Correlation Between Aerosolization Based Metrology and Enhanced Surface Scanning

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KANOMAX FMT
A Kanomax Company
NANOPARTICLE MEASUREMENT SOLUTIONS

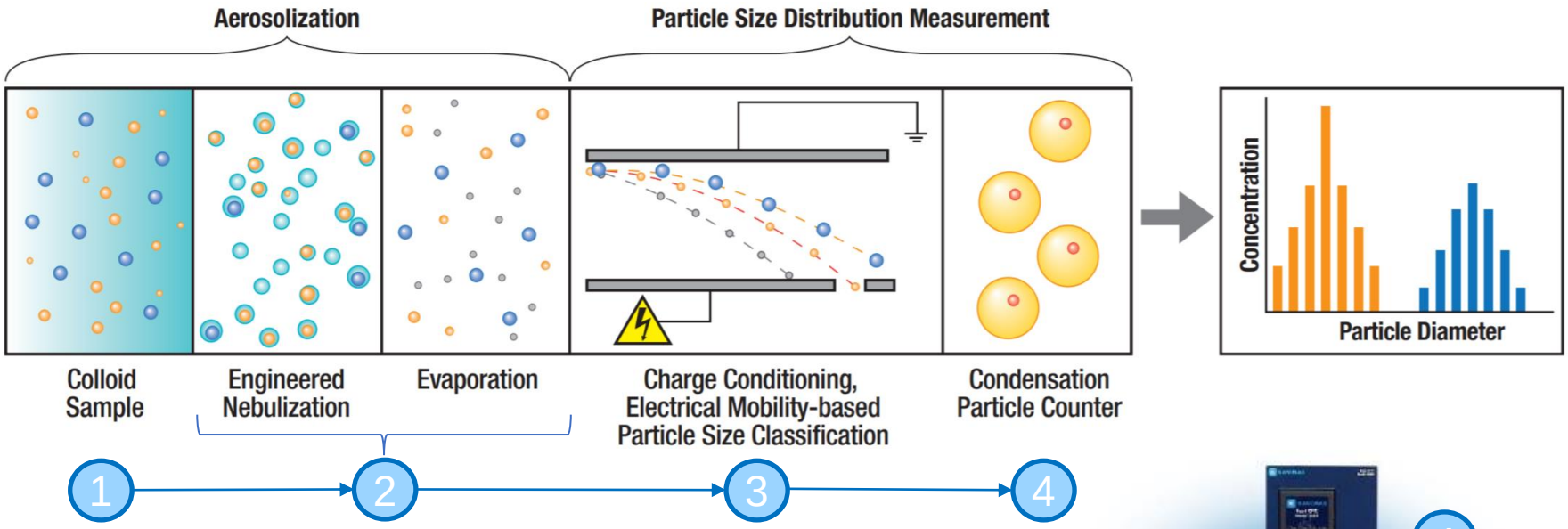
Creative Technology
CT Associates, Inc.

Objective

- Display the effectiveness of Aerosolization-Based and Surfaced Enhanced Particle Sizing technologies for quantifying the concentration of particles and particle precursors in Isopropyl Alcohol (IPA)
- Display the ability of these technologies to show variability in Semiconductor Grade IPAs supplied by different manufacturers
- Characterize the relationship of Aerosolization Based Metrology and UNISERS measured concentrations using controlled levels of added particles and particle precursors in IPA

Aerosolization + Ion Mobility Spectrometry

Operating Principle

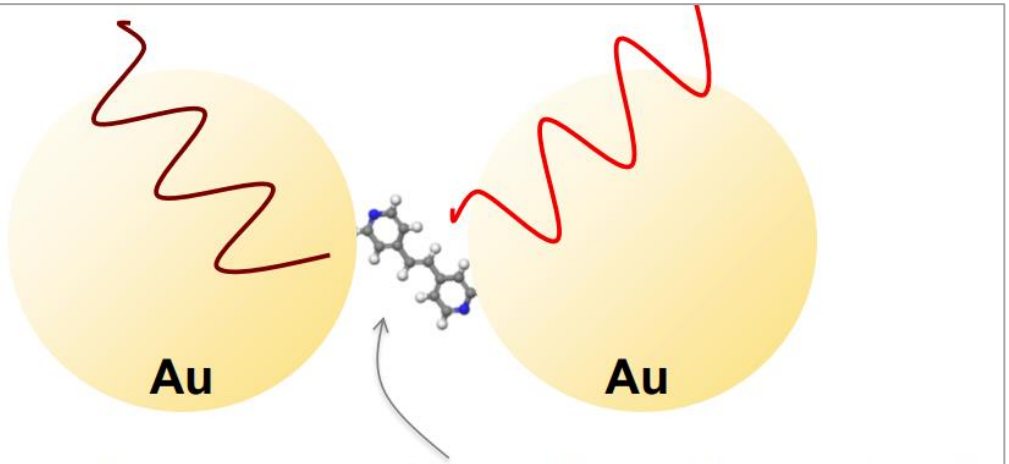


Injection ->Aerosolization -> Classification -> Counting

- **6 nm to 360 nm particles are individually measured regardless of shape or composition.**
- **64 size bins per decade are sequentially characterized.**
- **Complete concentration vs. particle size distribution in about 5 minutes.**
- **No *a priori* assumptions about the particles.**

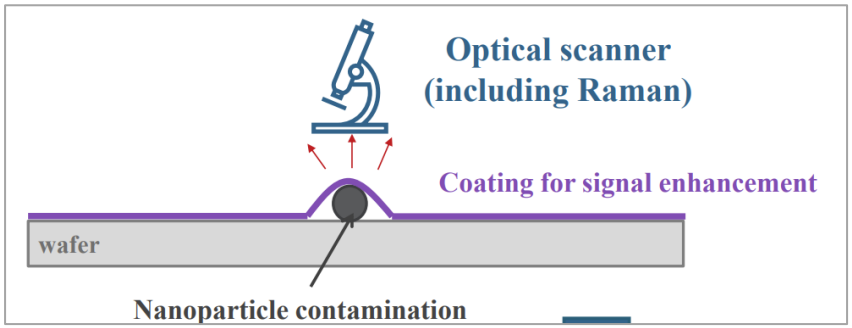
Surface Enhanced Particle Sizing (SEPS)

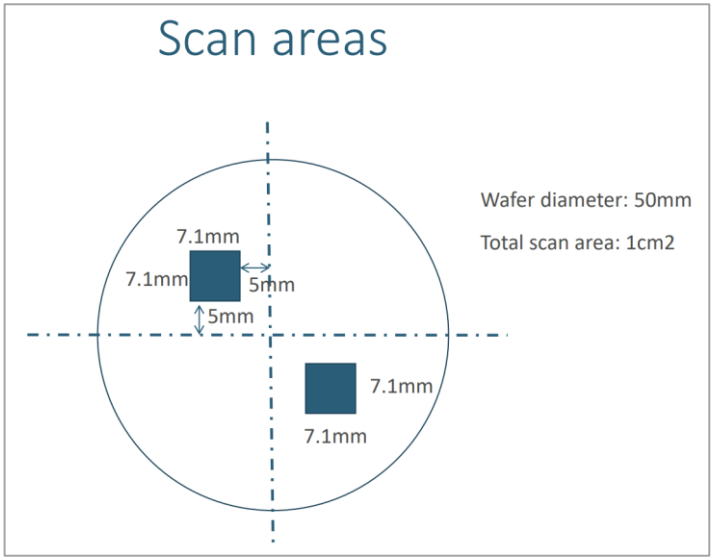
Operating Principle



Theory: Molecule resides in an electromagnetic hotspot (narrow gap btw noble metal nanoparticles). Excitation of localized surface plasmons of metal nanoparticles by incident light enhances the near field intensity. Raman signal amplification is proportional with the 4th power of the near field enhancement.

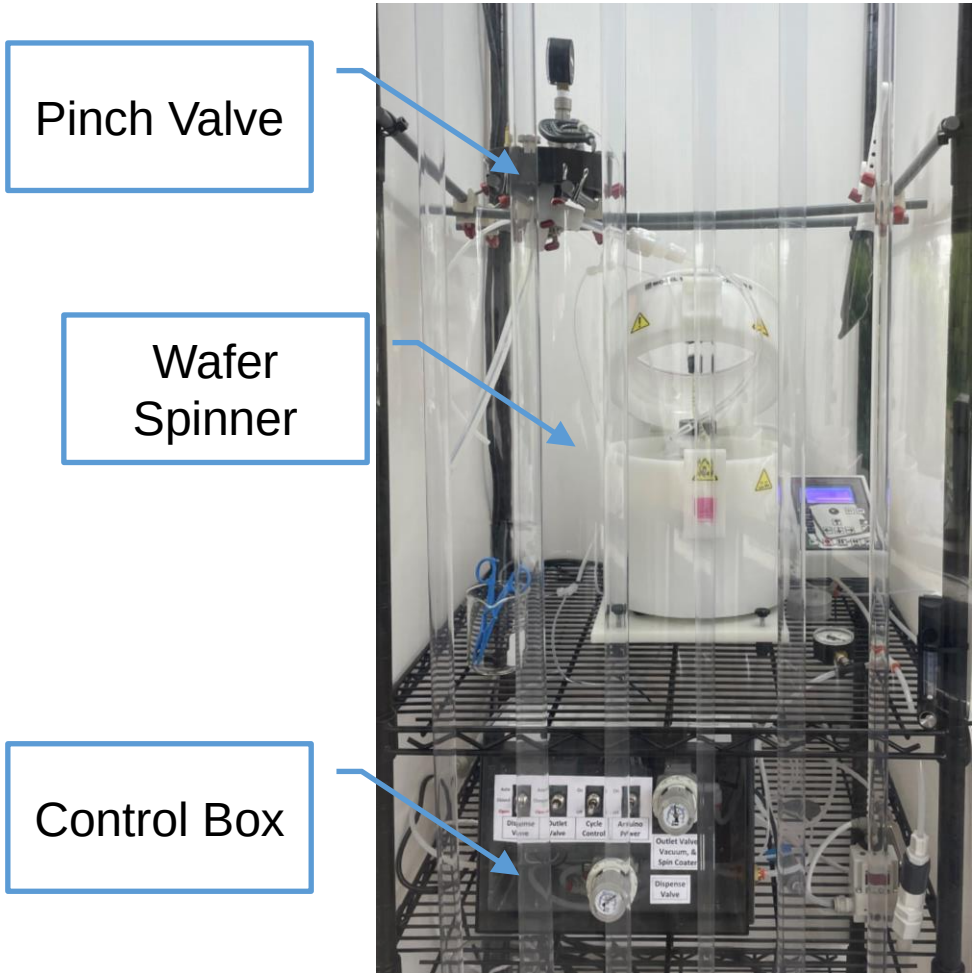
The coating creates optical resonance around the particles on the wafer, enhancing both total scattering and inelastic (Raman scattering).





Methodology cont. – Silicon wafer prep for SEPS/SERS

- Sequential Spin Coating used to deposit IPA onto wafers
- For each sample
  - IPA sample placed in pressurized vessel
  - Wafer transferred to spin coater
  - Wafer spun @ 500 RPM with drying at 2,500 RPM and 8,000 RPM.
  - 10-300 drops of the sample liquid applied to the center of the wafer every 18 seconds using an automated pinch valve
  - After wafer is visibly dry, remove and transfer to carrier

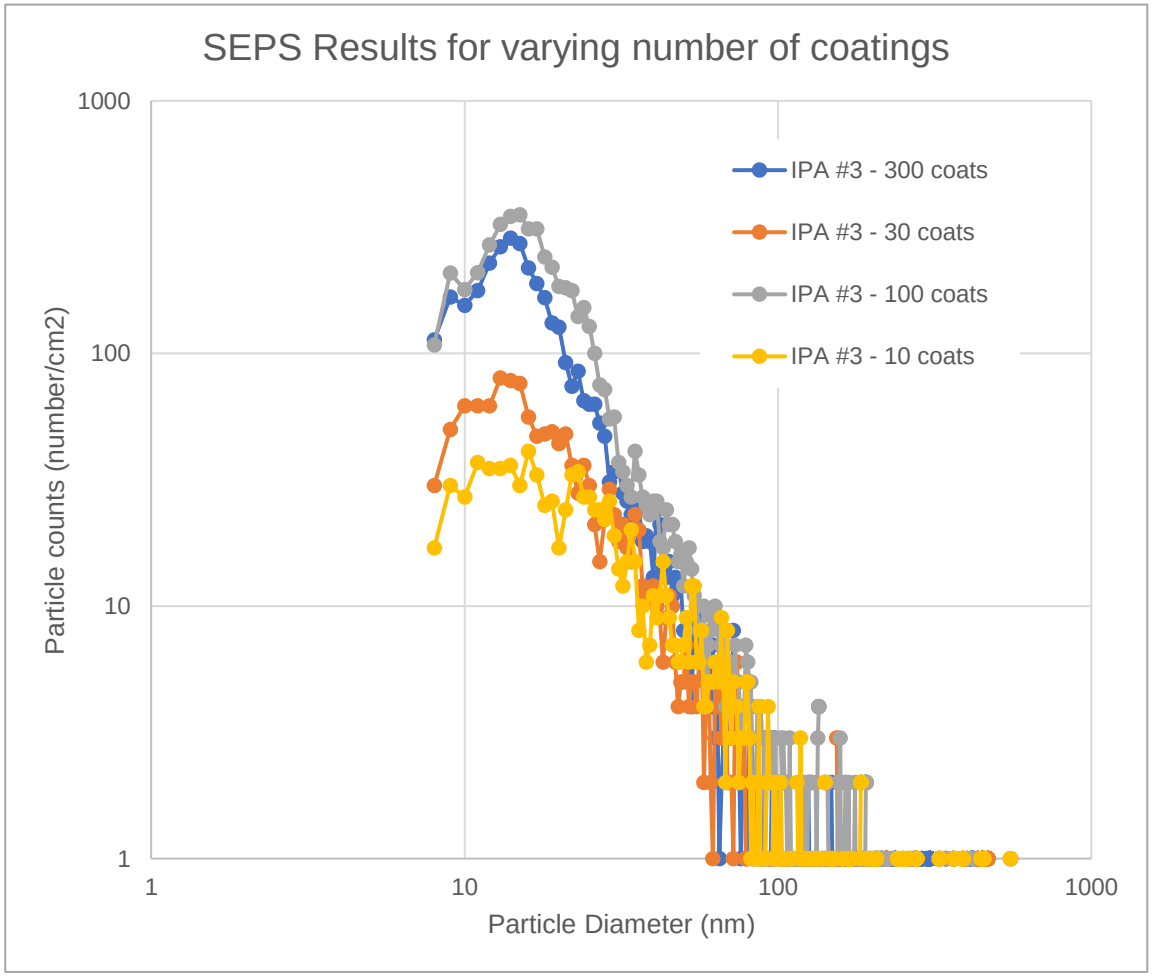


Experimental plan

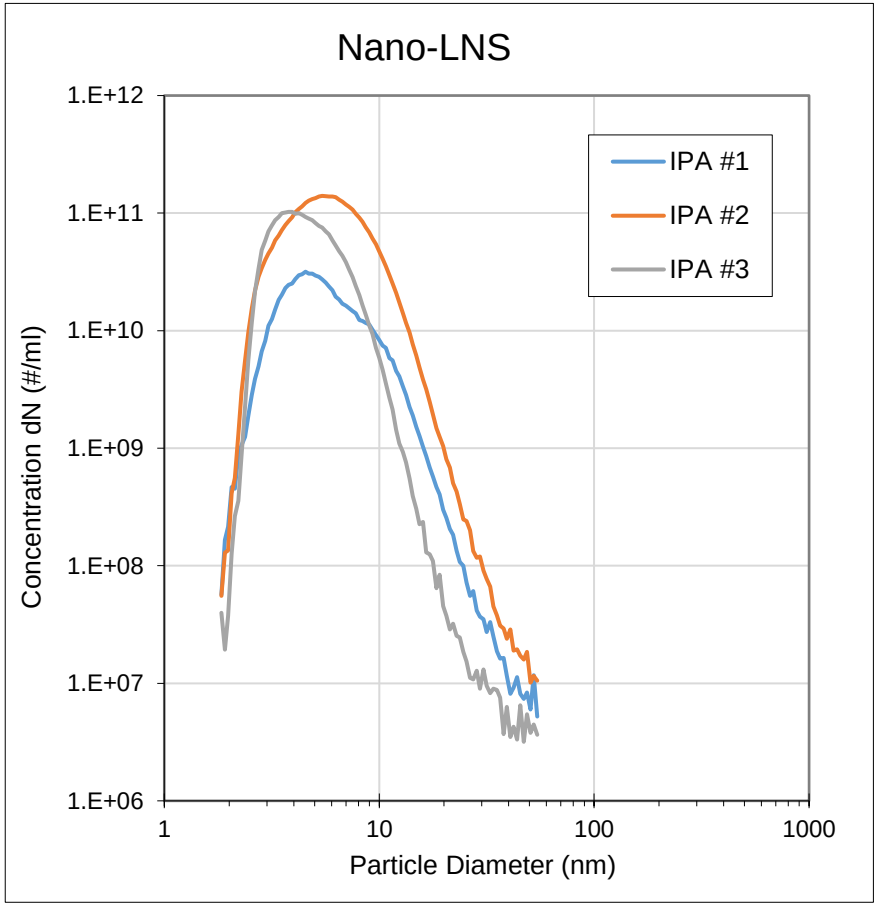
- Prepare all samples in high purity 125ml Nalgene FEP bottles
- Determine optimal number of drops to apply to wafer
- Measure the particle size distributions from three different IPA suppliers using Aerosolization + IMS (Kanomax FMT nano-AFIMS)
  - Pressurized sample chamber
  - KFMT nanoparticle nebulizer (direct injection)
  - Membrane dryer
  - KFMT nano-Annular Flow Ion Mobility Classifier (AFIMC)
  - KFMT FastCPC (boosted for lower detection limit)
- Identify the highest quality IPA to use for spiked samples
- Prepare the following samples:
  - Pure IPA from three vendors
  - IPA spiked with an organic acid at 0.1 and 1.0 mM
  - IPA spiked with colloidal silica at 1E8 and 1E9 #/ml
- Coat 2” diameter wafers using sequential spin coating and analyze using Surface Enhanced Particle Sizing (SEPS)
- Measure particle size distributions of IPA samples using Aerosolization + Ion Mobility Spectrometry

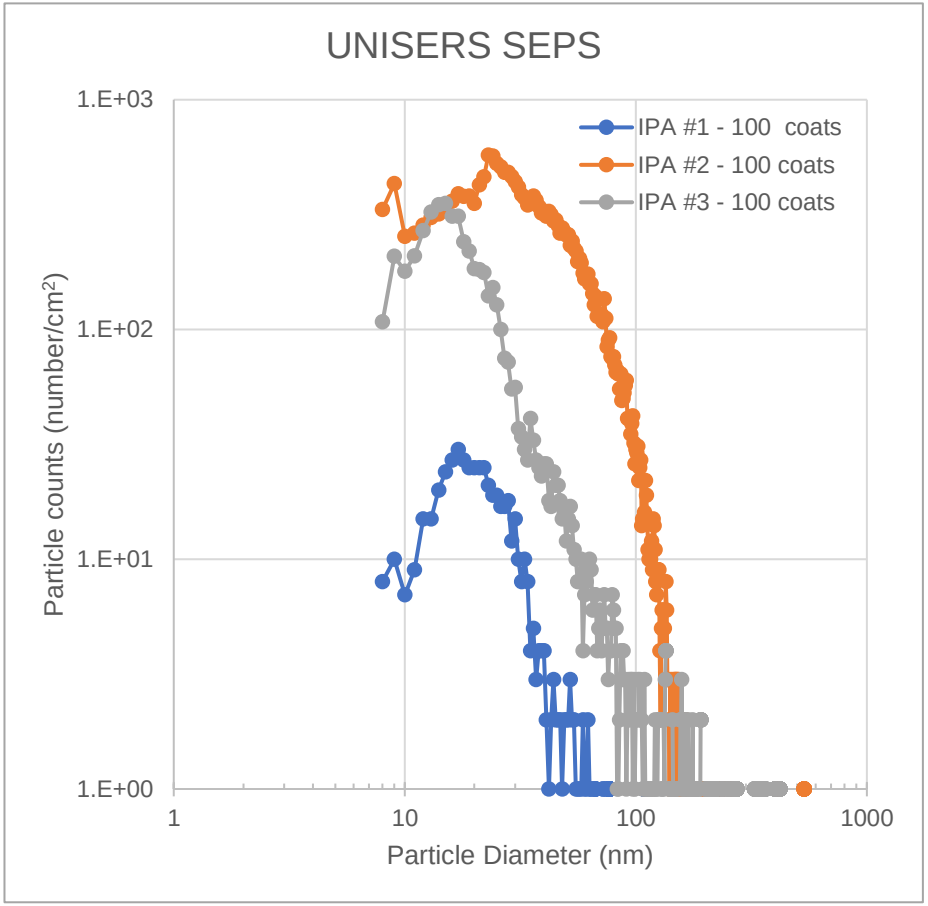
Results – Successive Spin Drying Optimization

- Wafers prepared at four different number of applied coats of the same IPA grade
- Total deposited particles showed little change above 100 coats

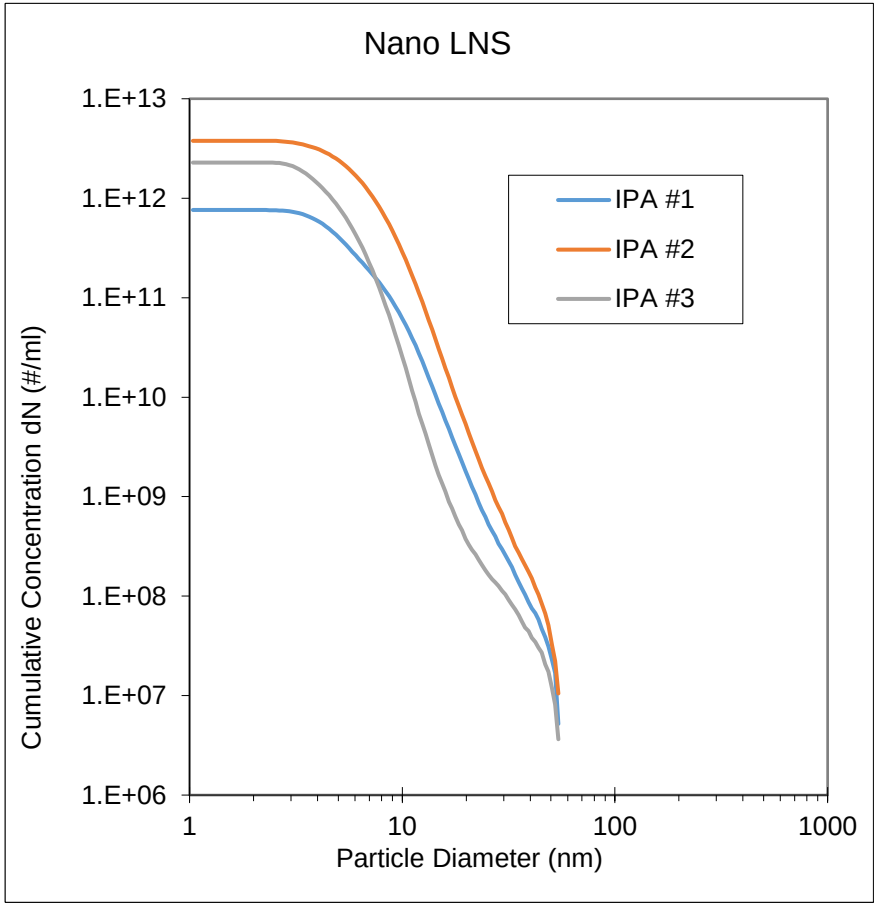


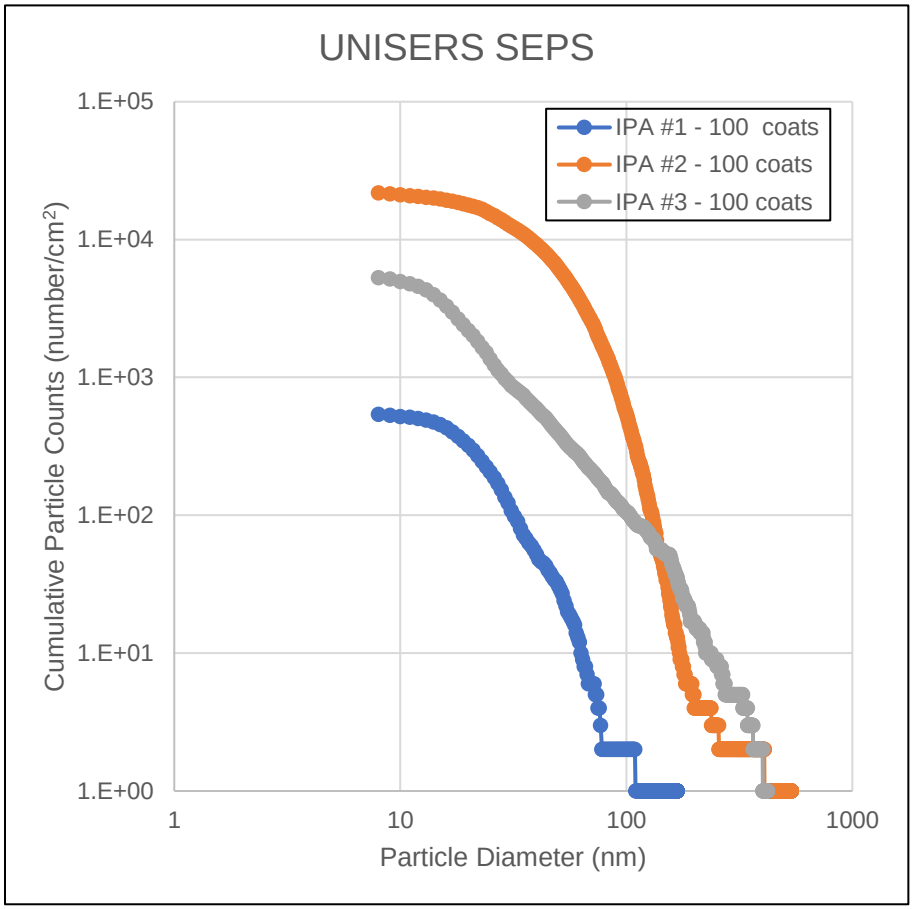
Results – Comparison of three IPA Grades





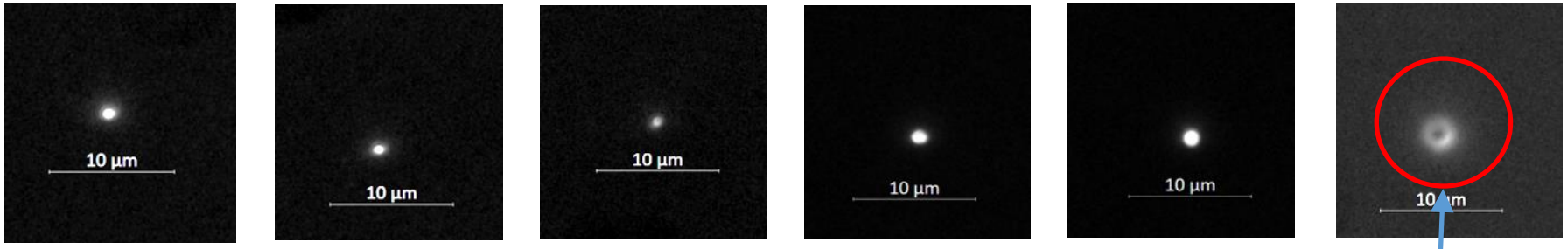
Results – Comparison of three IPA Grades



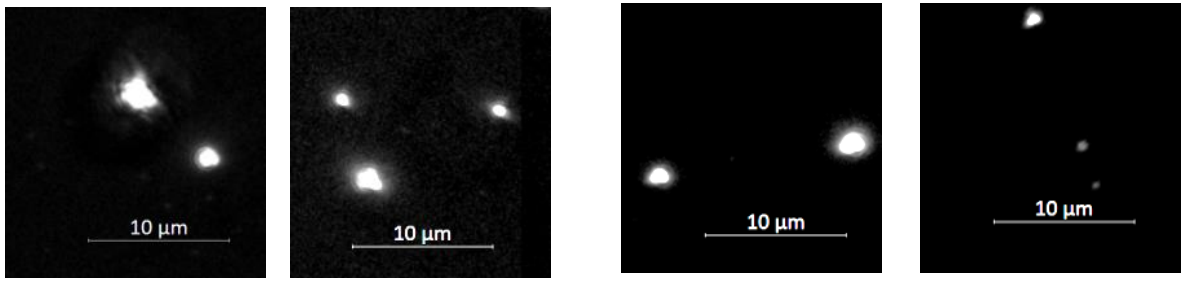


Results – comparison of three IPA grades (Images)

IPA #1 – 100 coats

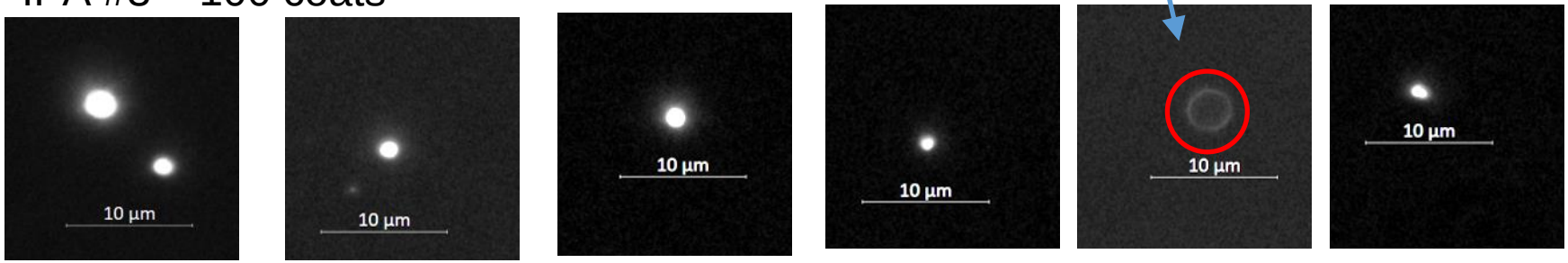


IPA #2 – 100 coats



Ring formation

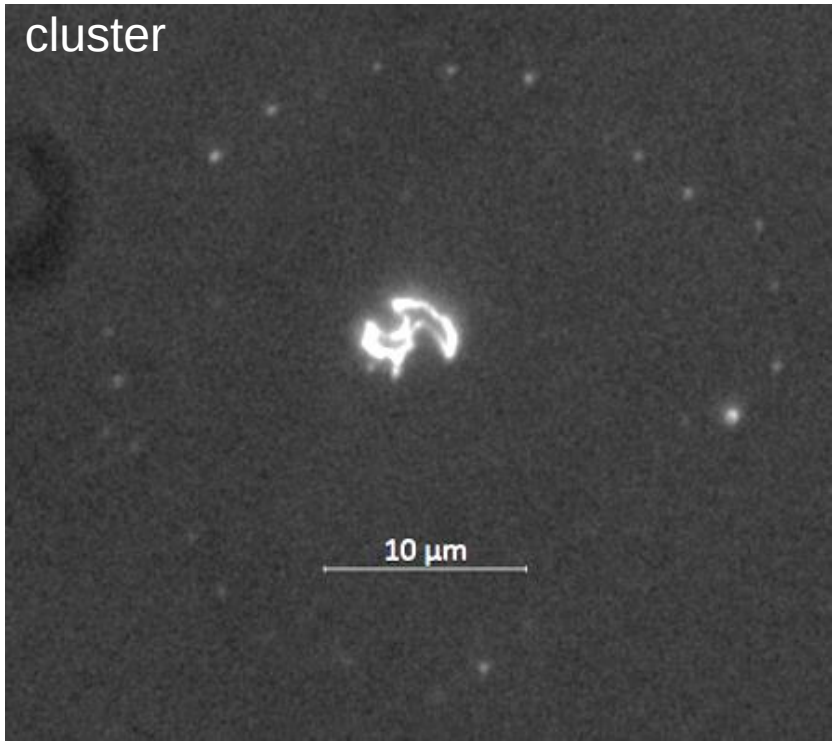
IPA #3 – 100 coats



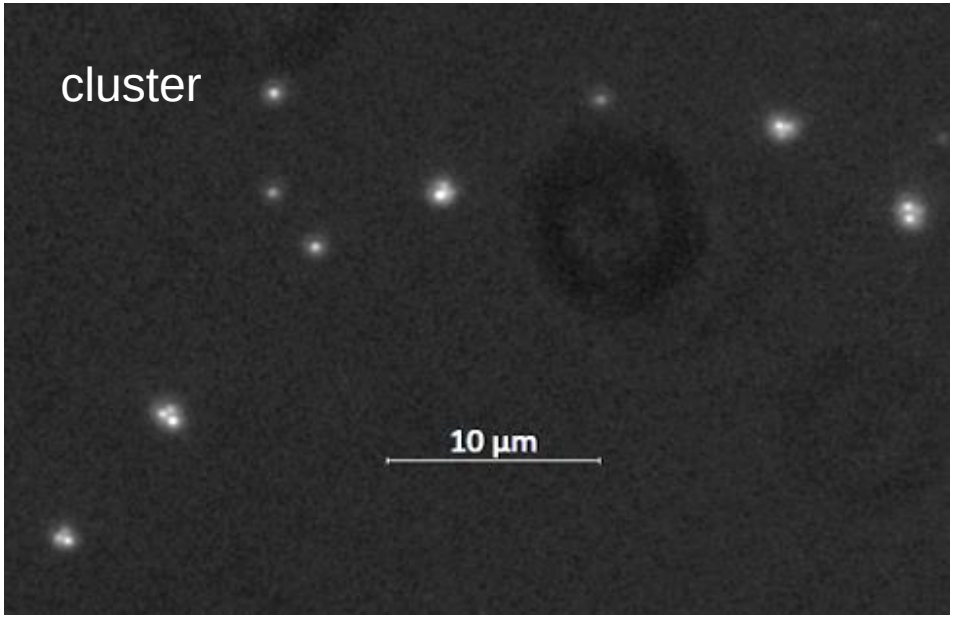
Note: Scale bars do not represent particle size. Particle diameters are 8-100 nm

Results – comparison of three IPA grades (Images)

IPA #2 – 100 coats



IPA #3 – 100 coats



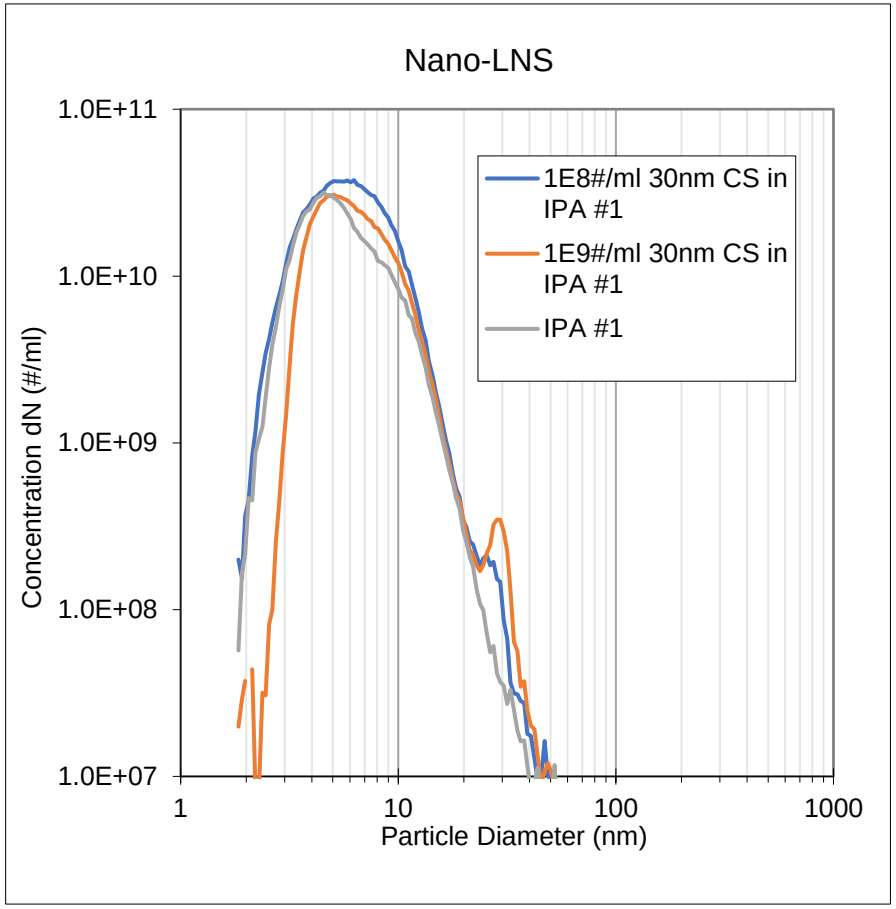
Note: Scale bars do not represent particle size. Particle diameters are 8-100 nm

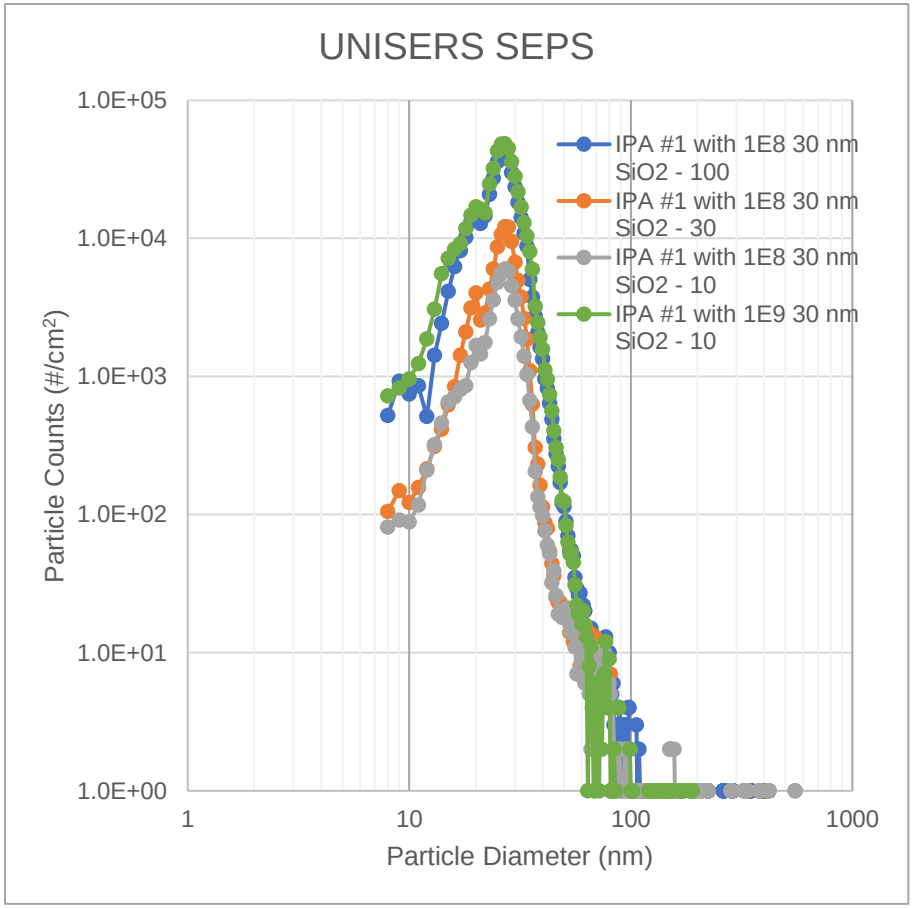
Discussion – comparison of three IPA grades

Sample	Nano-LNS > 8nm (#/ml)	Net Conc from SEPS > 8nm (#/cm^2)	Deposition Factor (#/cm^2)/(#/ml)
IPA #1	1.30E+11	411.5	3.2E-09
IPA #2	7.20E+11	21680.5	3.0E-08
IPA #3	1.00E+11	5148.5	5.1E-08

- Deposition factor for IPA #1 significantly lower than other IPA samples
- Nano LNS showed higher concentration for IPA #1 versus IPA #3 above 8nm; however, IPA #1 showed a significantly lower concentration across the entire Nano LNS scan range.

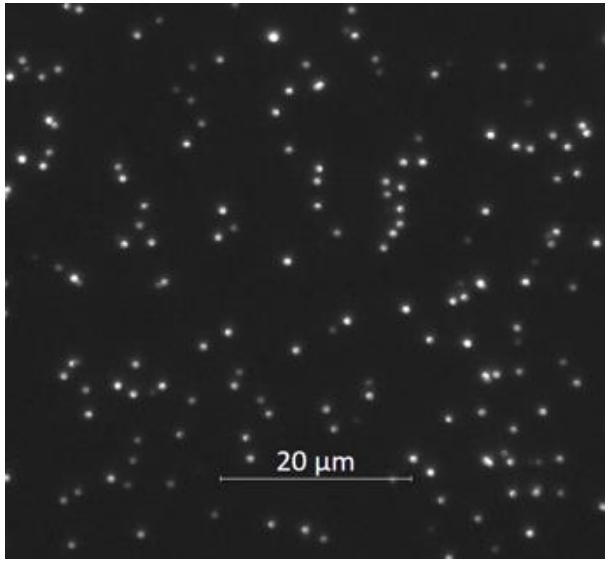
Results – IPA spiked with 30nm Colloidal Silica





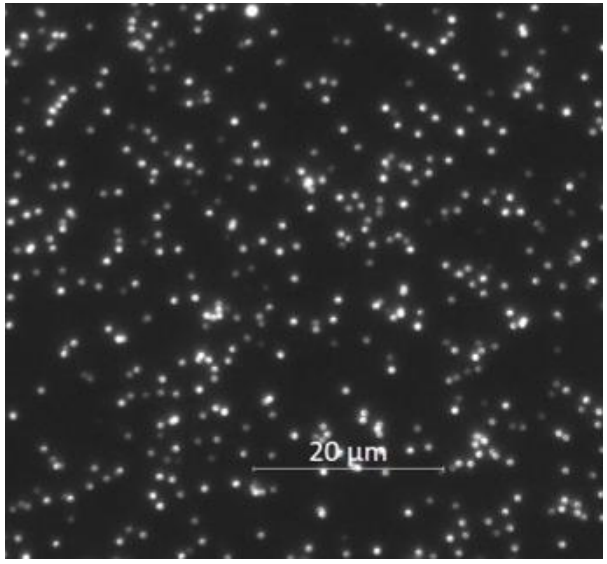
Results – IPA spiked with Colloidal Silica (images)

1E9 30nm SiO2, 10x coats



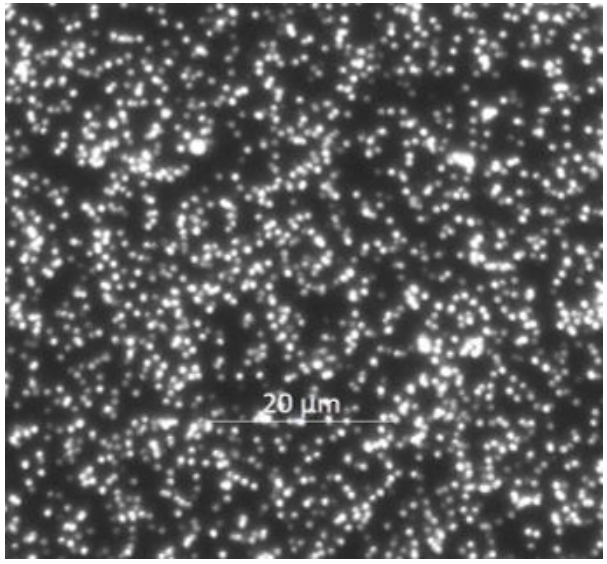
5E6 particles/cm^2

1E9 30nm SiO2, 30x coats



Overloaded

1E9 30nm SiO2, 100x coats



Overloaded

Note: Scale bars do not represent particle size. Particle diameters are 8-100 nm

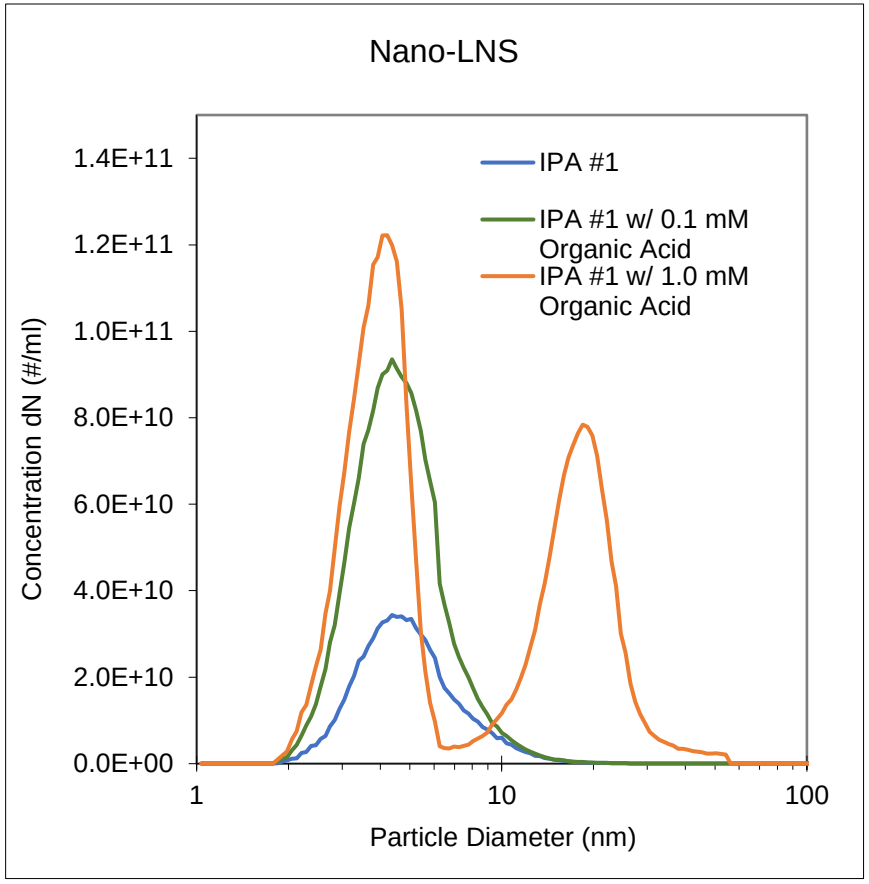
Discussion – IPA Spiked with 30nm Colloidal Silica

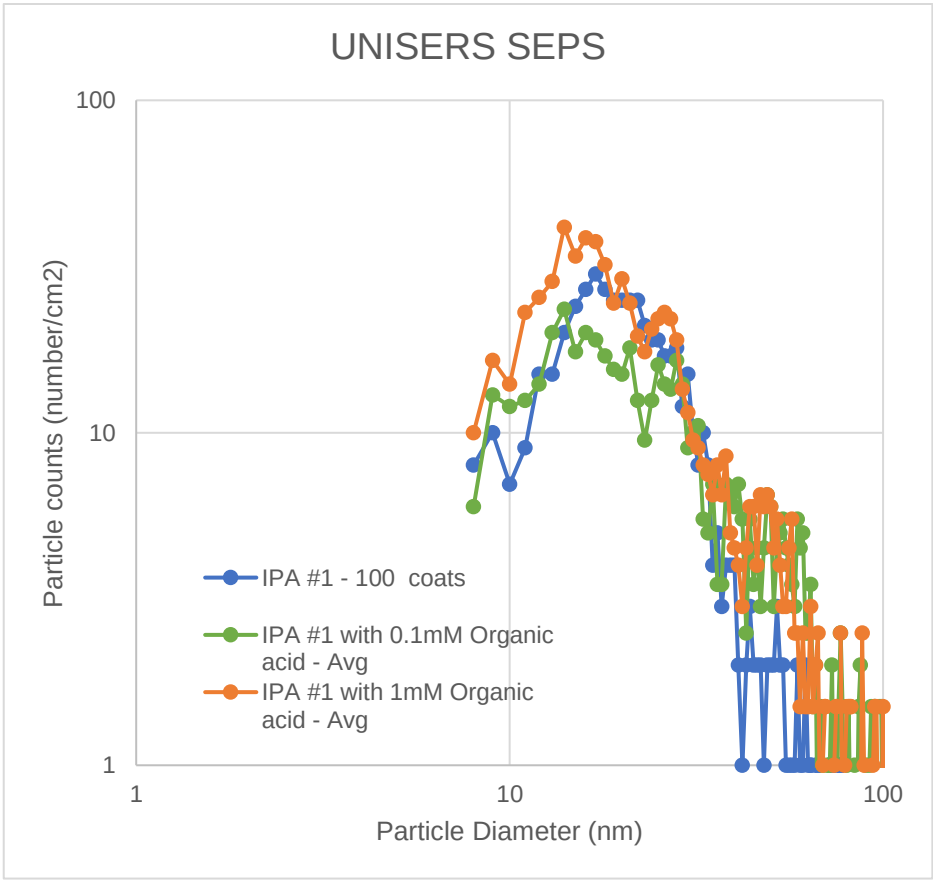
Sample	Nano-LNS > 8nm (#/ml)	Net Conc from SEPS >8nm (#/cm^2)	Deposition Factor (#/cm^2)/(#/ml)
1E8 CS	2.40E+11	420502.5	1.8E-06
1E9 CS	1.78E+11	508471.5	2.9E-06

Used value from 10 coats for 1E9 CS due to overloading

- Deposition factor significantly higher for CS compared to clean IPA
- Nano LNS had significant interference from precursor material in the stock colloidal silica
- Deposition observed by SEPS appears to correlate with the product of the number of coats and the concentration.

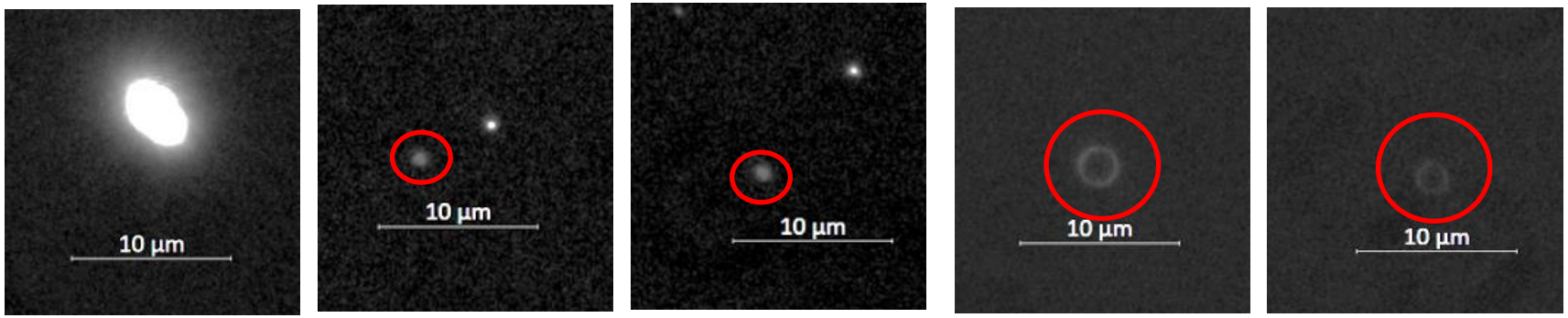
Results – IPA spiked with Organic Acid



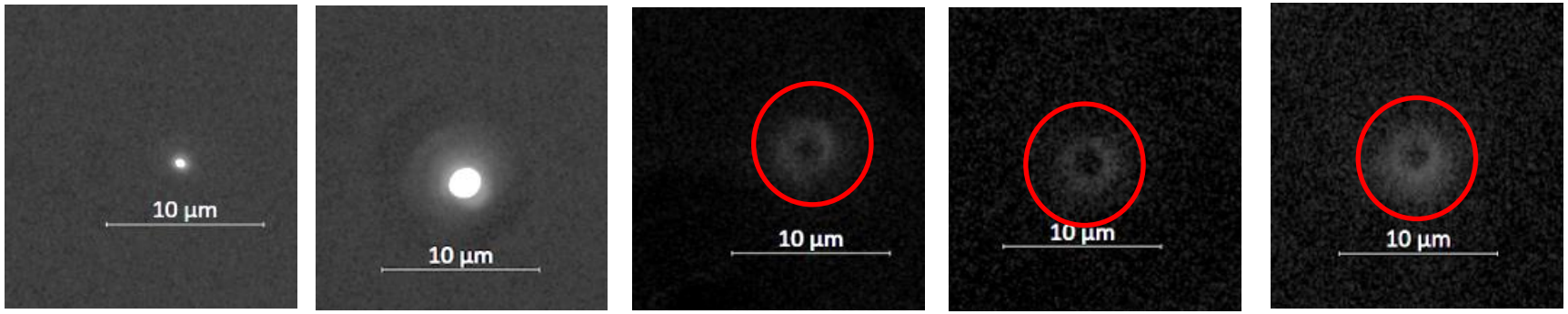


Results – IPA spiked with Organic Acid

1mM Organic acid



0.1mM Organic acid



Organic acid showed additional “drying rings”

Note: Scale bars do not represent particle size. Particle diameters are 8-100 nm

Discussion – IPA Spiked with Organic Acid

Sample	Nano-LNS > 8nm (#/ml)	Net Conc from SEPS > 8nm (#/cm^2)	Deposition Factor (#/cm^2)/(#/ml)
0.1 mM Organic Acid	1.21E+11	443	3.7E-09
1 mM Organic Acid	1.41E+12	651.5	4.6E-10

- Nano-LNS showed formation of a second mode at higher OA concentration. Possibly due to the formation of micelles.
- SEPS did not show a significant difference between the two concentrations.
- Possible causes of the difference with the SEPS measurement
  - Change in the OA concentration during deposition leading to the breakup of micelles or other types of meta-stable agglomerates
  - Collapse of micelle leading to a large, faint ring that isn't counted as a particle

Takeaway

- Impurities in IPA samples may have different deposition factors onto wafers
- Particle and Particle Precursor concentrations may not correlate for some IPA samples
- Organic acid appears to form an additional mode above a threshold concentration.
- Organic acid leads to a high number of “drying rings”

Follow on work

- Repeat study using additional metrologies
  - –Optical particle detectors
  - –Aerosolization + Threshold Condensation Particle Counting
- Capture Surface Enhanced Raman Spectroscopy (SERS) data
  - –Was not possible for this presentation due to a laser failure
- Correct data for detection efficiency of SEPS and transmission efficiency for nano-LNS

Thank you!

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Supplemental Materials

Nano-LNS detailed schematic

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Liquid Nanoparticle Sizer (LNS) System

System Schematic: Nebulizer -> AFIMC -> Fast CPC

- 6 nm to 360 nm particles are individually measured regardless of shape or composition.
- 64 size bins per decade are sequentially characterized.
- Complete concentration vs. particle size distribution in about 5 minutes.
- No *a priori* assumptions about the particles.