Introduction Thermo Fisher Portfolio

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13-03-2024

US based company as world leader in serving science

ThermoFisher SCIENTIFIC

ThermoFisher SCIENTIFIC

thermo scientific | applied biosystems | invitrogen | fisher scientific | unity lab services | Patheon

$40 B
revenue

130,000+
employees

$2B
invested in R&D

Industry-leading scale

- Unparalleled commercial reach
- Unique customer access
- Expanding global footprint

Unmatched depth of capabilities

- Leading innovative technologies
- Deep applications expertise
- Premier productivity partner
- Comprehensive services offering

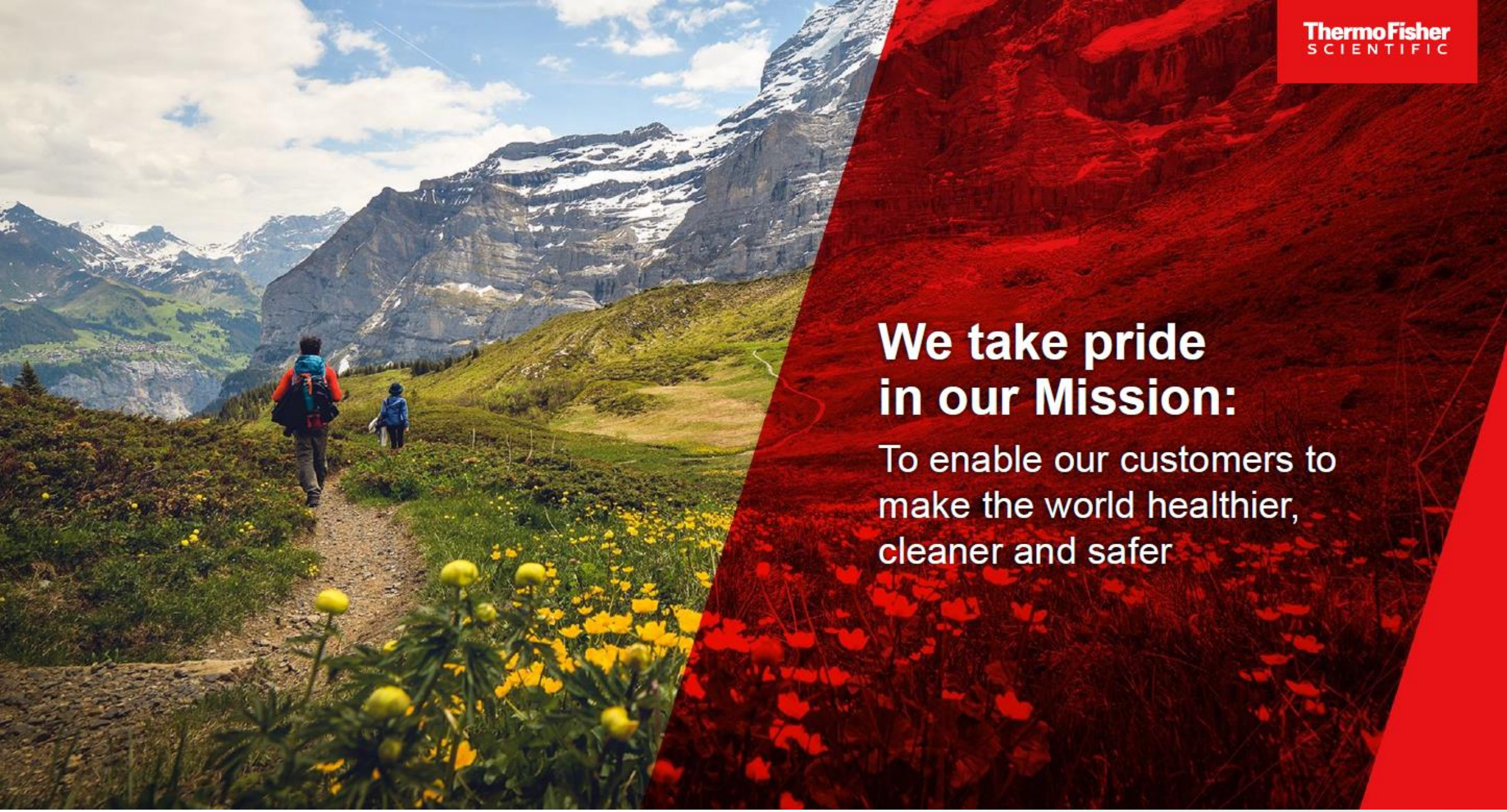
Powered by our Practical Process Improvement (PPI) Business System

Our leading scientific products, services and workflow solutions

ThermoFisher SCIENTIFIC

The world leader in serving science

thermo scientific	applied biosystems	invitrogen	fisher scientific	unity lab services	patheon	PPD
Analytical precision and diagnostics excellence	Inspiring meaningful genetic analysis	Accelerating discovery research	Products one-stop access for scientific supplies	Instrument and enterprise services	Pharma services	Drug development


ThermoFisher
SCIENTIFIC
We take pride
in our Mission:
To enable our customers to
make the world healthier,
cleaner and safer

MSD Locations

Analytical Instruments Group (AIG)

Chromatography & mass Spectrometry Division (CMD)

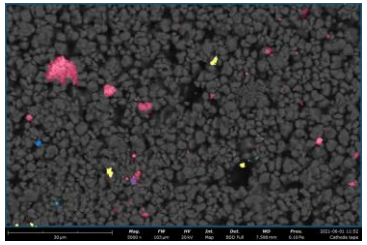
Chemical Analysis Divisions (CAD)

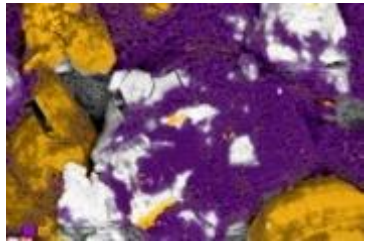
Materials and Structural Analysis Division (MSD)

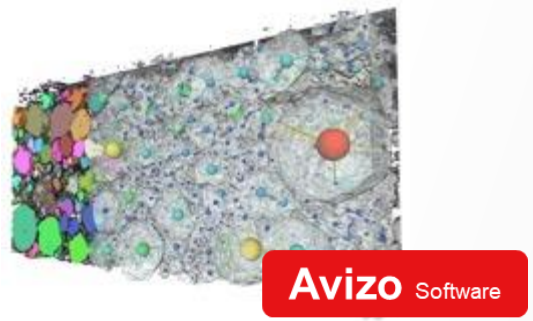
Instrument and Enterprise Services (IES)

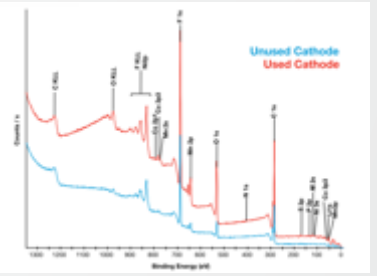
- Location of sites ≥ 5 R&D employees
- Location name called out for sites with ≥ 25 R&D employees

Thermo Fisher Materials MSD-EM









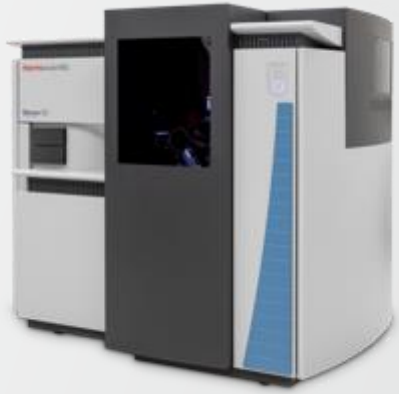
SEM | DualBeam | TEM | XPS

Thermo Scientific Phenom Desktop SEM

Thermo Scientific Apreo 2 and Axia SEM

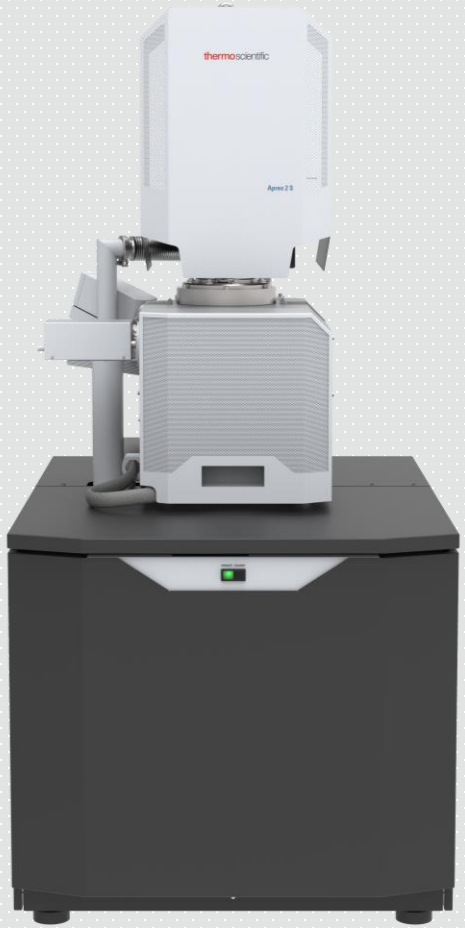
Thermo Scientific Scios 2 and Helios DualBeam

Thermo Scientific Talos and Spectra TEM



Thermo Scientific K-Alpha, Nexsa G2 And ESCALAB Qxi XPS

Contents



Thermo Scientific offers a complete SEM product portfolio

What samples do I have, and what do I want to see?

Desktop SEM

Phenom ProX

Phenom XL

Phenom Pharos

- **Time to image**
- **Ease of use**
- **Smallest footprint**

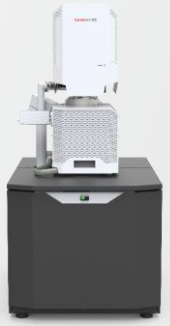
conventional SEM

- **Flexibility**
  - Large samples
  - Difficult samples
  - Accessories
- **Performance**
  - Ultimate resolution
  - Best contrast

Axia ChemiSEM

Prisma E

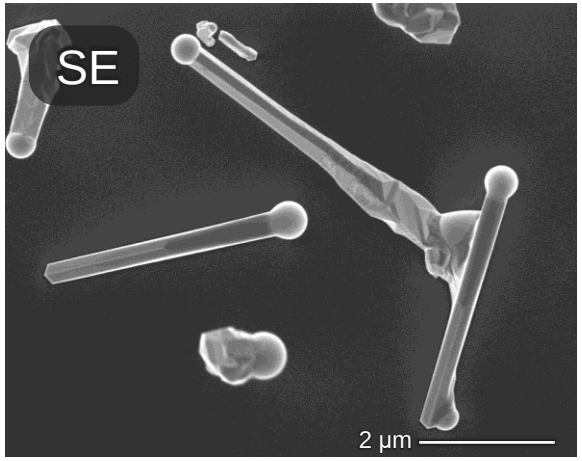
Quattro

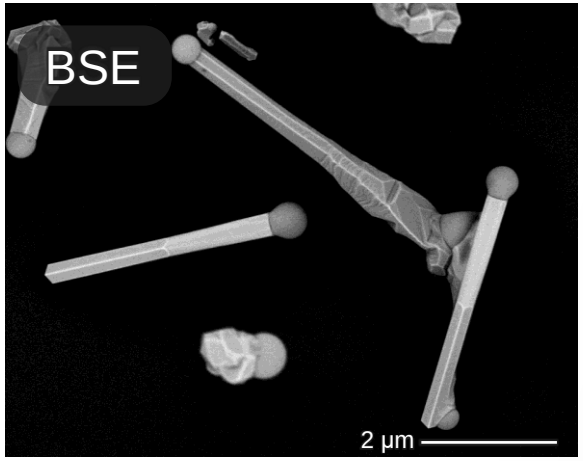


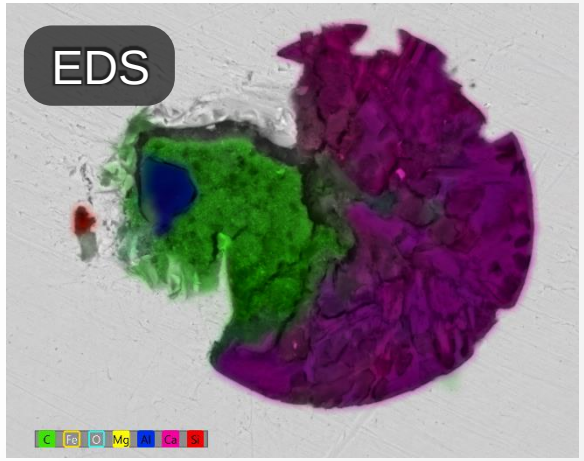
Apreo

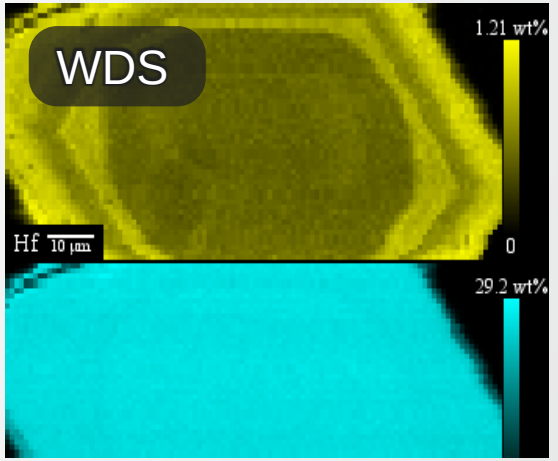
Verios

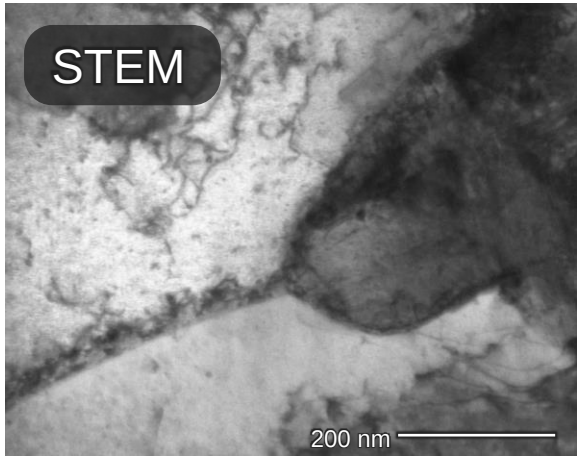
Type of information required – depends on the detector selection

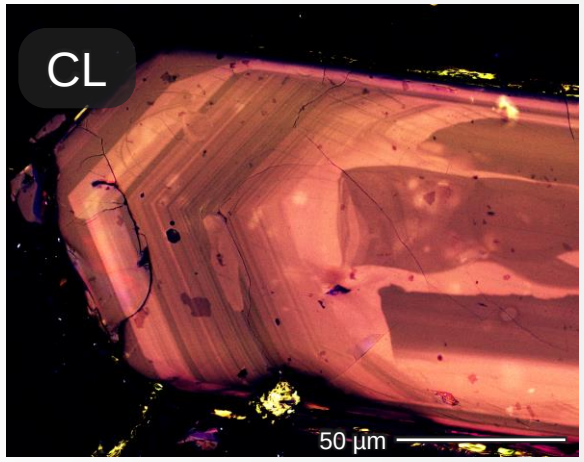


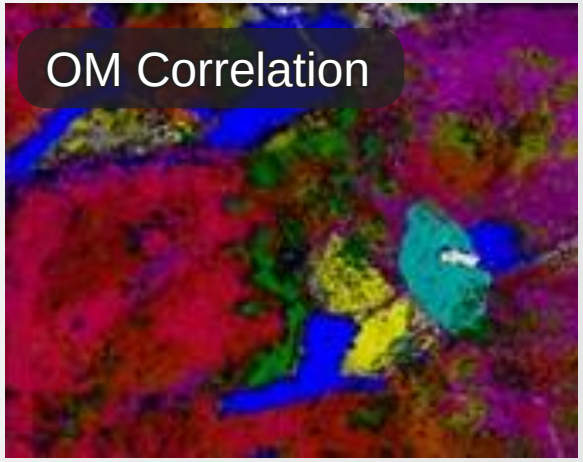






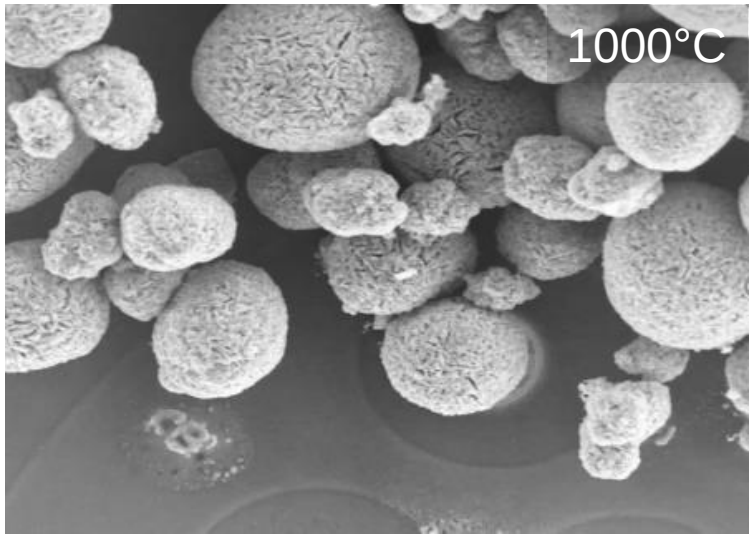


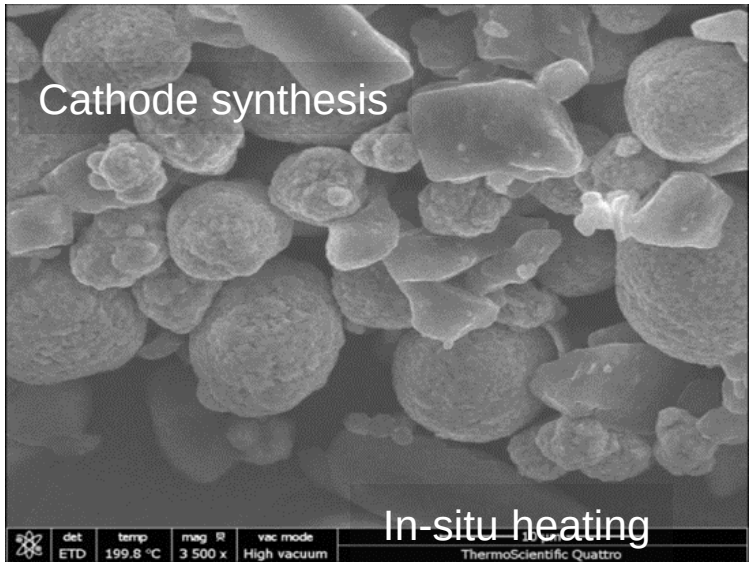


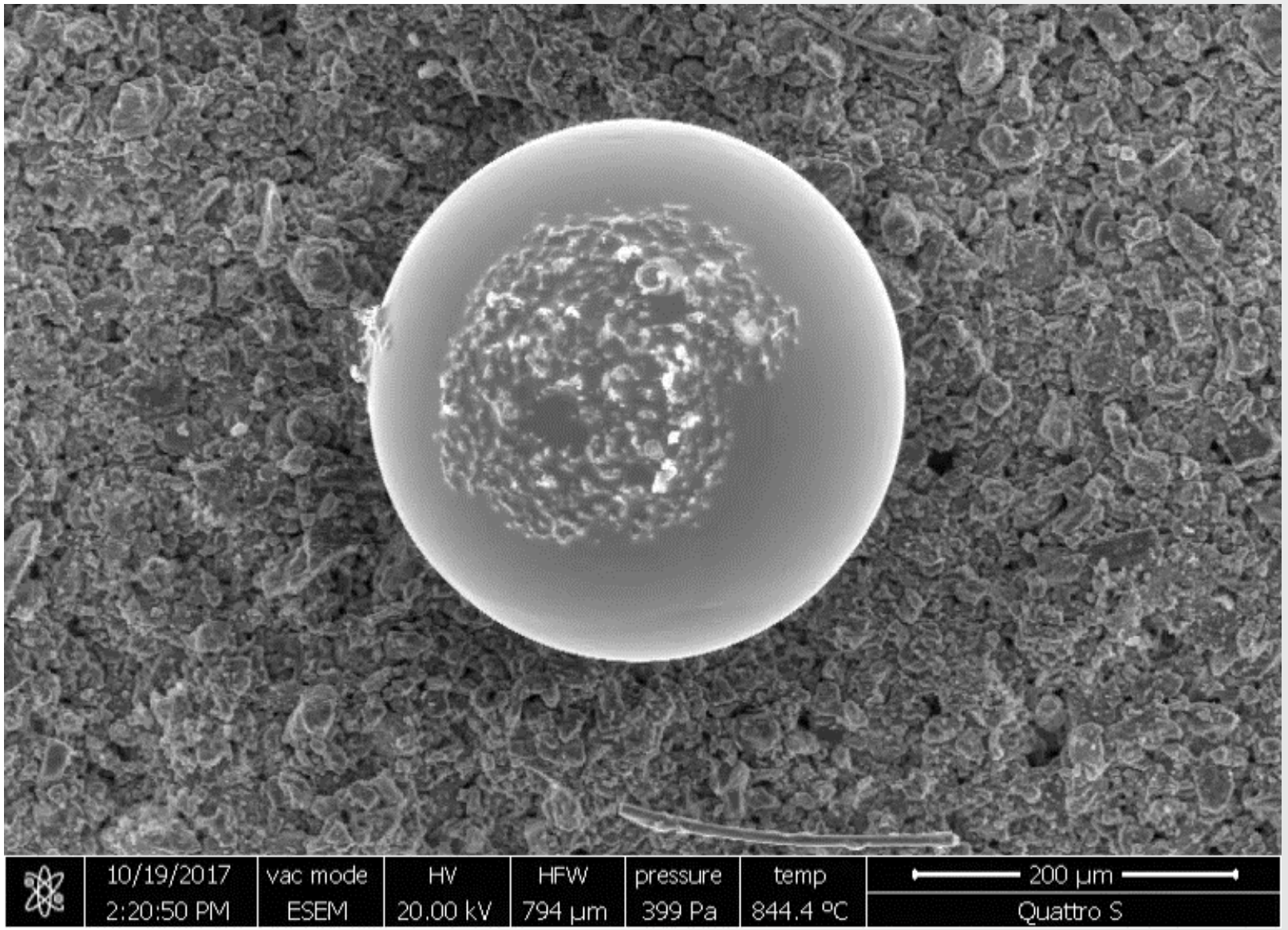


→ **All these types of information, and more, are available on all conventional Thermo Scientific SEMs**

In situ heating in SEM

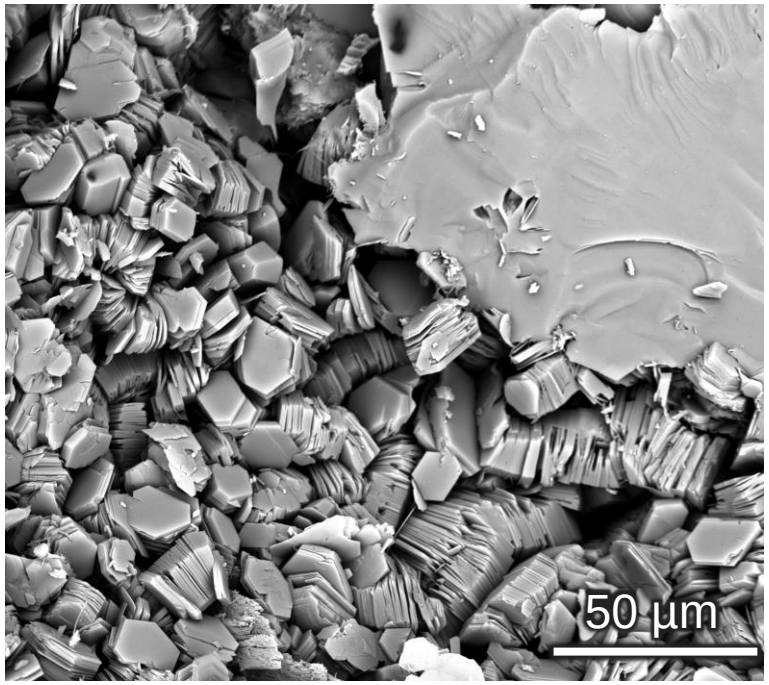






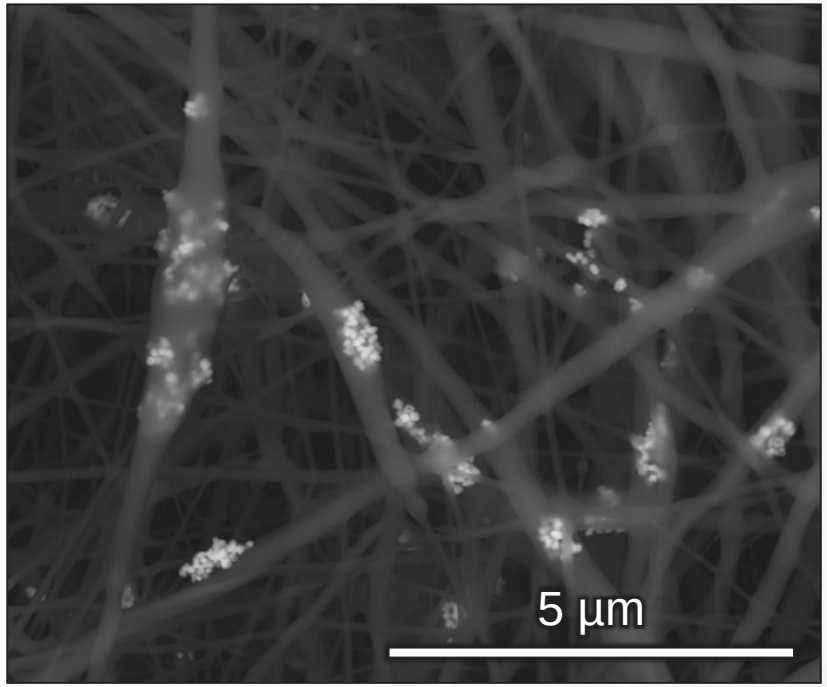
Feature size and resolution

Large feature sizes (> 1 µm)



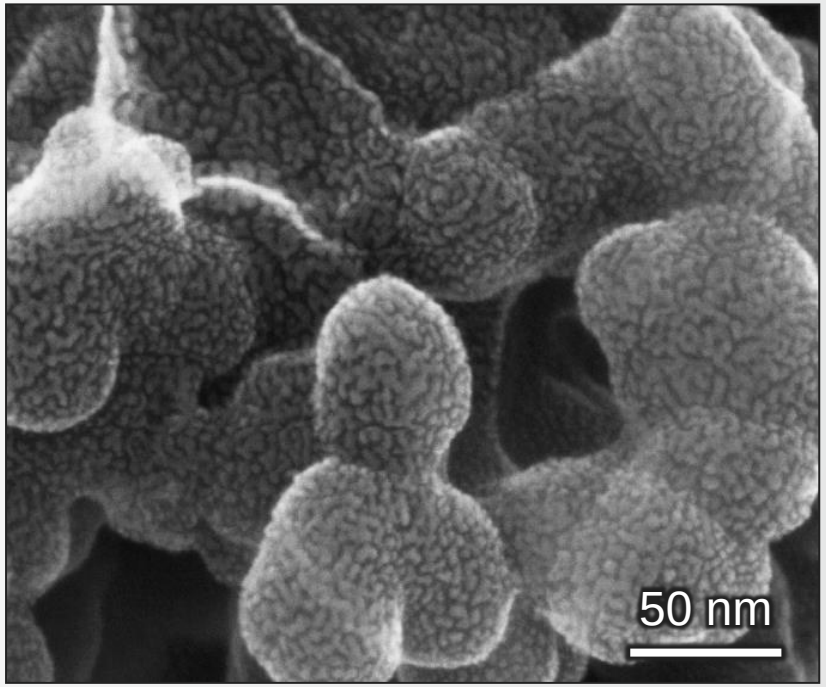
Aluminum fracture

Small feature sizes (<< 1 µm)



Electrospun fibers with Fe_3O_4 particles

Nanomaterials (<10 nm)



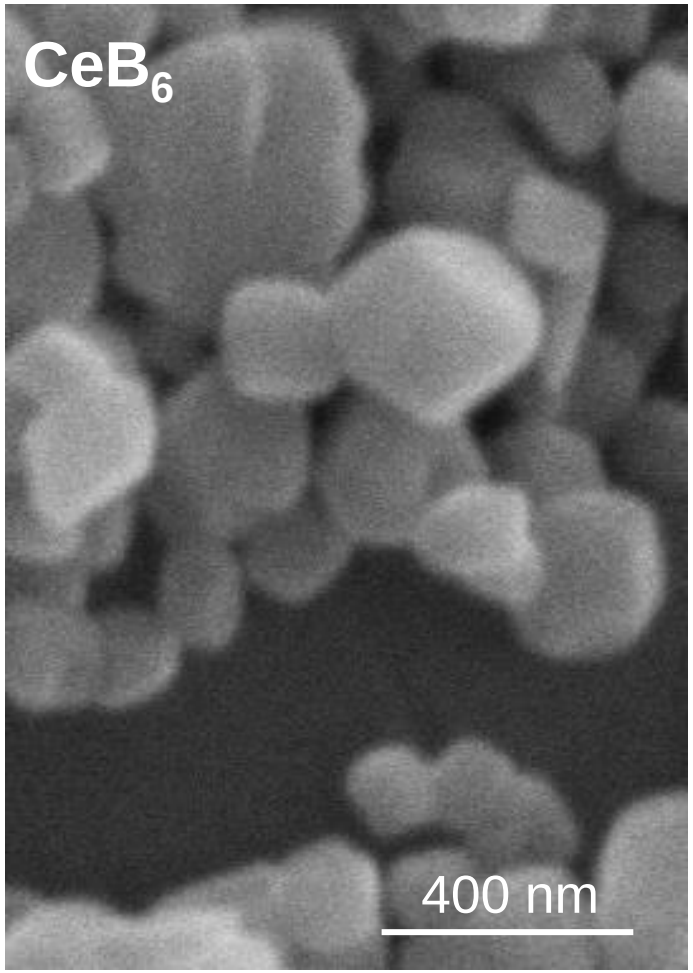
Sputtered Pt on C Film
Courtesy Sandia National Lab, USA

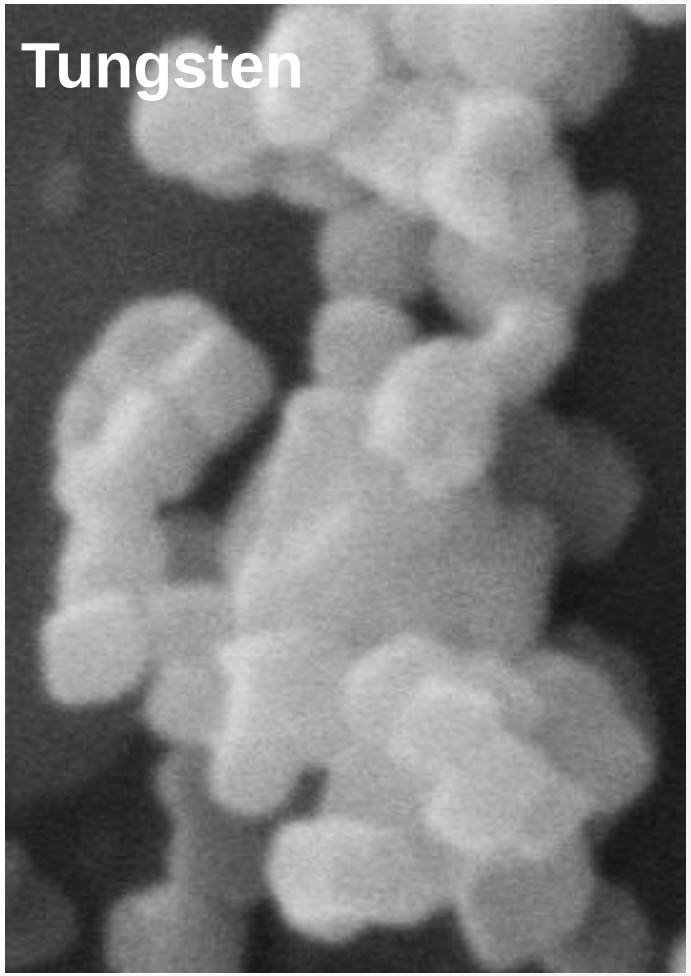
Tungsten SEM
a.k.a.
Conventional SEM
Thermionic source

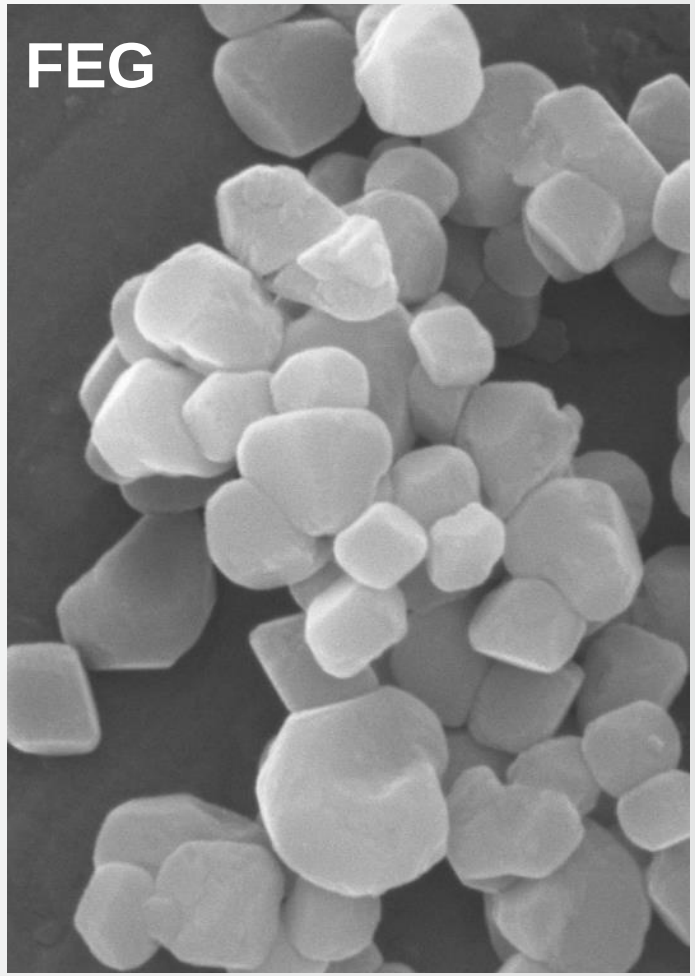
Field Emission SEM
a.k.a.
FEG (field emission gun)
Conventional FEG
Schottky FEG

In-lens SEM
a.k.a.
In-lens FEG
Acceleration tube FEG
Immersion SEM

FEG on desktop SEM enables outstanding results

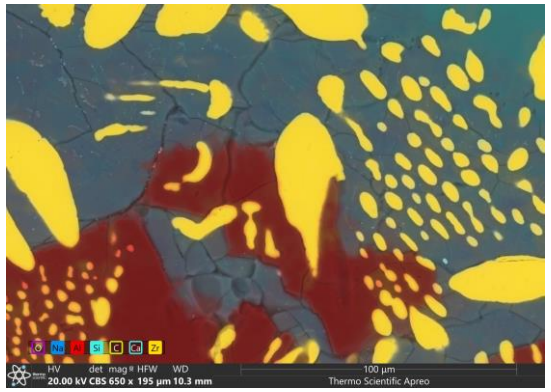


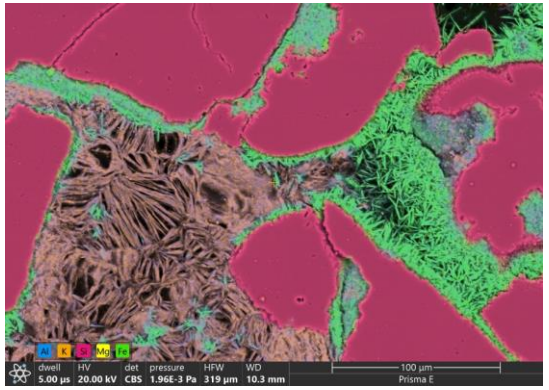


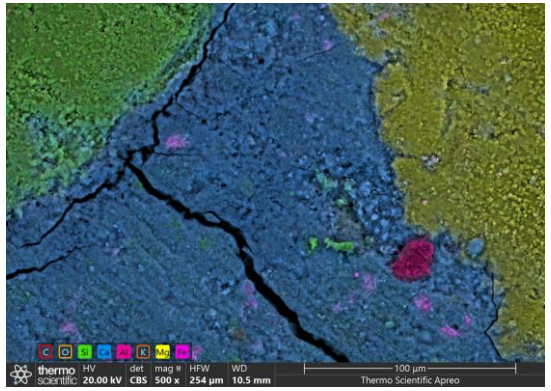


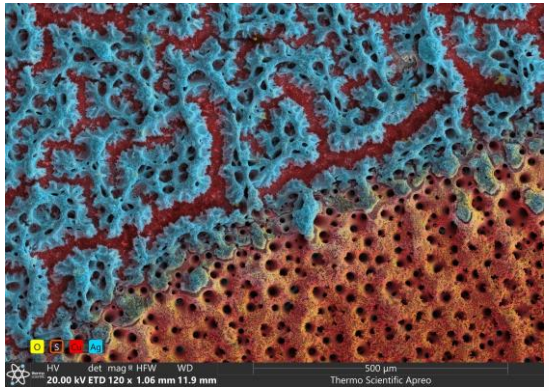
$CaCO_3$ particles, Pt coated, imaged at 10 kV

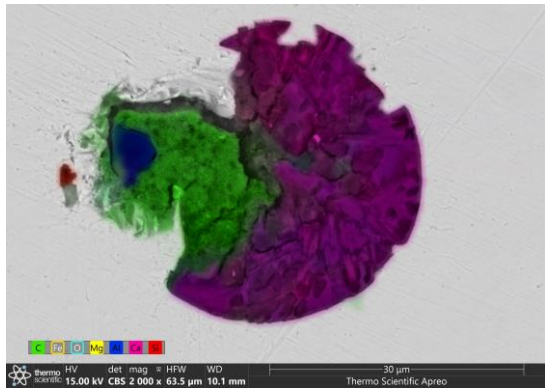
Live Quantitative Elemental Mapping with ColorSEM Technology

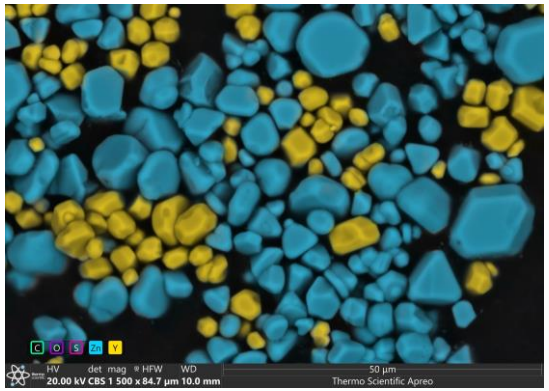












Fast

Get full compositional information up to 10 times faster with background X-ray collection, unique data processing, TV-rate pixel scanning, and an integrated user interface.

Intuitive

Color is switched on with a single click, all relevant functions are nearby, contrast is optimized with smart coloring, and live composition is shown with a tooltip.

Complete

ColorSEM Technology is always on, showing information that might otherwise be missed.

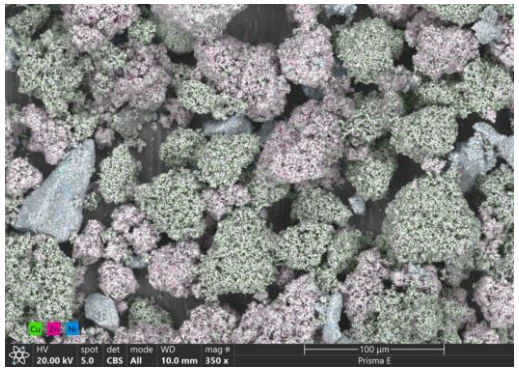
Reliable

Image coloring is based on live elemental quantification. The quant algorithms are based on decades of knowledge and experience to ensure reliable results.

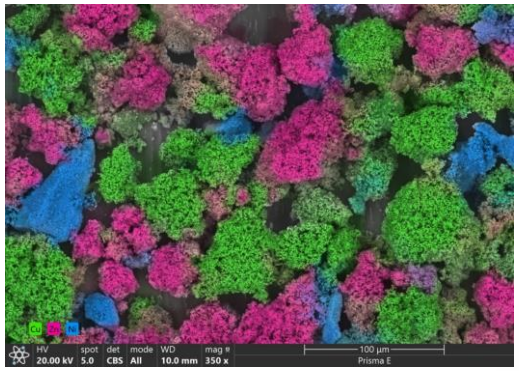
ColorSEM benefits

It’s fast

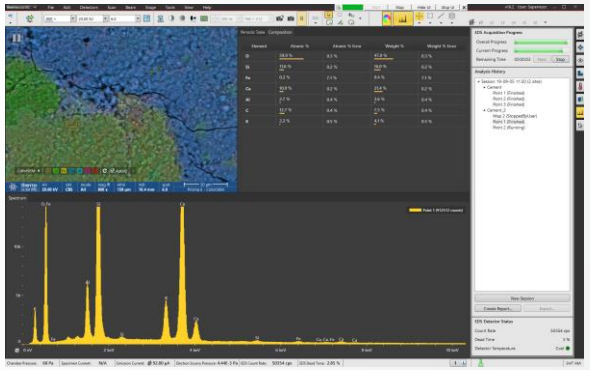
Traditional mapping (30 sec)



Live ColorSEM (30 sec)

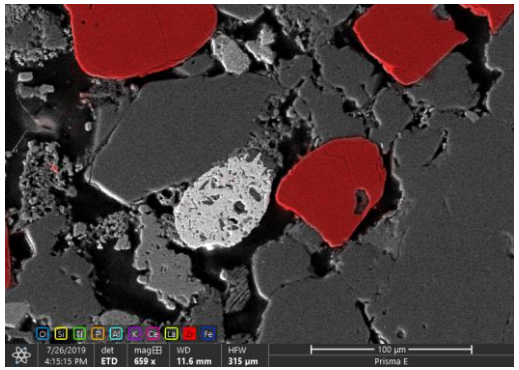


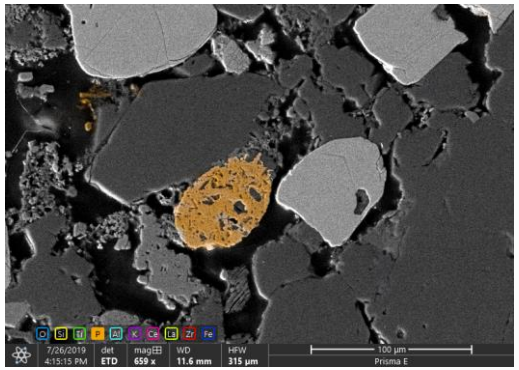
It’s intuitive



- Single UI, no switching
- No setup required
- Colors show elements
- Work directly on the SEM image for point & ID, etc
- All functions are one mouse click away

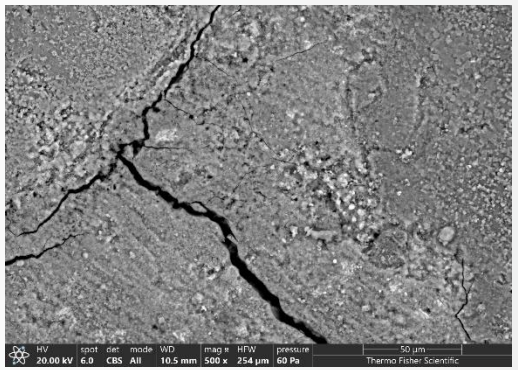
It’s reliable



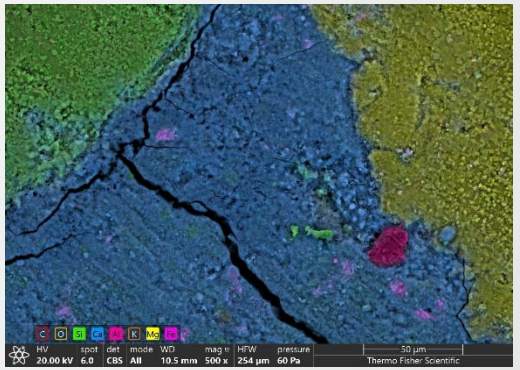


Materials correctly labeled, even when peaks overlap (Zr / P).
→ Live coloring is based on live quantification.

It’s complete



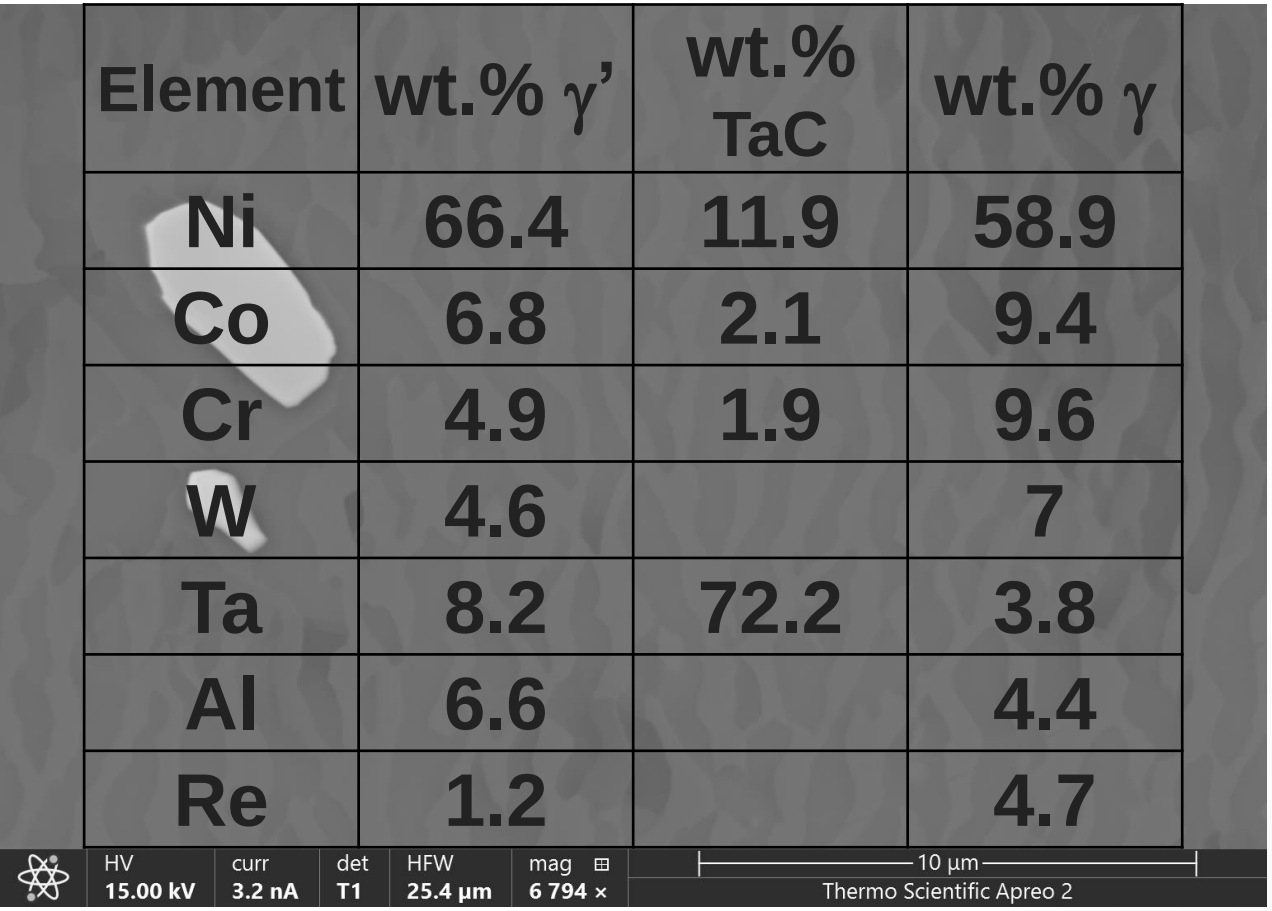
Materials contrast not clear, even in backscatter mode



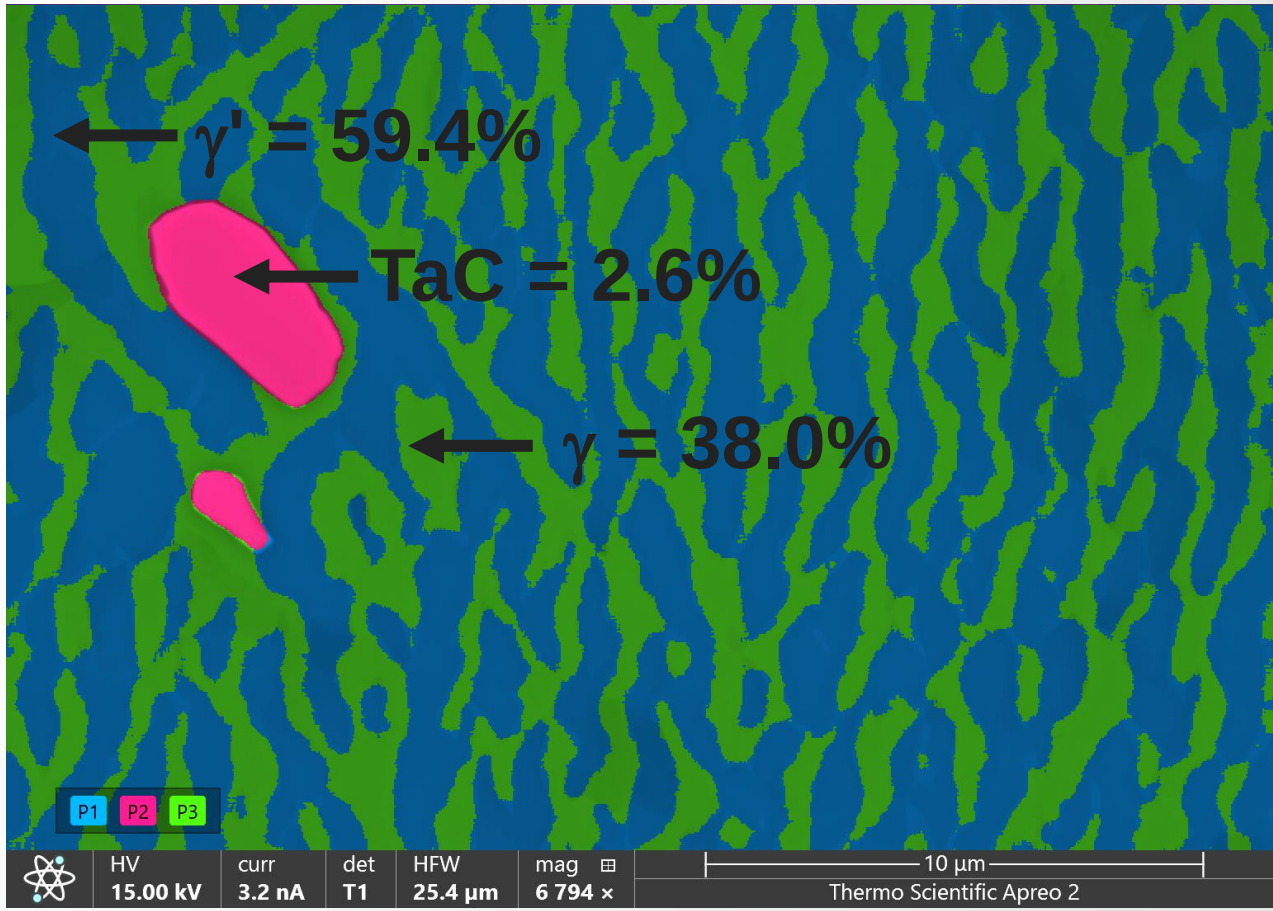
ColorSEM: highlight materials contrast you would otherwise miss

Identify and quantify metallic phases

Element	wt.% γ’	wt.% TaC	wt.% γ
Ni	66.4	11.9	58.9
Co	6.8	2.1	9.4
Cr	4.9	1.9	9.6
W	4.6		7
Ta	8.2	72.2	3.8
Al	6.6		4.4
Re	1.2		4.7



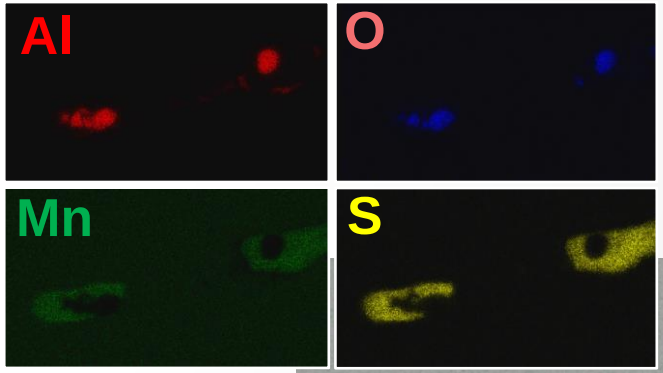
SEM image



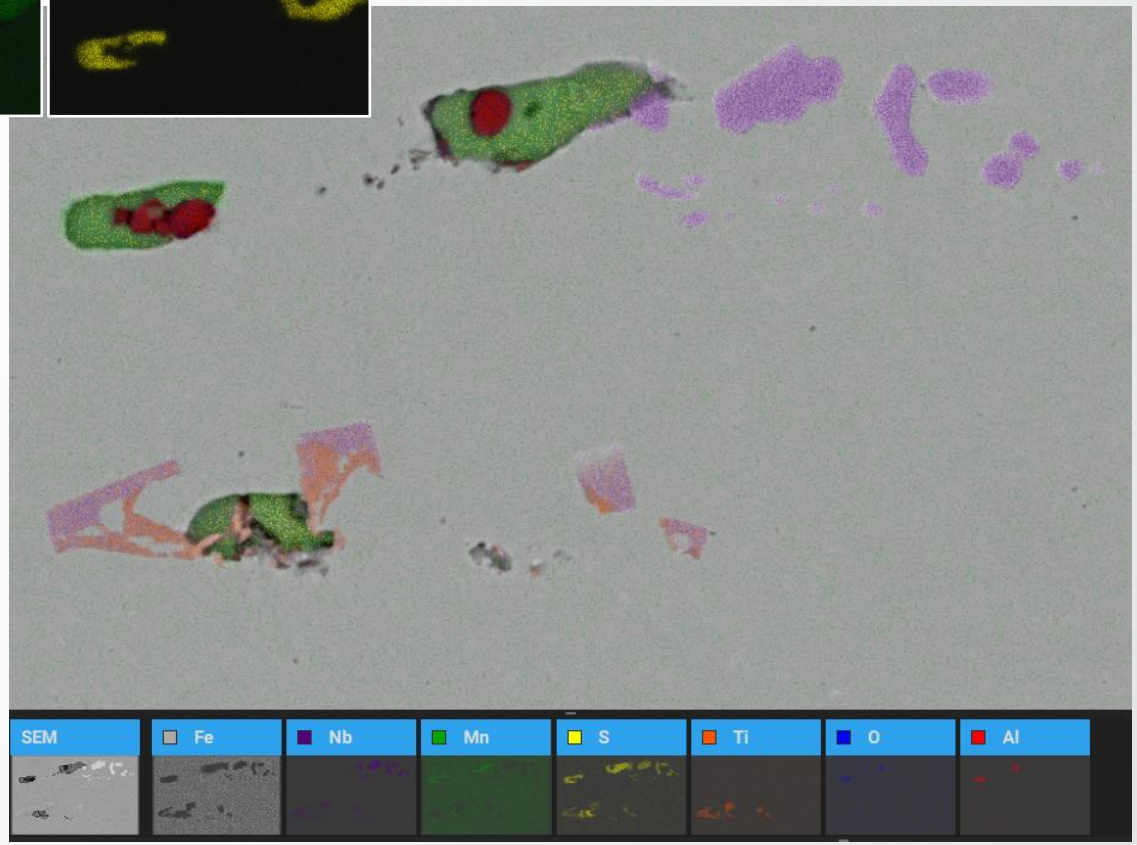
Quant map Phase analysis SEM image

Phenom ParticleX Steel – with Phenom User Interface

New Phenom User Interface for regular imaging and EDS work

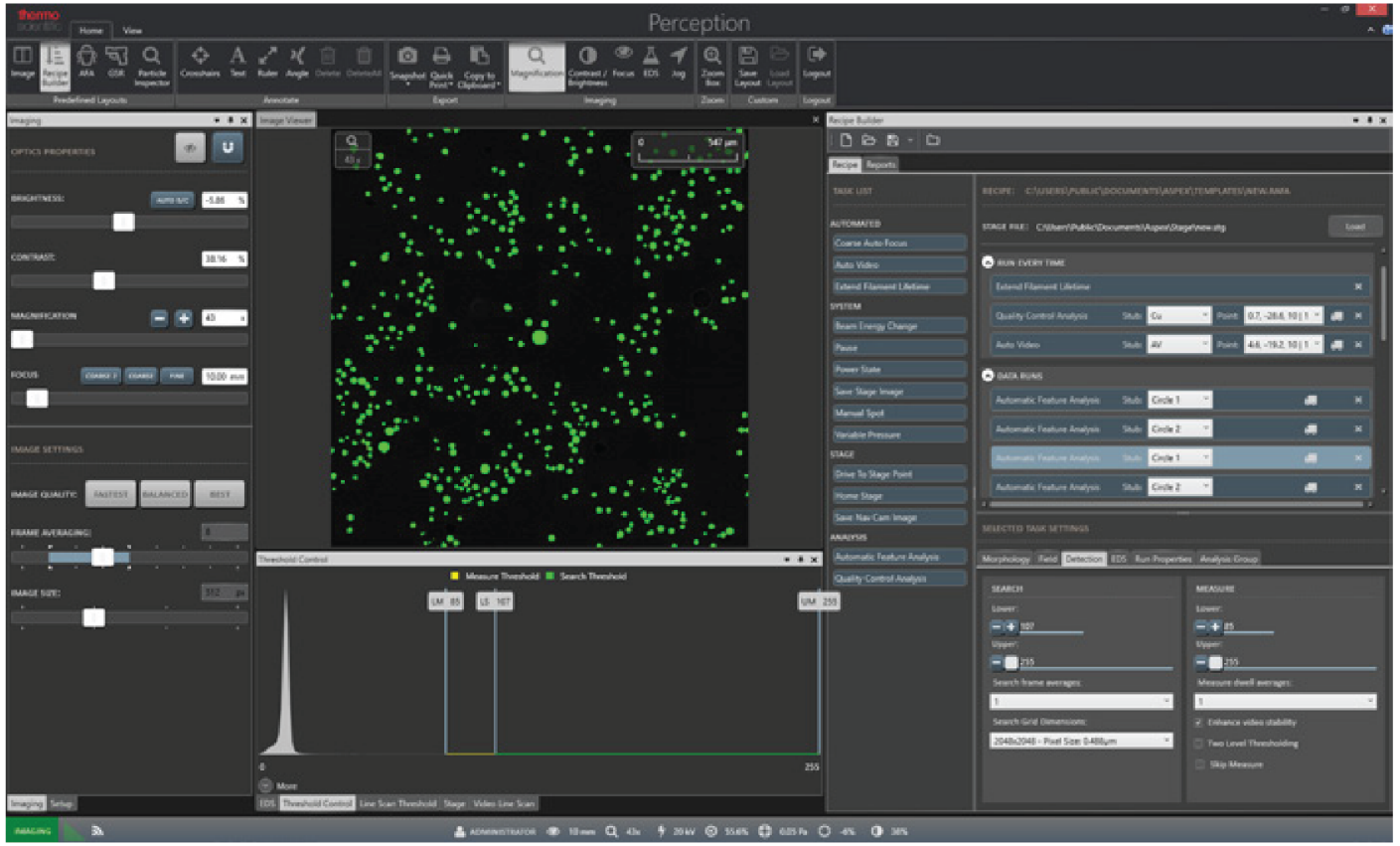


EDS mapping included



Particles Analysis Software

- Perception and Particles X for all our SEMs

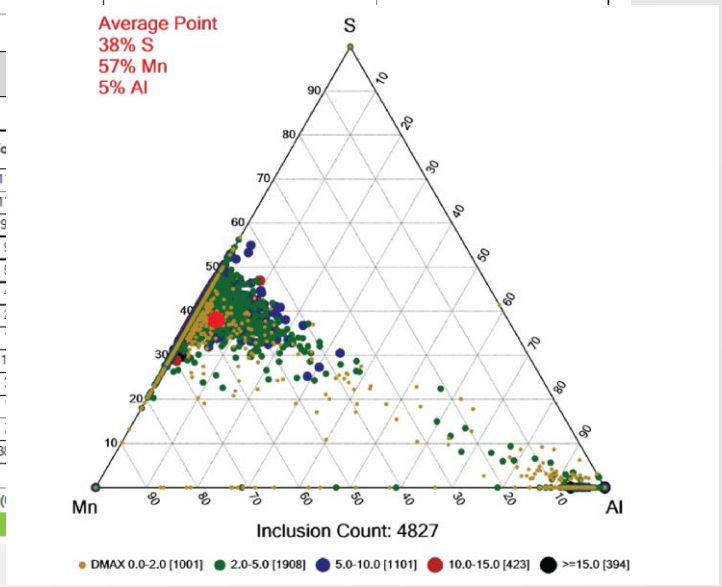


Inclusion Classification Summary

Classification	Particles	[%]	N	Mg	Al	Si	S	Ca	Ti	Mn	Features/ mm²	Area%	Incl.Index
Mn Sulfide	4130	84.8	1.5	1.2	1.3	0.6	36.2	2.1	2.2	54.0	20.1	94.4	0.02154
Ti Nitride	511	10.5	30.7	0.2	0.5	0.7	5.5		51.2	10.5	2.5	3.0	0.00068
Spinel Pure	201	4.1	0.1	32.1	56.5	2.5	1.3	0.9	1.4	4.7	1.0	0.9	0.00021
Spinel Rich	23	0.5	4.6	19.3	39.2	1.4	7.4	1.0	12.0	14.4	0.1	0.1	0.00001
Alumina	7	0.1	1.9	0.4	82.7	2.4	1.0	0.3	4.9	5.6	0.0	1.6	0.00037
Mg Rich	1	0.0		49.8	1.4	1.5	18.8			28.6	0.0	0.0	0.00001
All above combined	4873	100.0	4.5	2.5	3.8	0.7	31.3	1.8	7.3	47.2	23.7	100.0	0.02283

Sample Information		Sample Prep Information		Run Information	
Company:		Component Surface By:	Volume	Magnification:	220x
Operator Name:		Volume of Extraction(cm³)	100	Number of Stage Fields:	61
Part # / Sample ID:		Projected Volume(cm³)	100	Area Scanned(mm²):	36.42
Analysis Date:	3/29/2021 1:30:40 PM	Filter Size (mm):	300	RunID's Present:	Run_1

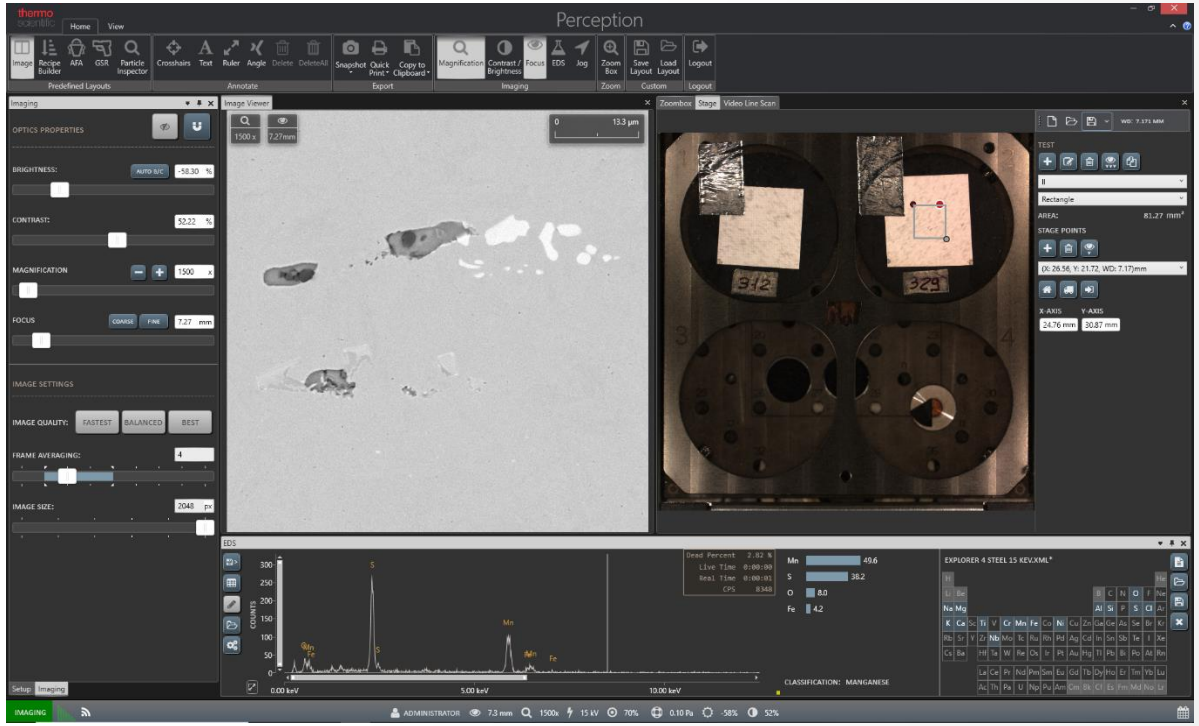
Size Class	
Size Range (um)	To
Glass	1
Al2O3	1
Si Rich	9
SiO2	
Ca-Aluminosilicates	
Aluminosilicates	
Steel	
Oxidized Al	
Misc	1
Mineral	
Non-Ferrous Metal	
Misc Salts	
Total Counts	3
Cleanliness Level	
Component Cleanliness Code (	
Specification:	



Automated analysis with Perception

Sample

Perception

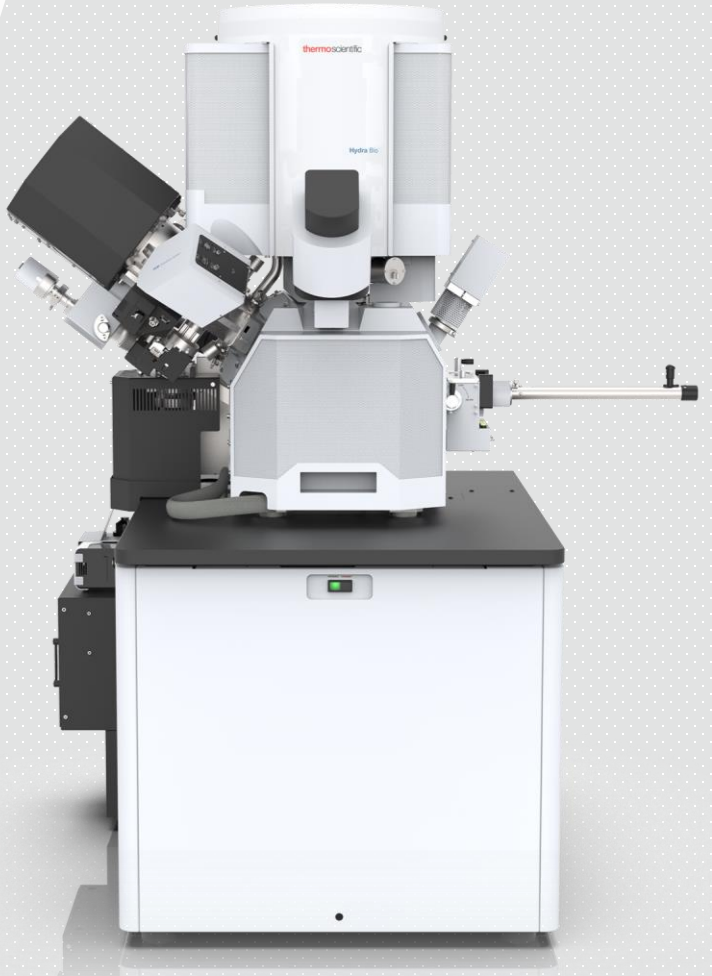


Automated inclusion **detection**
Automated **image** acquisition
Automated **EDS** analysis
Automated inclusion **classification**

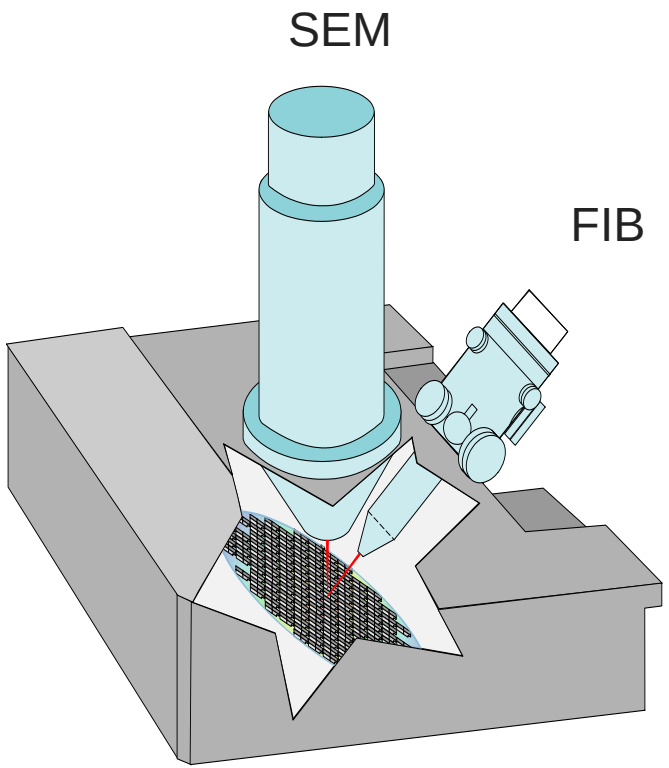
Conventional SEM product portfolio for Materials Science

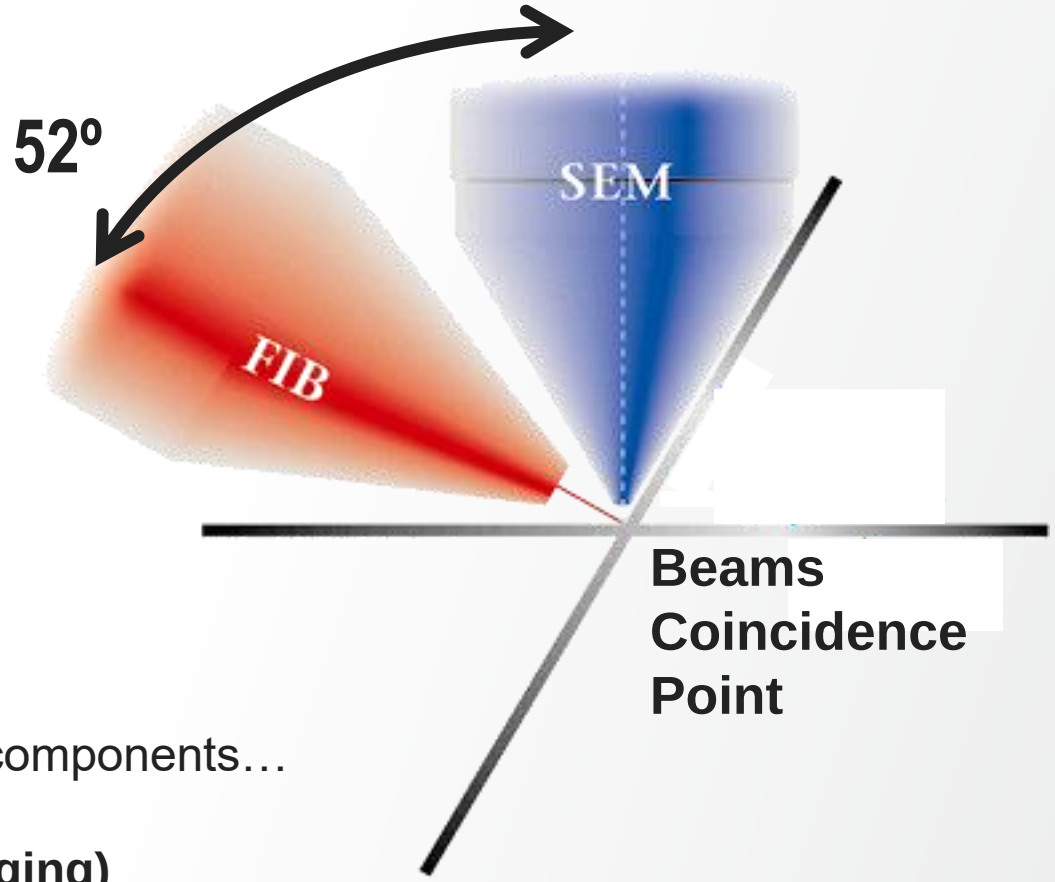
		Winning factor	Resolution	Low Vacuum	ChemiSEM	Automated Alignments & Settings	SE, BSE, STEM, CL, EDS, EBSD, Heating, Maps, Autoscript
Axia		Easy to use and maintain	3 nm @ 30 kV 8 nm @ 3 kV	Optional Or Prisma E	✓	✓	✓
Quattro		High resolution of any sample	1 nm @ 30 kV 3 nm @ 1 kV	ESEM	✓	✓	✓
Apreo		Excellent low-voltage contrast & hi-res analytics	0.8 nm @ 1 kV	Optional	✓	✓	✓
Verios		Nanoscale characterization	0.7 nm @ 1 kV	-		✓	✓

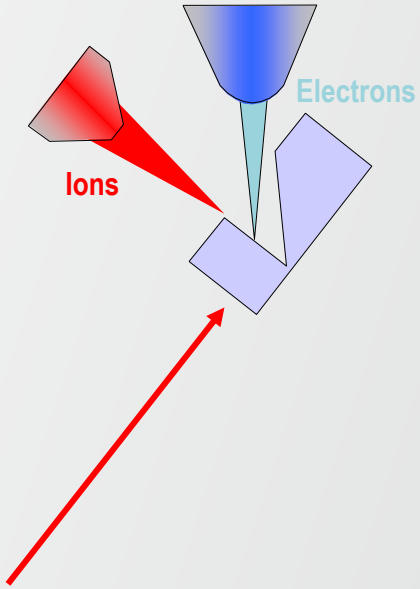
Contents



What is DualBeam (FIB-SEM)?





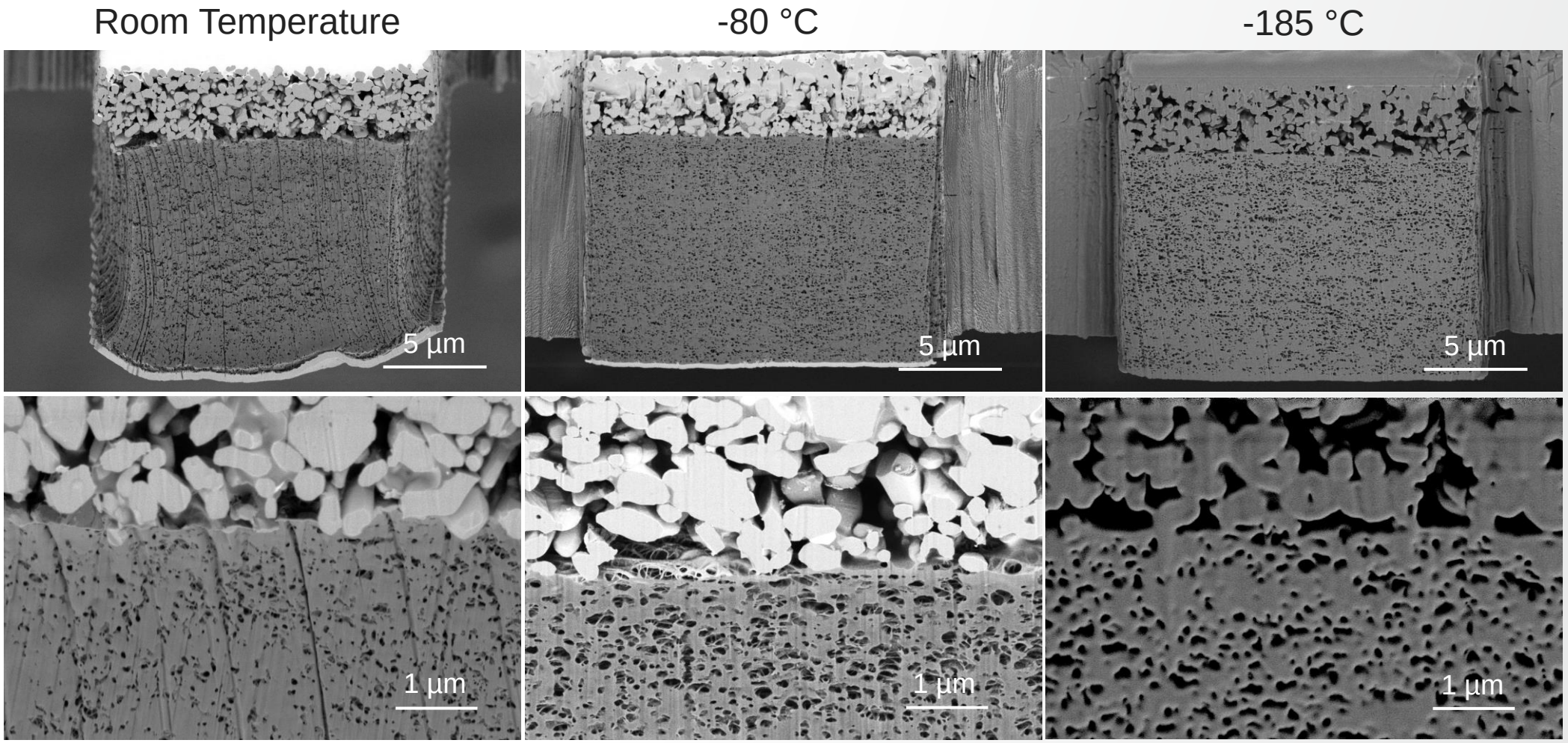


A DualBeam is defined by of two primary components…

Scanning Electron Microscope (for imaging)
+
Focused Ion Beam (milling)

The sample and stage are maneuvered beneath the beams to optimize imaging and milling

2D Cross-section: Separator Analysis

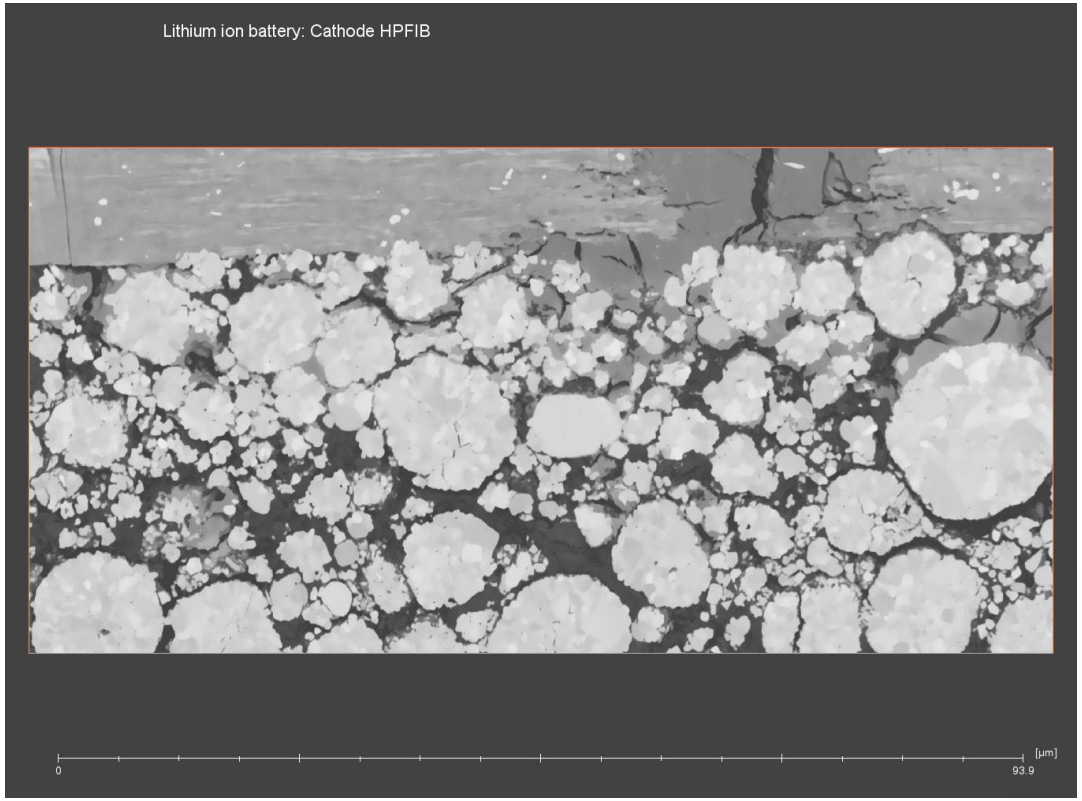


- Optimized FIB milling and imaging temperature at -80 ˚ C
  - Pore structures are well preserved
  - Clear contrast among separator, pore, alumina particles and binder

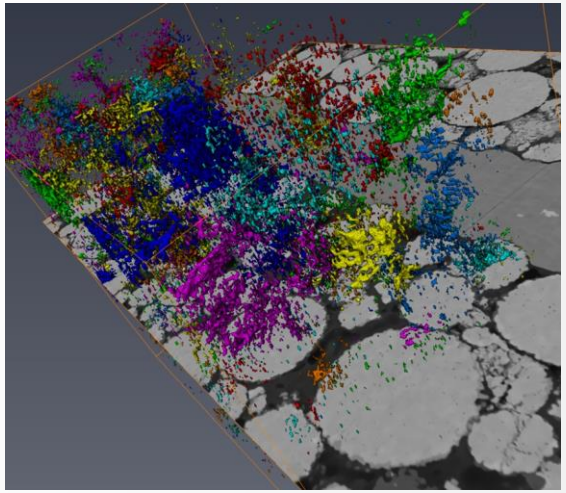
Courtesy: Devin Wu, Thermo Fisher

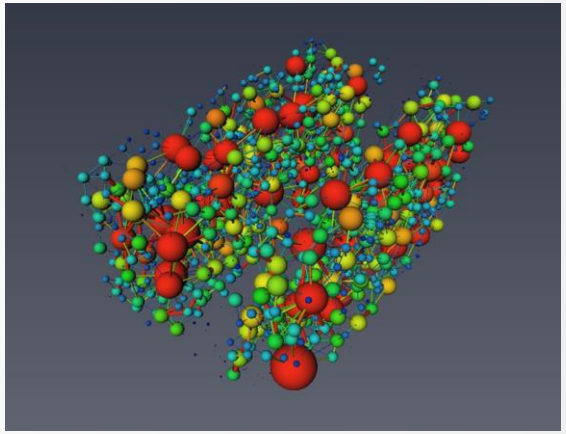
3D Imaging Analysis on NMC cathode via PFIB

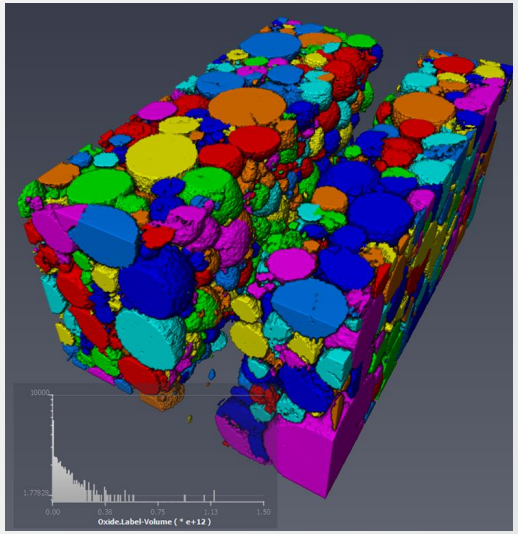
3D Imaging via Auto Slice & View



Structural Quantification via Avizo

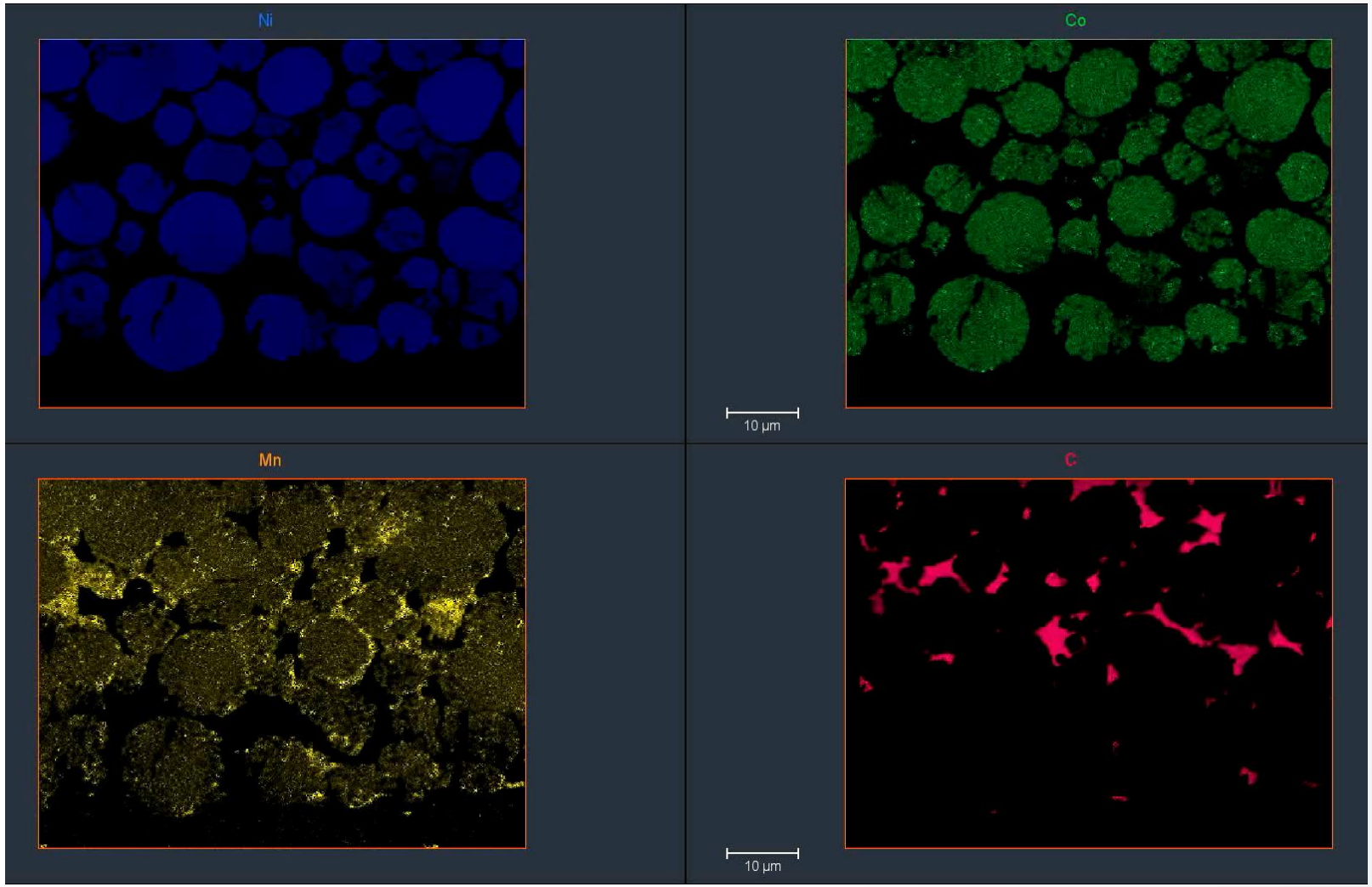






Porosity and impregnation assessment

LiNiCoMnO (811) EDS3 slice movie

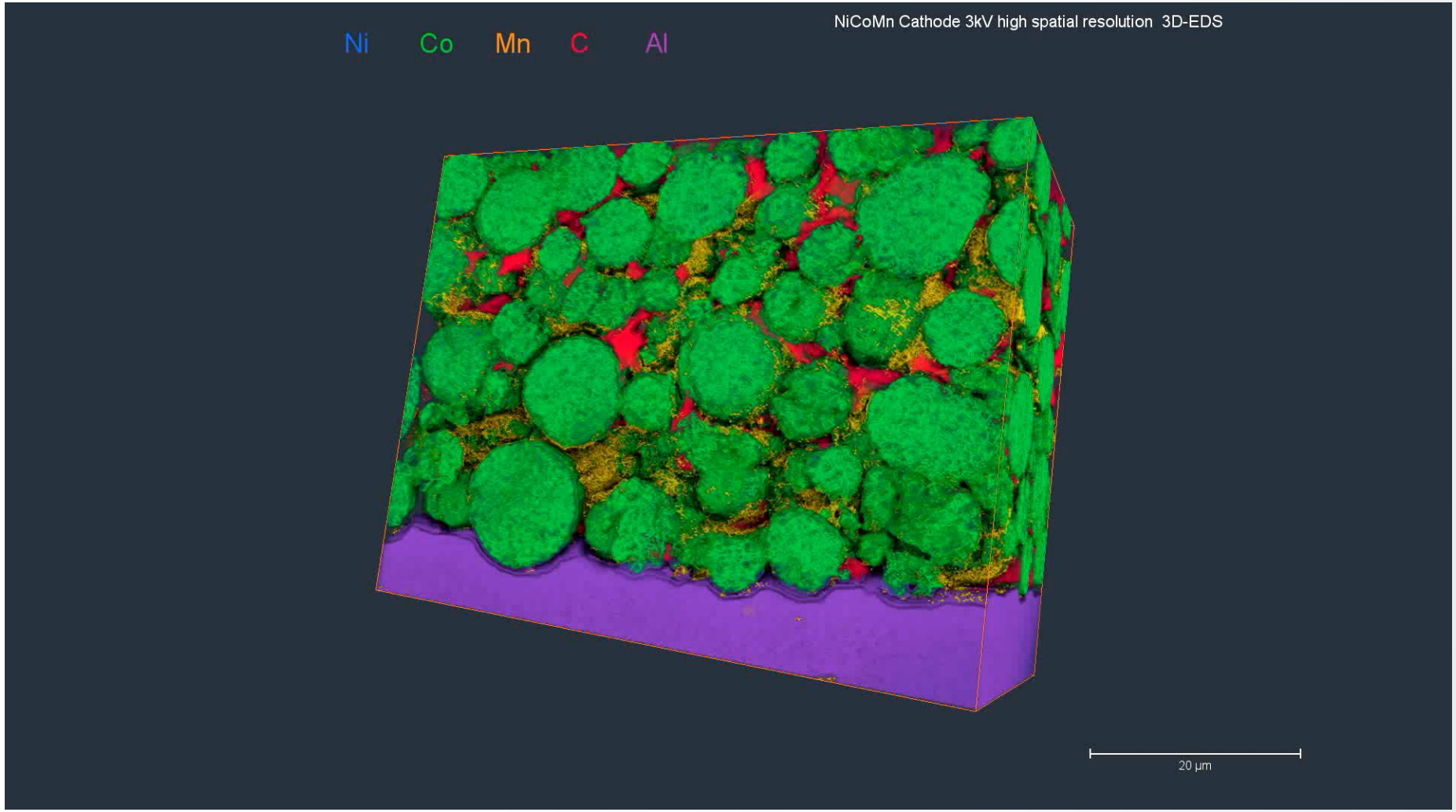


- The ratio of nickel to cobalt manganese 8:1:1 is the development trend. The cathode particles should be doped and coated to increase the charge and discharge cycle life of the battery.

Data acquired: Devin Wu

Porosity and impregnation assessment

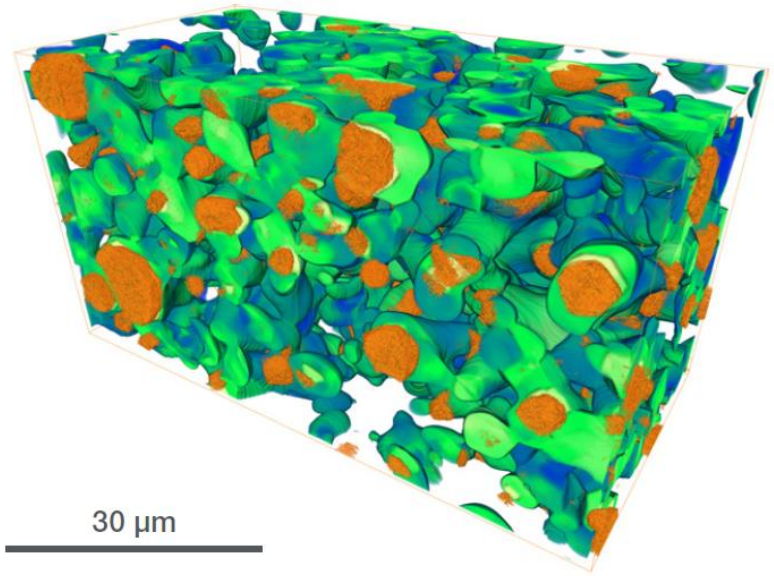
LiNiCoMnO (811) EDS3



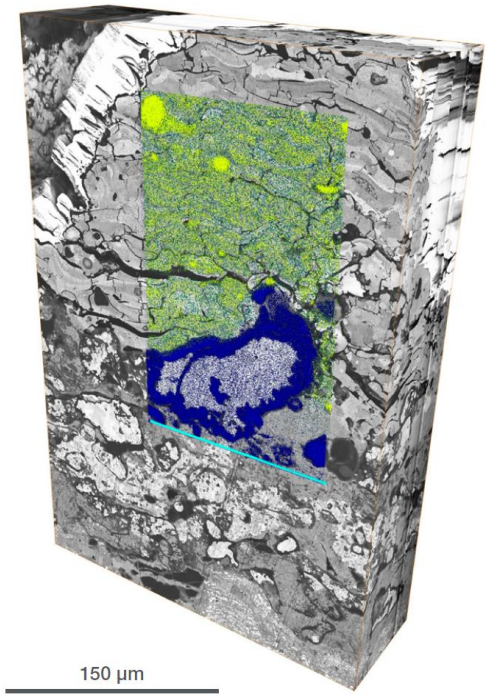
Data acquired: Devin Wu

What can we do with a Dual Beam ?

→ Structural analysis – 3D reconstruction

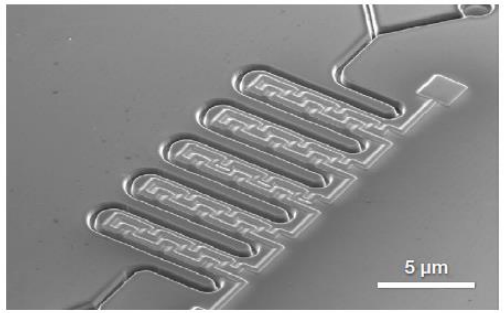


3D reconstruction of W-Mo-Cu sample using a combination of backscattered electron (green-blue) and energy dispersive X-ray (orange) data. Generated using a Scios DualBeam, Auto Slice and View Software, and visualized with Avizo Software.

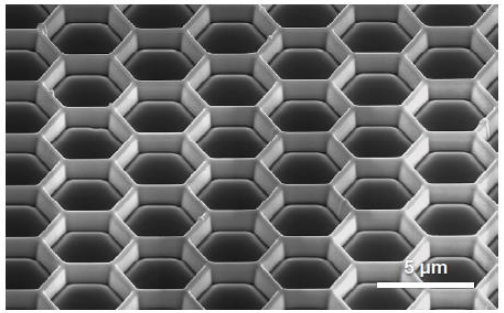


3D reconstruction of thermal barrier coating from an afterburner nozzle of a ramjet aero engine showing the microstructure state at end-of-life of the engine exhaust system. Overlaid EDS map shows elemental distribution on the top coat and bond coat interface: blue is aluminum, yellow is magnesium, and turquoise is yttria. The data have been produced with a Thermo Scientific Helios™ 5 PFIB, Auto Slice & View 5 Software and Avizo Software for Materials Science.

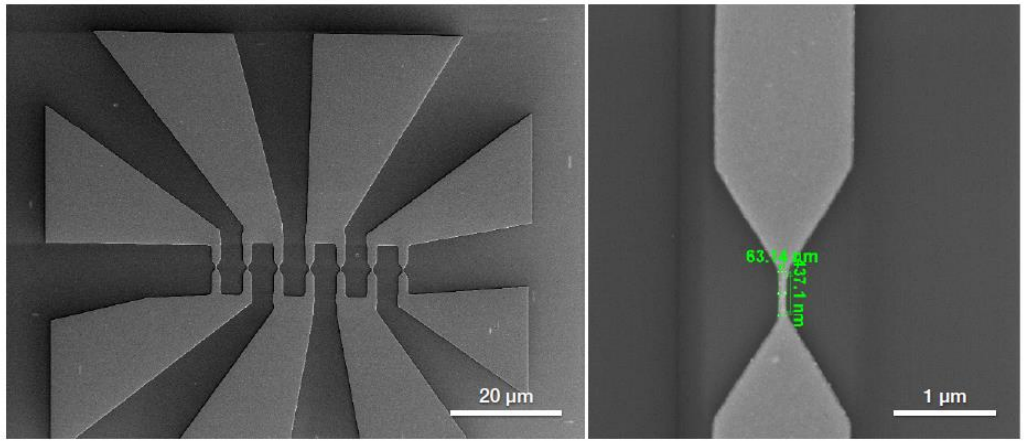
→ Nanoprototyping



A nano-fluidic system made with a multi-layered pattern in NanoBuilder Software. Each layer may consist of a deposition or milling sequence to selectively add or remove material.



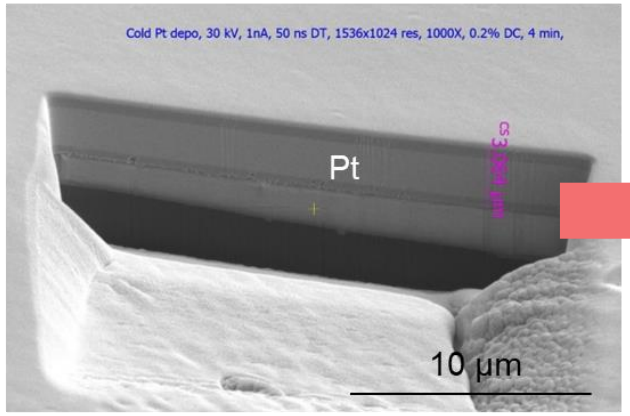
Secondary electron image of a honeycomb structure milled into a silicon substrate using a focused ion beam.



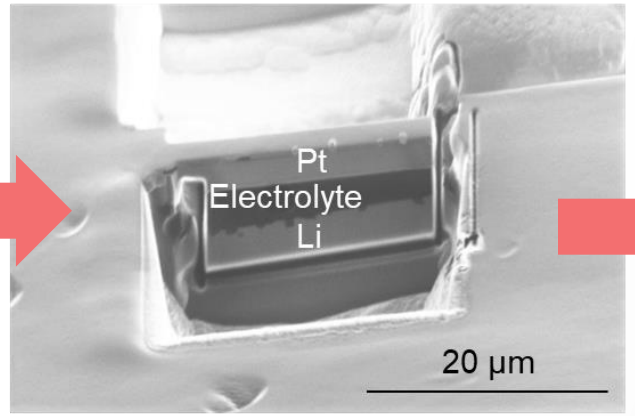
Left: Electromigration structure created with NanoBuilder Software. Right: Close up of a single channel. Critical dimensions as low as 10 nm are possible.

TEM Sample Preparation:
Cryo Lift-out Process on a Li-metal Battery

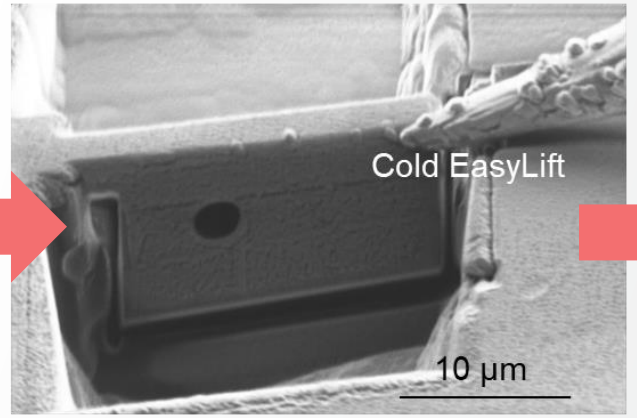
Cold Pt deposition and milling



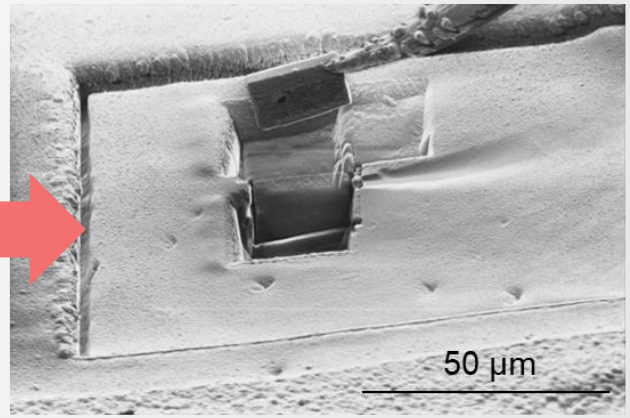
J-cut



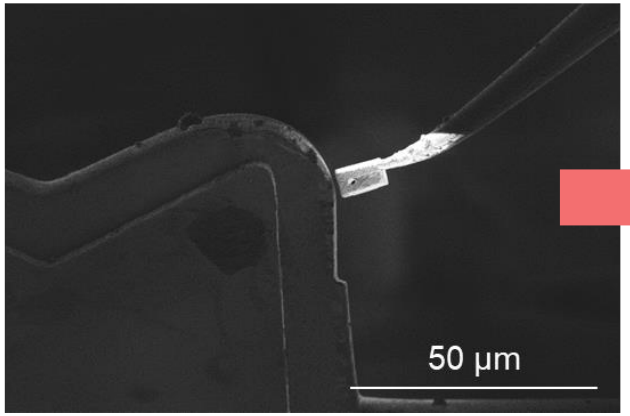
EasyLift attach



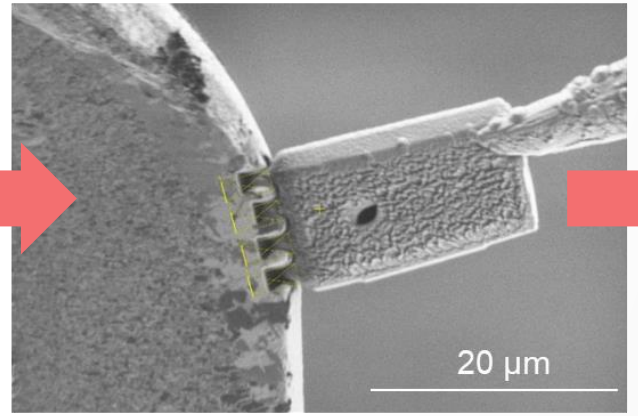
Lamella pull out



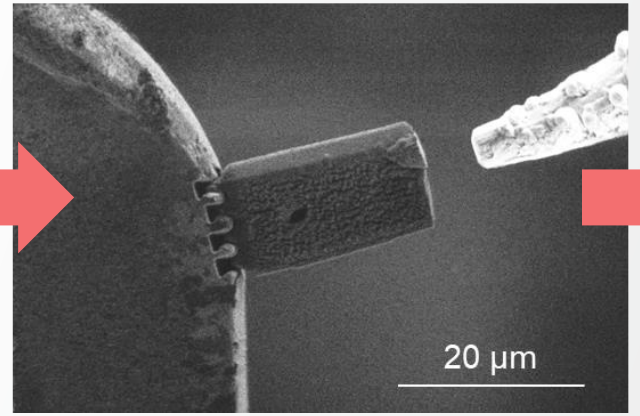
Cold lamella to grid



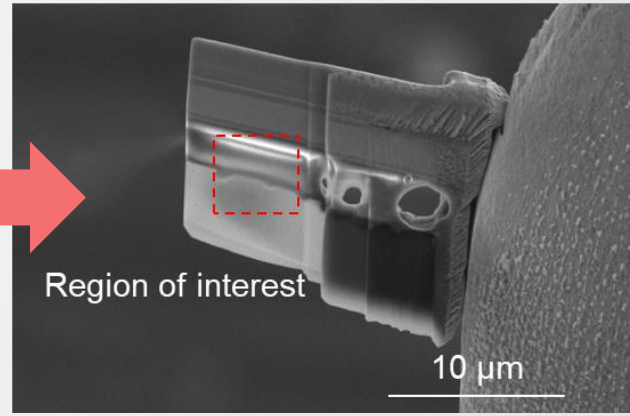
GIS free attach



EasyLift cut free

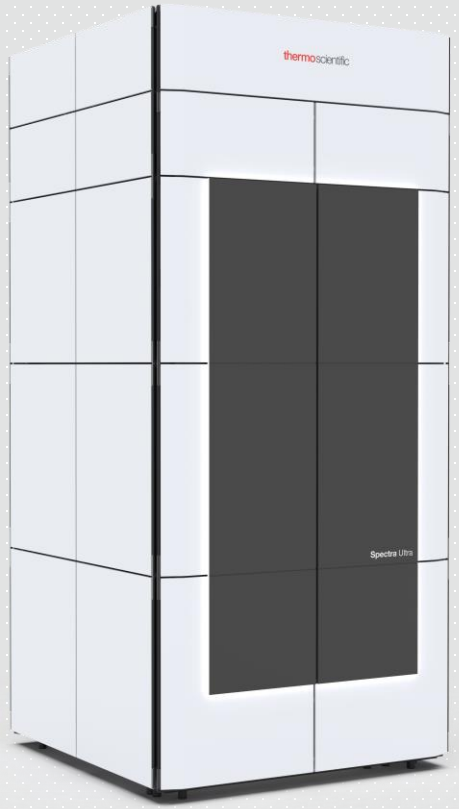


Further thinning at 100 pA



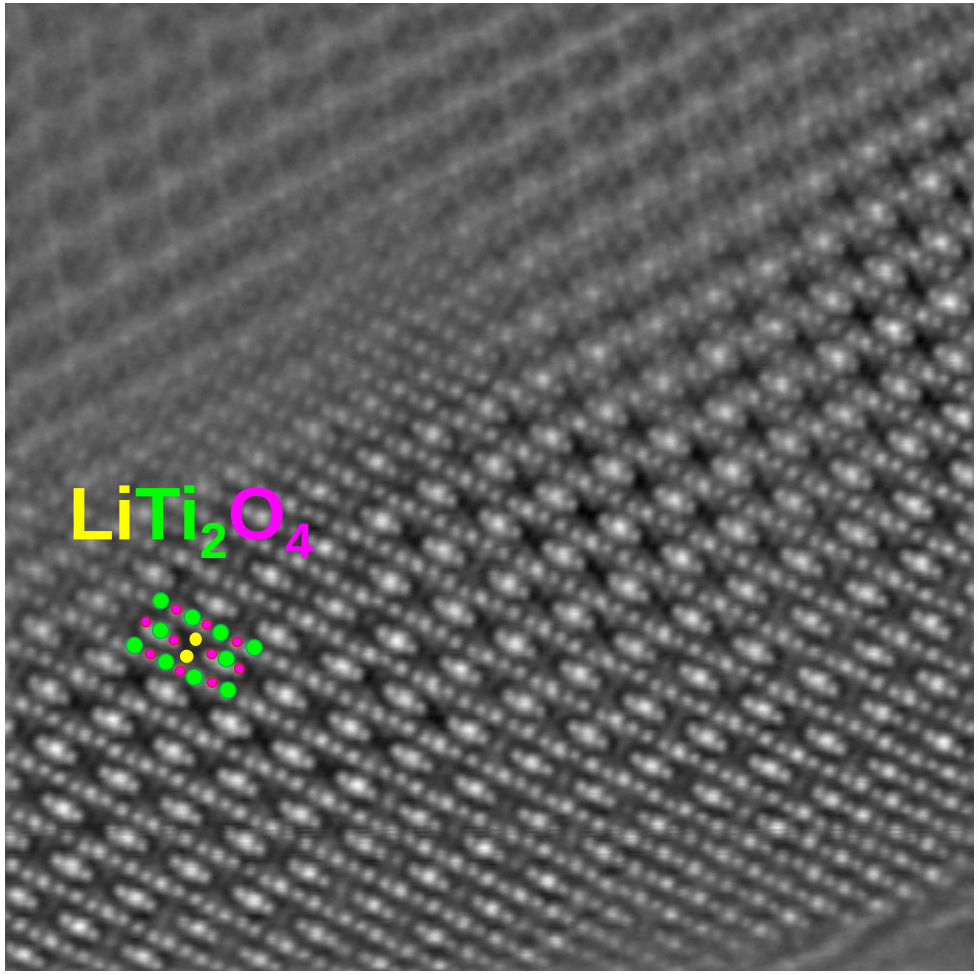
Courtesy: HBO NNP, Thermo Fisher

Contents

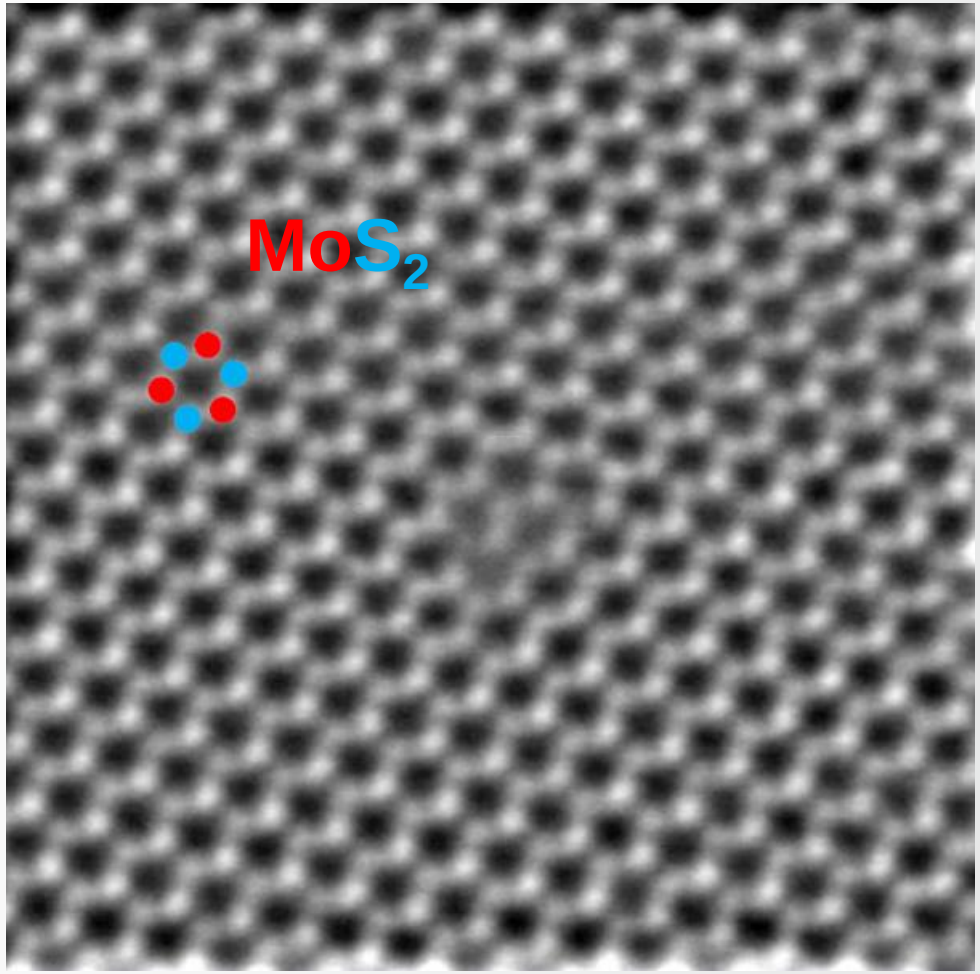


Imaging analysis of battery materials via TEM: iDPC

$LiTi_2O_4$ iDPC @ 300kV

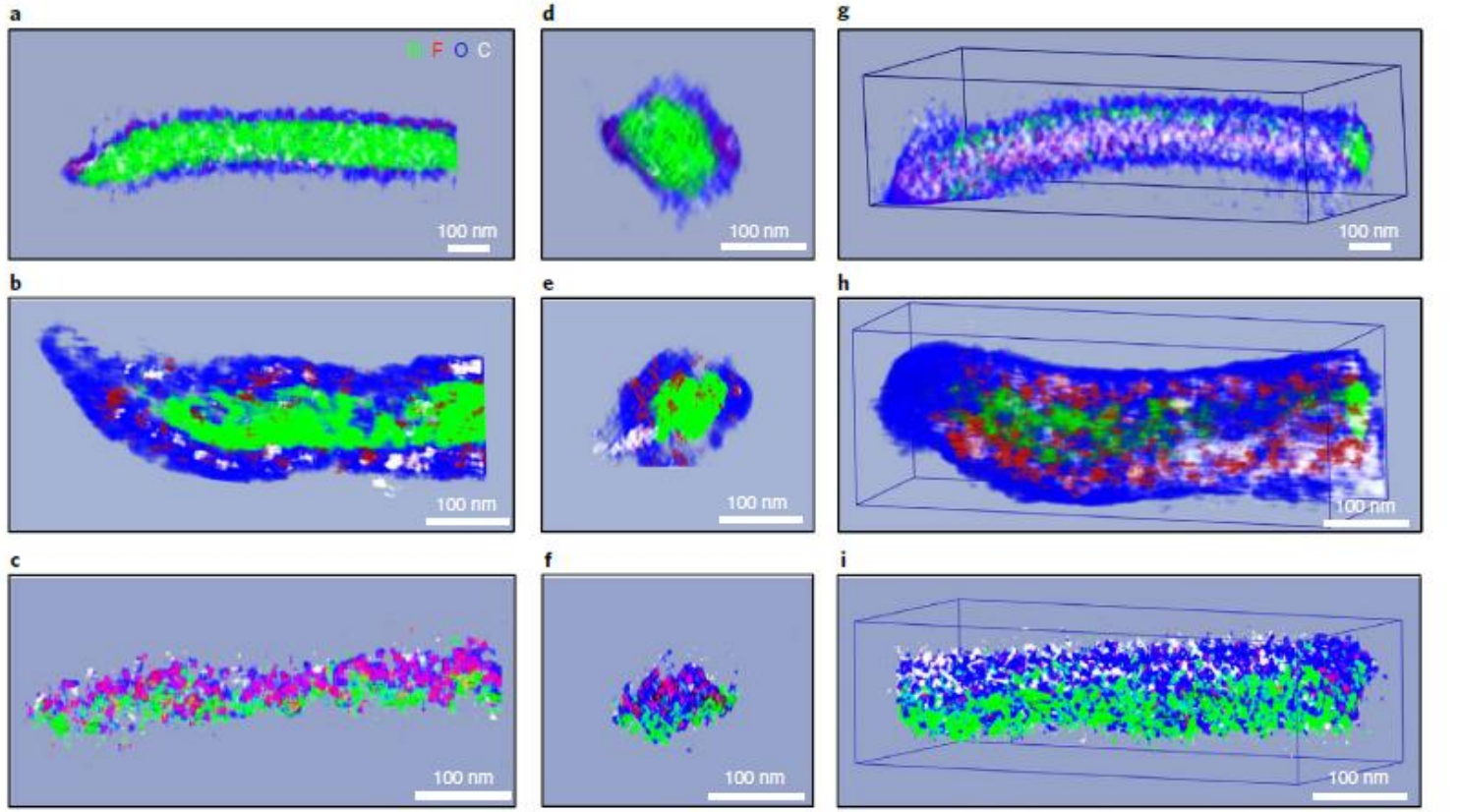


MoS_2 bi-layer iDPC @ 30kV



Sample Courtesy: Emrah Yucelen

Capacity fading due to SEI growth into the anode, studied in a Transmission Electron Microscope



nature nanotechnology
ARTICLES
https://doi.org/10.1038/s41565-021-00947-8
Check for updates

Progressive growth of the solid-electrolyte interphase towards the Si anode interior causes capacity fading

Pacific Northwest NATIONAL LABORATORY

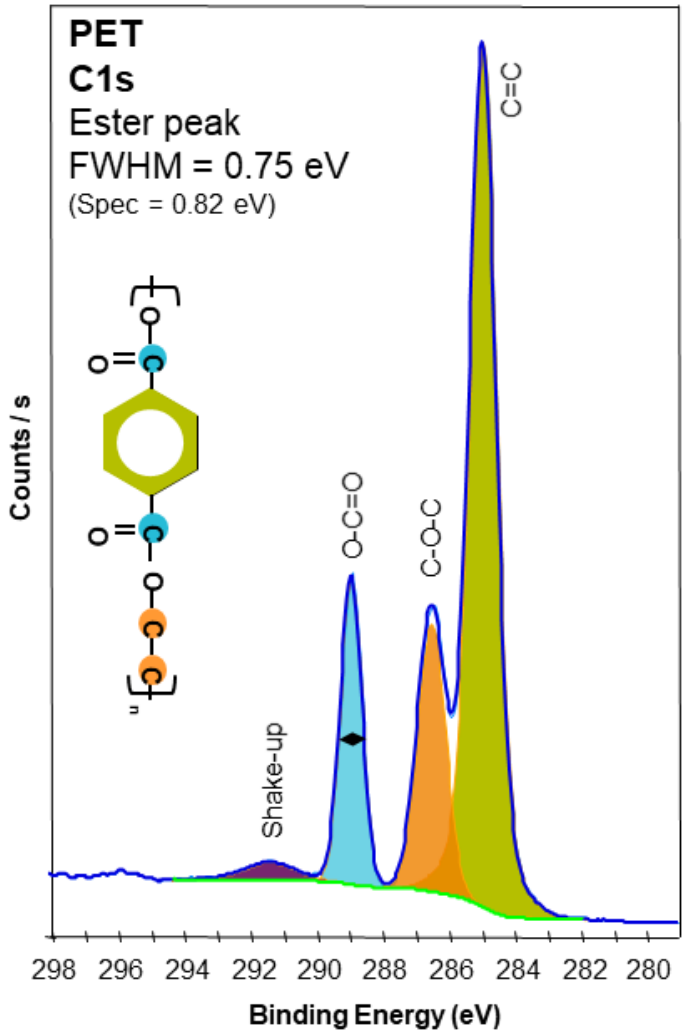
TEM

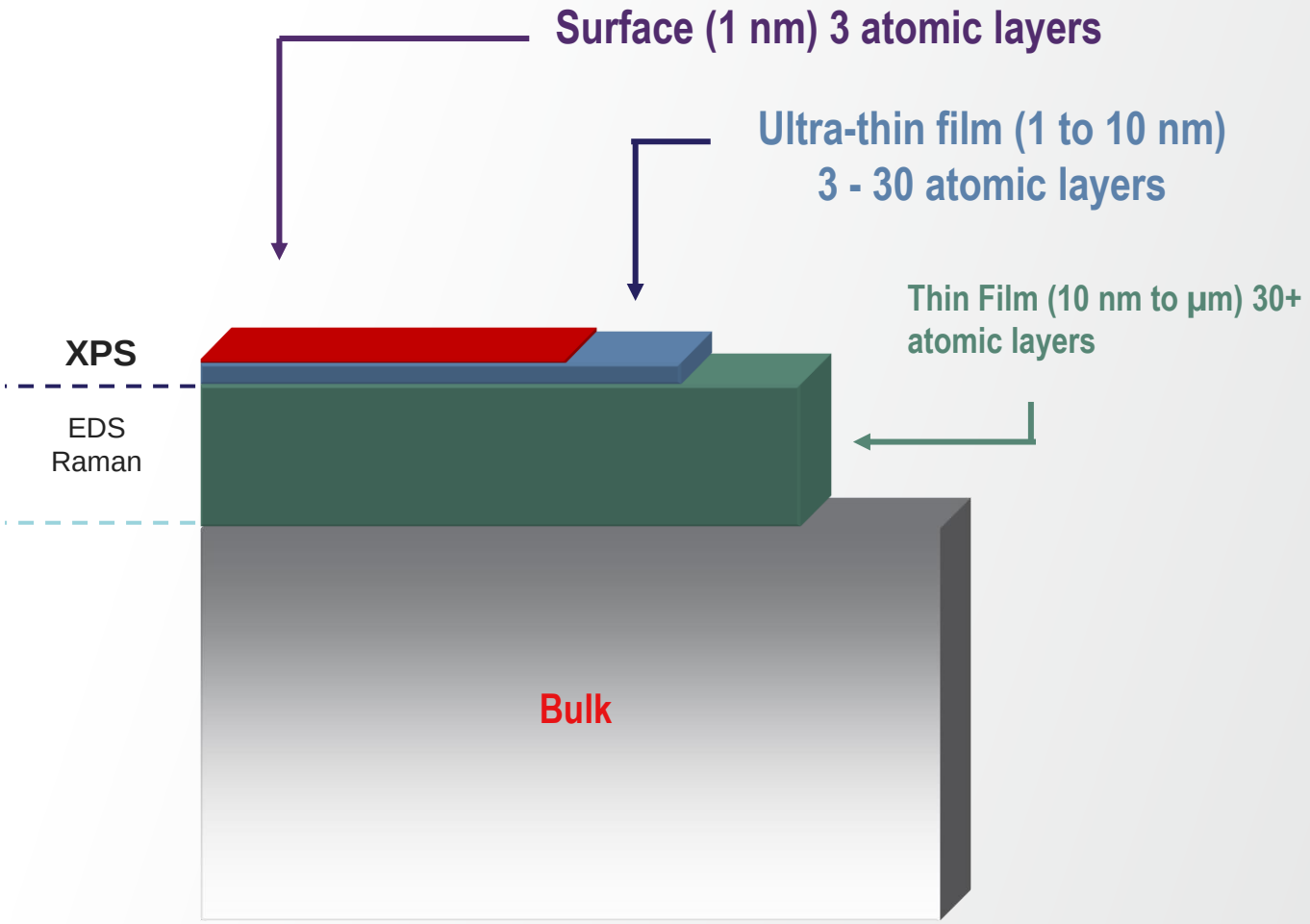
Cryo-STEM-EDS 3D Tomography to probe the SEI growth mechanism on Si-anode

In collaboration Dr. Chongmin Wang, PNNL

Contents

XPS: Elemental & Chemical State Analysis of Surfaces

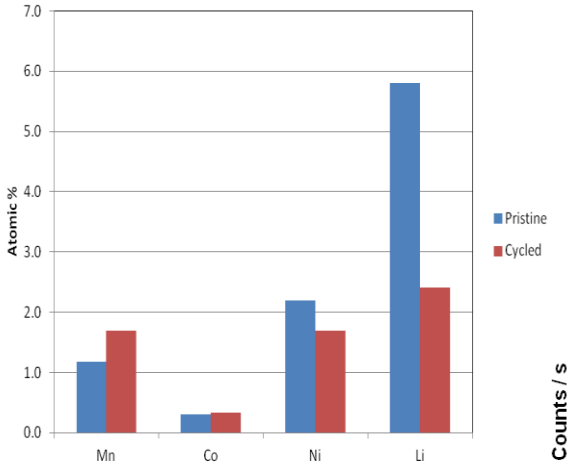




Note: Approximate layer thickness only. Actual values depend upon materials

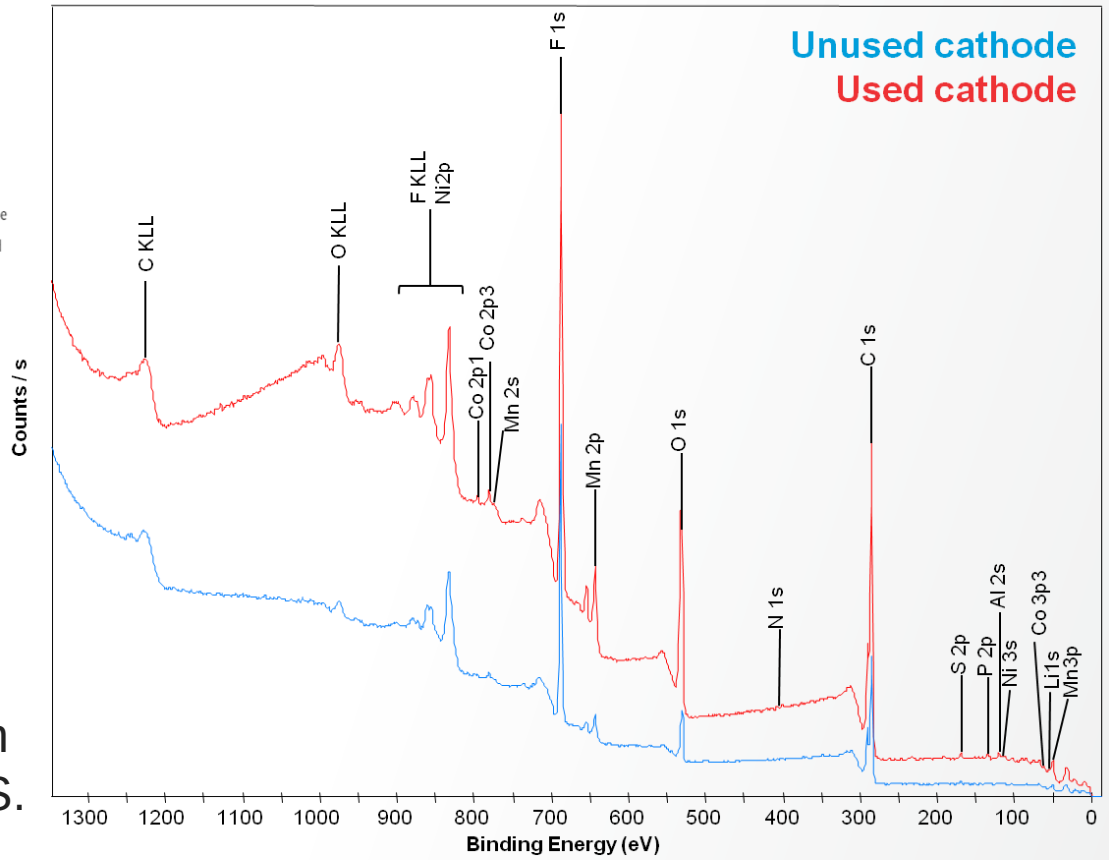
High Sensitivity XPS: Vital for Lithium Detection

- NexsaG2 has incredibly high sensitivity (6.5 million counts/s) with low X-ray power
  - Lithium is an element hard to detect, and surfaces could easily be damaged with X-rays



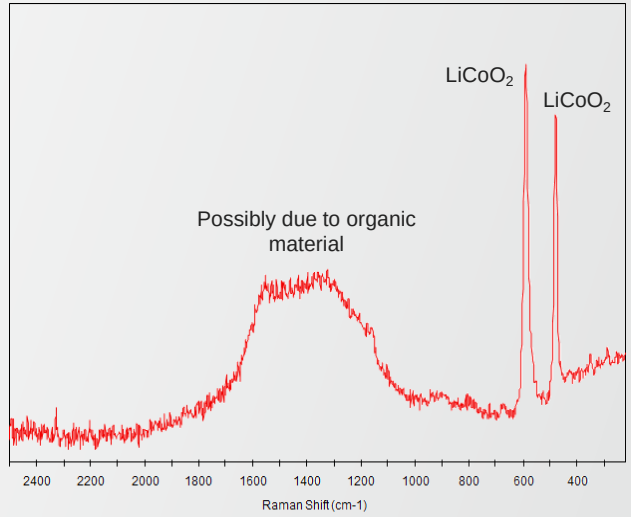
Comparison of pristine and cycled cathode material.

Variation in Li concentration is clearly evidenced by XPS.

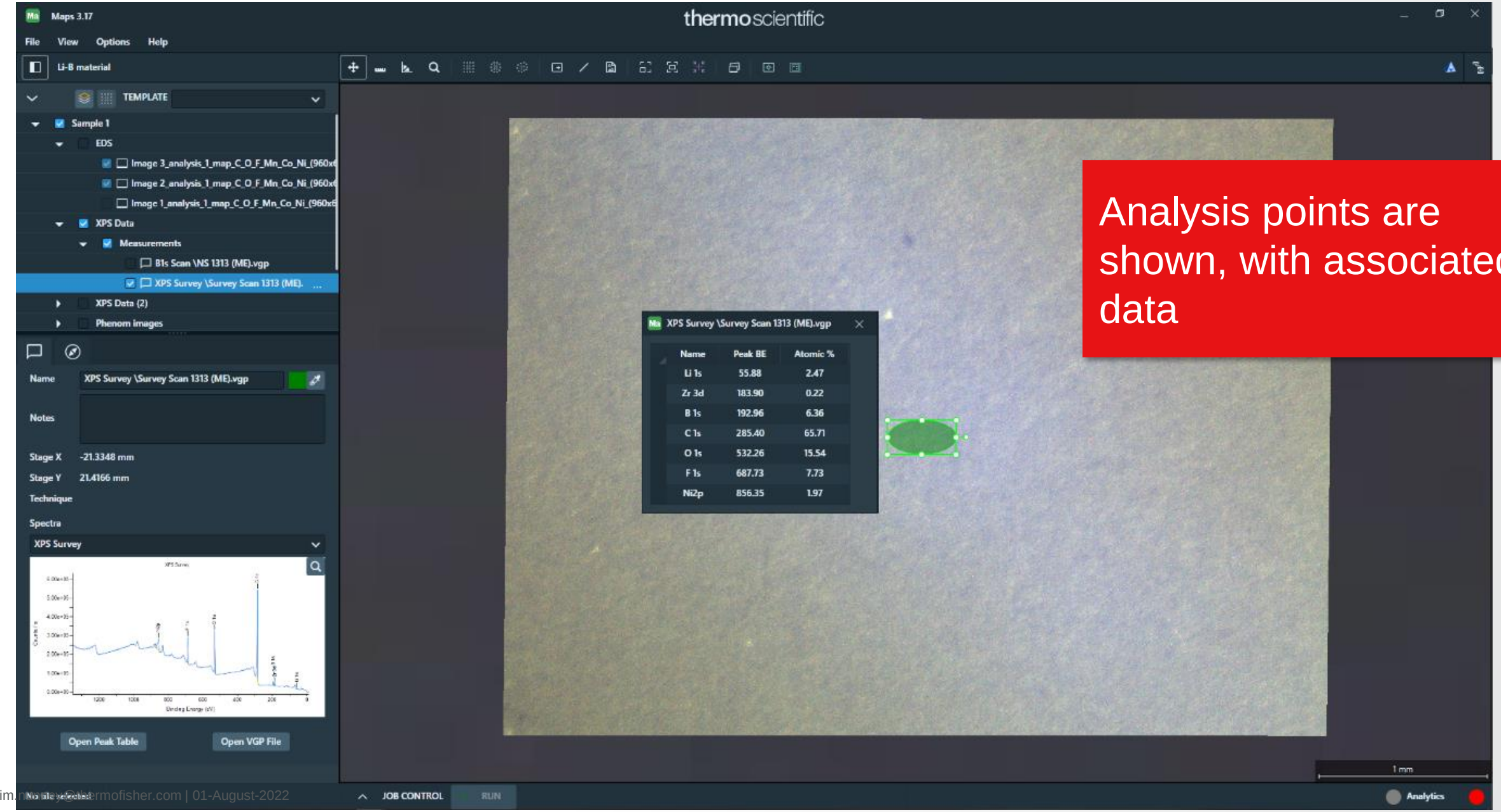


Co-incident Raman on NexsaG2 allows for bulk structural analysis of cathode material!

785nm Raman spectrum of $LiCoO_2$ cathode

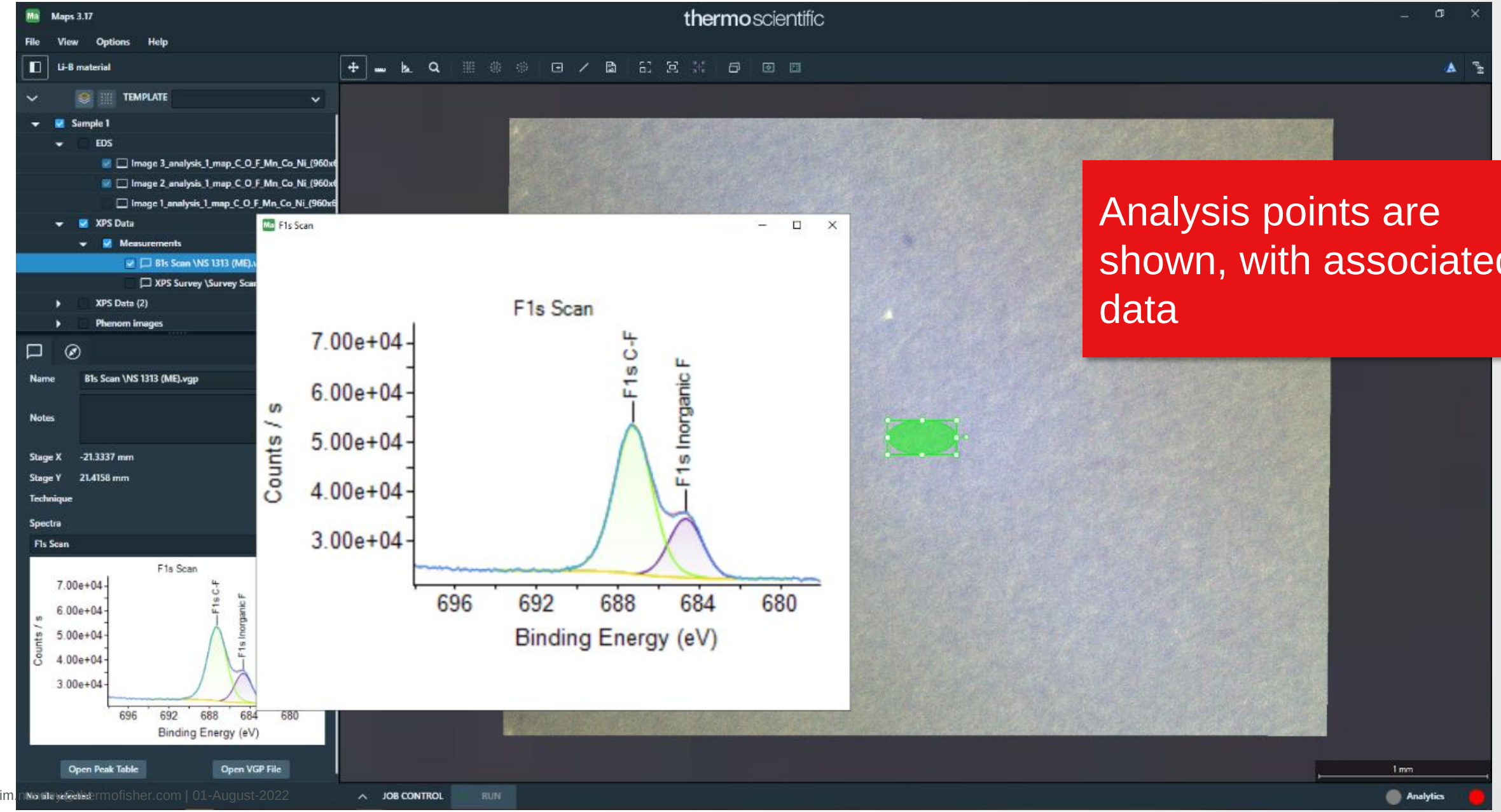


CISA: Correlative Microscopy → XPS x SEM



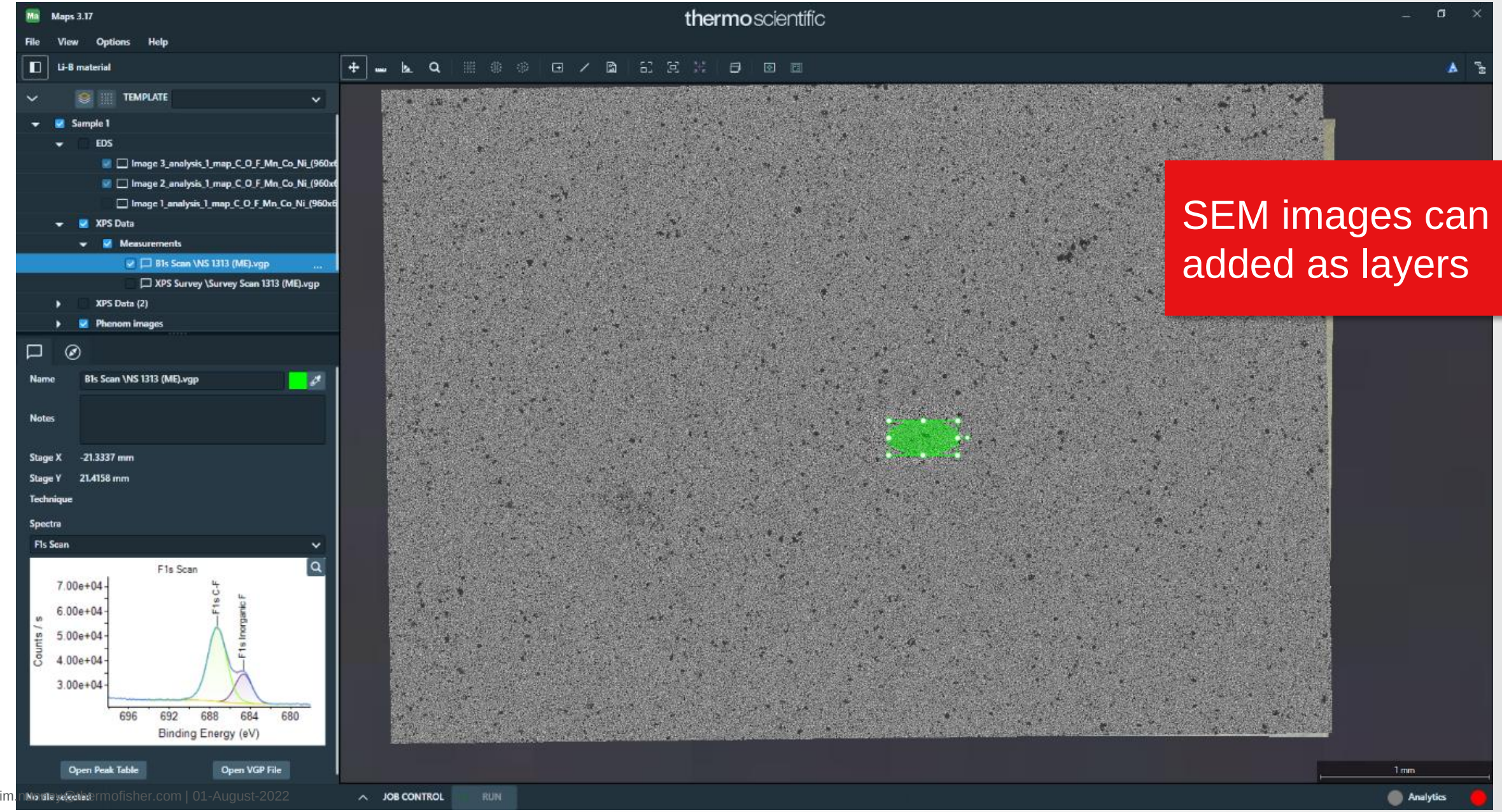
Analysis points are shown, with associated data

Example: Li-ion battery cathode



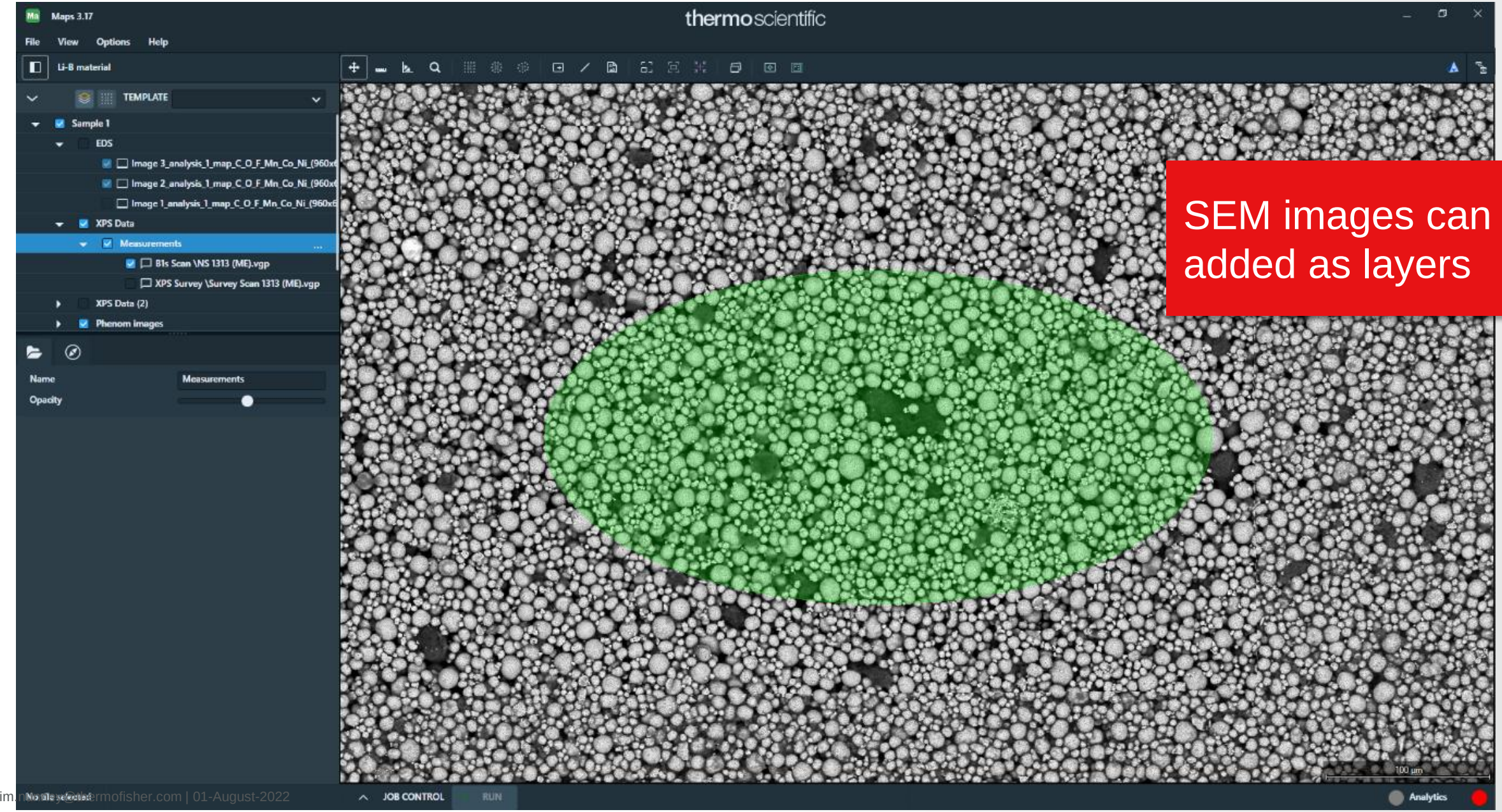
Analysis points are shown, with associated data

Example: Li-ion battery cathode



SEM images can be added as layers

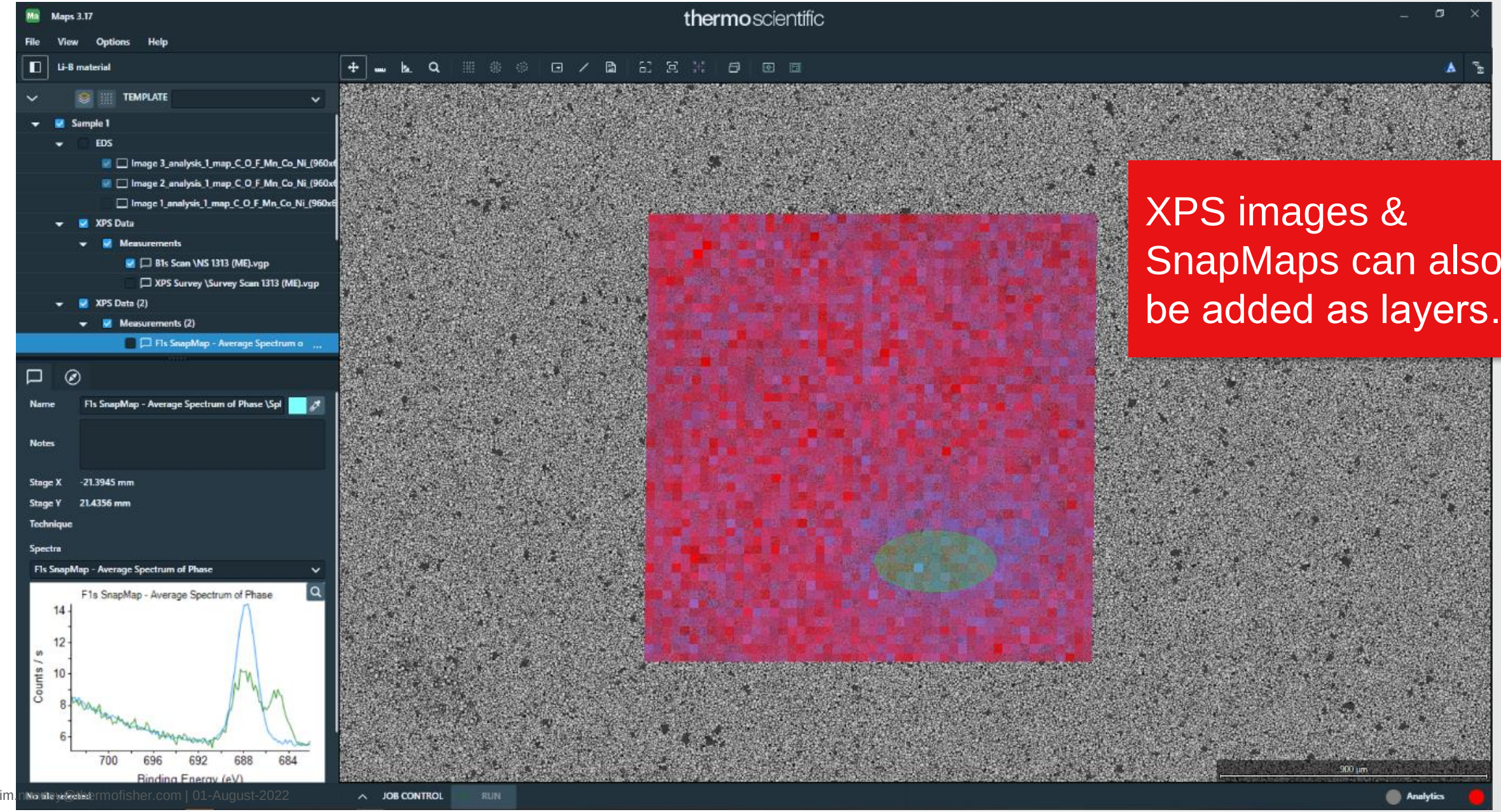
Example: Li-ion battery cathode



SEM images can be added as layers

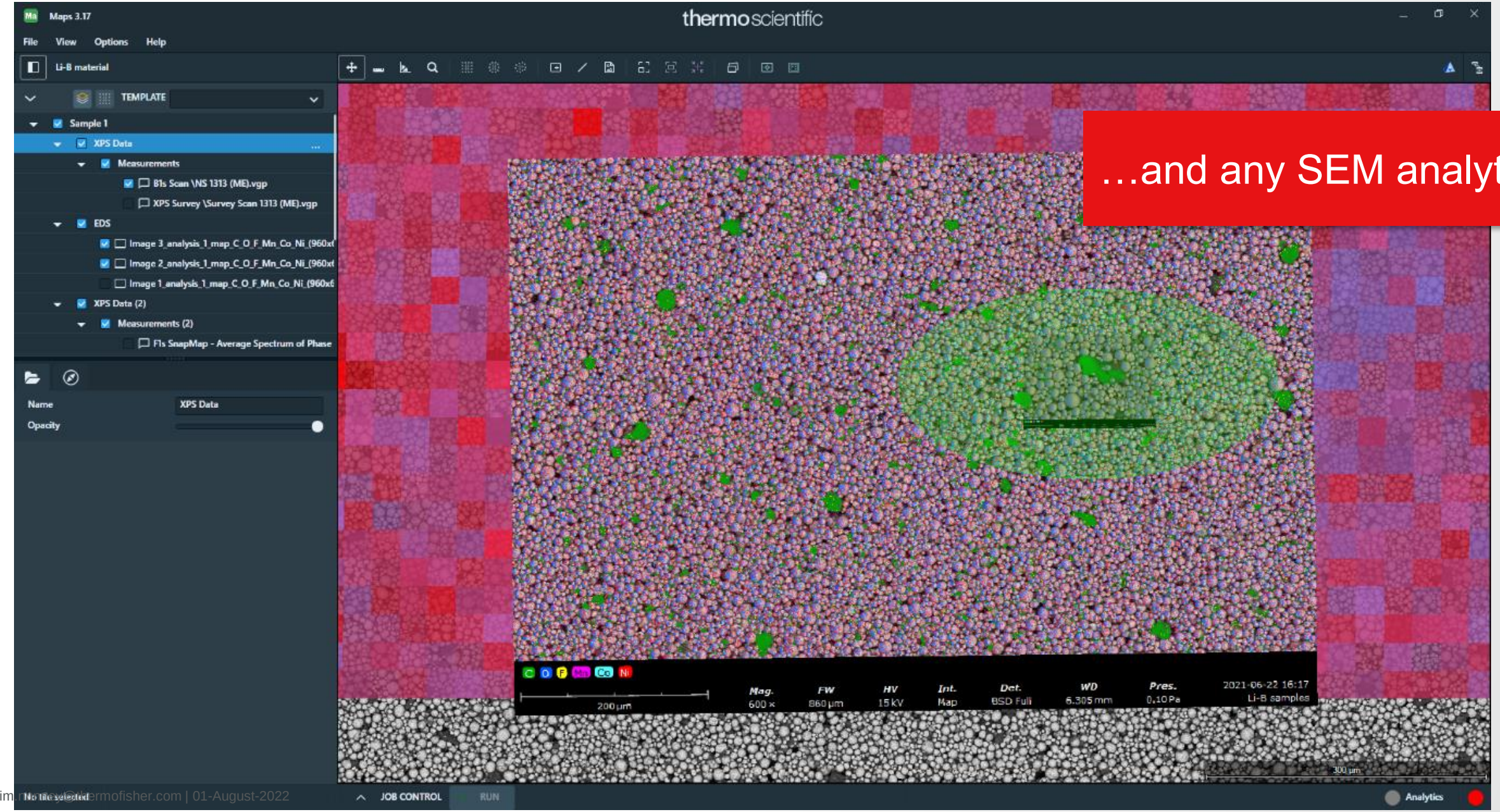
tim.nunney@thermofisher.com | 01-August-2022

Example: Li-ion battery cathode



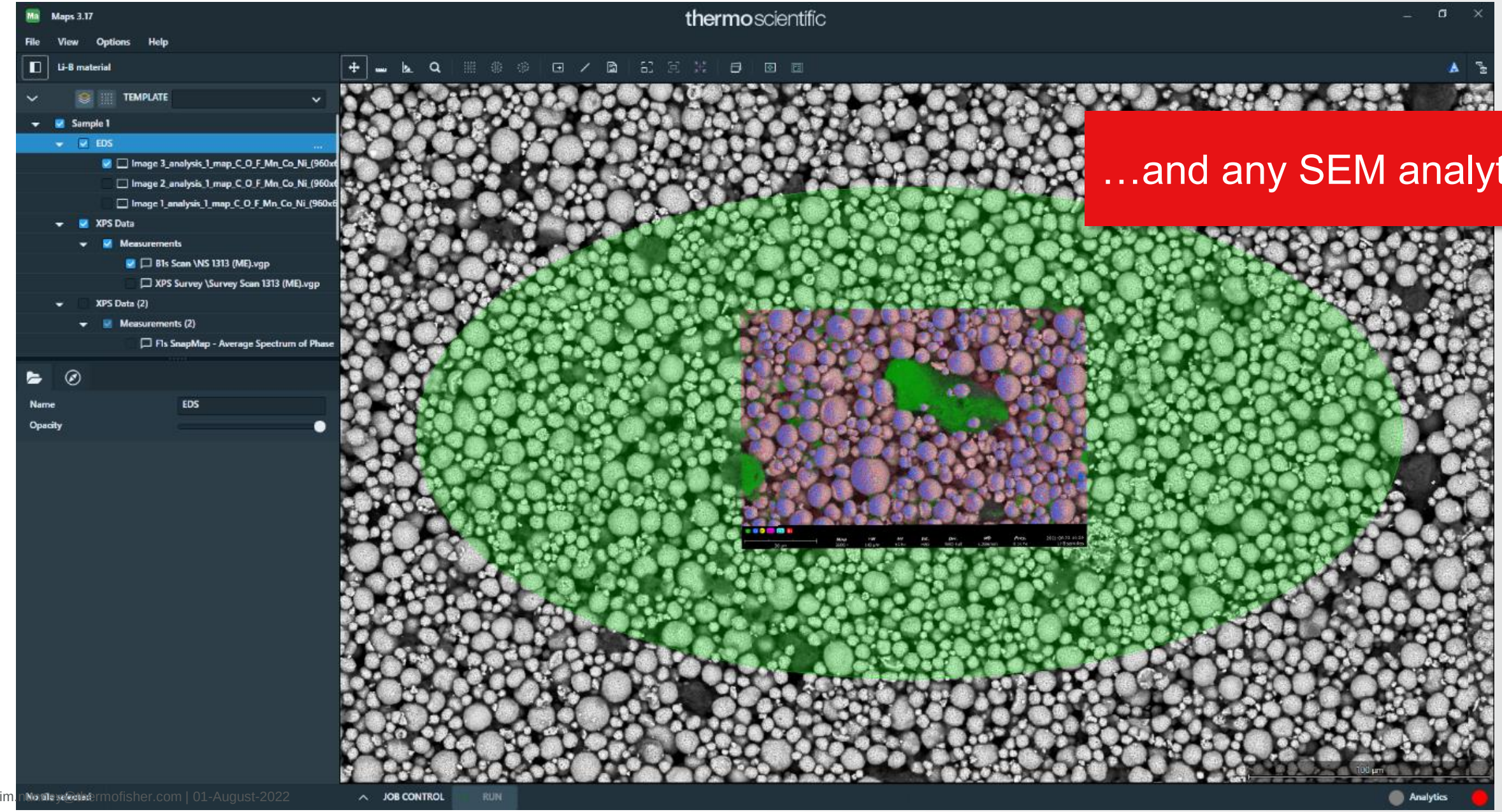
XPS images & SnapMaps can also be added as layers…

Example: Li-ion battery cathode



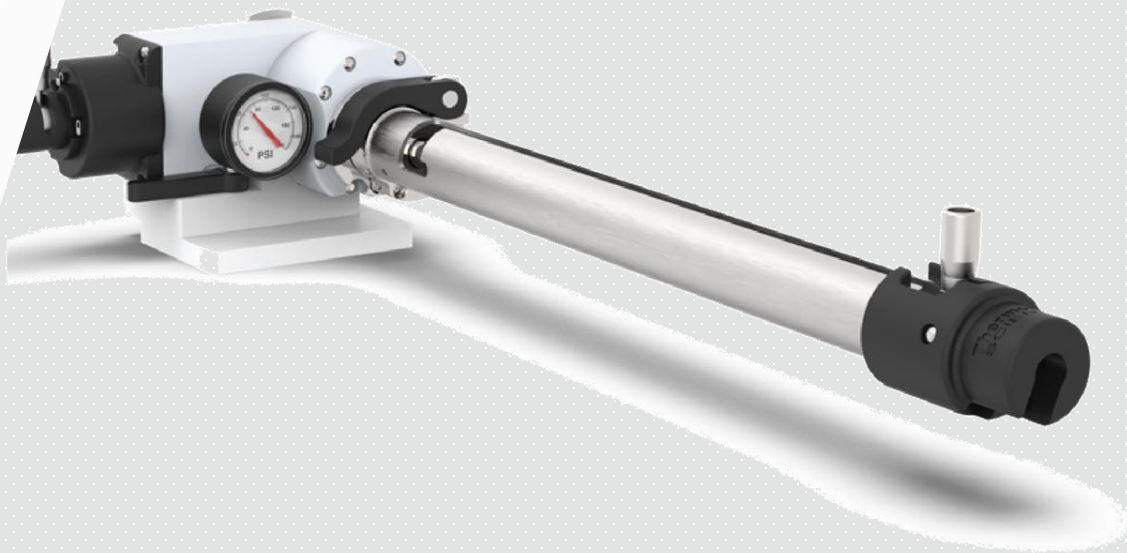
…and any SEM analytics

Example: Li-ion battery cathode

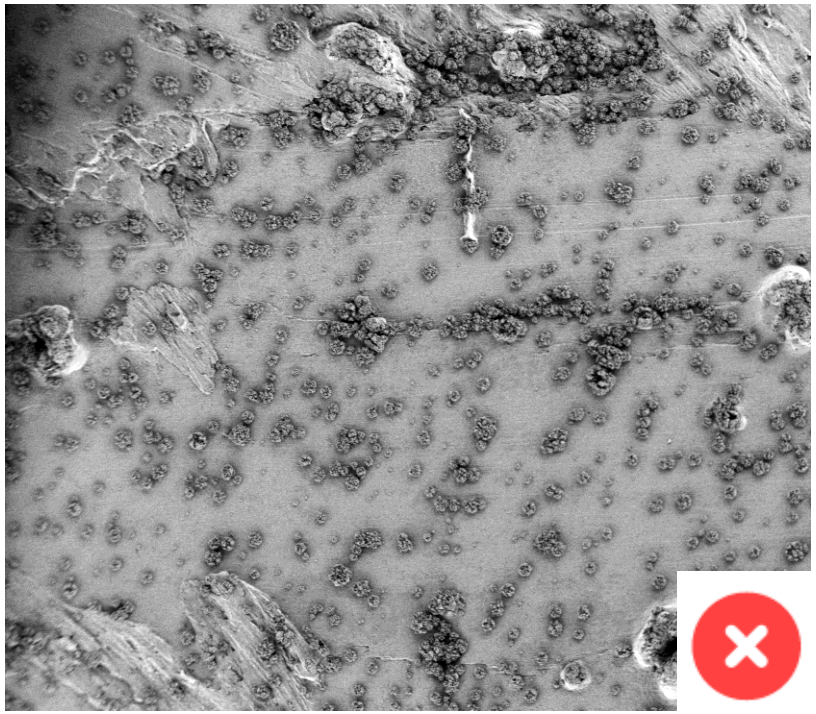


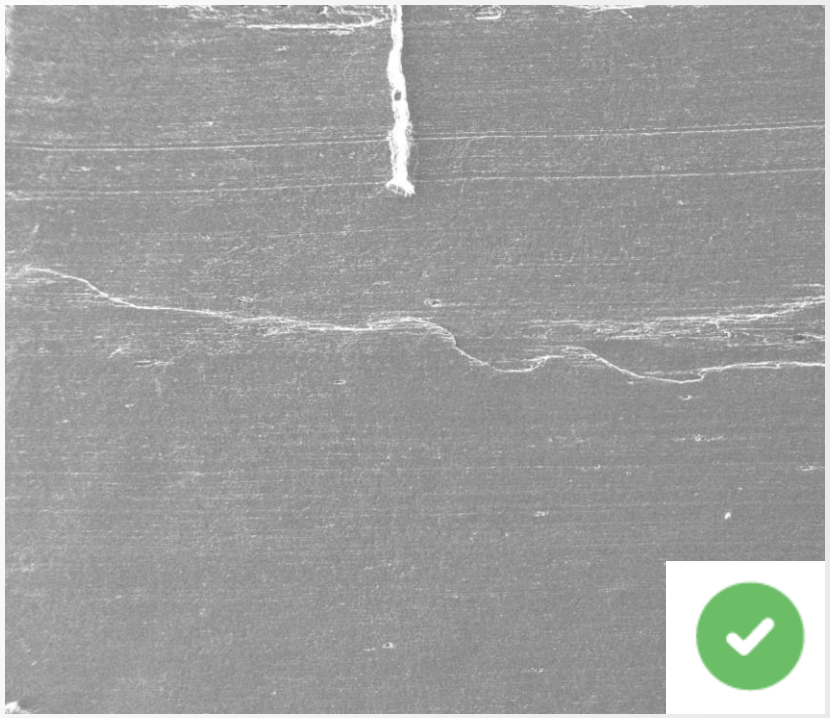
…and any SEM analytics

Contents



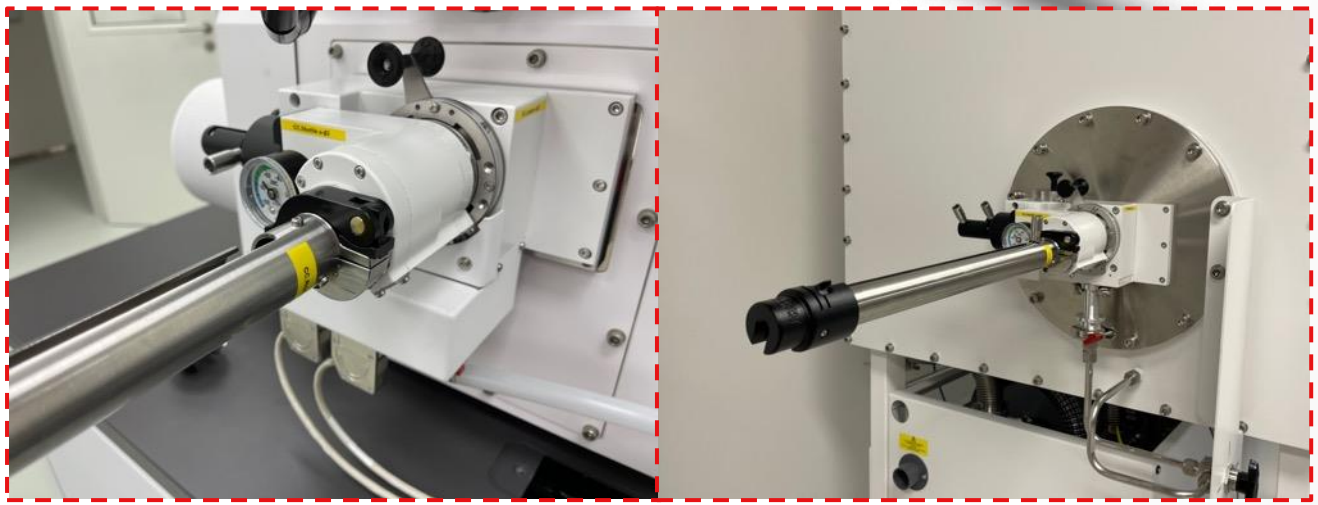
How to observe the sample at its native state in the **electron microscope**?





We want to observe
initial state → Inert Gas Condition

CleanConnect™ key benefits



Microscope side

Glovebox side

Key Benefits

Sample integrity is preserved by the use of inert gas sample transfer

Seamless, automatic integration to a variety of Thermo Scientific SEM/DualBeams

Compatible with Cryo-stage with no any changes in system or microscope

Ergonomic and modular design enables uncomplicated sample handling

Compatible with most glove box systems and available with optional direct glove box connectivity

Thermo Scientific Inert Gas Sample Transfer workflow

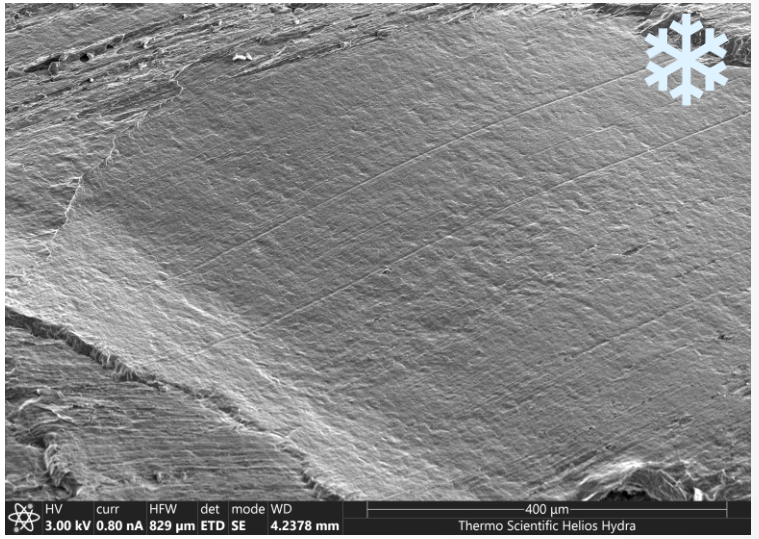
That's the theory, but in practice…?

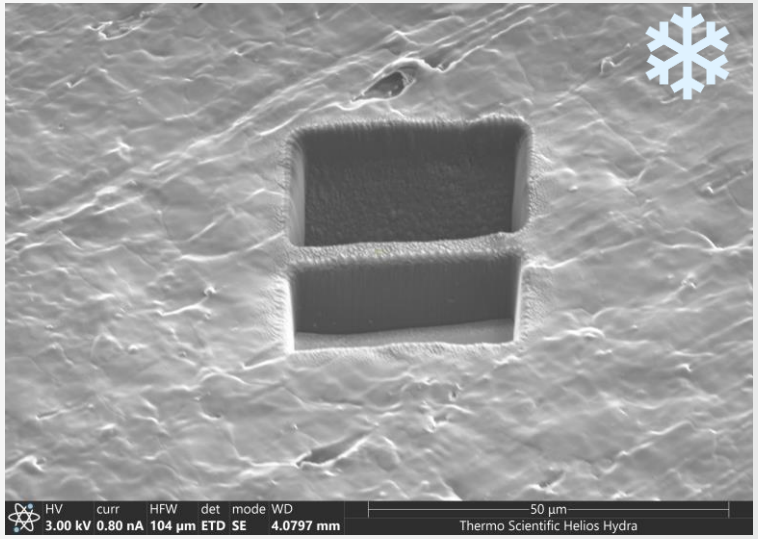
Application of IGST workflow on air-sensitive material

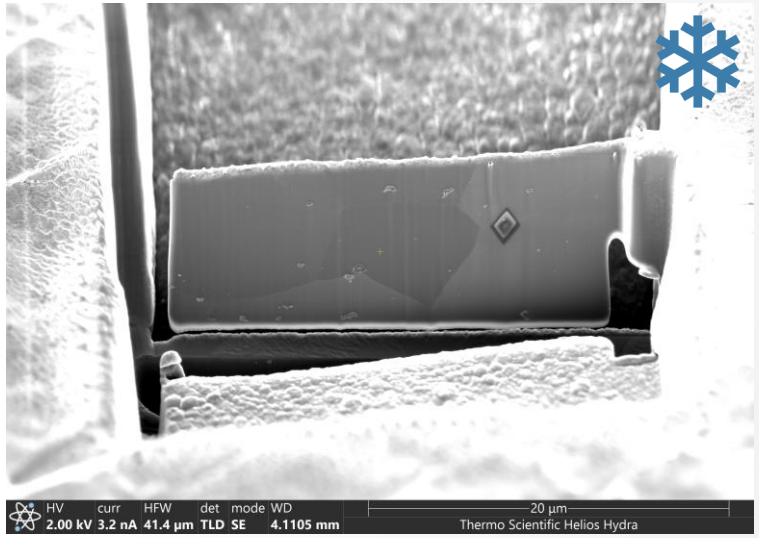
Proof Material #3: Li-metal lamellae preparation (Ar^+ ion) and imaging

Helios Hydra PFIB

Whole process under cryogenic condition at -178 ˚C







Lamellae not fine enough for SEM observation

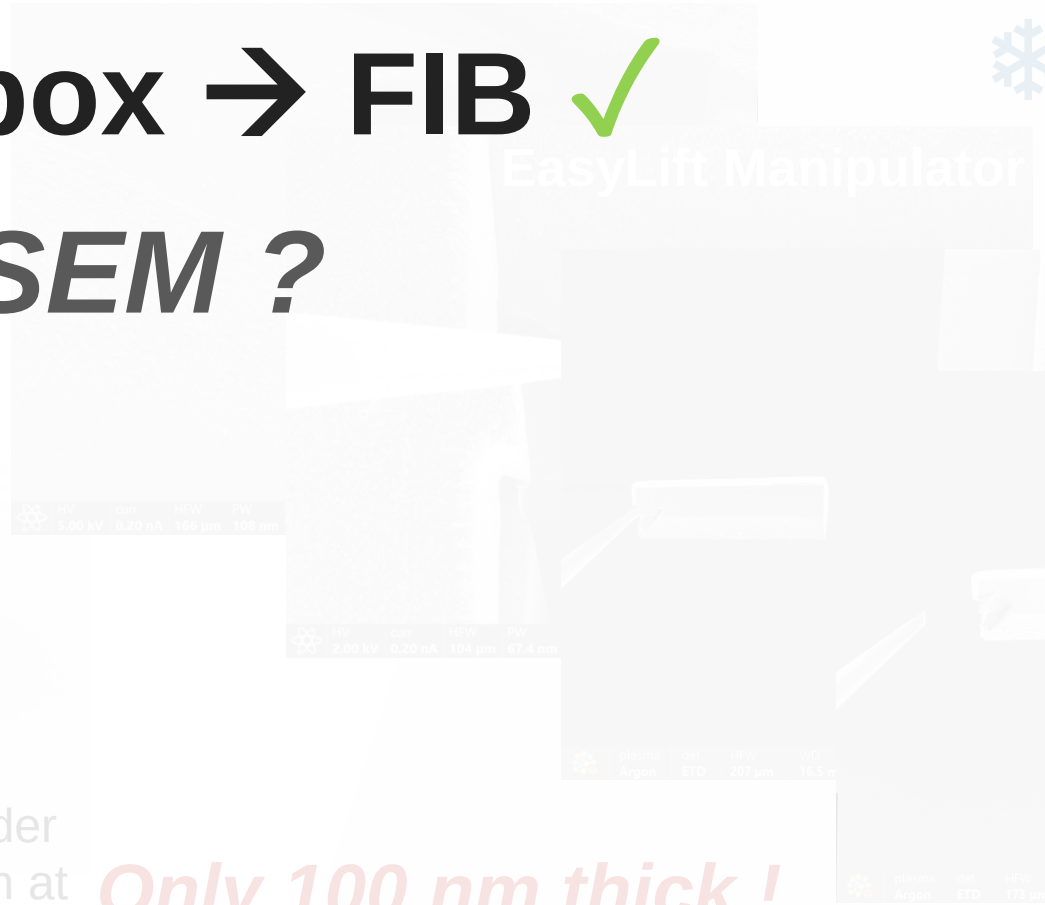
→ slim down with Arg milling beam

Sample preparation using cryo PFIB (~ -180 ˚C)

Proof Material #3: Li-metal lamellae preparation (Ar^+ ion) and imaging

- **Glove box → FIB ✓**
- ***FIB → SEM ?***

Helio Hydra PFIB



EasyLift Manipulator

Manipulator was held at cryogenic temperatures during the lift out

Whole process under cryogenic condition at -178 ˚C

Only 100 nm thick !

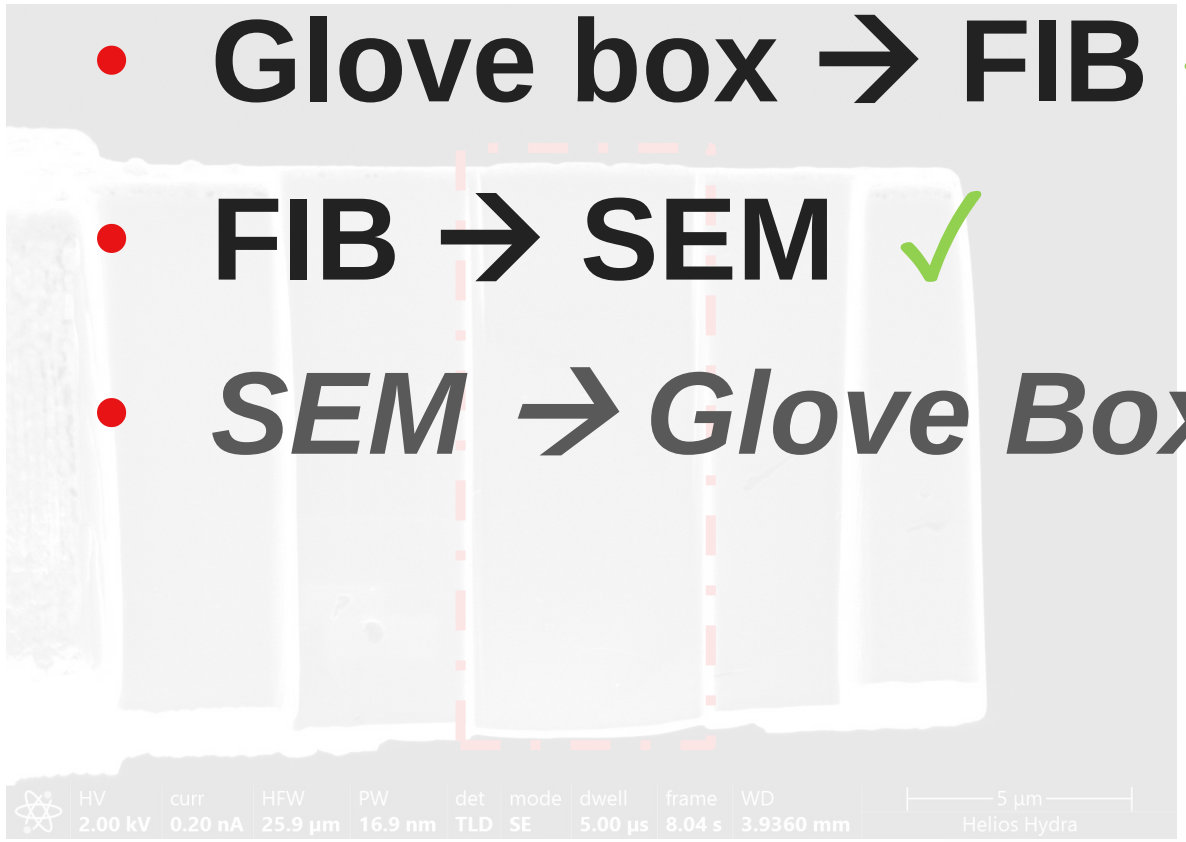
Huge surface x volume ratio

Application of IGST on air-sensitive materials

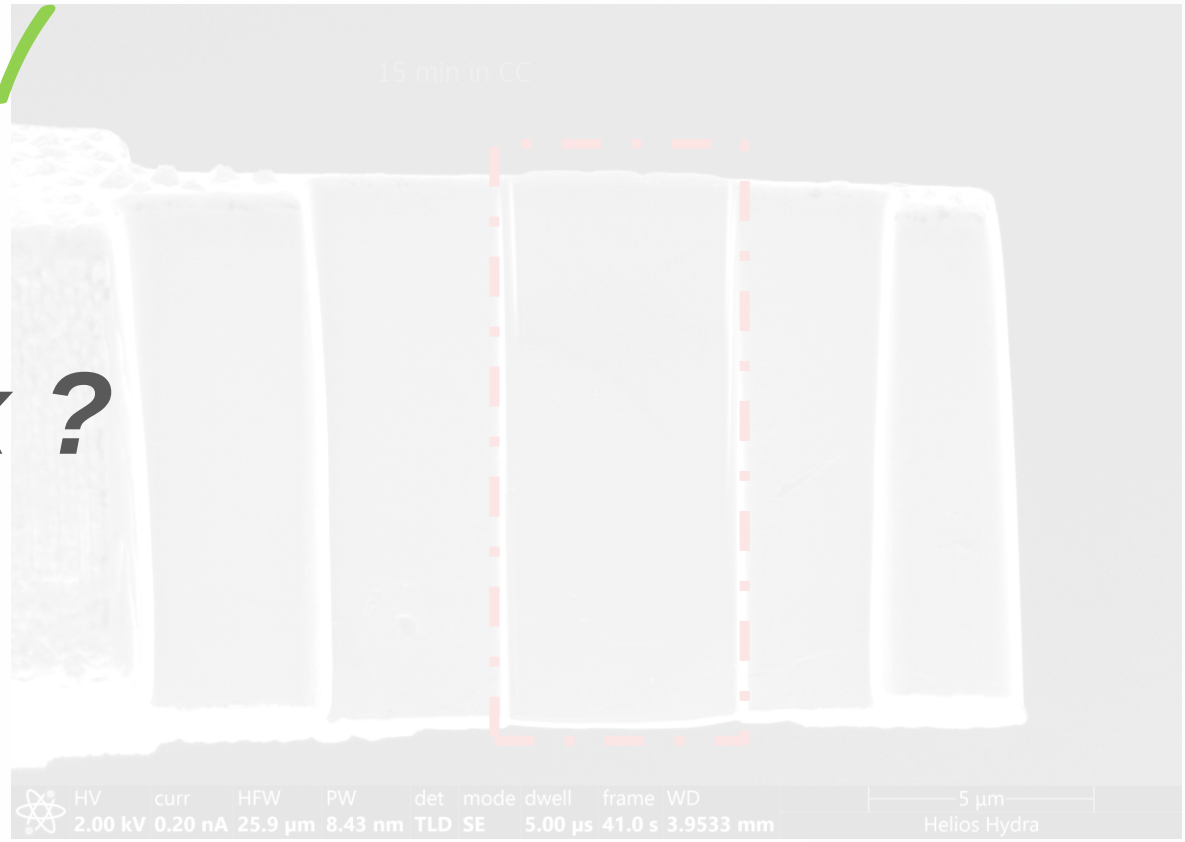
Proof Material #3: Li-metal lamellae preparation (Ar^+ ion) and imaging

- **Glove box → FIB** ✓
- **FIB → SEM** ✓
- ***SEM → Glove Box ?***

1st image inside the Helios chamber



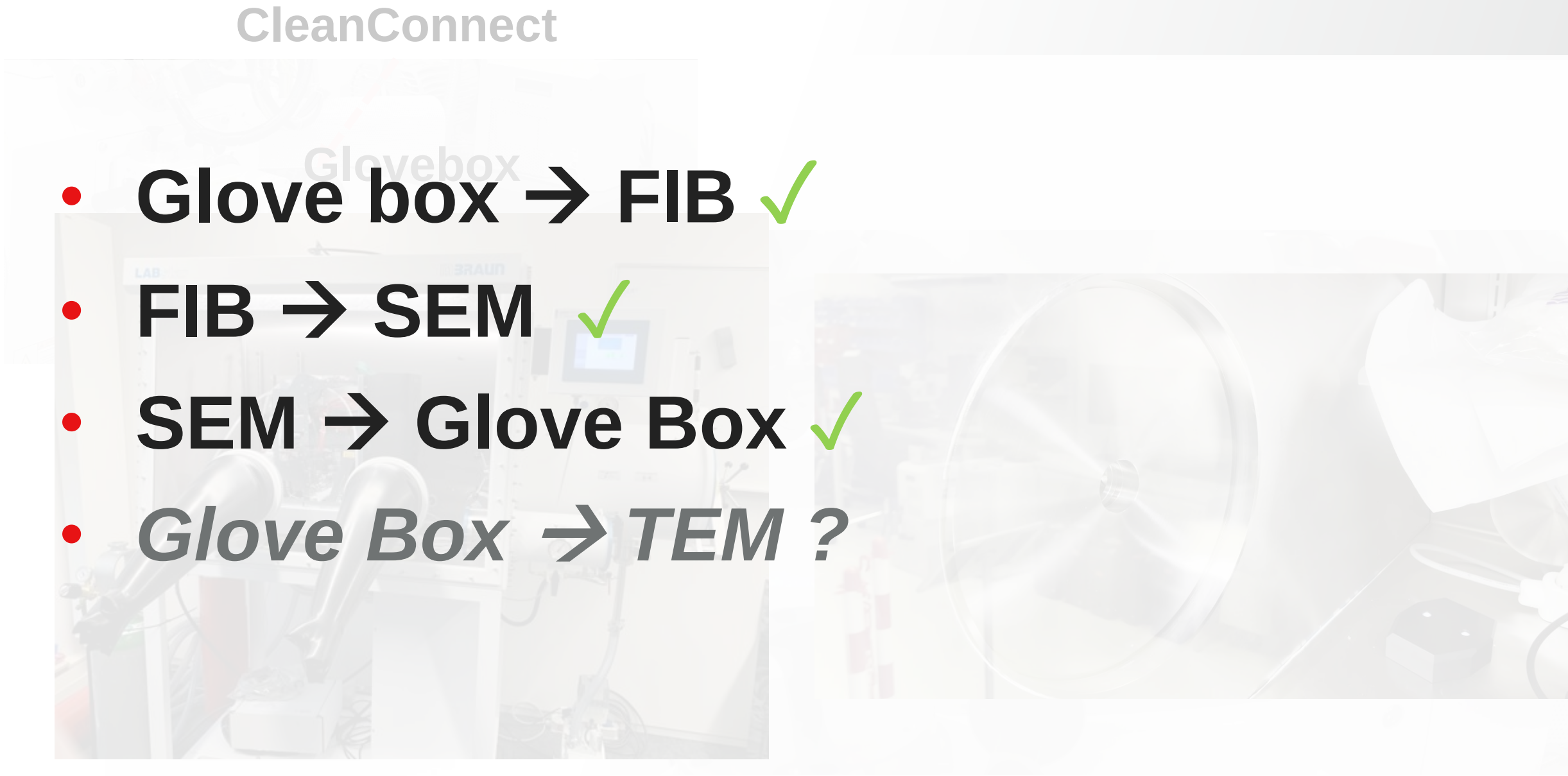
2nd image inside the Helios chamber after 15 minutes in CleanConnect



Excellent protection of Li-metal lamellae via CleanConnect

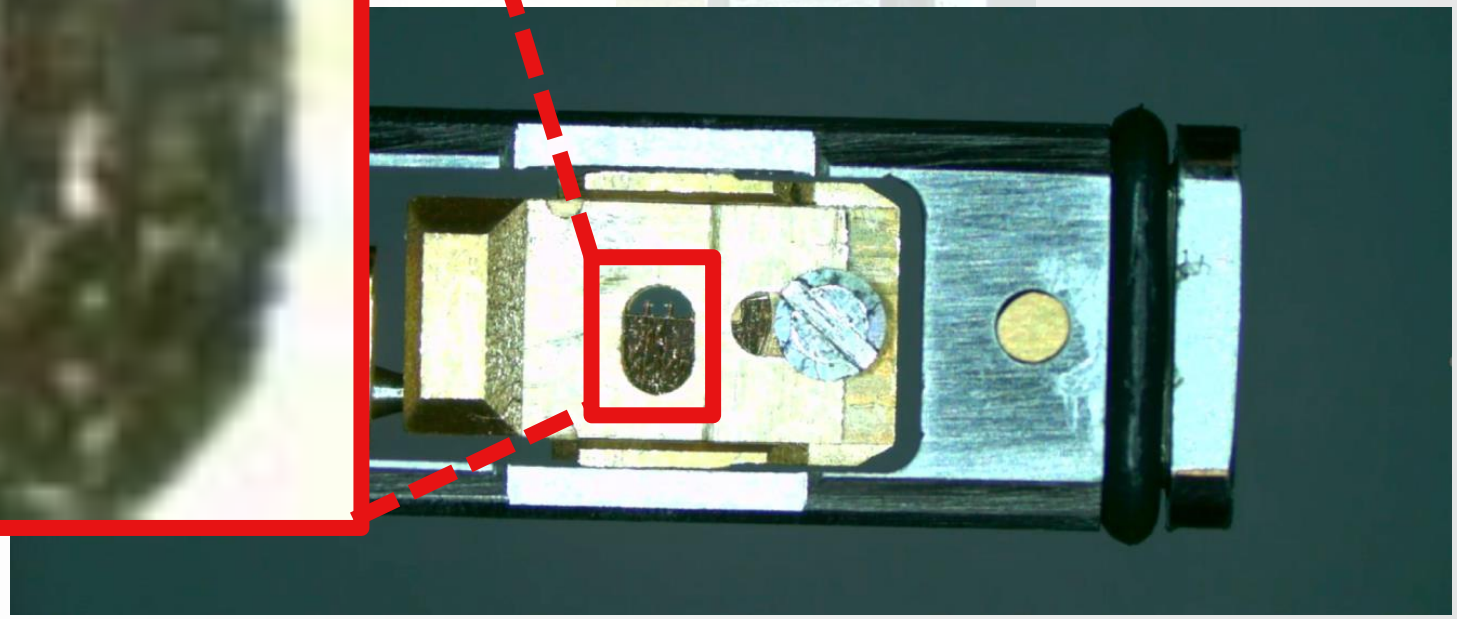
Image courtesy of Brandon van Leer, Letian Li, Thermo Fisher Scientific

Sample transfer to glovebox after preparation at the PFIB



- Glove box → FIB ✓
- FIB → SEM ✓
- SEM → Glove Box ✓
- *Glove Box → TEM ?*

Transfer to Cryo-IGST TEM holder

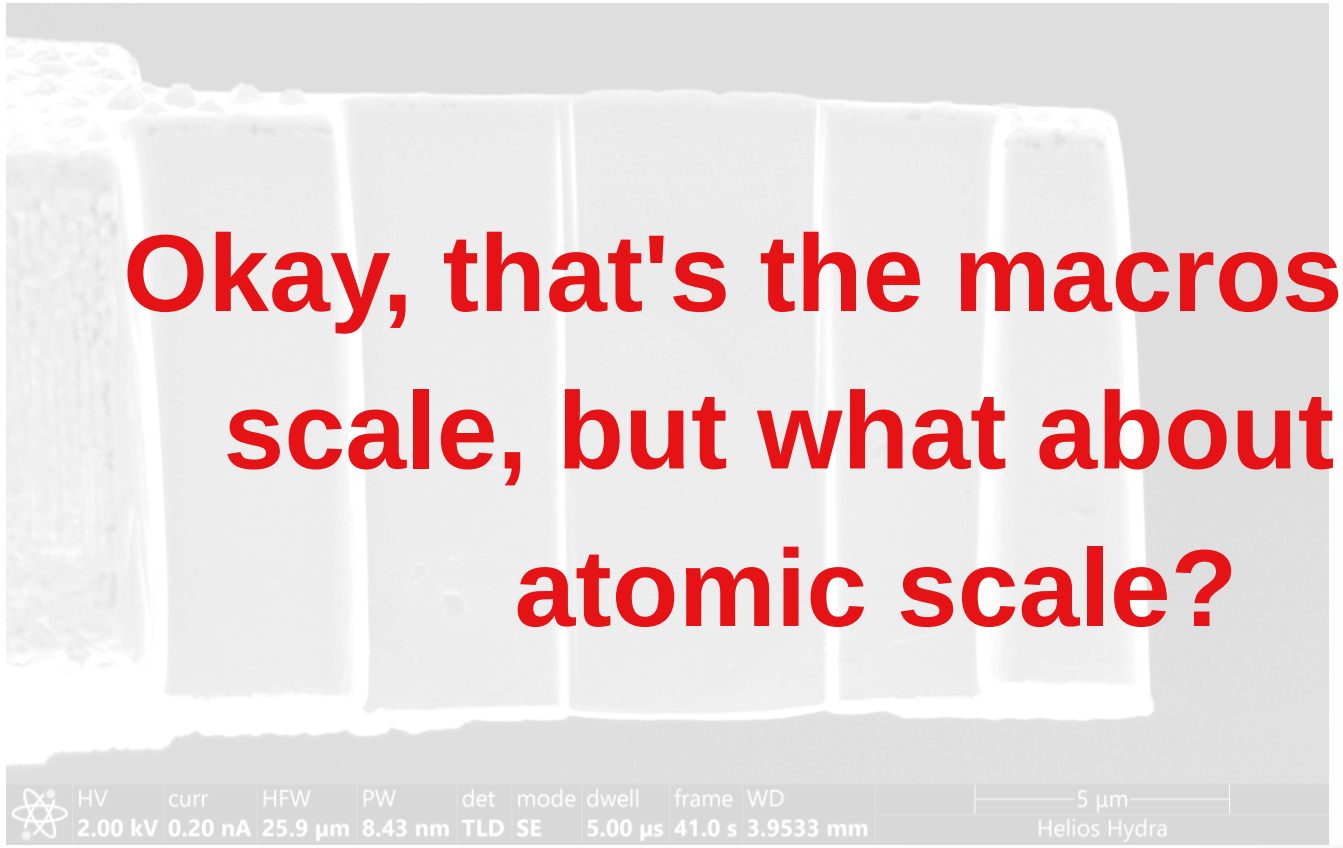


TEM Imaging

Acquired in Velox software using Ceta

Helios Hydra PFIB

Talos F200X G2 TEM



Okay, that's the macroscopic scale, but what about the atomic scale?

Application of IGST workflow on air-sensitive material

Proof Material #3: Li-metal lamellae preparation (Ar^+ ion) and imaging

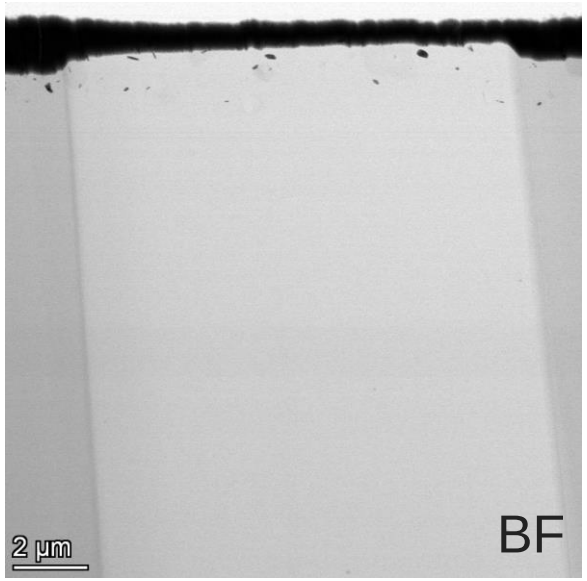
- Glove box → FIB ✓
- FIB → SEM ✓
- SEM → Glove Box ✓
- Glove Box → TEM ✓

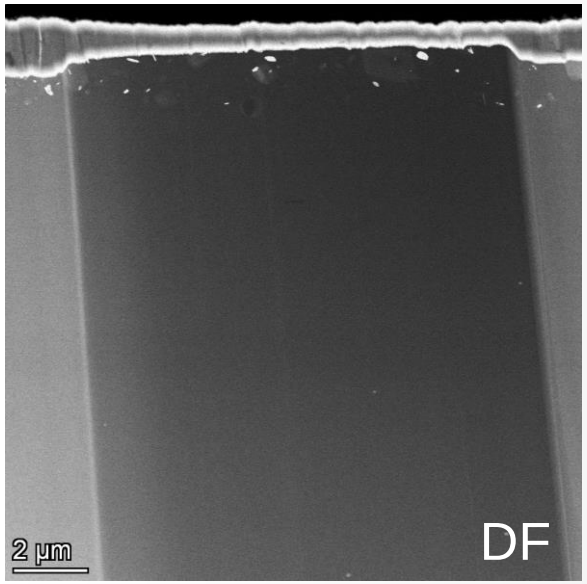
Image courtesy of Lin Jiang, Thermo Fisher Scientific

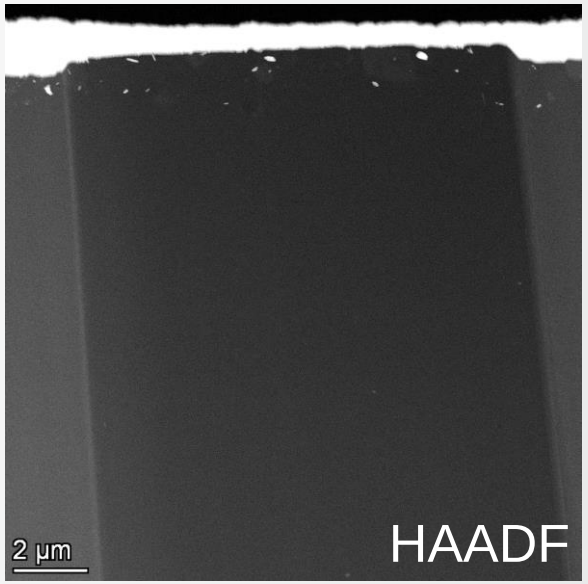
Clean connect solution Vs Conventional workflow

Li metal STEM imaging comparison with and without air exposure (15 s)

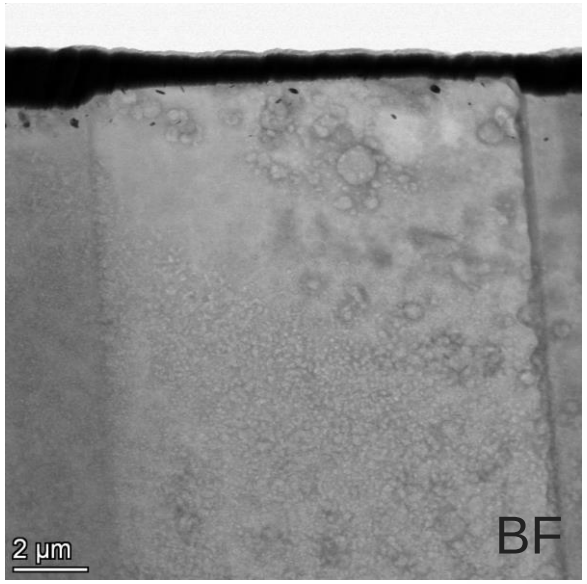
Lamella #1 by clean connect solution

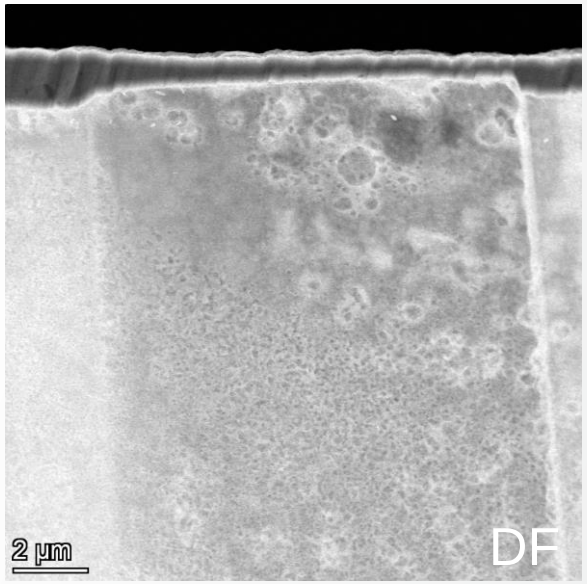


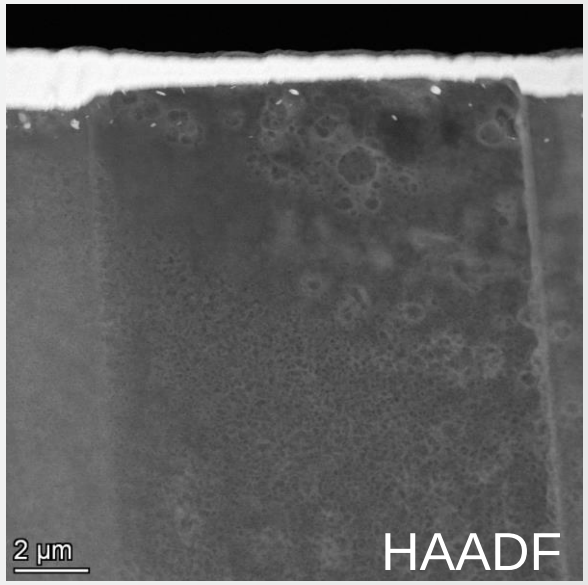




Lamella #1 after 15s air exposure



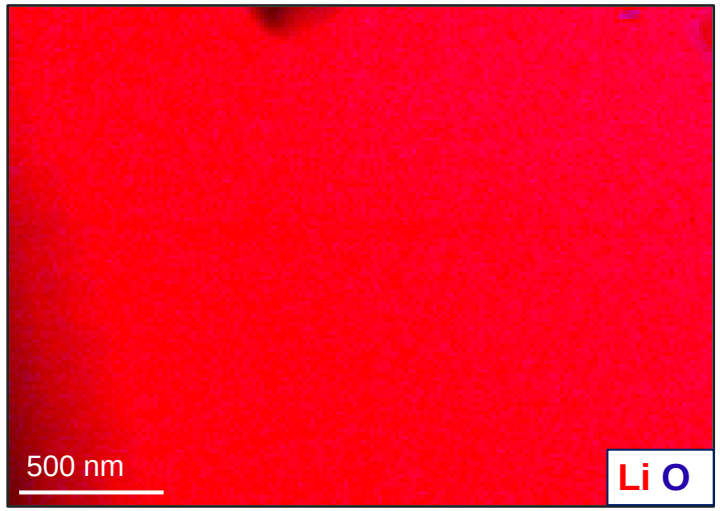


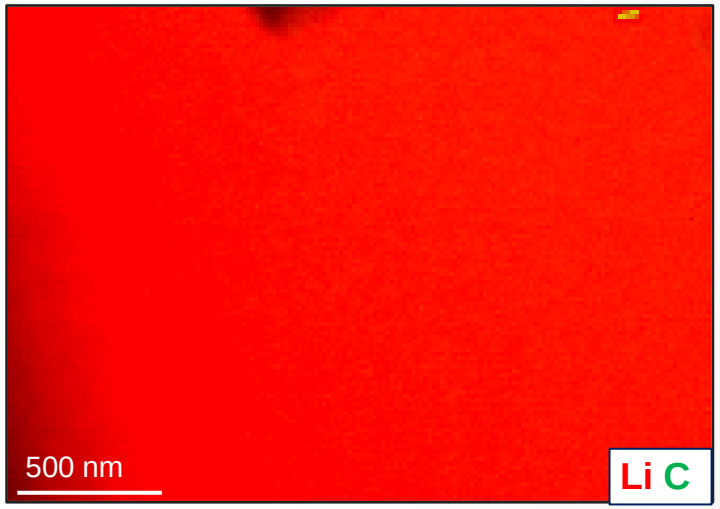


Clean connect solution Vs Conventional workflow

Li metal EELS analysis comparison with and without air exposure (Lamella #1)

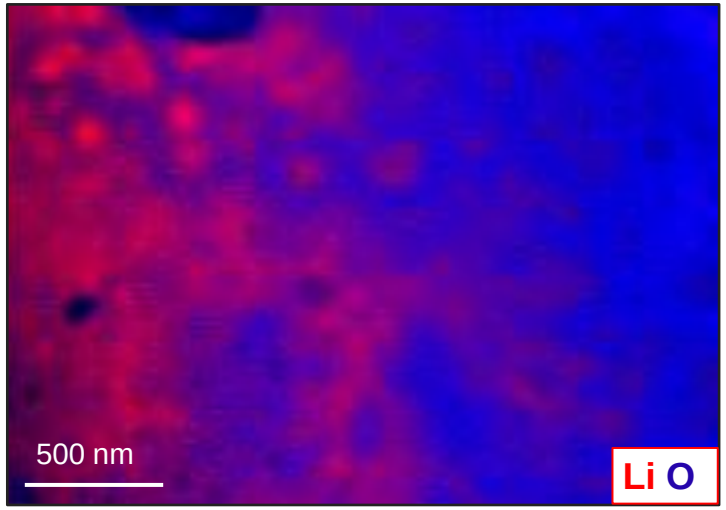
Without air exposure

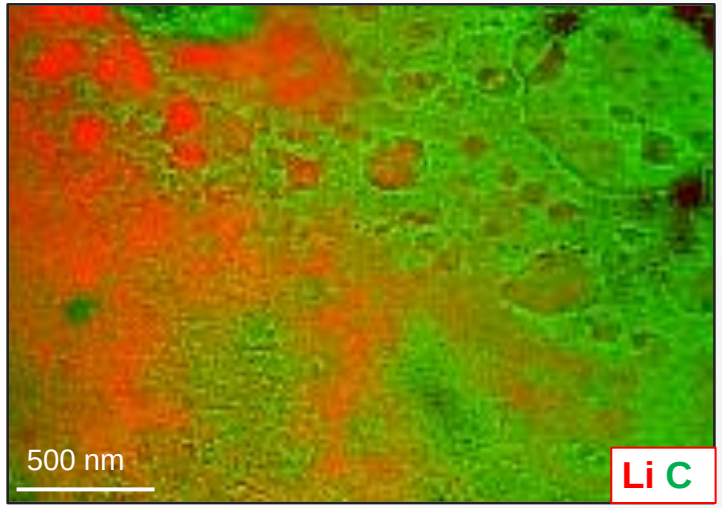




Element	Shell	Signal (Counts)	Comp. (at.%)
Li	K	6.768032e+11	94 ± 4
C	K	5.9267e+09	2.98 ± 0.14
O	K	9.3609e+08	2.92 ± 0.14

After 15s air exposure





Element	Shell	Signal (Counts)	Comp. (at.%)
Li	K	7.20142e+10	41.6 ± 1.2
C	K	1.83907e+10	22.0 ± 0.6
O	K	9.61318e+09	36.4 ± 1.1

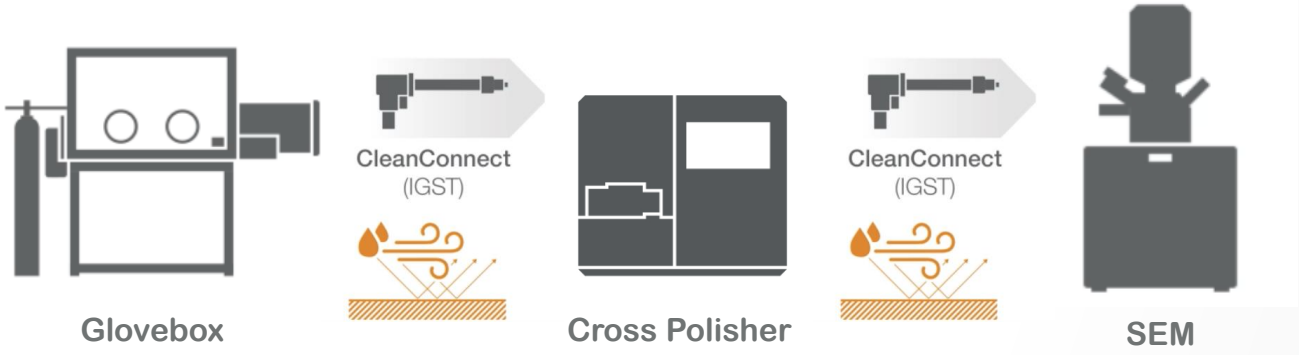
→ *Oxidation effects*

Contents

CleanMill and CleanConnect

CleanMill and CleanConnect compatible enabling the Thermo Scientific IGST workflow

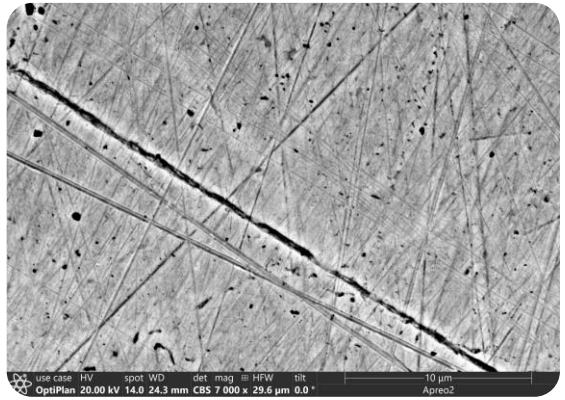
Enabling observation of materials in their native state



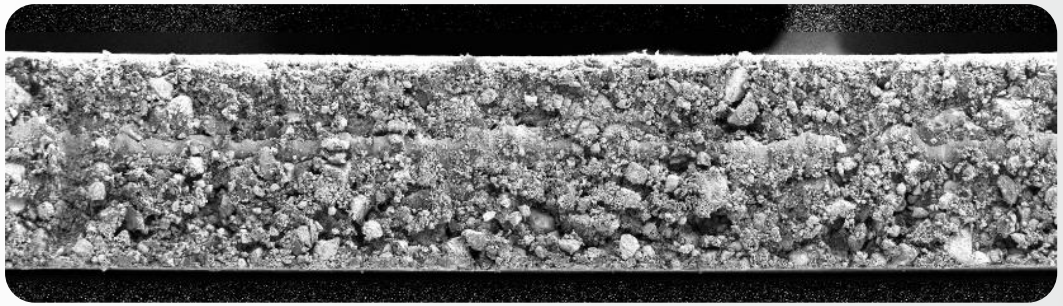
Sample Preparation for Nanoanalysis

Reliable & Repeatable sample prep is required for success in any surface examination

Common Issues



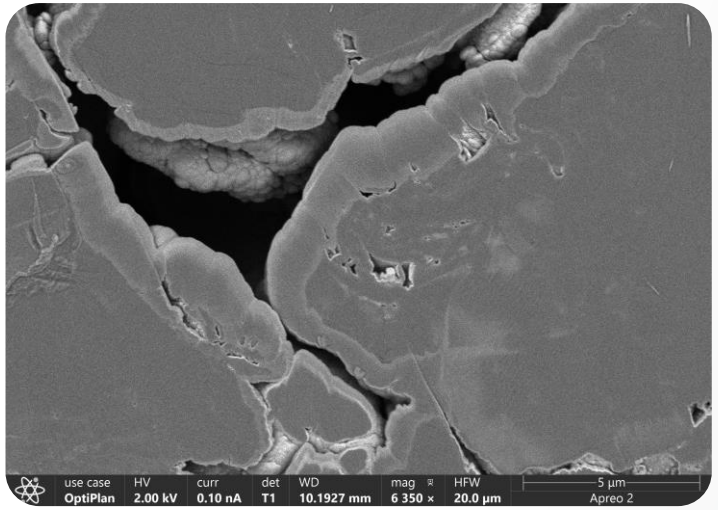
Mechanical Polishing



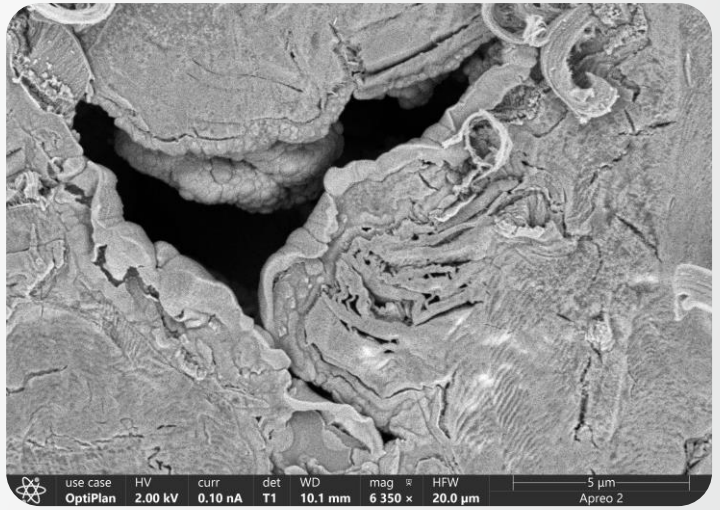
Composite Materials & Polymers

Energy &

Next Gen Materials



Lithiated Graphite Anode

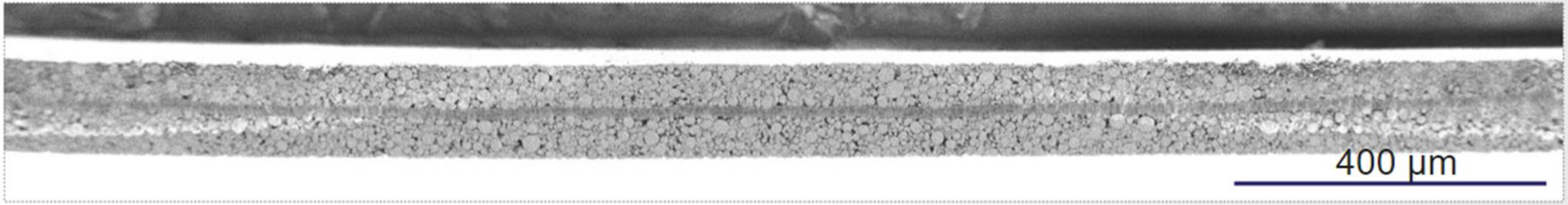


After 1 min air exposure

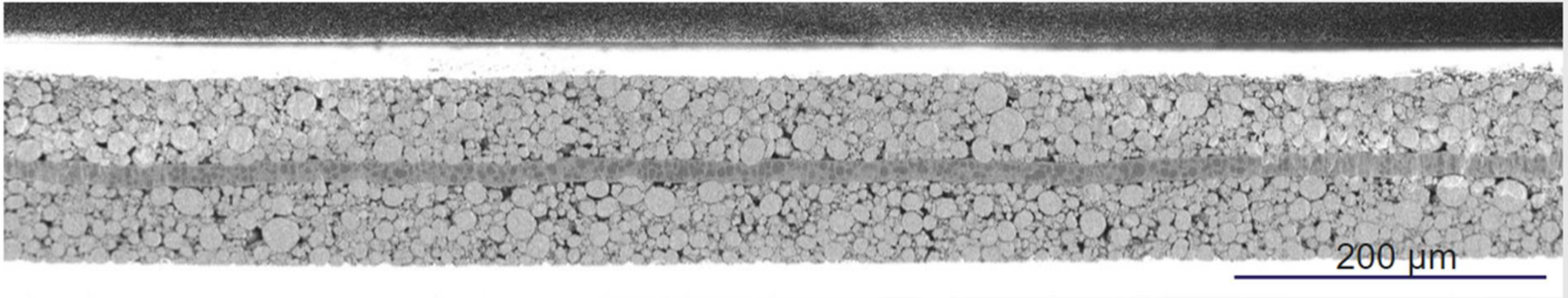
CleanMill Results: Cathode NMC

40º Oscillation, 1.5° tilt, **1 hour** polishing; 16 kV

HFW: 2 mm

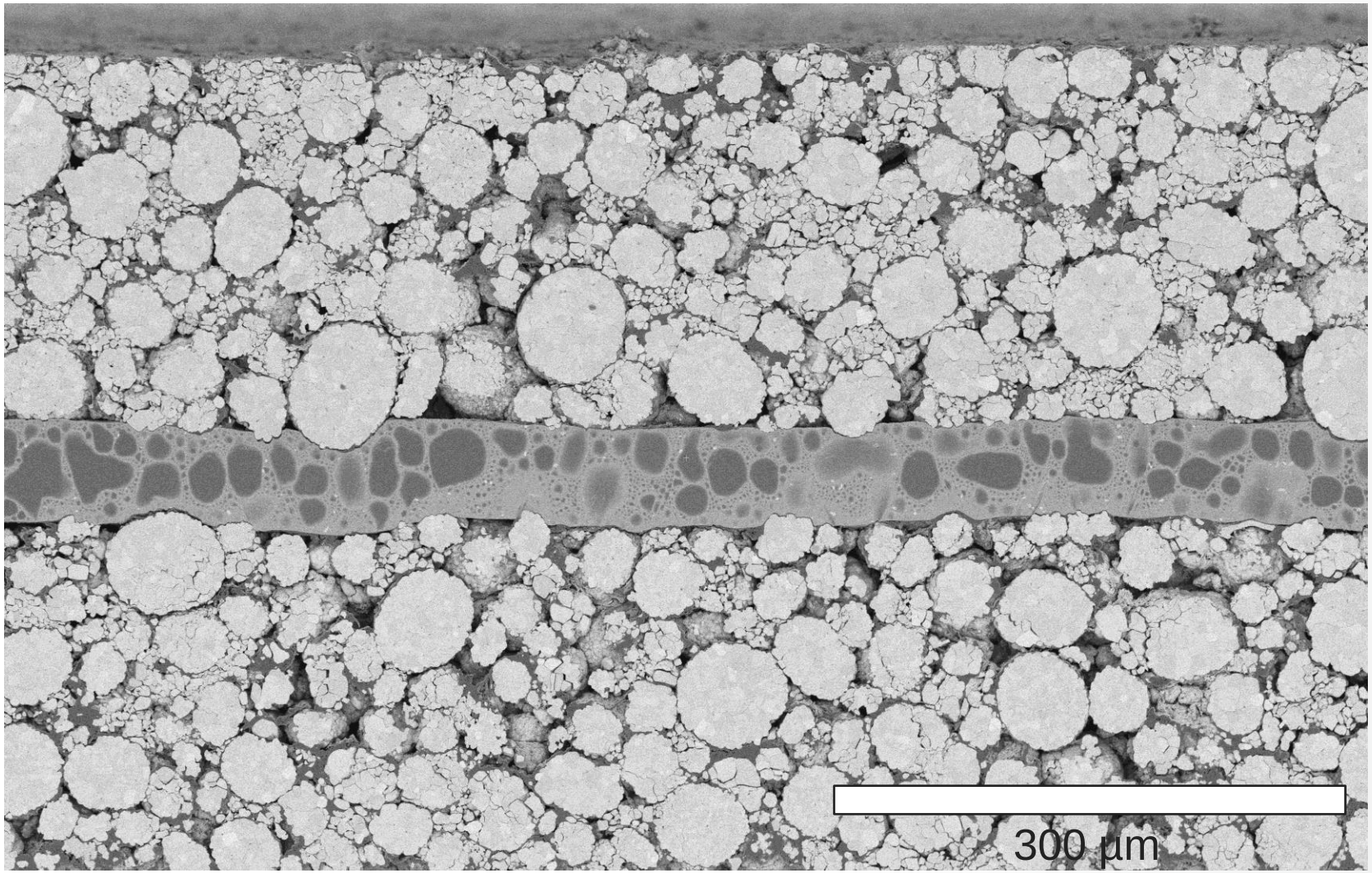


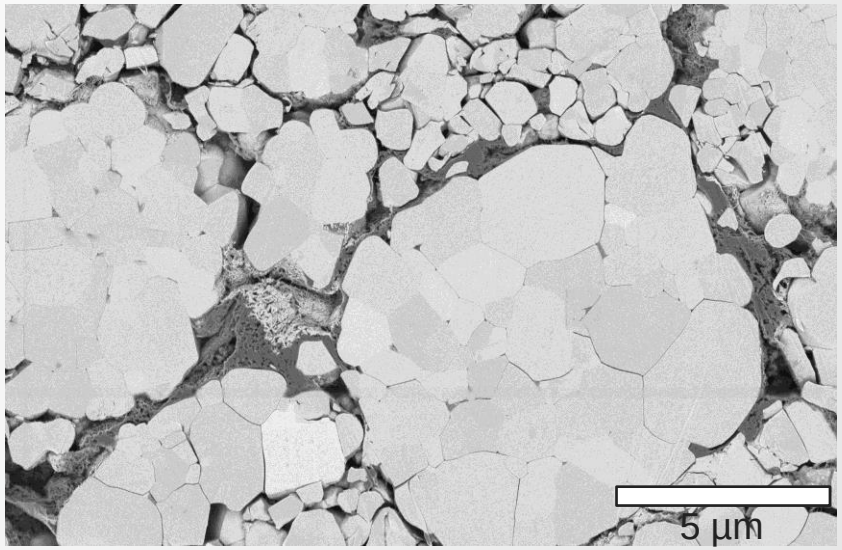
HFW: 1 mm

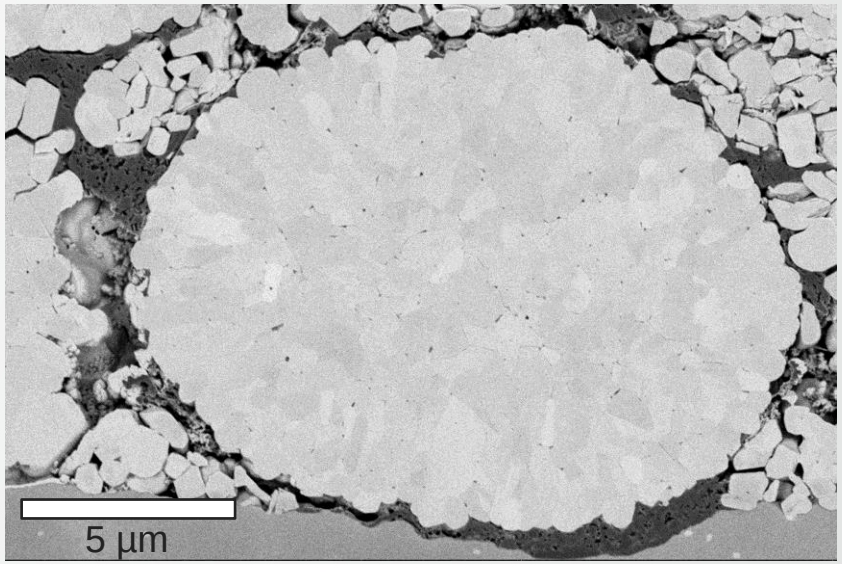


CleanMill Results: Cathode NMC

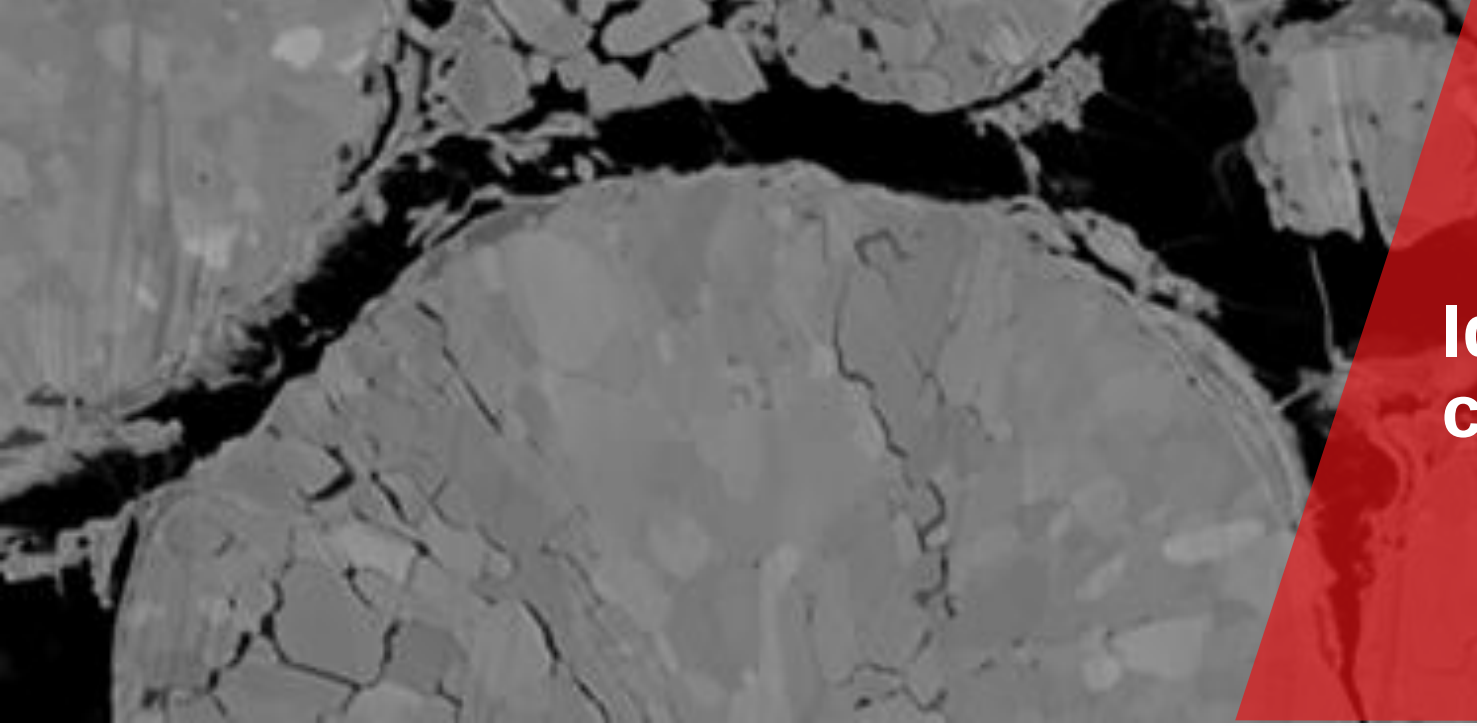
40º Oscillation, 1.5° tilt, **1 hour** polishing; 16 kV







Contents



Identification of micro cracks cathode

Challenge

Improve performance of batteries by better understanding of degradation mechanism

Problem

Hard and tedious to be able to correctly analyze micro-cracks without being an image processing expert.

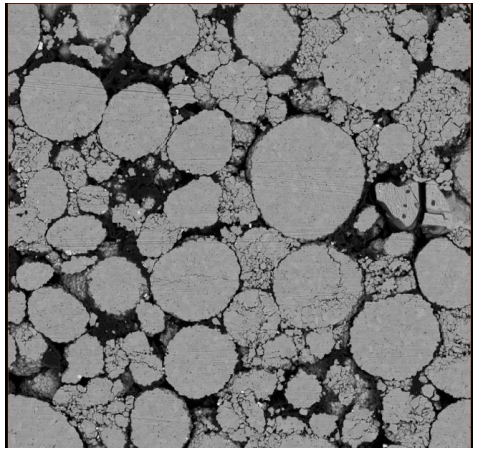
Need

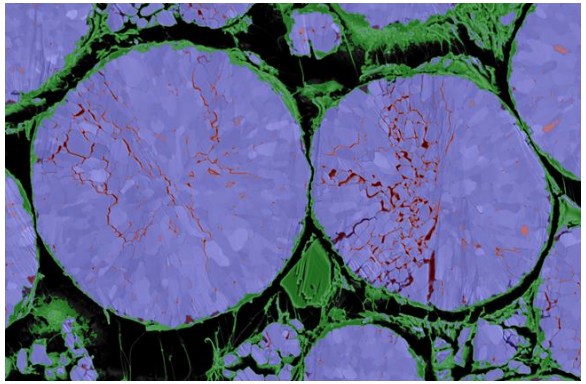
Automated detection of micro cracks playable by non image processing experts

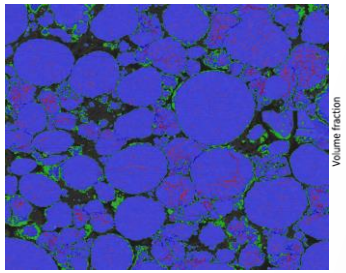
Impact

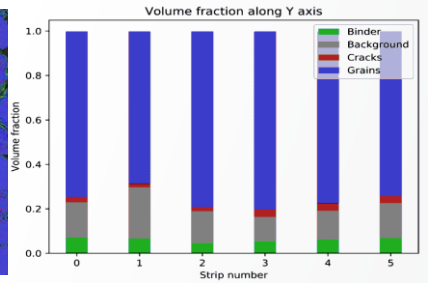
Improve existing electrode manufacturing process validation and new electrode architecture development.

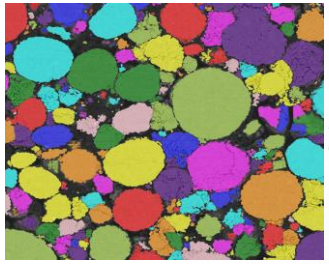
Identification of Micro Cracks Cathode

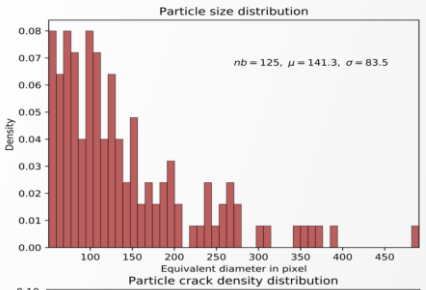


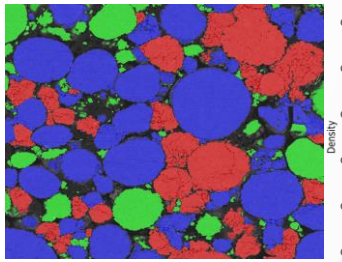


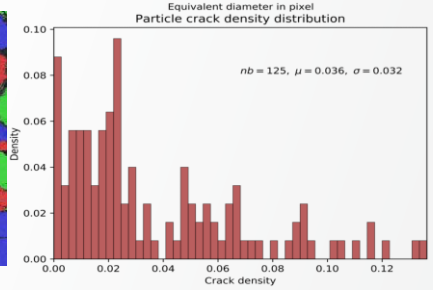








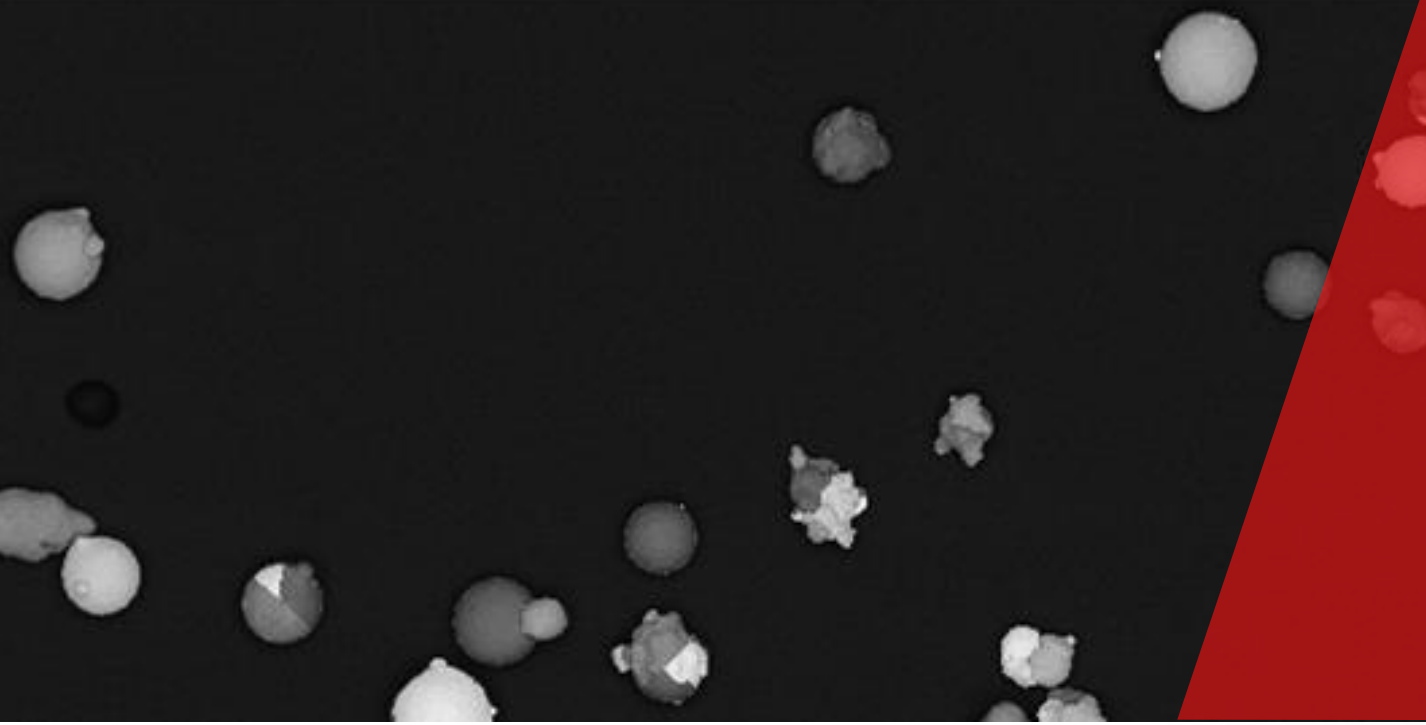




- Fully automatable workflows for crack detection and measurements
- Advanced segmentation tools including complete deep learning environment
- Report generation

Impact:
Advanced image processing packaged in an intuitive way, with simple GUI and which can be played by non experts

Data collection courtesy of Ron Kelley, Thermo Fisher Scientific. Sample provided by Prof. Shirley Y. Meng, University of California, San Diego



Unattended identification of particles on a tiled dataset

Challenge

Improve output and precision of particle analysis in cathode materials in an unattended way

Problem

Not easy to master different software environments and trigger automation on a large tiled dataset without being an image processing expert

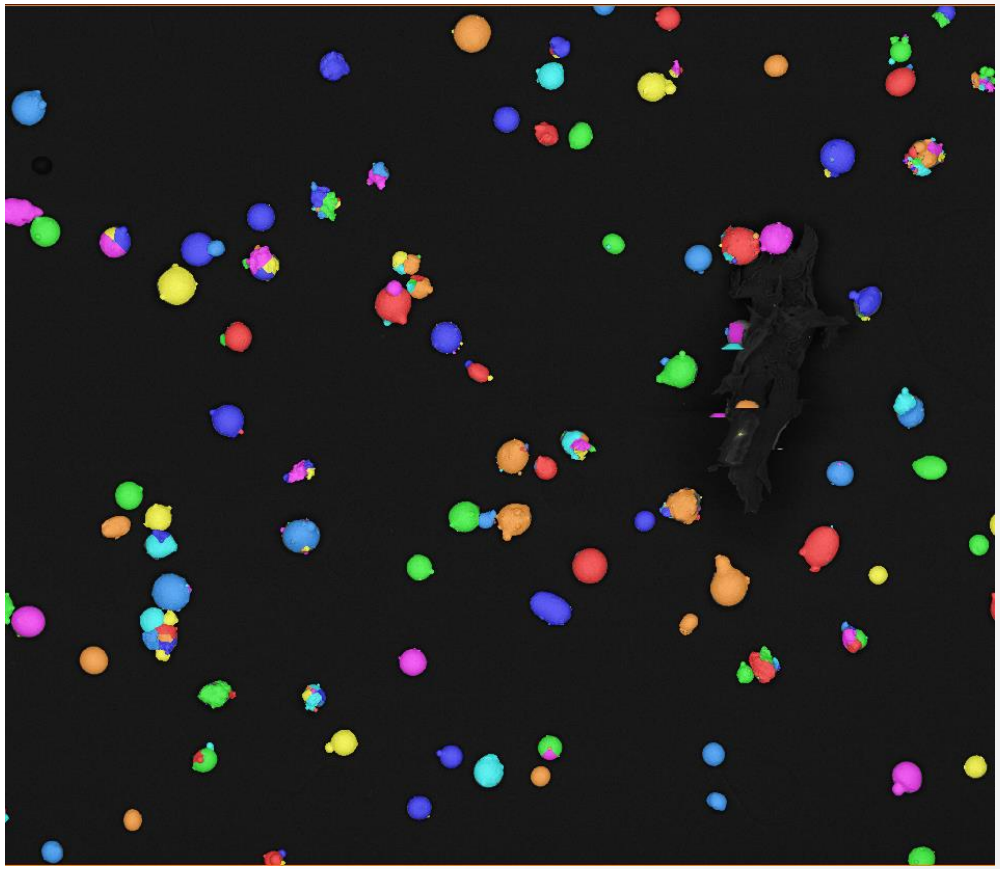
Need

Analyze different particle morphologies, chemical composition, density and association and so on …

Impact

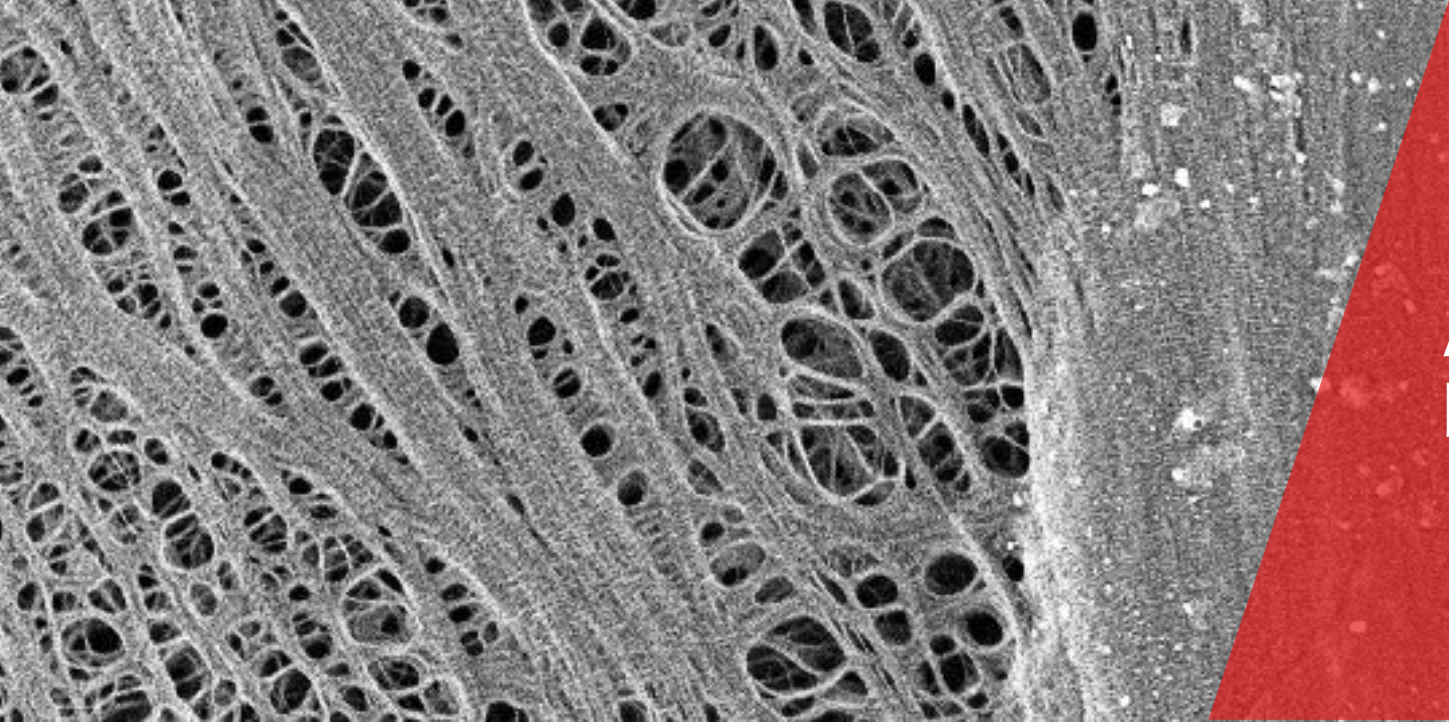
Dramatically Improve output of analysis in the electrode manufacturing process validation and new electrode architecture development.

Unattended identification of particles on a tiled dataset



- Fully automatable workflows for crack detection and measurements
- Advanced segmentation tools including complete deep learning environment
- Report generation

Impact:



Advanced analysis of a battery separator

• Challenge

- Suboptimal performances in charging / discharging
- Unable to scale analysis

Problem

- Tedious
- Highly manual
- Prone to errors

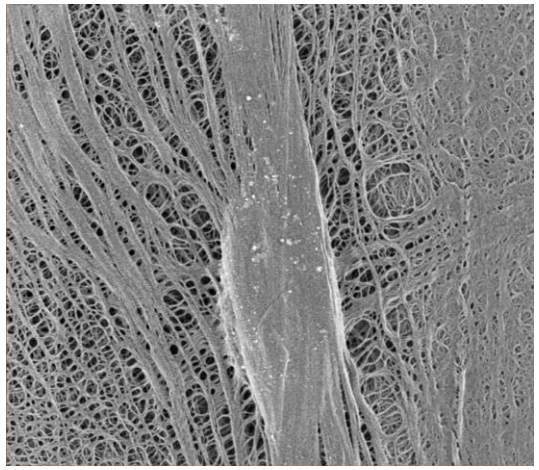
Need

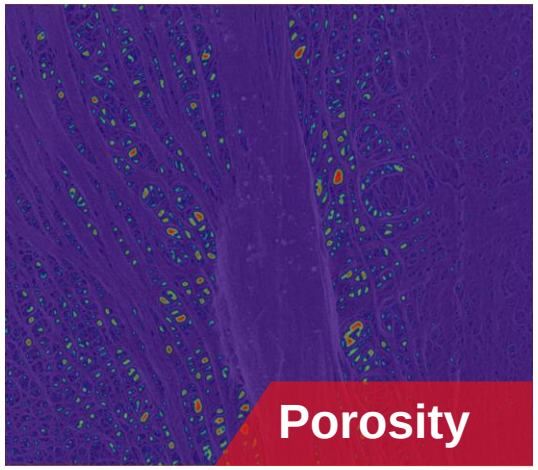
- Automated process
- Fast and reproducible

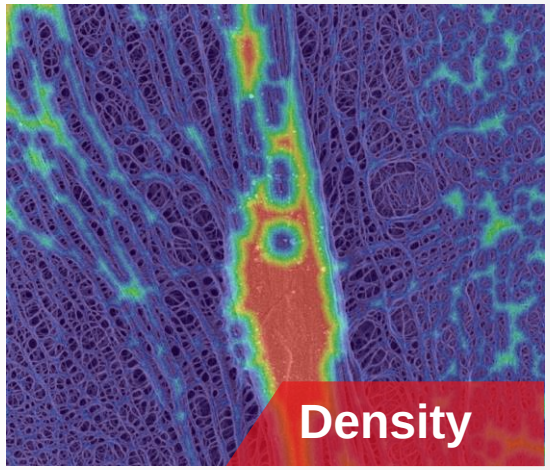
Impact

- Better understanding of transport mechanisms and separator performance
- Better throughput thanks to automation of recipes

Identification of Micro Cracks Cathode



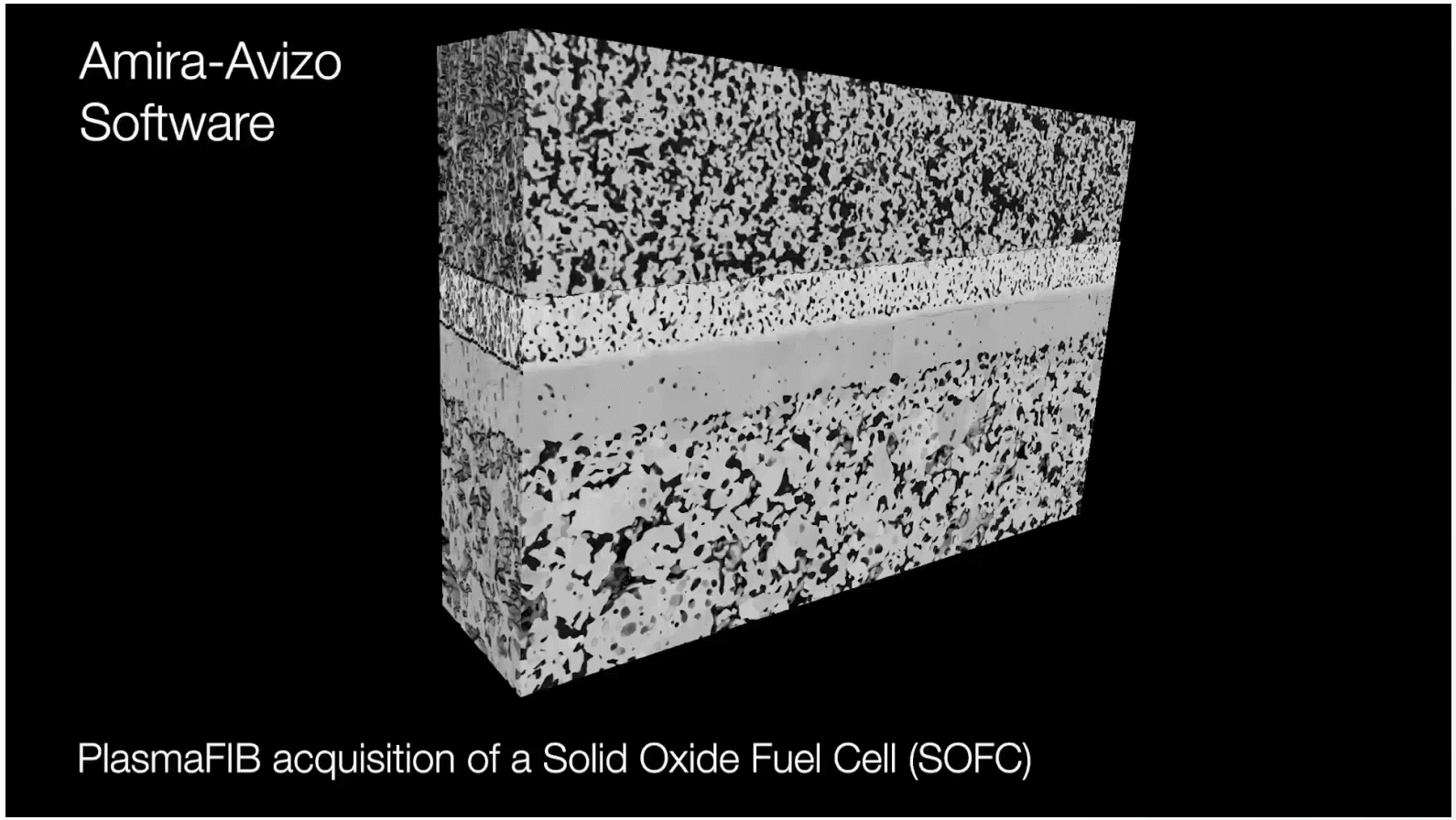




- Fully automatable workflows getting density and porosity information
- Advanced segmentation tools including complete deep learning environment
- Report generation

Impact:
Design better performing batteries with less scrap parts through better understanding of separator performances

Fuel Cell structure and tortuosity

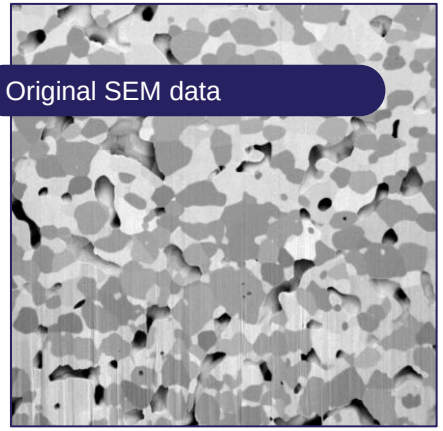


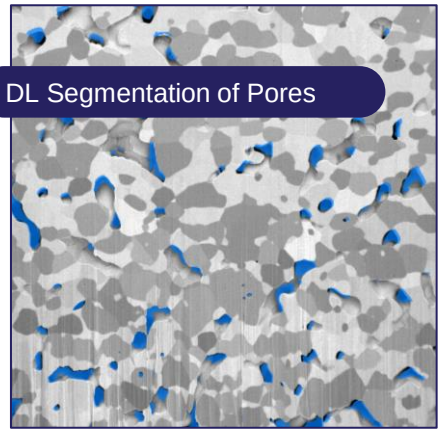
- Pores/particles extraction
- Pore network modeling
- Tortuosity estimation
- Triple phase boundaries
- Absolute permeability
- Molecular diffusion simulation

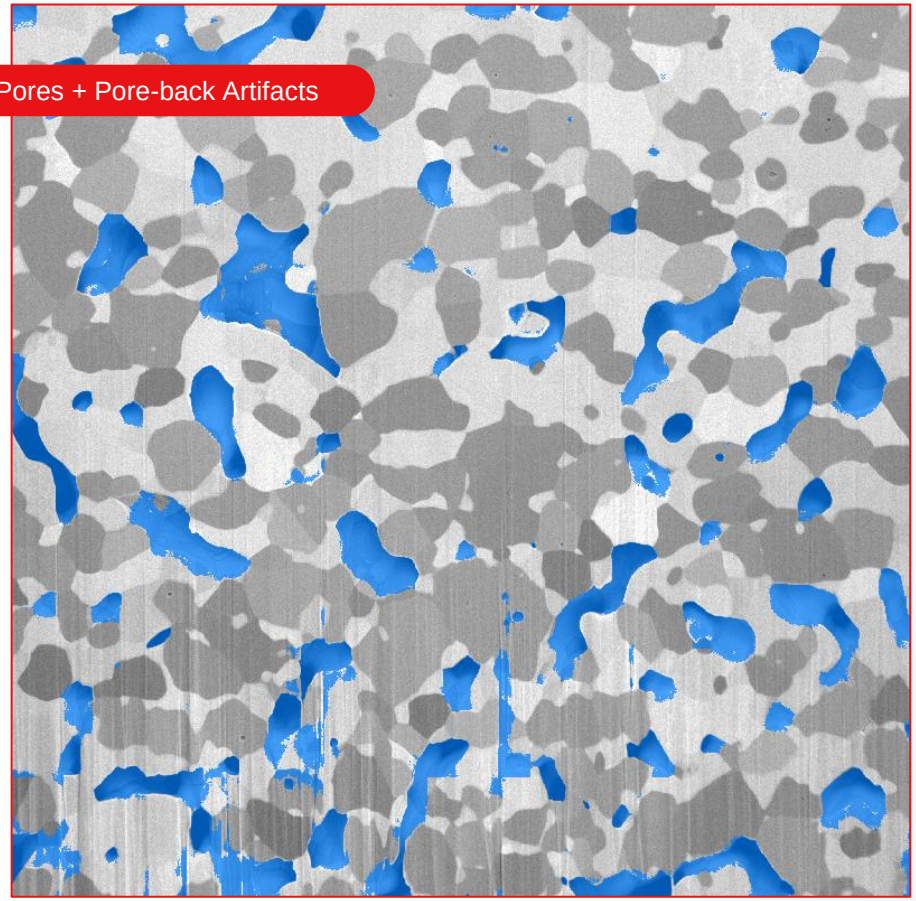
Impact :
Need to perform simulation faster and easier in a digital way so that it becomes part of the design workflow of battery materials and Cell Manufacturing.

Data courtesy of Jochen Joos, Institut für Angewandte Materialien Werkstoffe der Elektrotechnik (IAM-WET), Karlsruher Institut für Technologie (KIT)

Deep learning for better accuracy





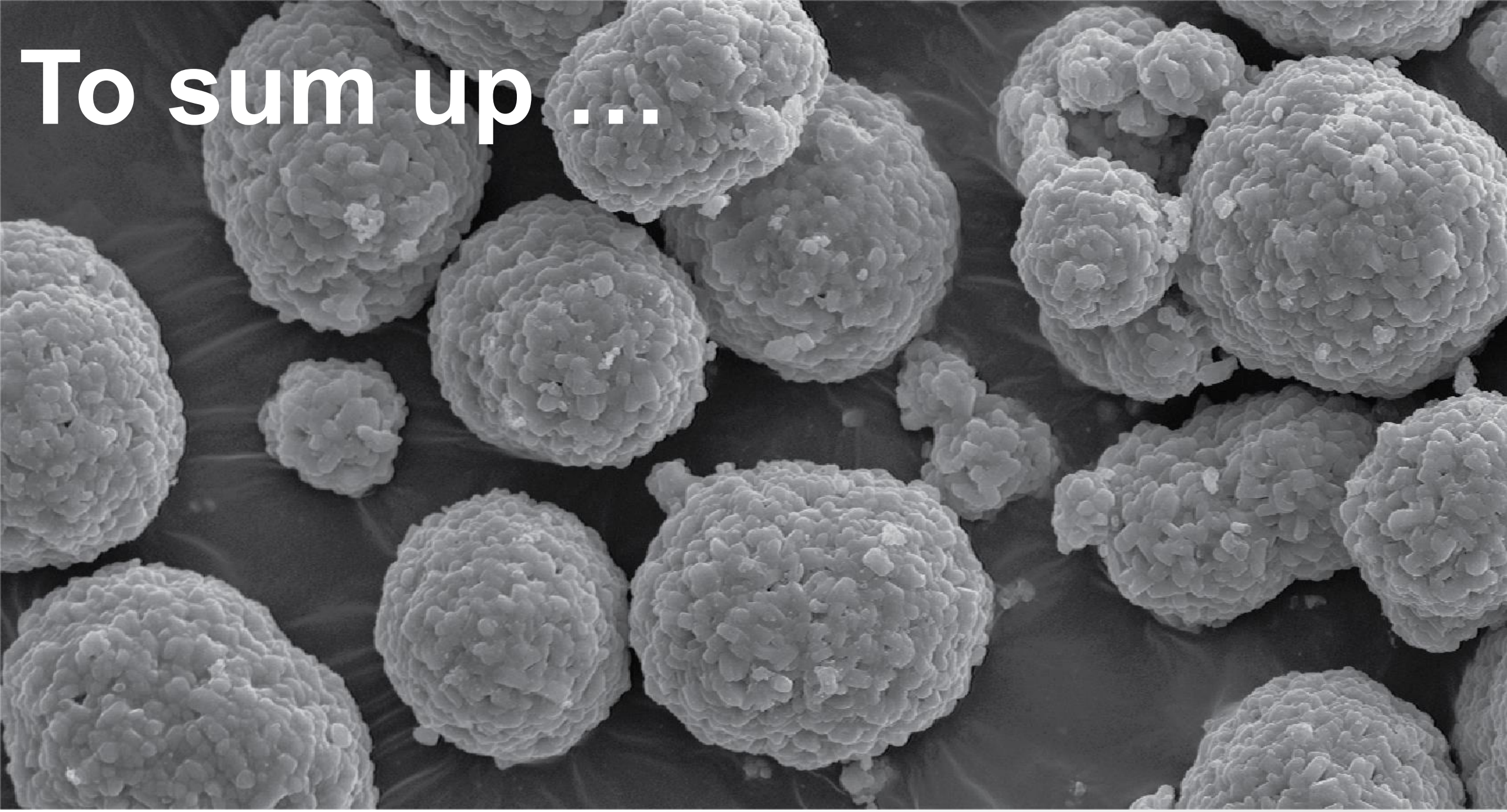


Phase	Pore Fraction		
	Min	Mean	Max
True Porosity (DL)	3.69 %	4.23 %	4.84 %
Porosity + Pore-Back	9.17 %	10.86 %	12.38 %

Impact :
Prevent overcounting porosity by 250%, usable by non-experts

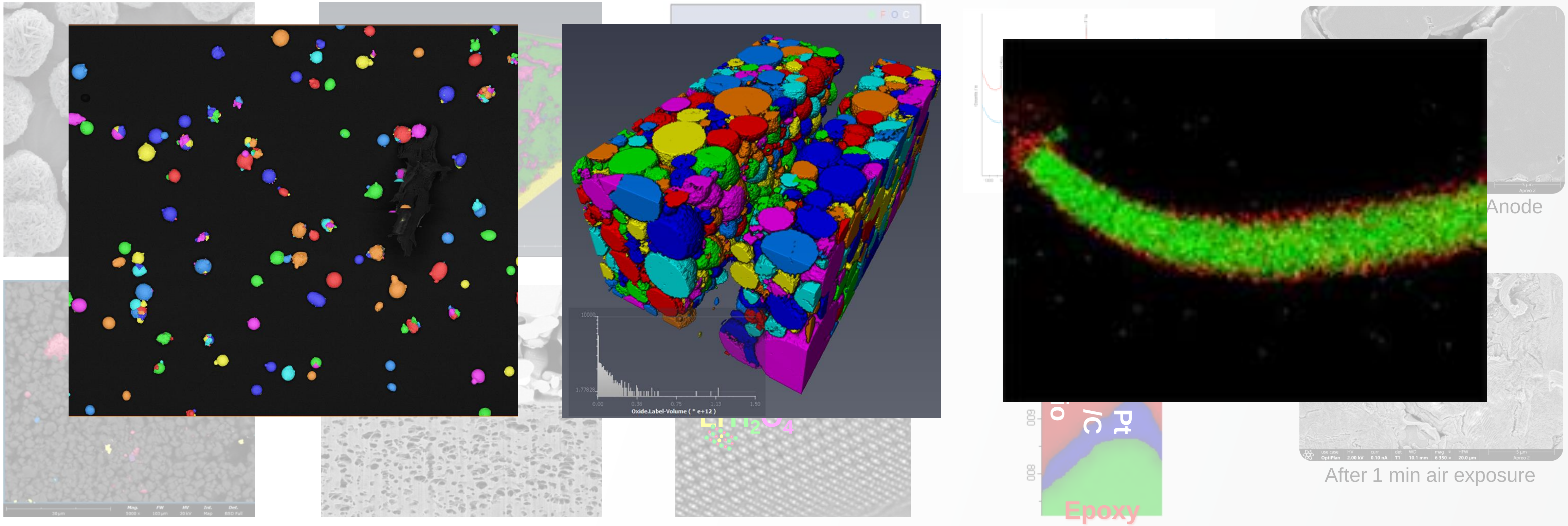
Data Courtesy of Sabanci University, Turkey

To sum up …



Thermo Fisher Materials MSD-EM portfolio

Avizo Software



Thank you