THE USE OF FOCUSED AEROSOL DEPOSITION (FAD) TO CAPTURE, IDENTIFY AND QUANTIFY KILLER DEFECT PARTICLES IN UPW

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KANOMAX FMT
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INTRODUCTION

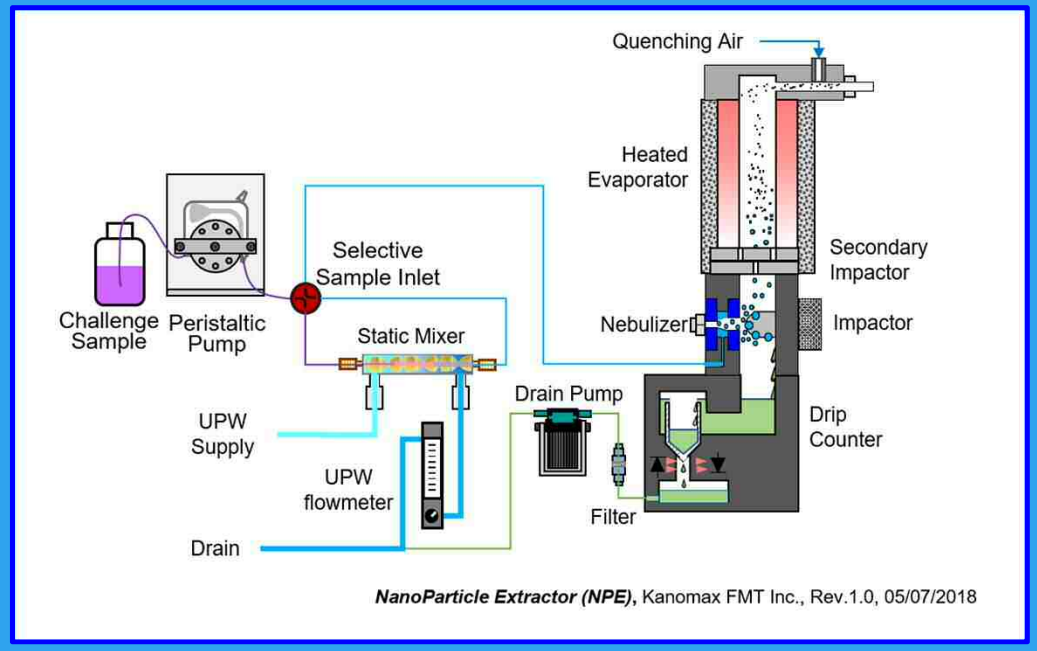
- DETECTING, COLLECTING AND IDENTIFYING "KILLER" PARTICLES (< 20 NM) IN HIGH-PURITY LIQUIDS IS VERY CHALLENGING AND TIME CONSUMING.
- DEVICE GEOMETRIES AND FEATURE SIZES WILL CONTINUE TO PRESS THESE SIZES TOWARD 5 NM IN THE COMING YEARS.
- TRADITIONAL FILTER-BASED CAPTURE TECHNIQUES HAVE BOTH PORE-SIZE AND FLOWRATE LIMITATIONS, AND MAY TAKE WEEKS FOR ONE SAMPLE.
- NEW, FASTER METHODS FOR EFFICIENTLY COLLECTING PARTICLES AS SMALL AS 5 NM ARE NEEDED.
- THIS PRESENTATION WILL REVIEW THE ADVANCEMENTS MADE IN FOCUSED AEROSOL DEPOSITION AS A TOOL FOR CAPTURING LIQUID-BORNE NANOPARTICLES FOR OFF-LINE SIZING AND COMPOSITIONAL ANALYSIS BY ELECTRON MICROSCOPY (SEM AND TEM) AND ENERGY DISPERSIVE X-RAY (EDX)

PRESENTATION OUTLINE

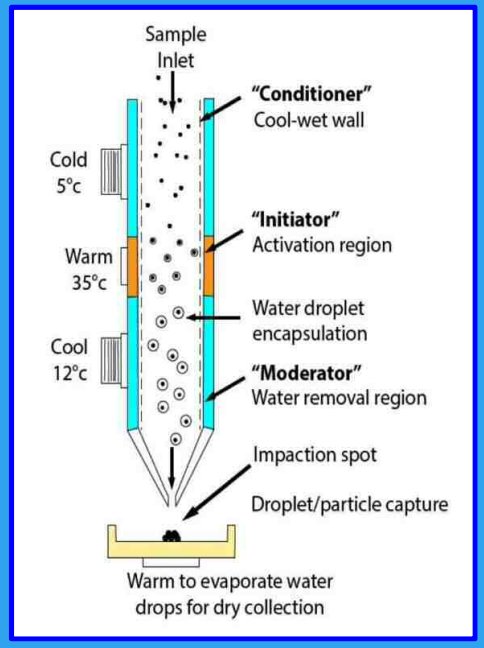
- FOCUSED AEROSOL DEPOSITION (FAD) THEORY OF OPERATION
- FAD TECHNOLOGY DEVELOPMENTS AND CAPABILITIES
- TEST RESULTS AND POTENTIAL APPLICATIONS
- NEXT STEPS
- SUMMARY

THEORY OF OPERATION – FAD[3]

Ultrafine Aerosolization[1]



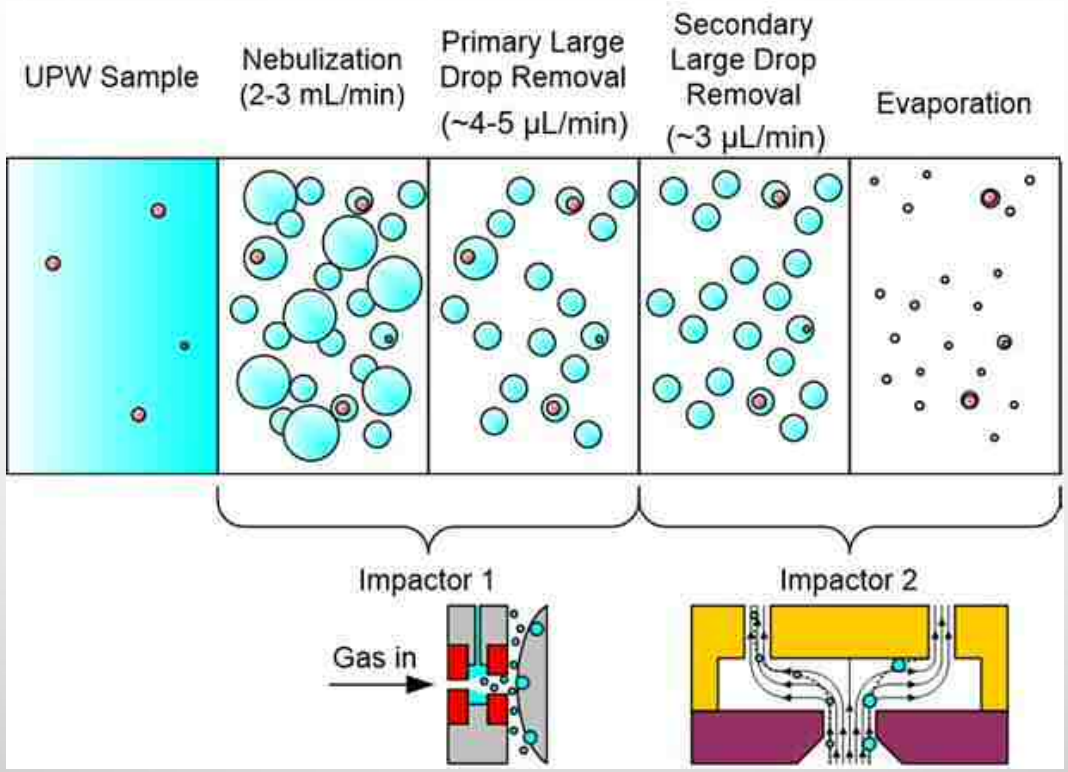
Nanoparticle Collection[2]



[1] Kanomax FMT NanoParticle Extractor
[2] Kanomax FMT NanoParticle Collector
[3] Kanomax FMT NanoSpotLight™

Van Schooneveld, et al., Focused Aerosol Deposition, Ultrapure Micro 2018, Austin, Texas

THEORY OF OPERATION – NANOPARTICLE EXTRACTOR



Large droplet removal limits the formation of large precipitated non-volatile residue (PNVR) particles.

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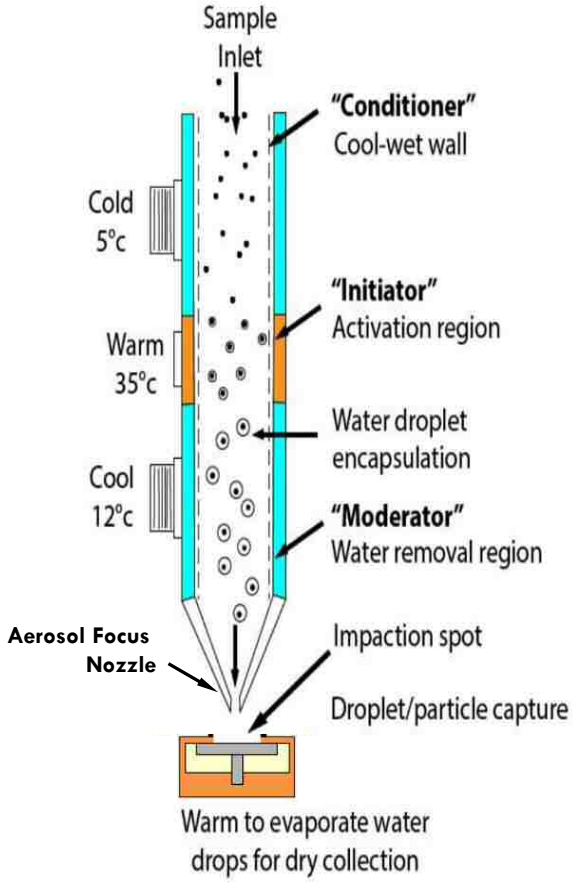
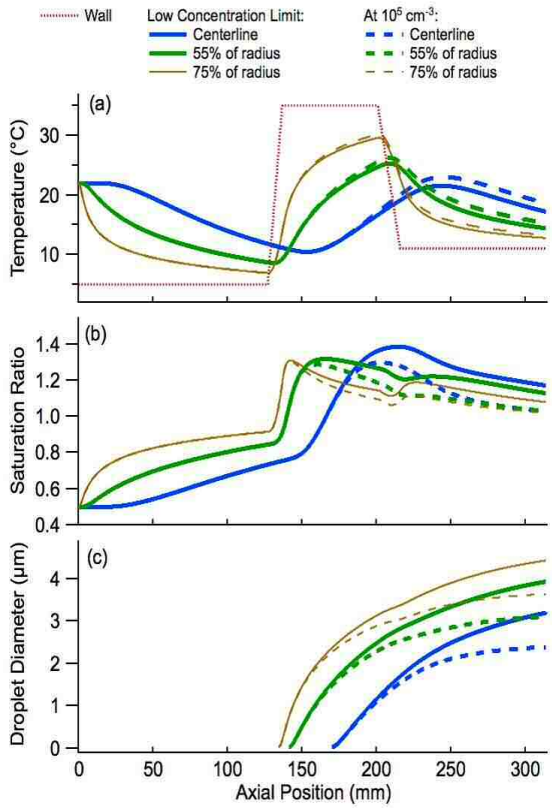
THEORY OF OPERATION – NANOPARTICLE COLLECTOR





Moderate sample flow temperatures never exceed 30 ˚C. Exit flow temperature <18 ˚C; dewpoint < 20 ˚C.

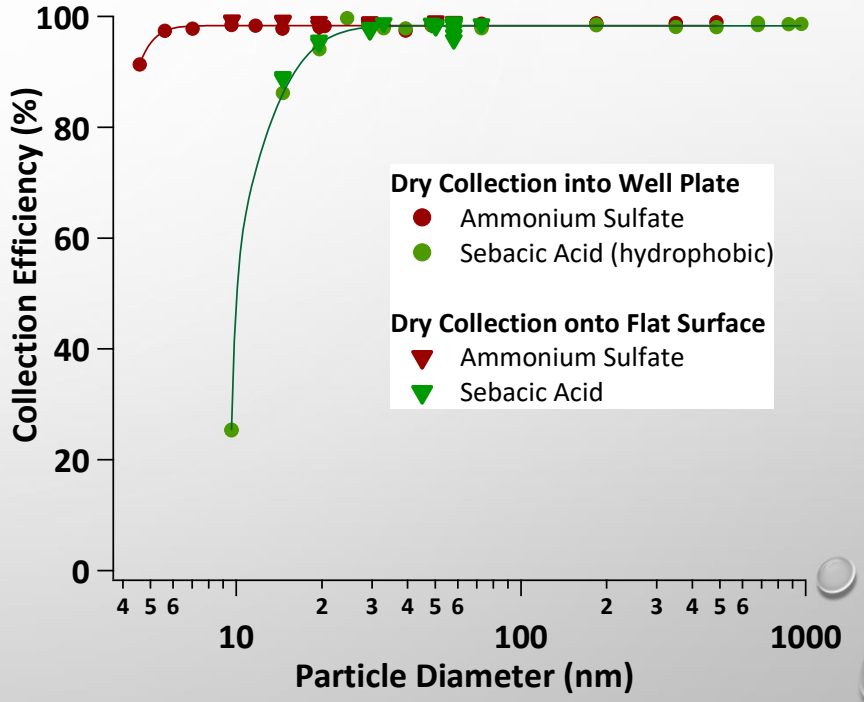
Supersaturation levels of 120-140% activate condensation growth on particles as small as 5 nm.

Droplets grown to nominal 3µm diameter are easily captured by bounce-free, soft inertial impaction.

ACTIVATION EFFICIENCY AND SIZE IMPLICATIONS

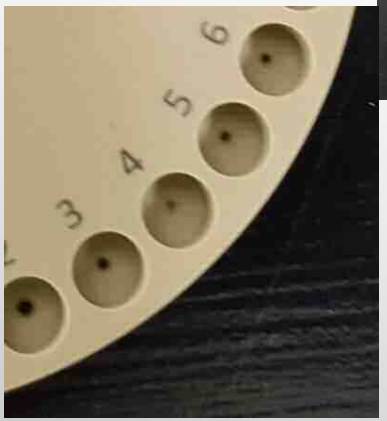
- **Material Effects:**
  - Hydrophilic vs. hydrophobic particles
- **Condensation Growth Tube:**
  - Temperature, flowrate, bore size, gas
- **Aerosol Concentration :**

Parameter	< 1E4/cc	> 1E4/cc
Minimun activation size	Smaller (5nm)	Larger
Droplet size	Larger (3µm)	Smaller
Collection efficiency	Higher (> 98%)	Lower
Media heating requirements	Lower (<40°C)	Higher



FAD Improvements

ORIGINAL – deposition ~ 1mm diameter "spot" deposits into multi-well PEEK sample plates



NOW - focused deposition in the center of test silica wafers attached to 1" SEM stub on a 5-stub heated sample platen

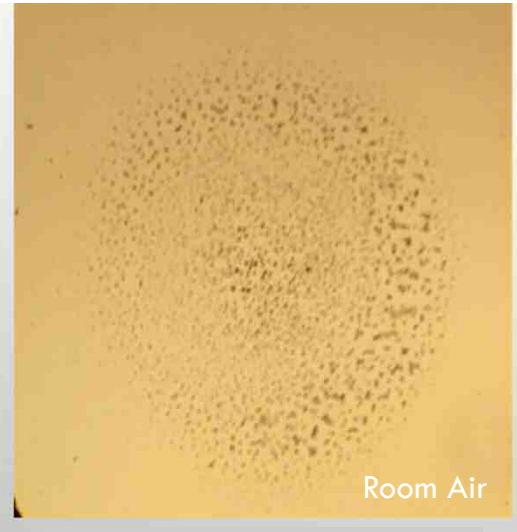
- **Increased aerosol particle concentration to collector 5- 10X.**
- **Tighten aerosol focus nozzle.**
- **Reduced focus distance between nozzle and media.**
- **Optimized aerosol flow parameters.**
- **Collection focused directly on SEM ready stub.**

Collection of atmospheric particles for visual illustration

FAD Improvements – Spot Size Reduction

Typical deposit spot - 2017

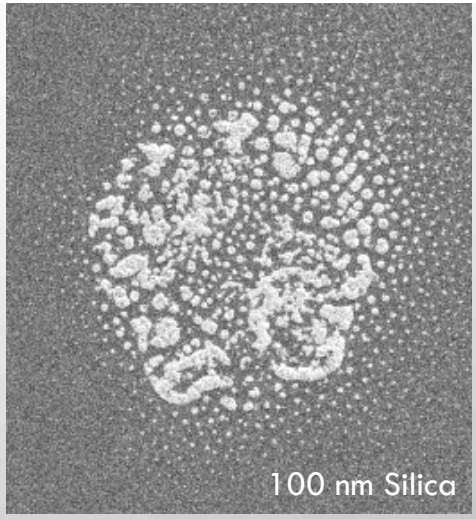
Spot pattern at 50 X magnification



1 mm

Typical FAD deposit - 2018

Spot pattern at 50 X magnification



0.1 mm

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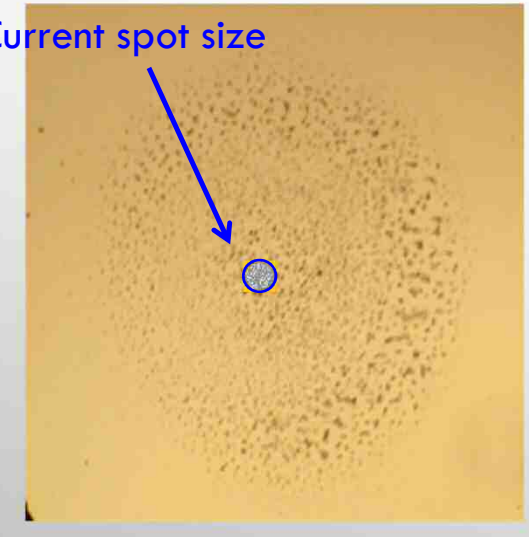
Spot Size Reduction Implications

99% reduction in spot size with a 5 to 10X increase in deposition rate.



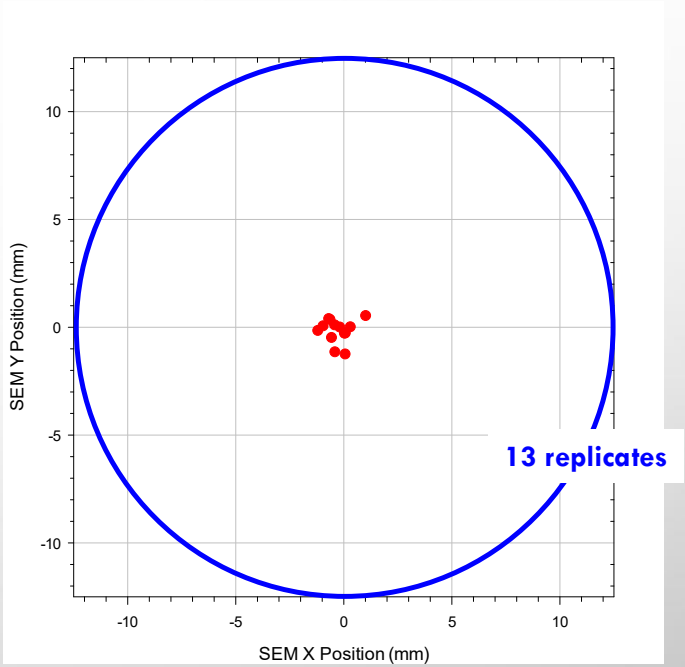
	Minimum Particle Size (nm)	Inspection Area (mm^2)	Typical flow rate (mL/min)	Depostion Rate (mL/mm2)	Relative Sampling Time (to FAD)
FAD	5	0.01	0.003	0.3	1
Al_2O_3 Filter	20*	346	37.3	0.11	2.8
Track Etch Filter	50	415	10.0	0.02	12.5

** 10 nm Al_2O_3 have been demonstrated but are not commercially available.*

FAD Improvements – Direct Deposition on SEM-Ready Stubs

Two Deposition Media:

- 25mm Silicon Wafer
- 25mm Polycarbonate Track Etch



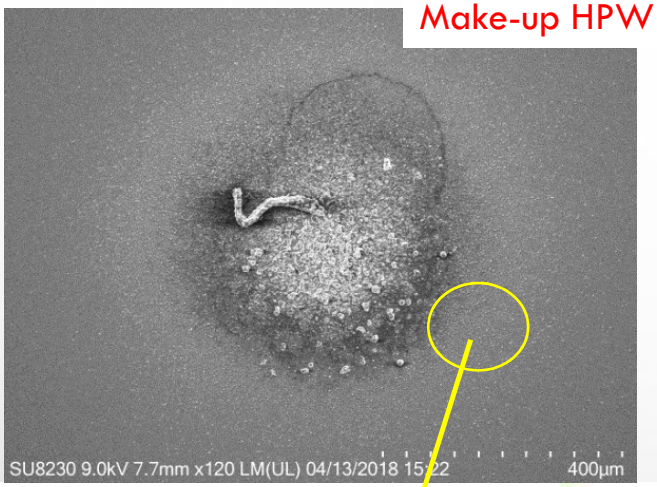
Depositing on SEM-ready stub with alignment pin provides the ability to rapidly locate spot and begin SEM/EDX analysis.

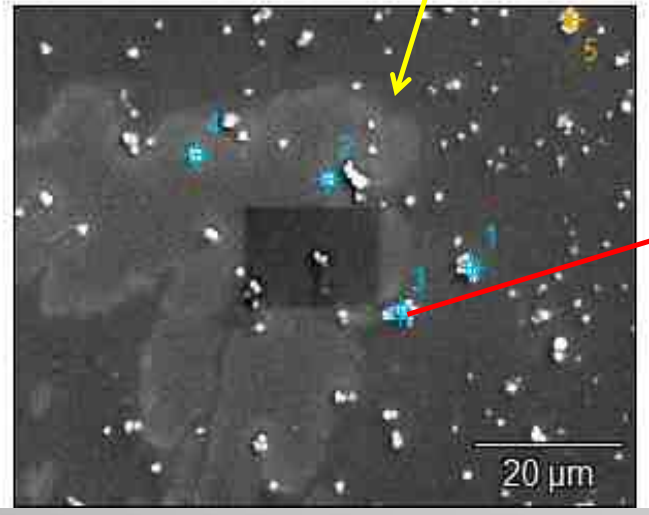
POTENTIAL APPLICATIONS

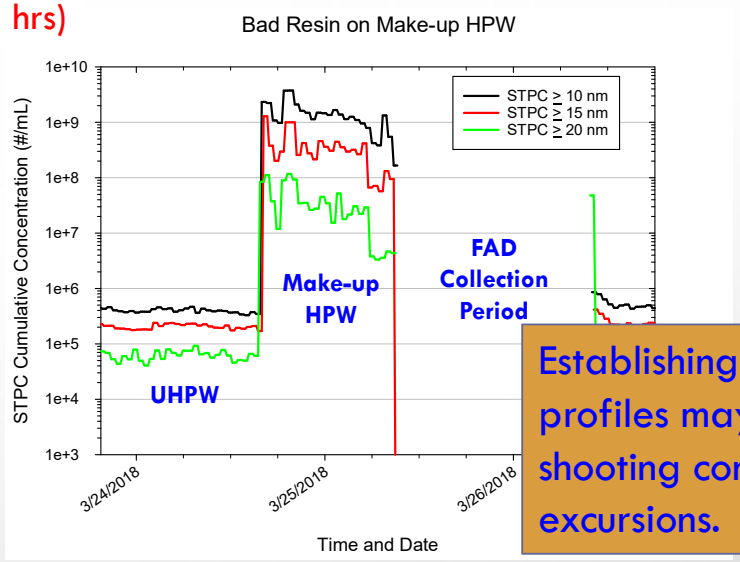
- UPW SYSTEM CONTAMINATION MAPPING
- FILTER RETENTION TESTING
- COMPONENT CONTAMINATION PROFILING
  - FILTER (MF AND UF) SHEDDING
  - IX RESIN RELEASE
  - MECHANICAL COMPONENTS (VALVES, REGULATOR, TUBING, ETC.)
  - MEMBRANE CONTACTORS
- PARTICLE MEASURING INSTRUMENT TO PARTICLE CORRELATIONS

UPW System Contamination Mapping

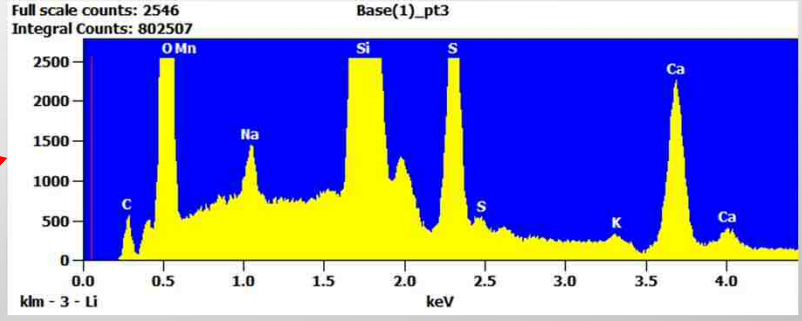
Make-up HPW (25 hrs)







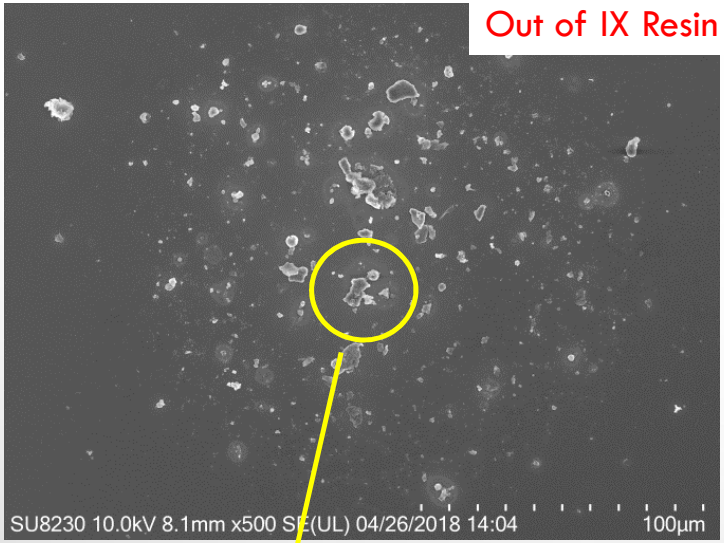
Establishing contamination profiles may assist in trouble shooting contamination excursions.

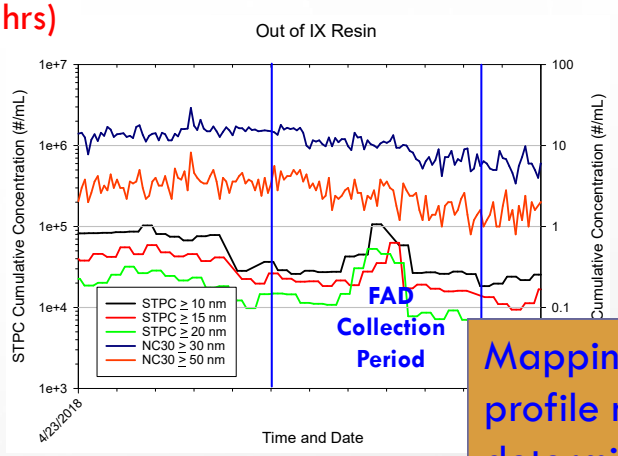


* STPC - Scanning Threshold Particle Counter (KFMT)

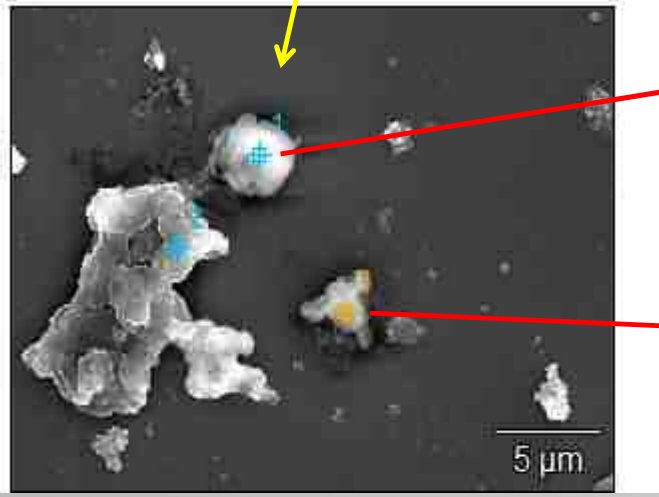
UPW System Contamination Mapping

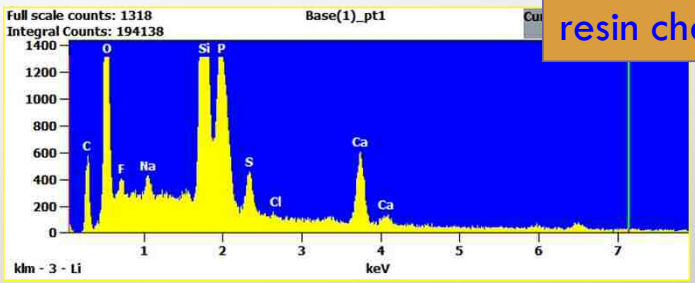
Out of IX Resin (11.5 hrs)

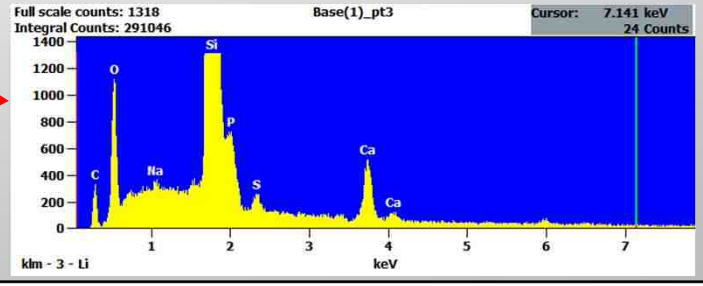




Mapping IX release profile might assist in determining timing for resin changes.

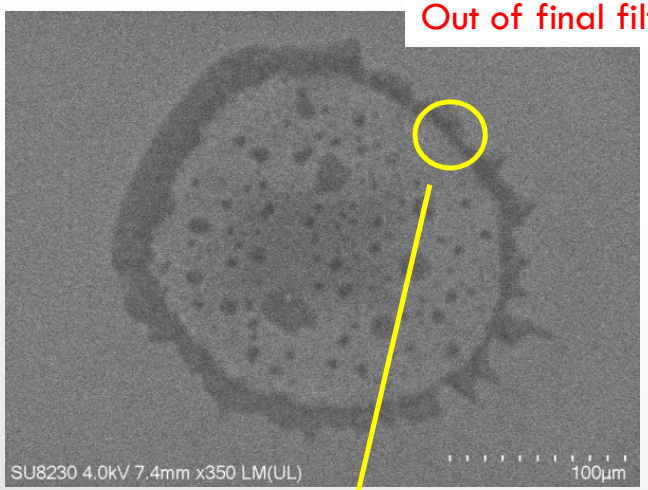


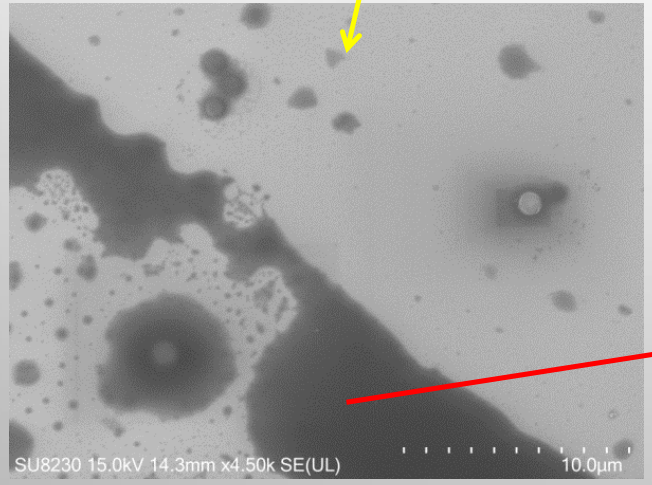


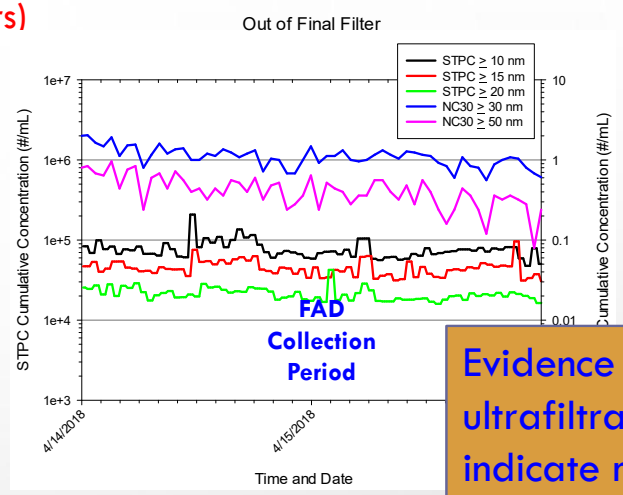


UPW System Contamination Mapping

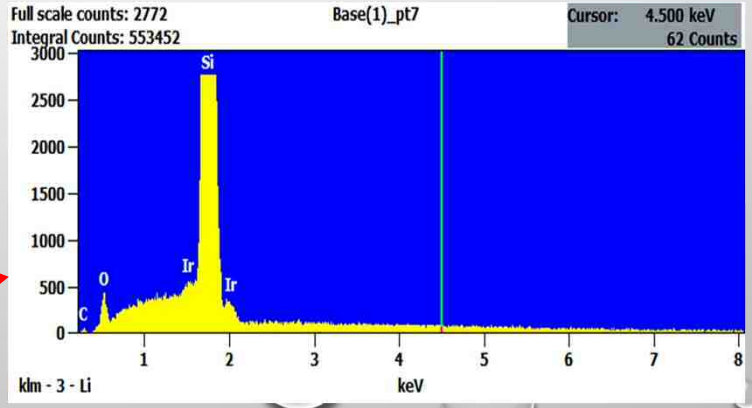
Out of final filter (37 hrs)



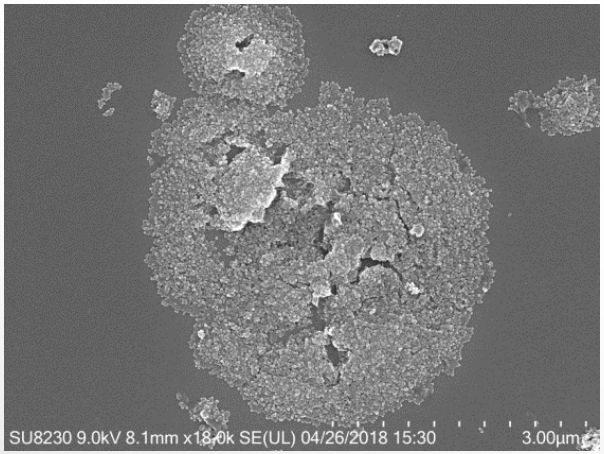


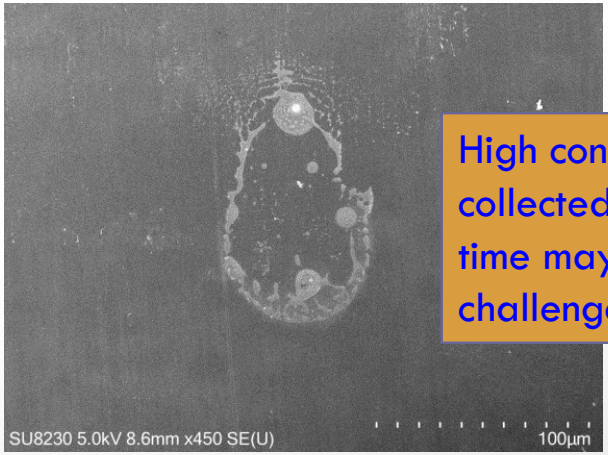


Evidence of micro and ultrafiltration media may indicate media compromise or deterioration.

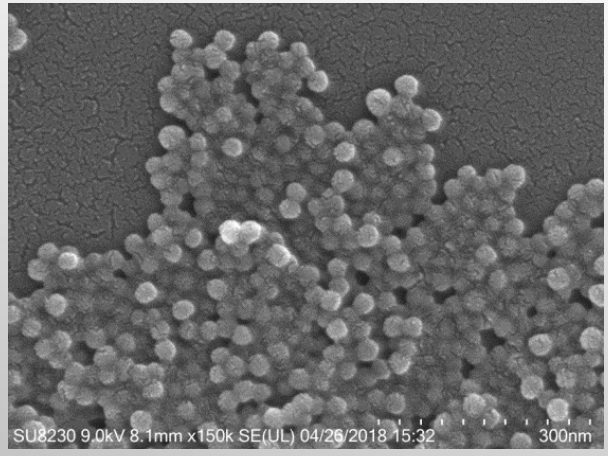


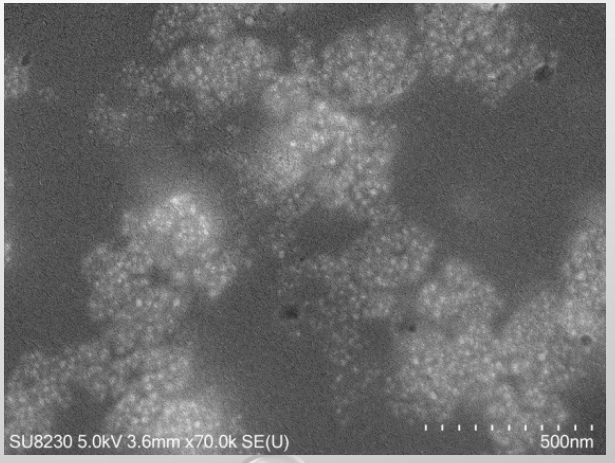
Filter Retention Testing





High concentration of particles collected in a short period of time may allow for lower filter challenge concentrations.





30 nm SiO_2 at 1.8E8/mL for 100 minutes

20 nm SiO_2 at 1E9/mL for 60 minutes

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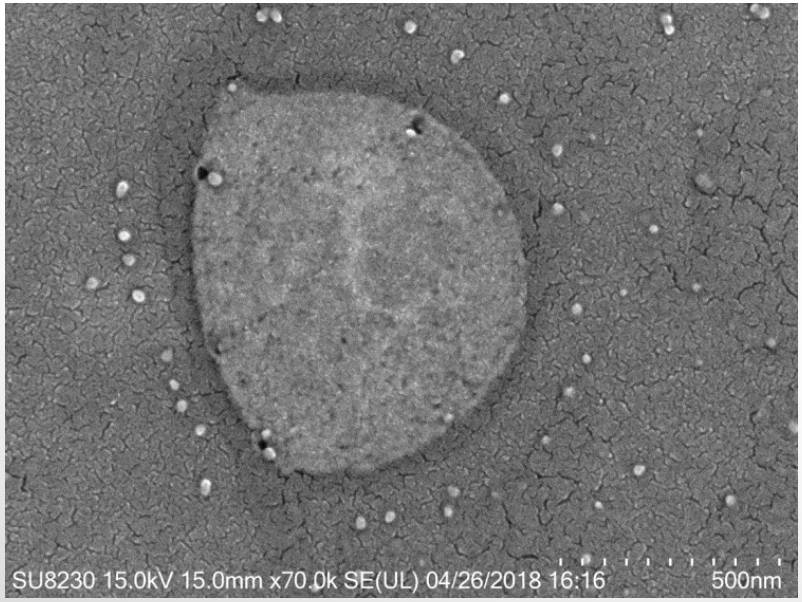
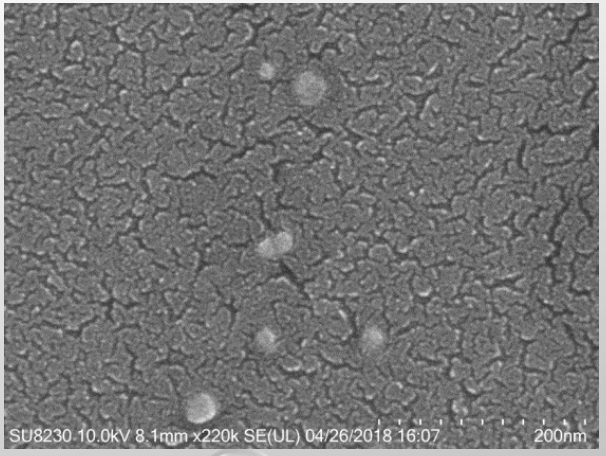
Component Contamination Profiling – IX Resin Effluent



Profiling individual contamination contributors will be beneficial in identifying or eliminating potential sources in the event of a contamination event.



- **Semiconductor grade virgin mixed IX resin.**
- **Triple rinsed in HDPE bottle in UPW.**
- **Agitated for 1 hour on rotary shaker table.**
- **Serial filtration with 100 and 20 nm Anodisk aluminum oxide filter.**
- **1000:1 online dilution for 1.6 hours.**

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NEXT STEPS

- CONTINUE TO IMPROVE SPOT LOCATION REPRODUCIBILITY.
- DEVELOP METHOD FOR TEM-READY DEPOSITION FOR SUB 10 NM ANALYSIS.
- IMPROVED NON-SILICON BASED DEPOSITION MEDIA TO ALLOW FOR BETTER SILICA IDENTIFICATION.
- DEVELOP QUANTIFICATION (COUNTING) METHODOLOGY.
- EVALUATE THE APPLICATION OF THE METHOD FOR FILTER TESTING (NOT NECESSARILY LIMITED TO PARTICLES).

SUMMARY

- FOCUSED AEROSOL DEPOSITION IS A POWERFUL NEW TOOL FOR VISIBILITY OF SUB-20NM UPW PARTICLE CONTAMINANTS.
- MADE SIGNIFICANT IMPROVEMENTS TO THE METHOD:
  - TIGHTLY FOCUSED AND CENTERED PARTICLE SAMPLE ON READY-TO-ANALYZE SEM SUBSTRATE.
  - FAST SAMPLE COLLECTION.
  - RAPID SPOT LOCATION AND RESTRICTED PROXIMITY FOR HIGH EFFICIENCY SEM/EDX ANALYSIS.
- PRELIMINARY DEMONSTRATION OF UPW SYSTEM MAPPING CAPABILITIES.
- POTENTIAL TO IMPROVE UPW PURITY IN MANY APPLICATIONS.

THANK YOU FOR YOUR ATTENTION!

Creative Technology

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