#### THE USE OF FOCUSED AEROSOL DEPOSITION IN THE COLLECTION AND ANALYSIS OF NANOPARTICLES FROM ULTRAPURE WATER SYSTEMS USED IN SEMICONDUCTOR AND PHARMACEUTICAL INDUSTRY

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## INTRODUCTION

- PARTICLES AS SMALL AS 10 NM AND LESS HAVE THE POTENTIAL TO CAUSE "KILLER DEFECTS" IN THE LATEST AND NEXT GENERATION OF SEMICONDUCTOR DEVICES.
- DETECTING, COLLECTING AND IDENTIFYING THESE "KILLER" PARTICLES IN HIGH-PURITY LIQUIDS IS EXTREMELY 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.
- THIS PRESENTATION WILL REVIEW FOCUSED AEROSOL DEPOSITION (FAD) 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<sup>3</sup>

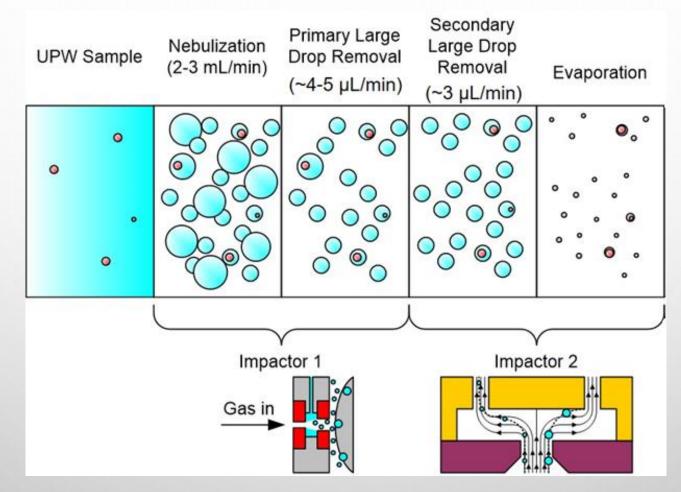
Ultrafine Aerosolization<sup>1</sup> Quenching Air Heated Evaporator Secondary Selective Impactor Sample Inlet Challenge Peristaltic Sample Pump Impactor Nebulizer Static Mixer Drain Pump Drip UPW Counter Supply UPW 0 flowmeter Filter Drain NanoParticle Extractor (NPE), Kanomax FMT Inc., Rev.1.0, 05/07/2018

Sample Inlet "Conditioner" Cool-wet wall Cold 5°c "Initiator" Activation region Warm 35°c Water droplet encapsulation Cool III 12°c "Moderator" 0 Water removal region 0 Impaction spot Droplet/particle capture Warm to evaporate water drops for dry collection

Nanoparticle Collection<sup>2</sup>

<sup>1</sup> Kanomax FMT NanoParticle Extractor (Model 9410-00)
<sup>2</sup> Kanomax FMT NanoParticle Collector (Model 9410-01)
<sup>3</sup> Kanomax FMT NanoSpotLight<sup>™</sup>

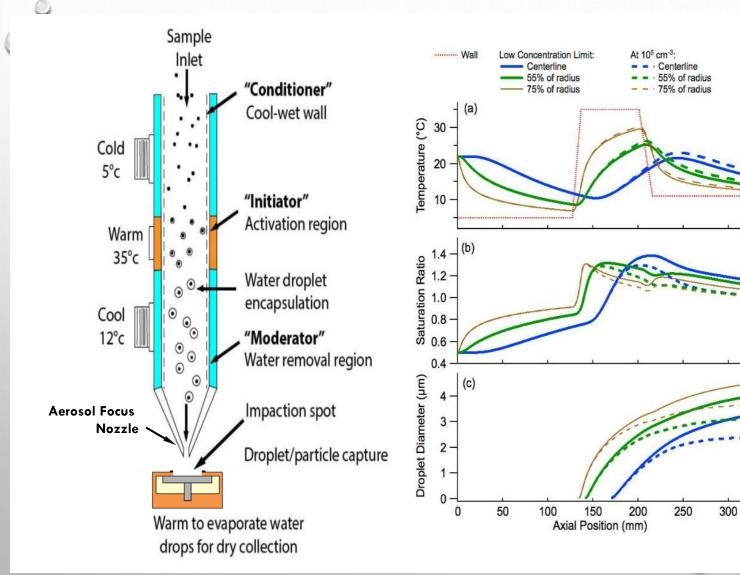
## THEORY OF OPERATION – NANOPARTICLE EXTRACTOR



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.

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#### Condensation Growth Tube:

ACTIVATION EFFICIENCY AND SIZE IMPLICATIONS

Temperature, flowrate, bore size, gas

- Hydrophilic vs. hydrophobic particles

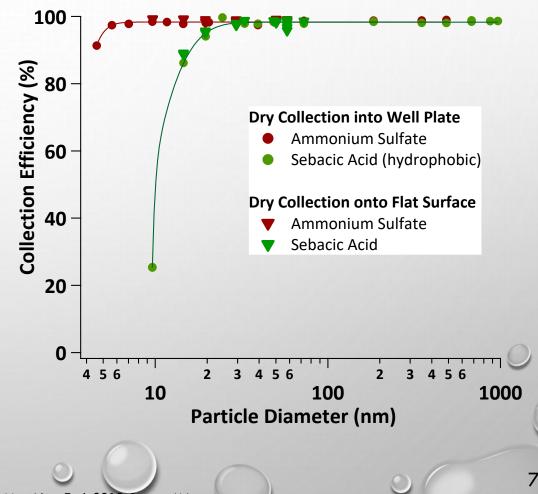
Aerosol Concentration :

**Material Effects:** 

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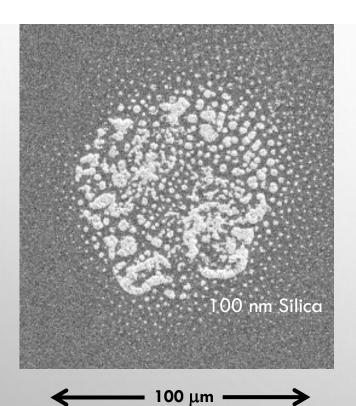
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Parameter	< 1E4/cc	> 1E4/cc
Minimun activation size	Smaller (5nm)	Larger
Droplet size	Larger (3µm)	Smaller
Collection efficiency	Higher (>98%)	Lower
Media heating requirements	Lower (<40°C)	Higher



### BENEFIT OF SPOT CONCENTRATION

Spot pattern at 50 X magnification



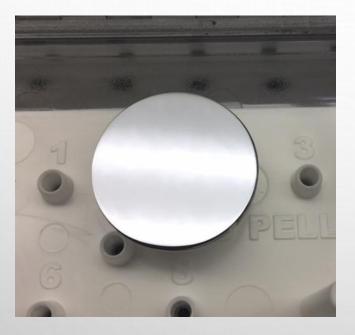
	Minimum Particle Size (nm)	Inspection Area (mm <sup>2</sup> )	Typical flow rate (mL/min)	Depostion Rate (mL/mm2)	Relative Sampling Time (to FAD)
FAD	5	0.01	0.005	0.64	1
Al <sub>2</sub> O <sub>3</sub> Filter	20	346	37.3	0.11	5.9
Track Etch Filter	50	415	10.0	0.02	26.5

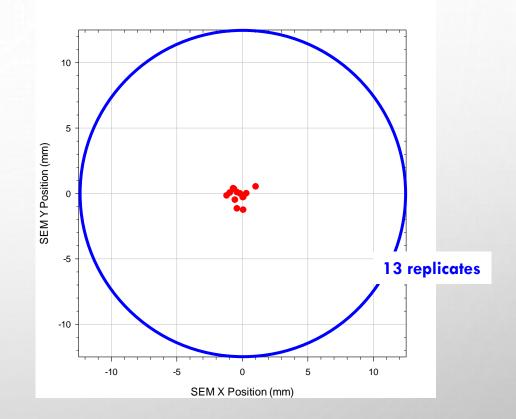
Assumes a 100  $\mu m$  spot diameter with a 5  $\mu L/min$  extraction rate.

### **Direct Deposition on SEM-Ready Stubs**

Two Deposition Media:

- 25mm Silicon Prime Wafer
- 12.5mm x 12.5mm Germanium



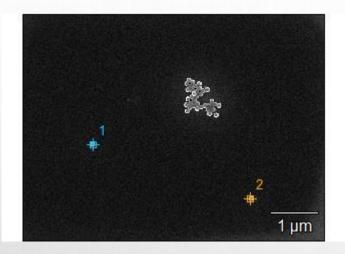


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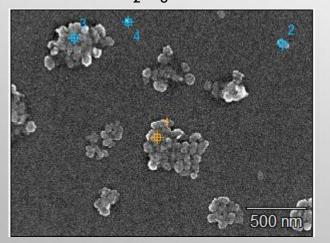
Depositing on SEM-ready stub with alignment pin provides the ability to rapidly locate spot and begin SEM/EDX analysis.

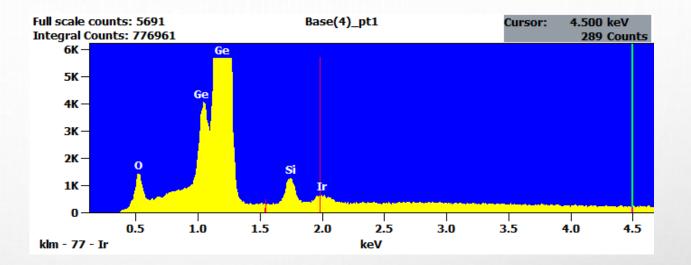
### MEDIA CAPABILITIES

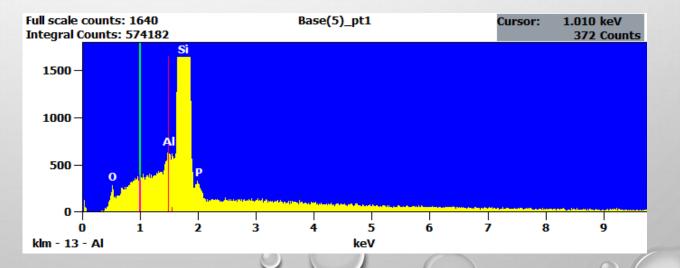
 $30 \text{ nm SiO}_2$  on Ge wafer



 $45 \text{ nm Al}_2\text{O}_3$  on Si wafer







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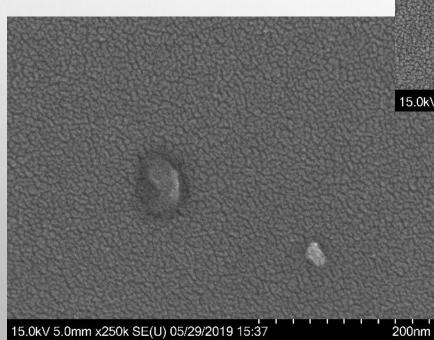
# ULTRAPURE WATER APPLICATIONS

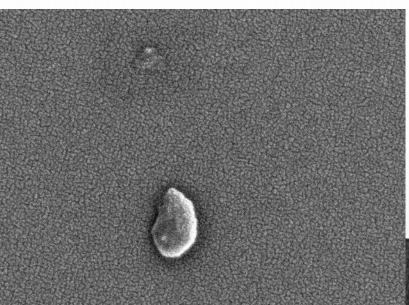
- UPW SYSTEM CONTAMINATION DETECTION AND IDENTIFICATION
- COMPONENT CONTAMINATION PROFILING
  - FILTER (MF AND UF) SHEDDING
  - IX RESIN RELEASE
  - MECHANICAL COMPONENTS (VALVES, REGULATOR, TUBING, ETC.)
  - MEMBRANE CONTACTORS
- RAPID BACTERIA COLLECTION AND CONCENTRATION

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# SEMICONDUCTOR UPW EXAMPLES\*

Source: 2019 IRDS Round Robin Testing Participating Companies: Global Foundries, IMFT, Intel, Micron, Samsung





15.0kV 8.0mm x200k SE(U) 05/29/2019 13:33

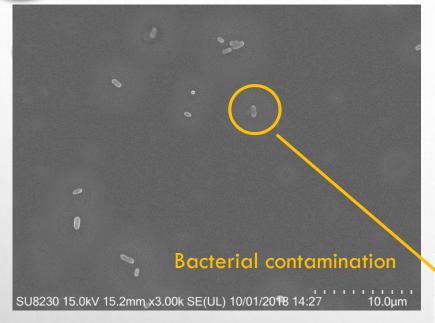
Predominately "soft" organic particles, some metal oxides Particles found downstream of the final distribution filters.

15.0kV 8.0mm x250k SE(U) 05/29/2019 13:20

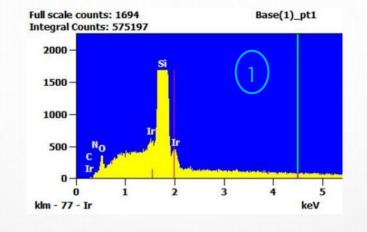
200nn

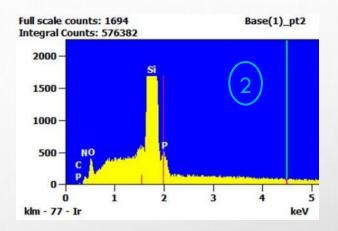
200nm

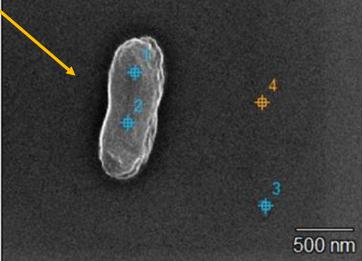
## SEMICONDUCTOR UPW EXAMPLES\*

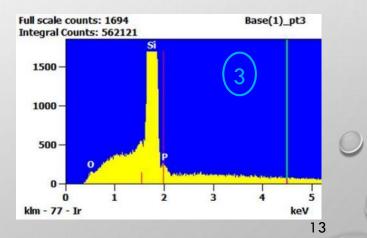


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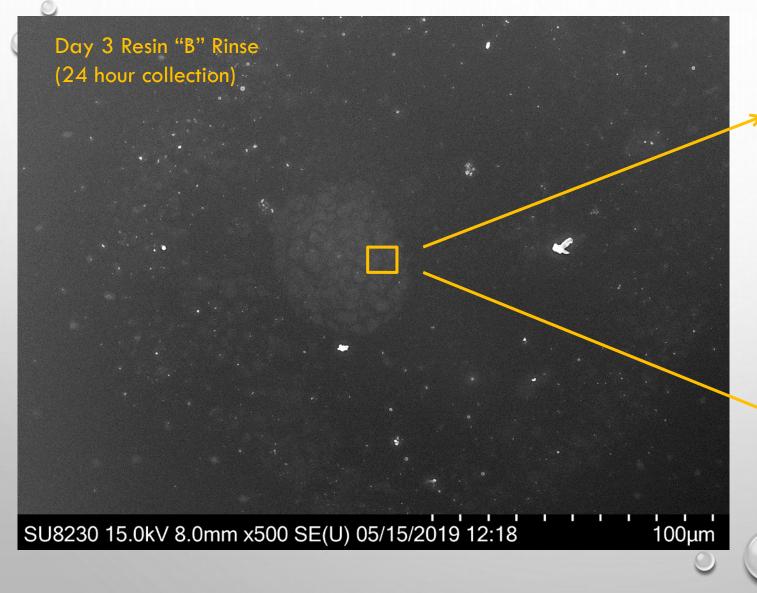


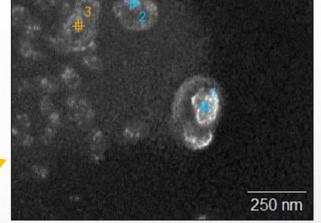




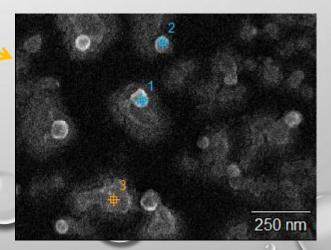


### ION EXCHANGE RESIN RELEASE DURING RINSE



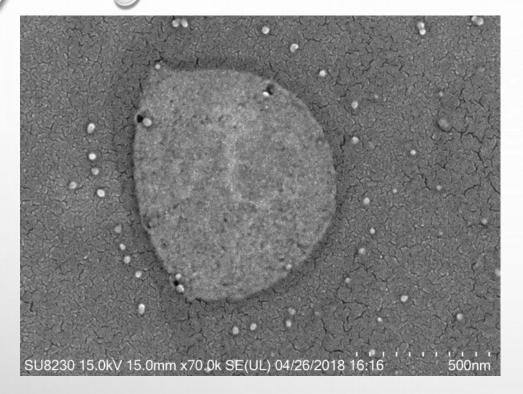


Location	Elements (less Si)		
T1	O,Na,S,Ba		
T2	O,Na,S		
Т3	O,Na,S		
L1	O,Na,S		
L2	O,Na,S		
L3	O,Na,S		



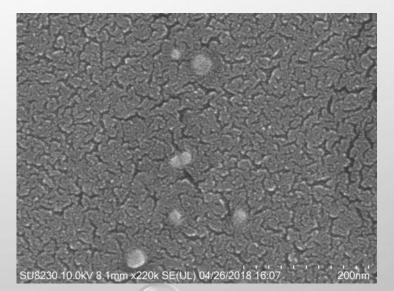
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#### Component Contamination Profiling – IX Resin Effluent



- 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.

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



### **DEVELOPMENT PRIORITIES**

- CHEMICALLY COMPATIBLE NEBULIZER.
- INCREASED PARTICLE EXTRACTION AND DEPOSITION RATES.
- IMPROVED SPOT POSITION ACCURACY AND REPEATABILITY.
- DEVELOP QUANTIFICATION (COUNTING) METHODOLOGY.
- METHOD FOR TEM-READY DEPOSITION FOR SUB-10NM ANALYSIS.
- EVALUATE ADDITIONAL IDENTIFICATION TECHNIQUES (AFM-IR, MALDI, SERS, TOF-SIMS)

### SUMMARY

- FOCUSED AEROSOL DEPOSITION IS A POWERFUL NEW TOOL PROVIDING RAPID COLLECTION OF SUB-50NM PARTICLE CONTAMINANTS FROM UPW.
  - TIGHTLY FOCUSED AND CENTERED PARTICLE SAMPLE ON READY-TO-ANALYZE SEM SUBSTRATE.
  - NEARLY 30 FOLD INCREASE IN PARTICLE COLLECTION EFFICIENCY COMPARED TO TRADITIONAL METHODS.
  - RAPID SPOT LOCATION AND RESTRICTED PROXIMITY FOR HIGH EFFICIENCY SEM/EDX ANALYSIS.

### THANK YOU FOR YOUR ATTENTION!

Van Schooneveld, et al., Focused Aerosol Deposition, NanoTech 2019, Boston, MA

Creative Technology *CTAssociates, Inc.*  Aerosol Devices Inc

