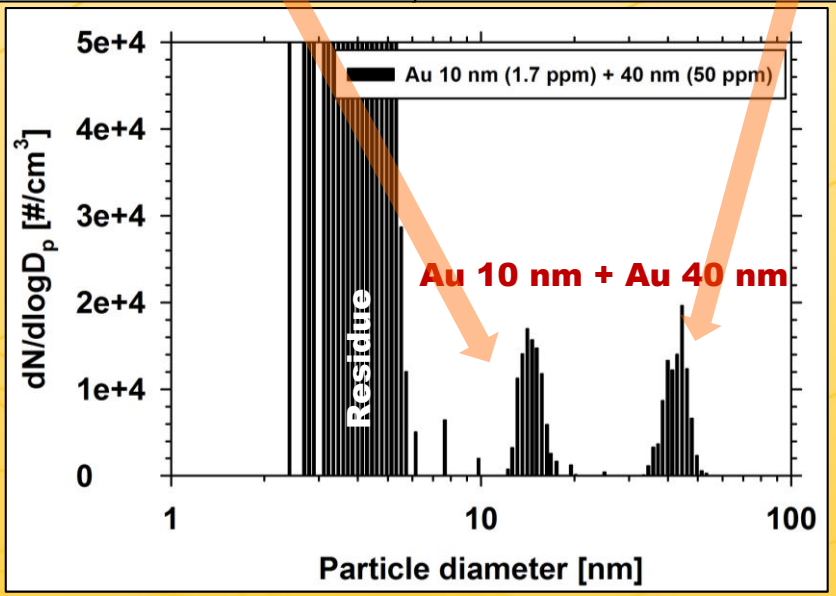












- **DLS cannot distinguish the two particle sizes.**
- **DLS output is dominated by the larger particle size, i.e., 40 nm.**
- **ES-SMPS is based on single particle counting, which enables to show completely distinguished 10 and 40 nm Au particles.**

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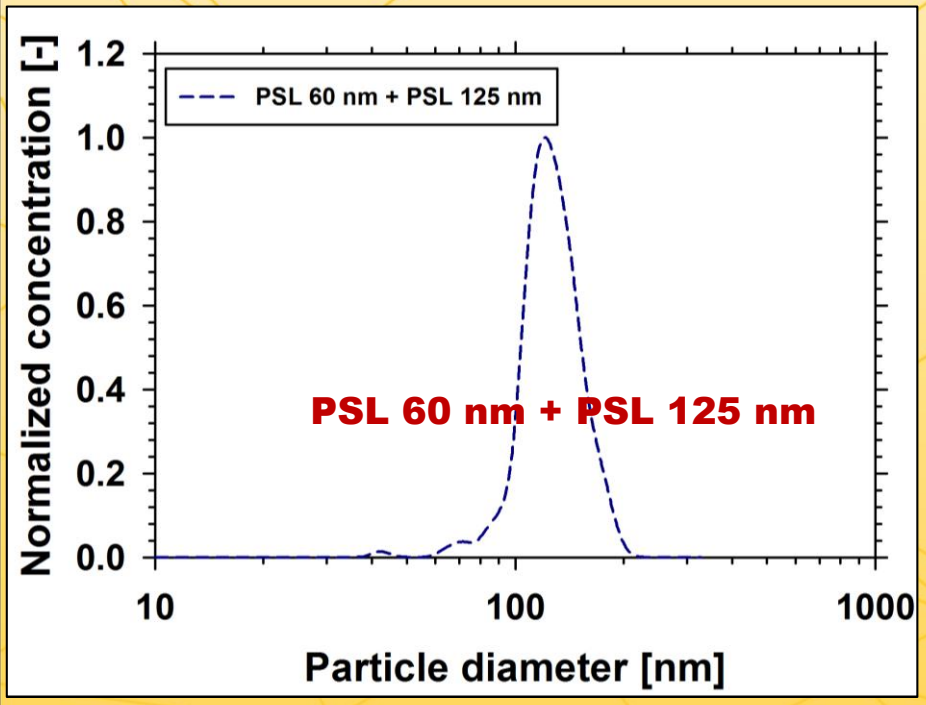
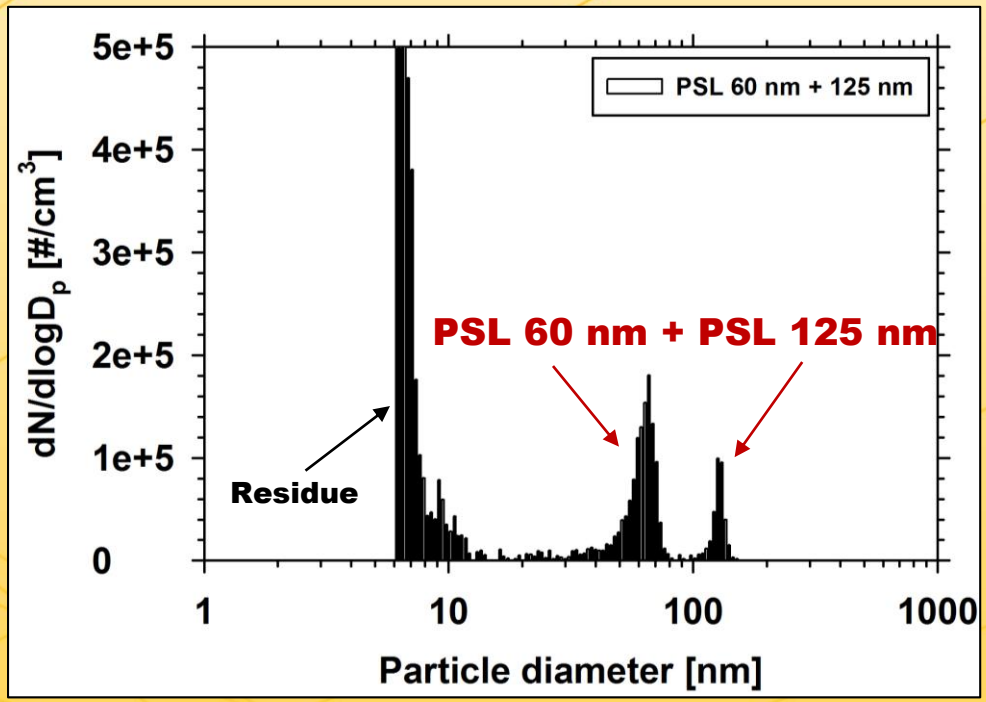
Mixtures of Different Particle Sizes
NTA vs ES-SMPS

NTA

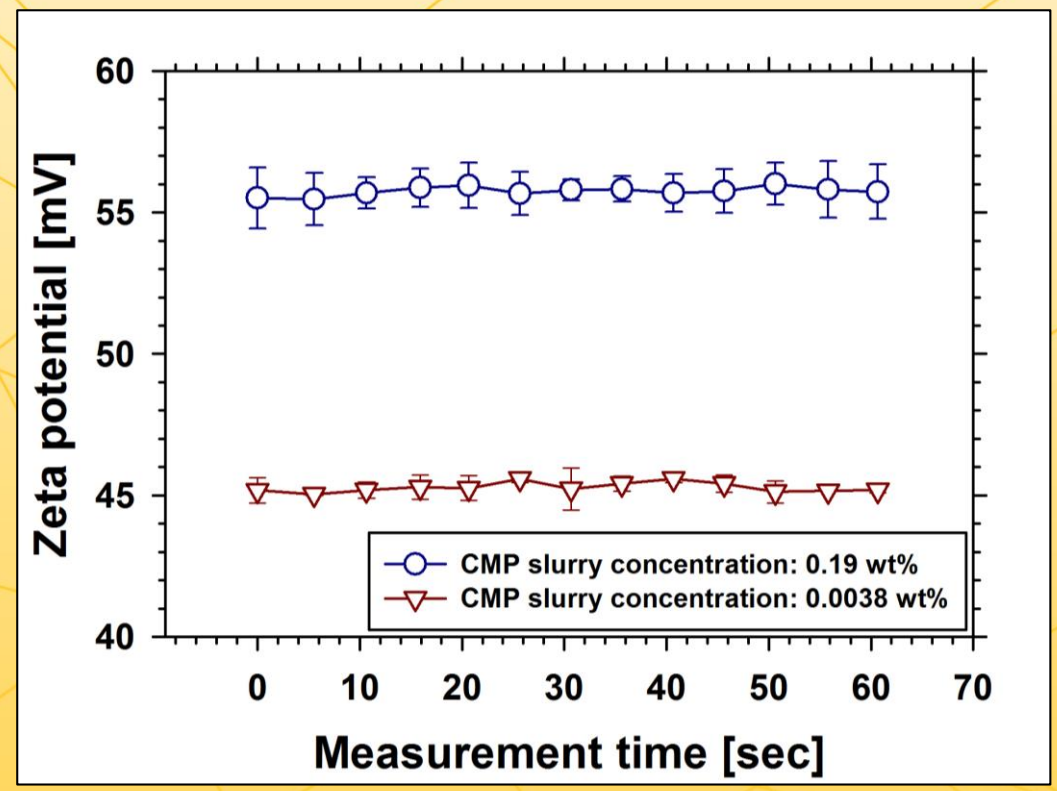


ES-SMPS

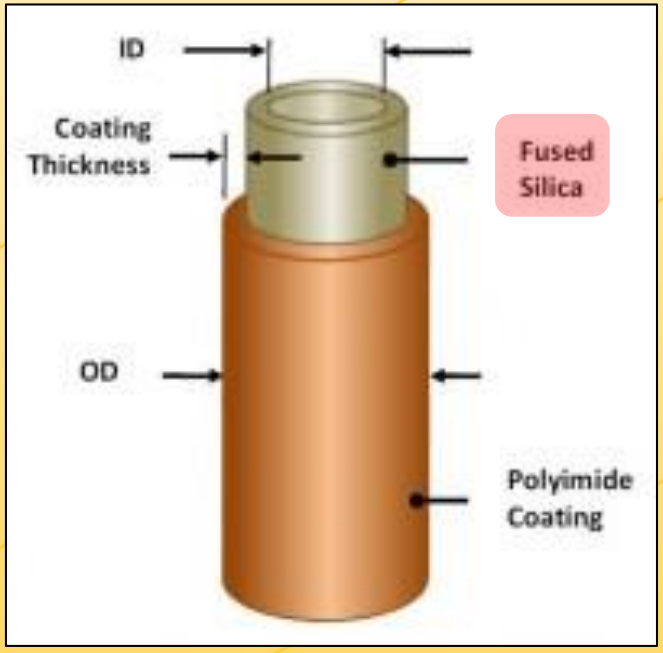


- **NTA cannot distinguish two particles.**
- **NTA is also based on intensity measurement.**
- **The NTA output is dominated by larger particles.**

Zeta Potential of CMP Particles



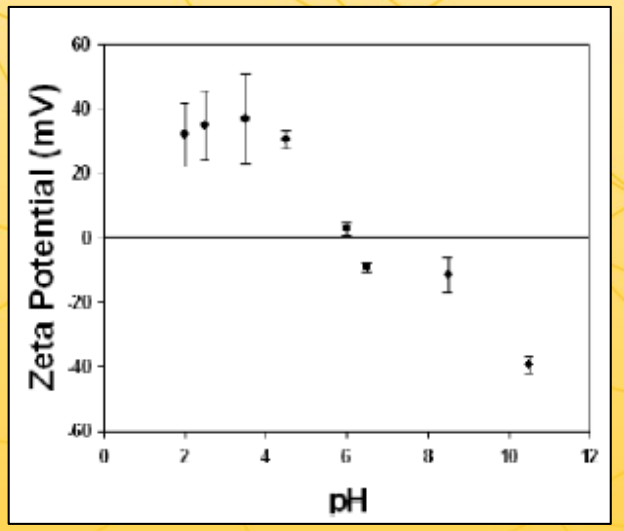
Capillary tube in electrospray



- **CMP slurry particles in ultrapure water have positive 55.7 mV zeta potential at pH 3.5 ~ 4.**
- **However, the silica surface inside capillary tube of ES has negative zeta potential, resulting in deposition of CMP particles during transport.**

Conclusion and Future Work

- **Intensity based measurements (DLS, NTA) show biased signals to larger particles.**
- **Aerosolization method is a promising technique to characterize polydisperse (or mixtures of different sizes) particles.**



- **In high pH (> 9) solution, CMP slurry particles tend to have negative zeta potentials.**
- **No deposition inside capillary tube will be expected when particles have negative zeta potentials.**

THANK YOU

Q/A