


# 3D characterization at every length scale

10. – 12. September

Hamburg

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Business Development Manager for Clean Energy DACH

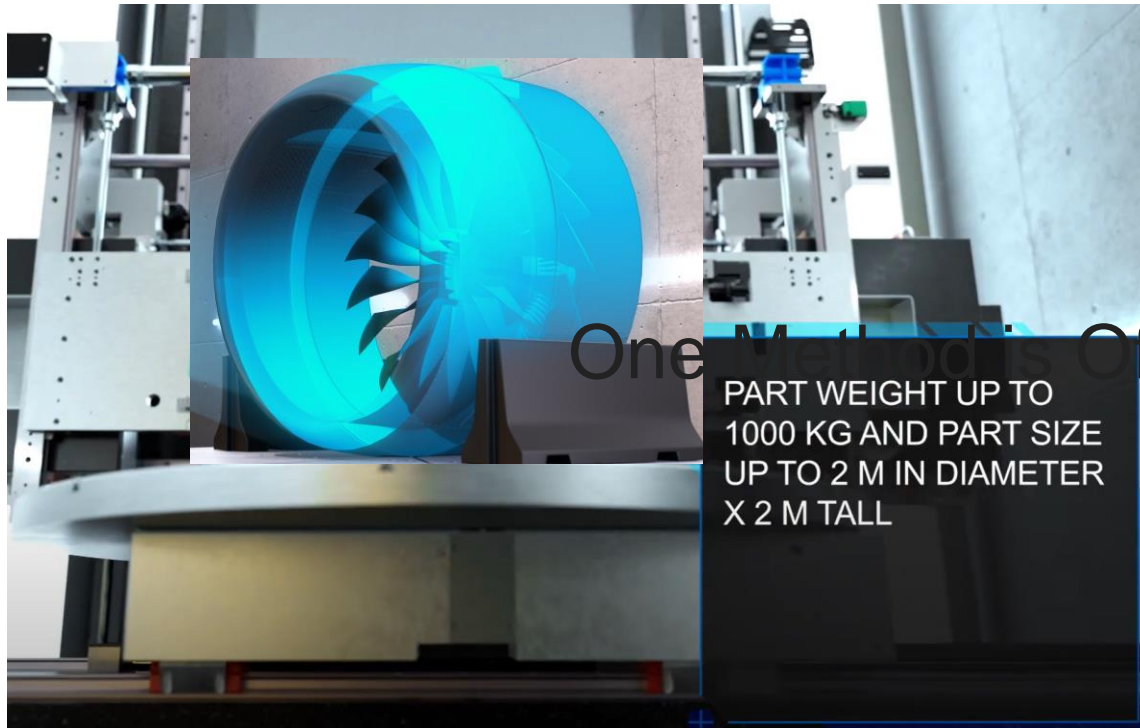
 The world leader in serving science

# Why is Microscale Analysis Important?

I need to analyze large parts...

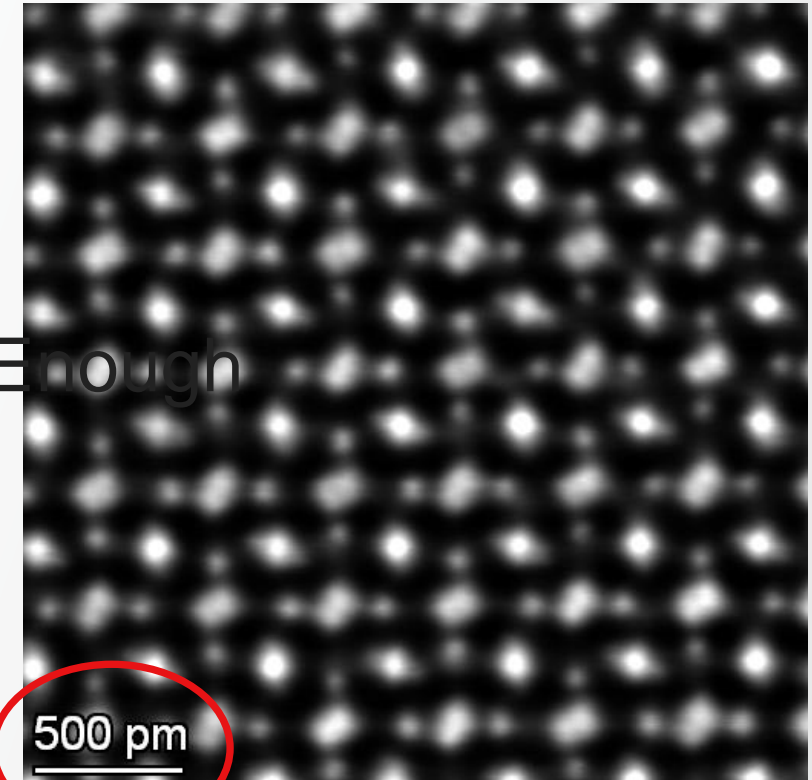
AND

I need to visualize small features...



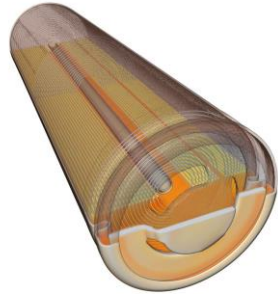
Waygate Technologies | High-Energy CT  
Phoenix Power|scan HE

One Method is Often Not Enough



Spectra 300 Scanning Transmission Electron  
Microscope (S/TEM)

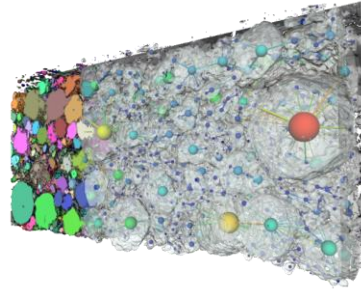
# Thermo Fisher Scientific MSD/EM Portfolio



(micro/nano) CT



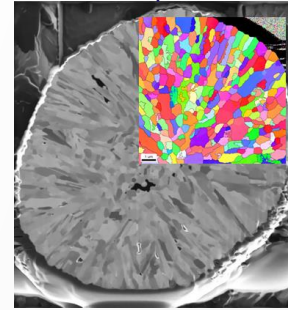
## SEM/DualBeam



SEM



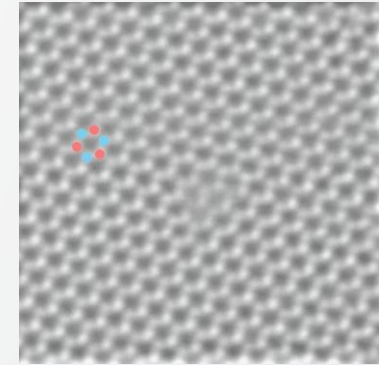
Axia, Apreo 2  
Phenom Desktop



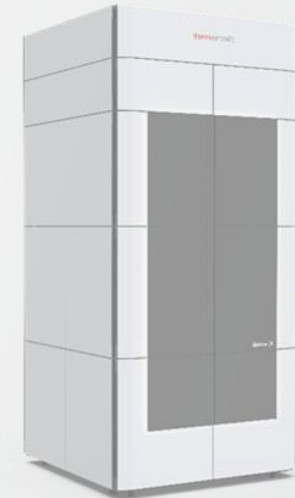
DualBeam



Scios 2  
Helios 5 PFIB (Laser)



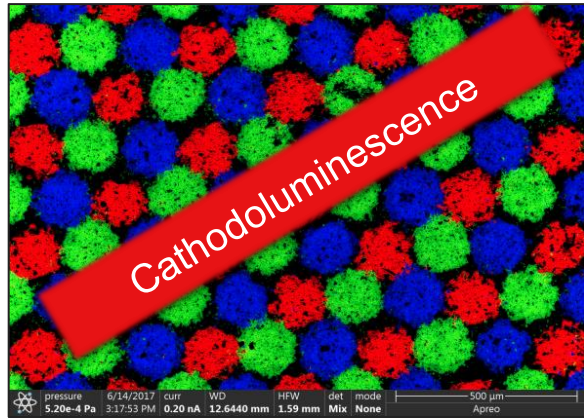
TEM



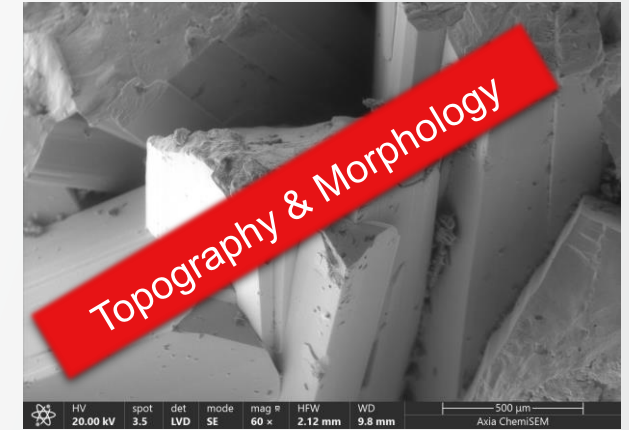
Talos  
Spectra

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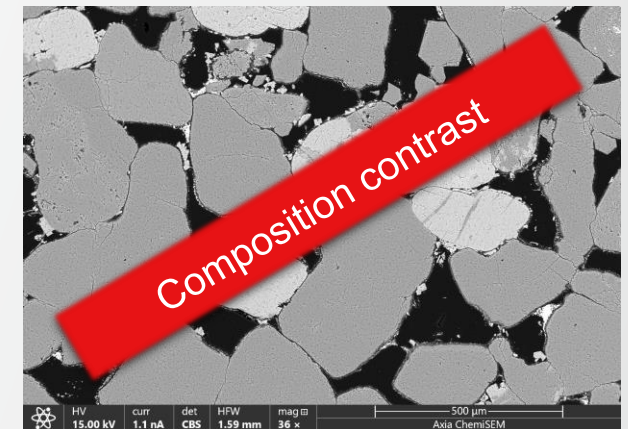
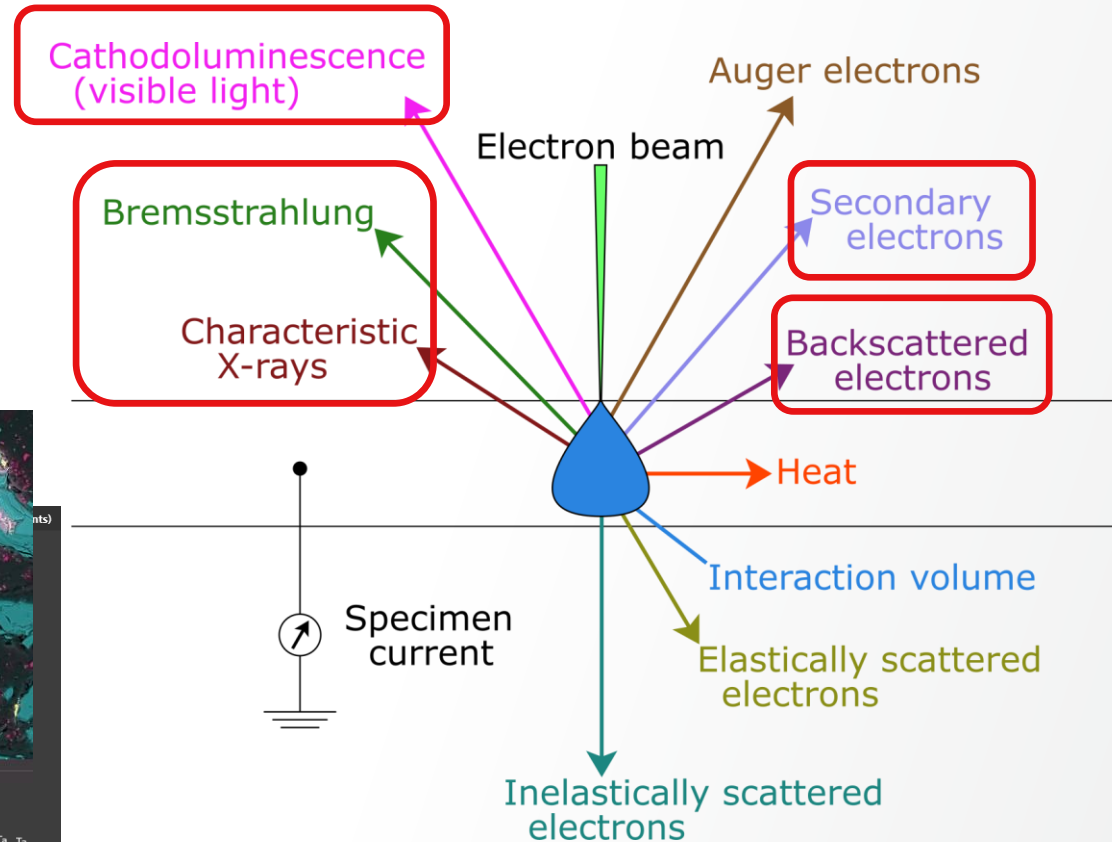
# SEM enables a vast variety of information available:



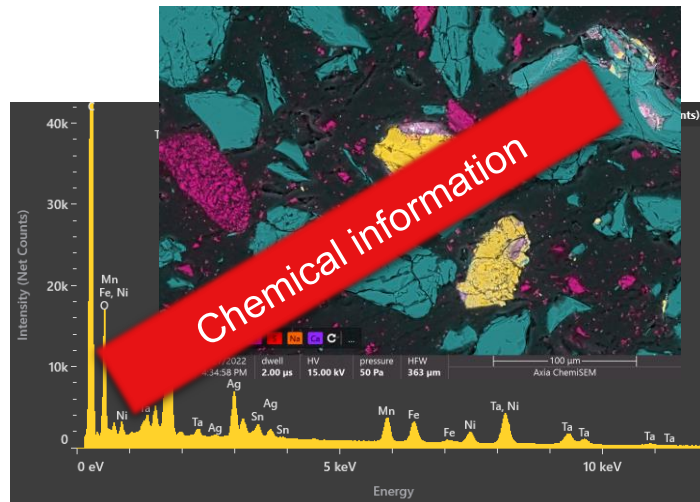
Luminescent materials in a display



Topography & Morphology



Composition contrast

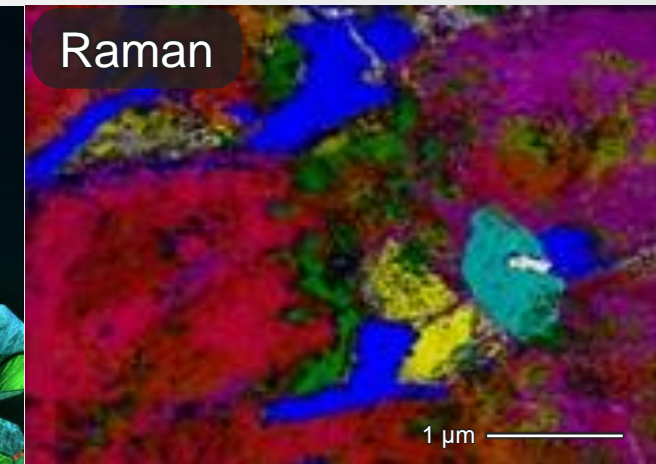
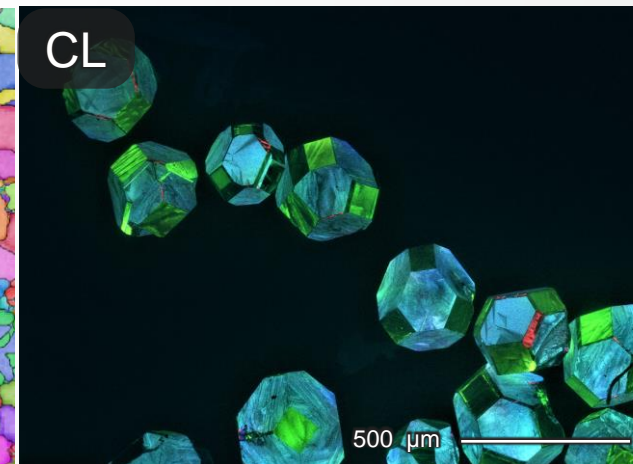
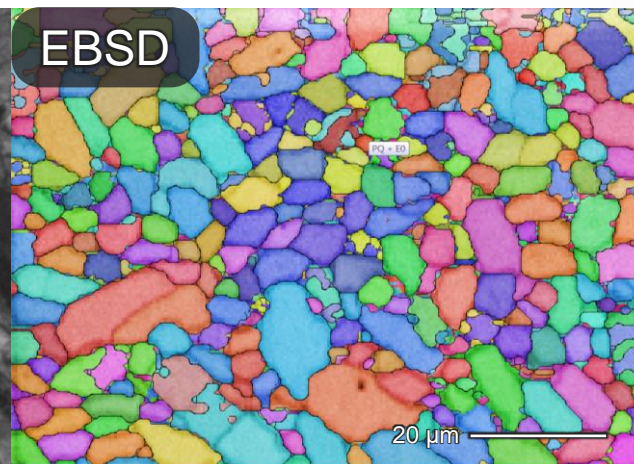
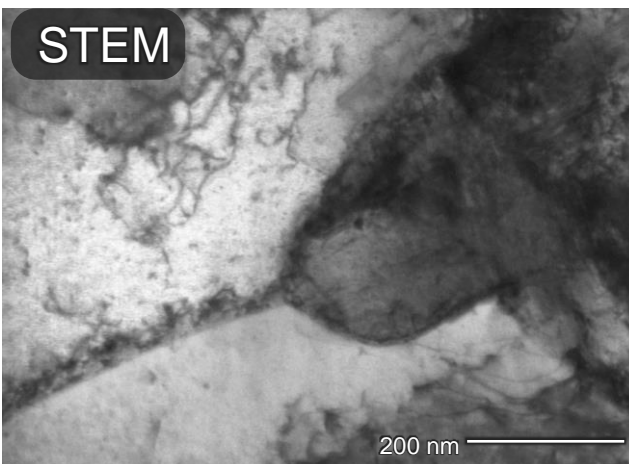
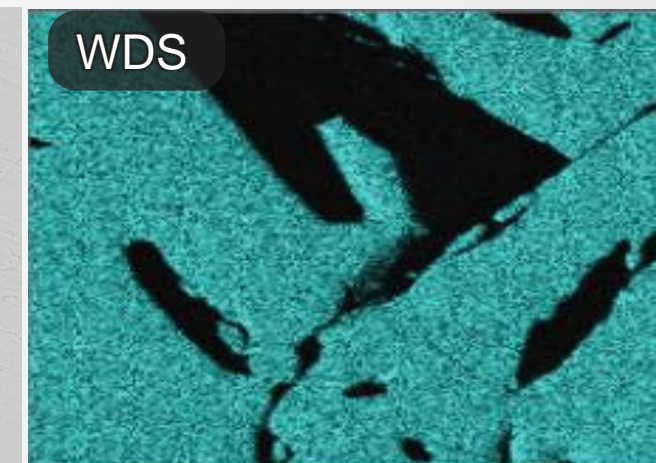
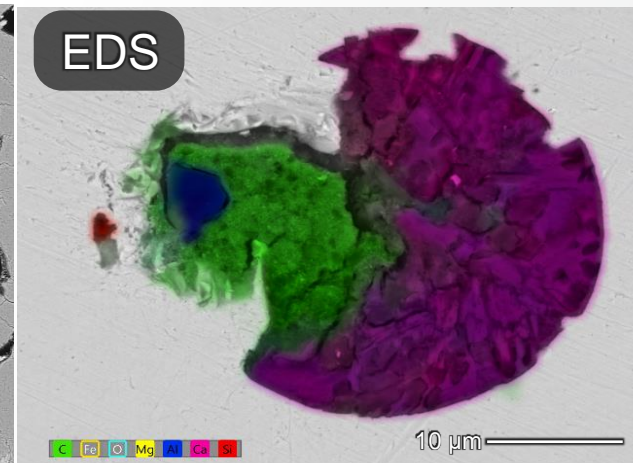
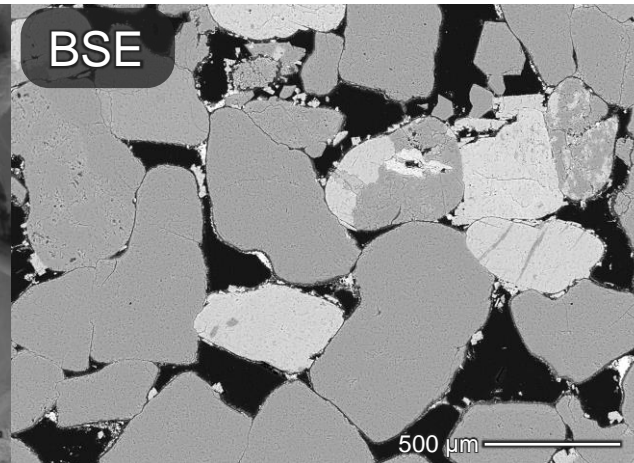
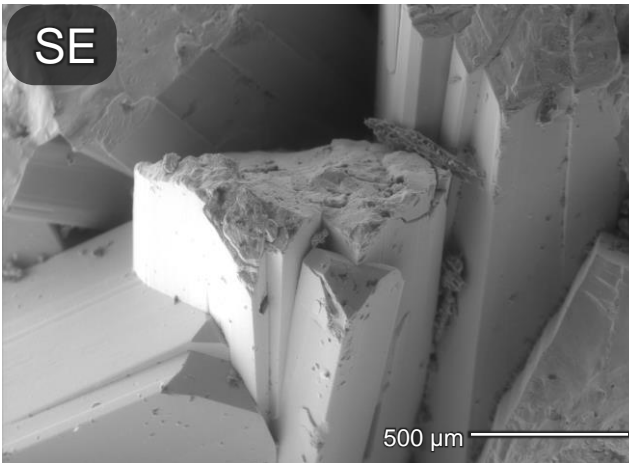


Chemical information

Today, EM is a multimodal instrument that doesn't just "take pictures".

# SEM enables a vast variety of information available

Floor model SEMs allow for a wide variety of information and analysis



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# Controlling Sample Humidity – Bulk Samples



Salt crystals dissolving and recrystallizing.

Conditions: 10 kV, GSED detector, sample temperature 2° C, chamber pressure 300 – 620 Pa

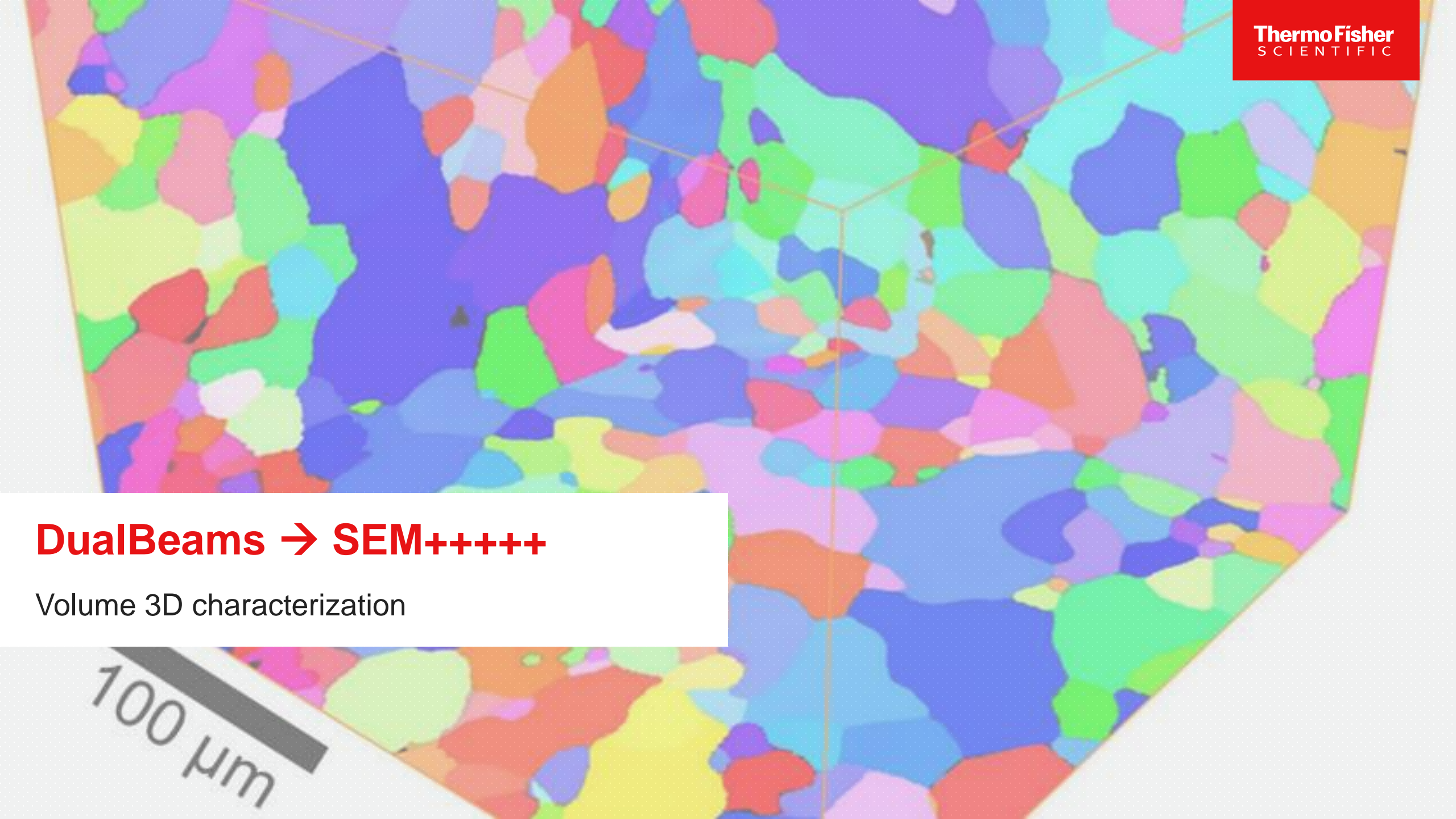
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Image courtesy: Yigit Oztan, Eindhoven NanoPort

**DualBeams → SEM+++++**

Volume 3D characterization

100  $\mu\text{m}$

The image shows a scanning electron microscope (SEM) micrograph of a material with a complex, multi-phase microstructure. The material is composed of numerous small, irregularly shaped grains or particles, each rendered in a different color (including shades of blue, green, yellow, orange, red, pink, and purple). These grains are densely packed and separated by thin, dark lines representing grain boundaries. The overall appearance is that of a polycrystalline or multiphase material. In the bottom-left corner, there is a white rectangular scale bar with the text "100 μm" written in black. The background of the image is a light, neutral color, likely the surface of the sample or the SEM stage.

# DualBeam for the 3<sup>rd</sup> dimension: SEM + FIB



FIB

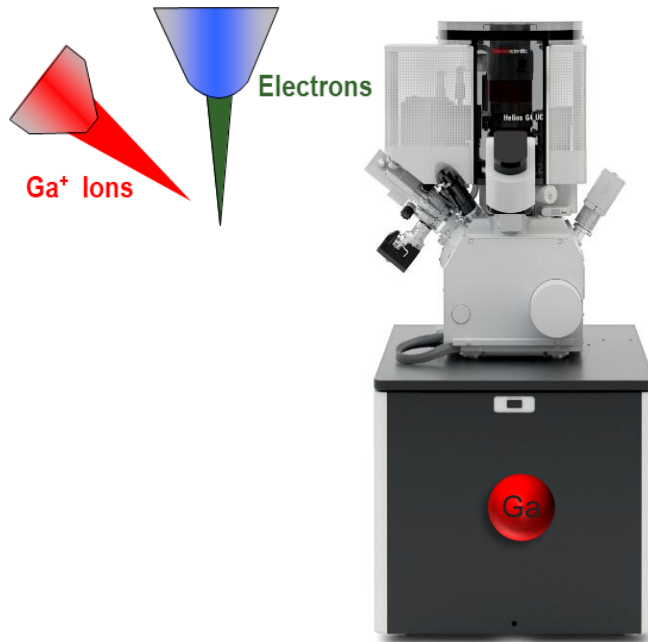
SEM



# DualBeam Technologies

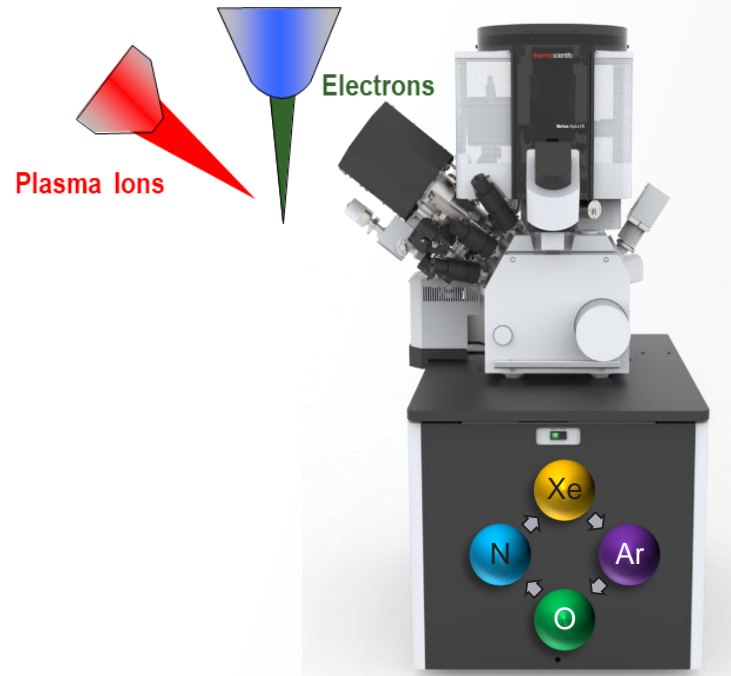
- Different ion sources (incl. laser) to cover wide number of applications in different materials system
- Increasing volume for analysis: Ga<sup>+</sup>-FIB → P-FIB → Laser PFIB

## Ga<sup>+</sup>-FIB



Ga<sup>+</sup> ion source

## Plasma-FIB



Plasma ion source  
(Xe, Ar, N, O)

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## Laser-PFIB



Femtosecond Laser +  
Plasma ion source

# DualBeam Technologies: Analyzed 3D Volume Comparison

3D volumes acquired within the same amount of time with FIB, Plasma FIB, and fs-laser:

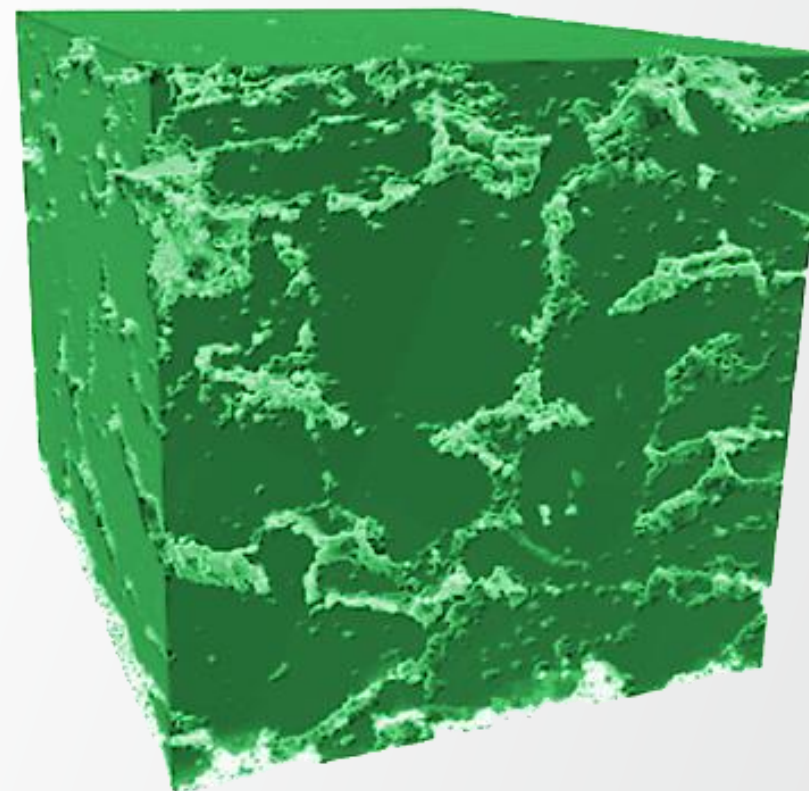
Example: Graphite anode (slow to mill with every FIB)



FIB (1x)



PFIB  
(~40x)



fs-Laser ~15000x

500  $\mu\text{m}$

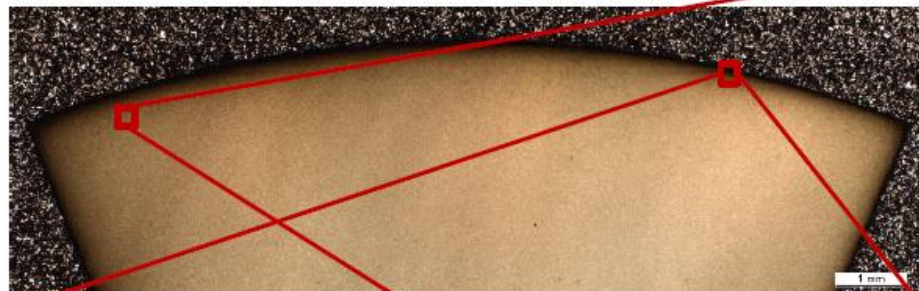
Representative length scale of DualBeam platforms:

- Ga-FIB: ~ 50  $\mu\text{m}$
- Plasma-FIB: ~ 50 - 200  $\mu\text{m}$
- Laser PFIB: 200  $\mu\text{m}$  to mm

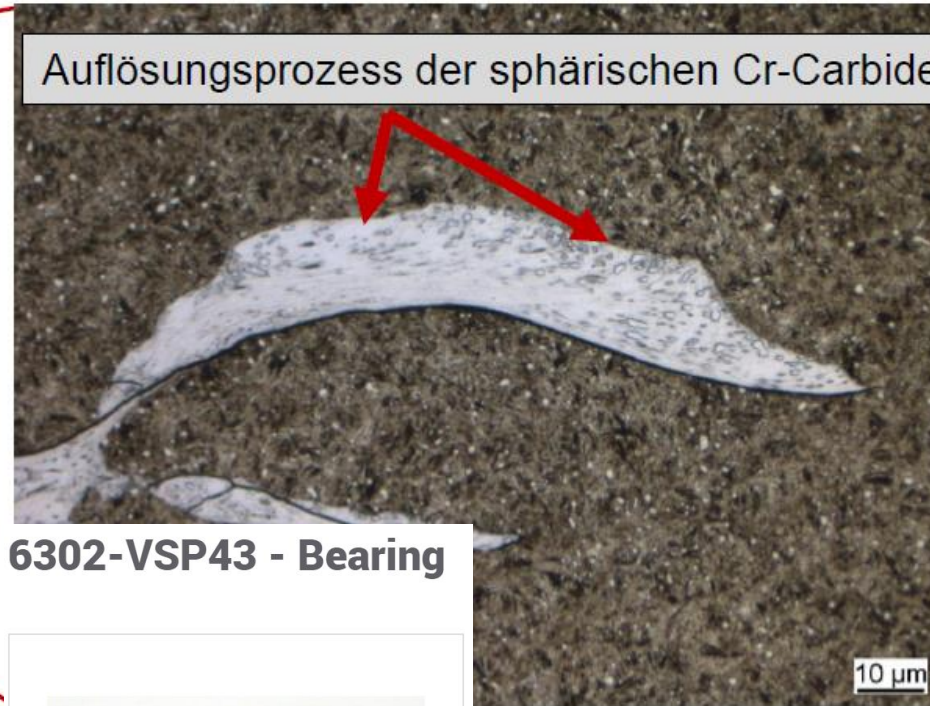
# Metallographische Untersuchung

## Voranalyse für Mikrostrukturuntersuchung - VSP 43 (16\_0245)

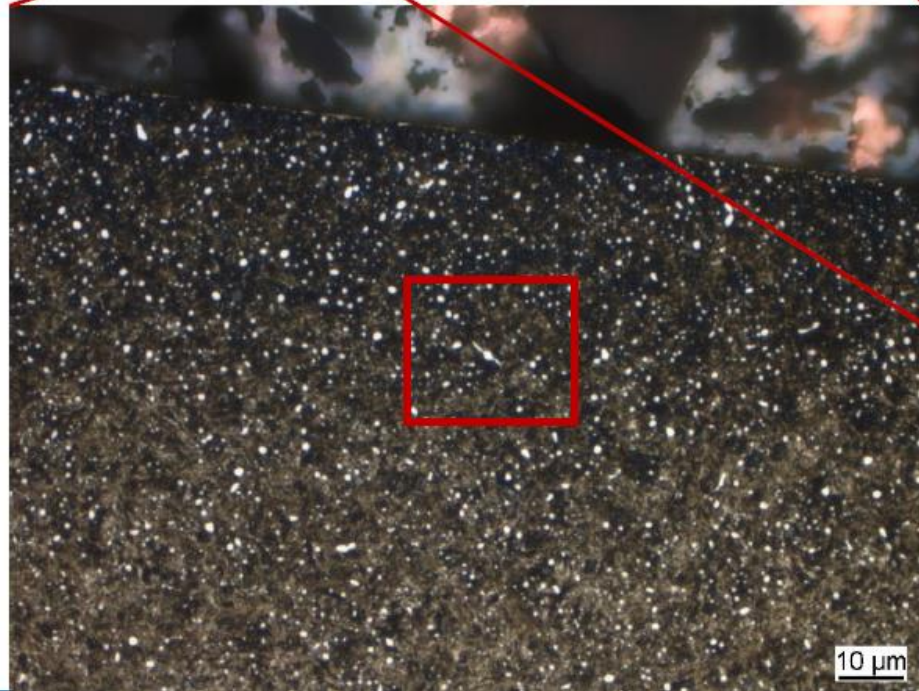
Metallografischer Schliff



Auflösungsprozess der sphärischen Cr-Carbide



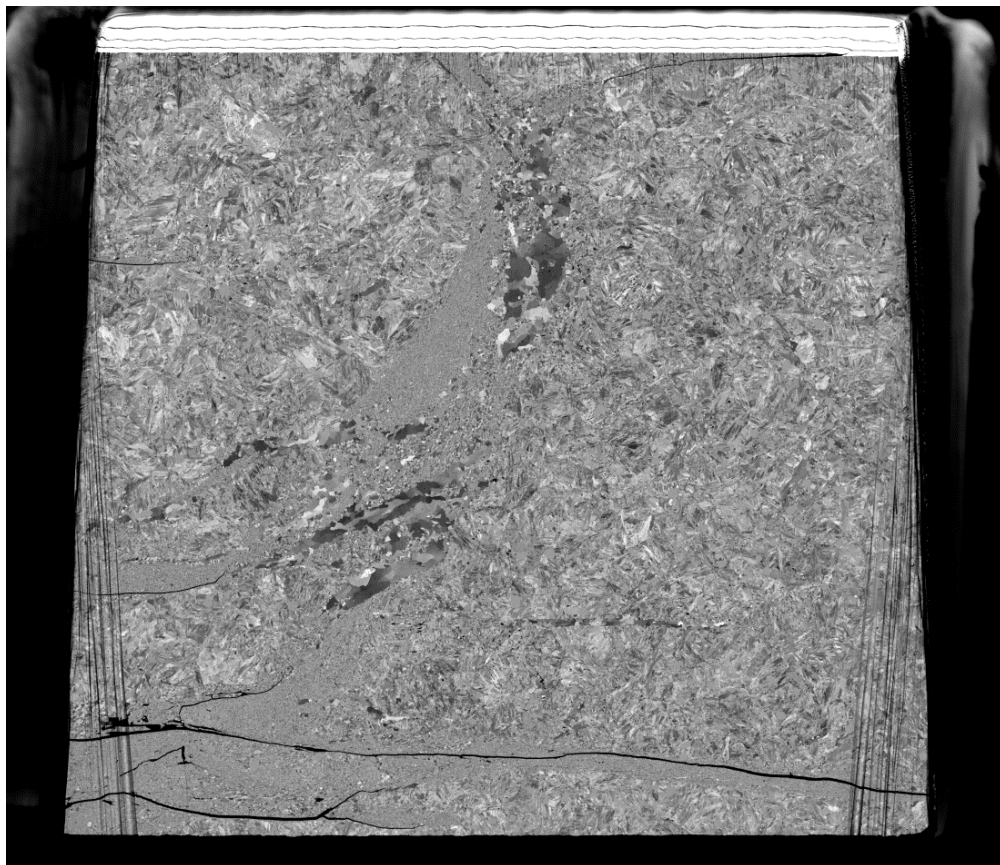
6302-VSP43 - Bearing



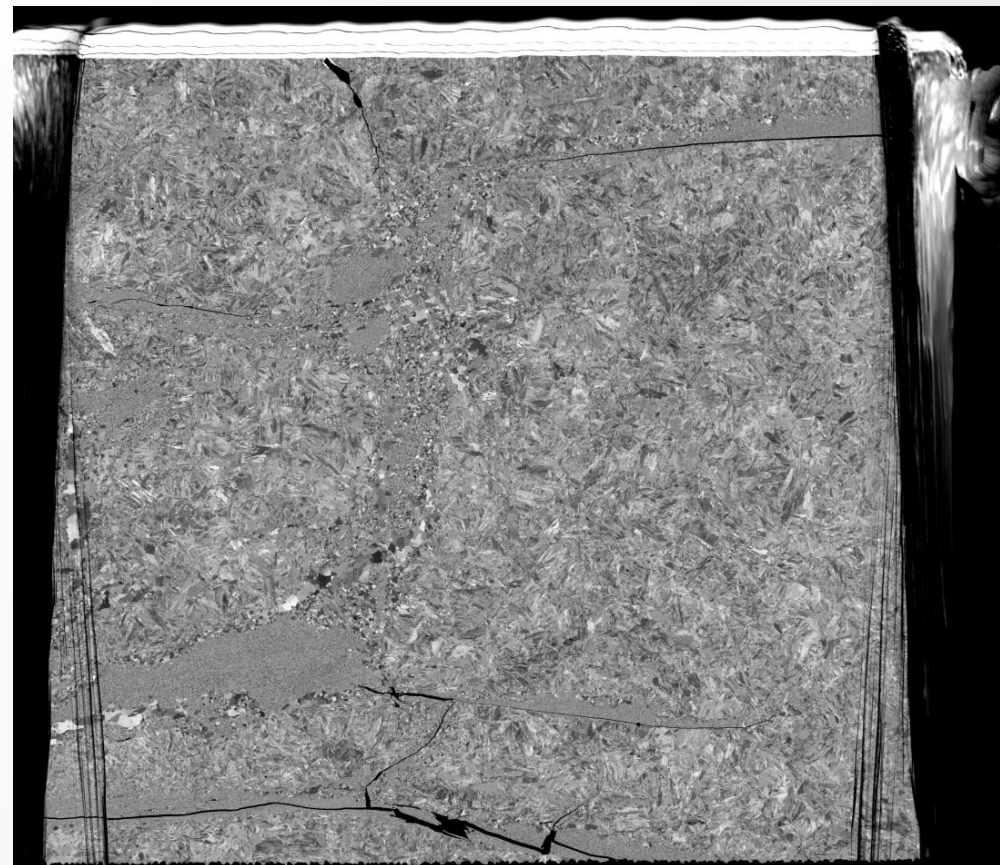
Ba

# GFE – Auto Slice & View Images

[100 nm slice thickness]



Slice 1



Slice 585

# GFE – Segmentation & Animation



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# Composition analysis in 3D with EDS

## Example: battery anode

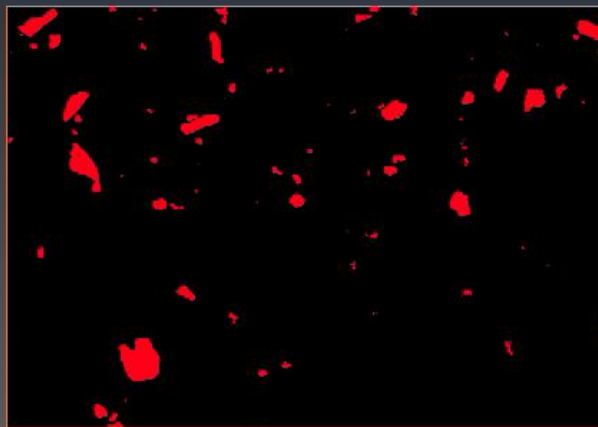
Adding Si to the graphite anode increases the electrochemical performance.

CMC (carboxymethylcellulose) is added as it has a higher binding strength to silicon and higher elastic moduli to withstand extremes volume changes than the traditional PVDF binder

EDS in 3D is used to see the distribution of Si and CMC

SiO<sub>x</sub>

Si

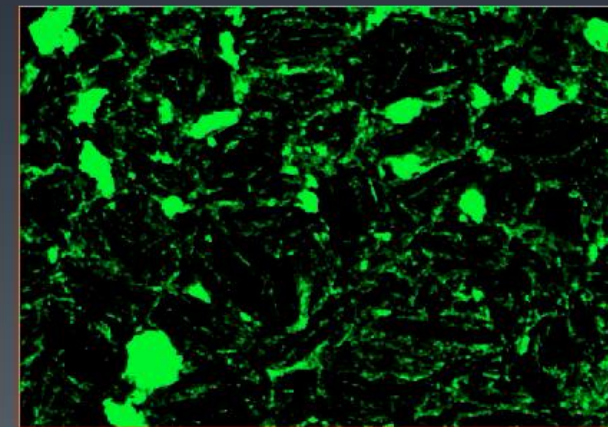


Avizo™

10 μm

CMC

O

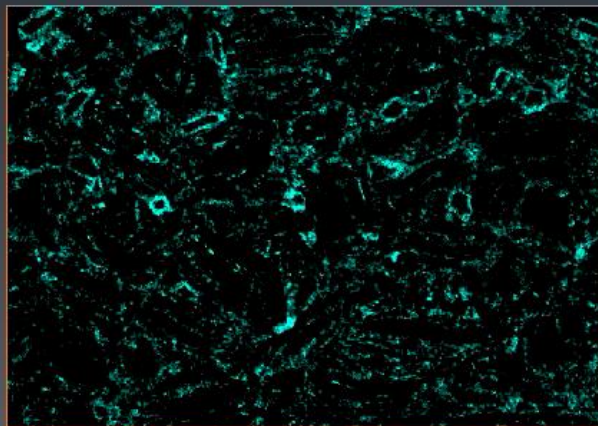


Avizo™

10 μm

PVDF

F

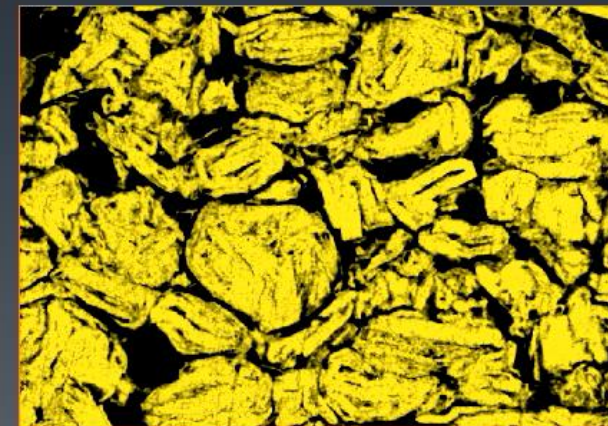


Avizo™

10 μm

Graphite

C

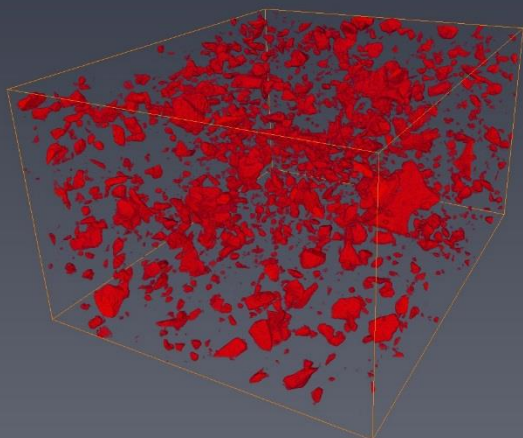


Avizo™

10 μm

# Anode composition in 3D with EDS

SiO



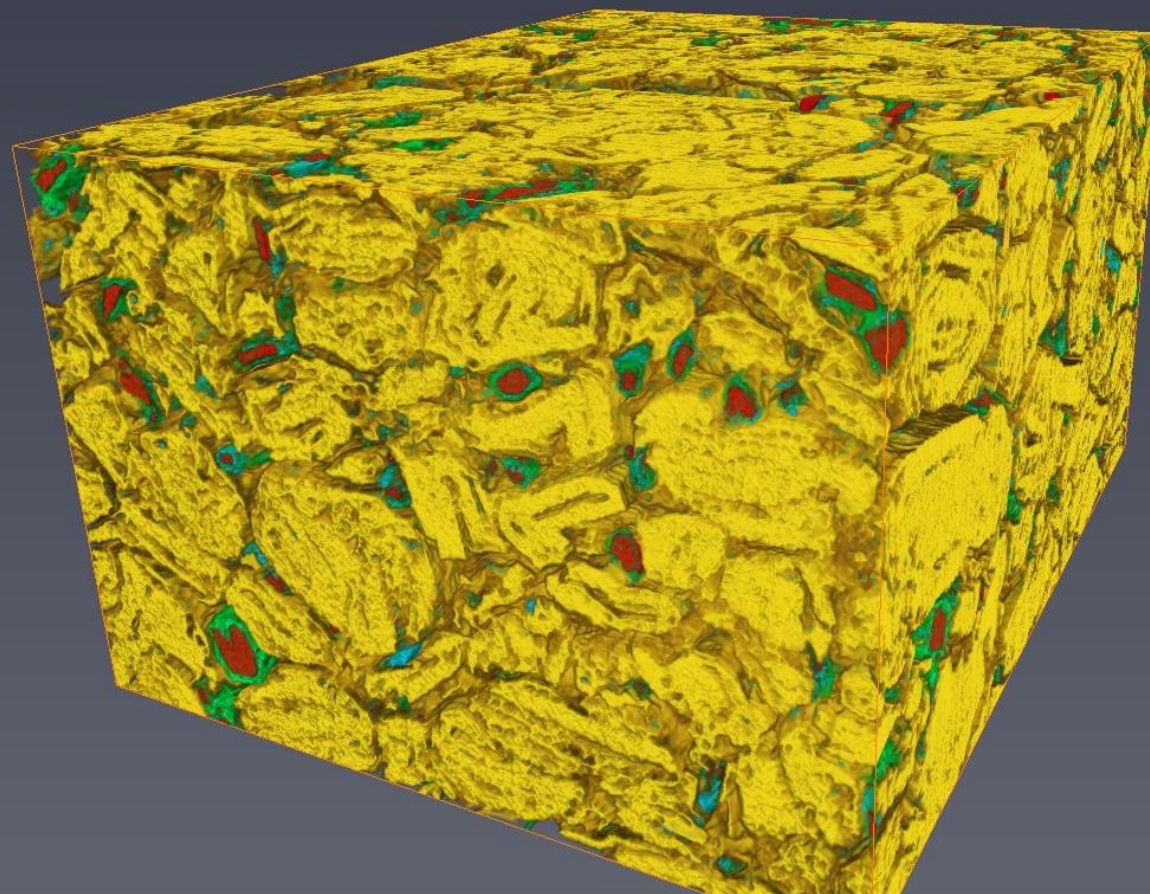
30 μm

SiO

CMC

PVDF

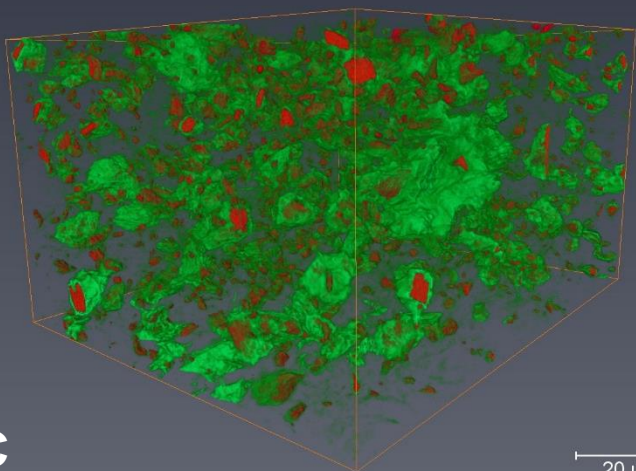
C



25.2 μm

SiO

CMC



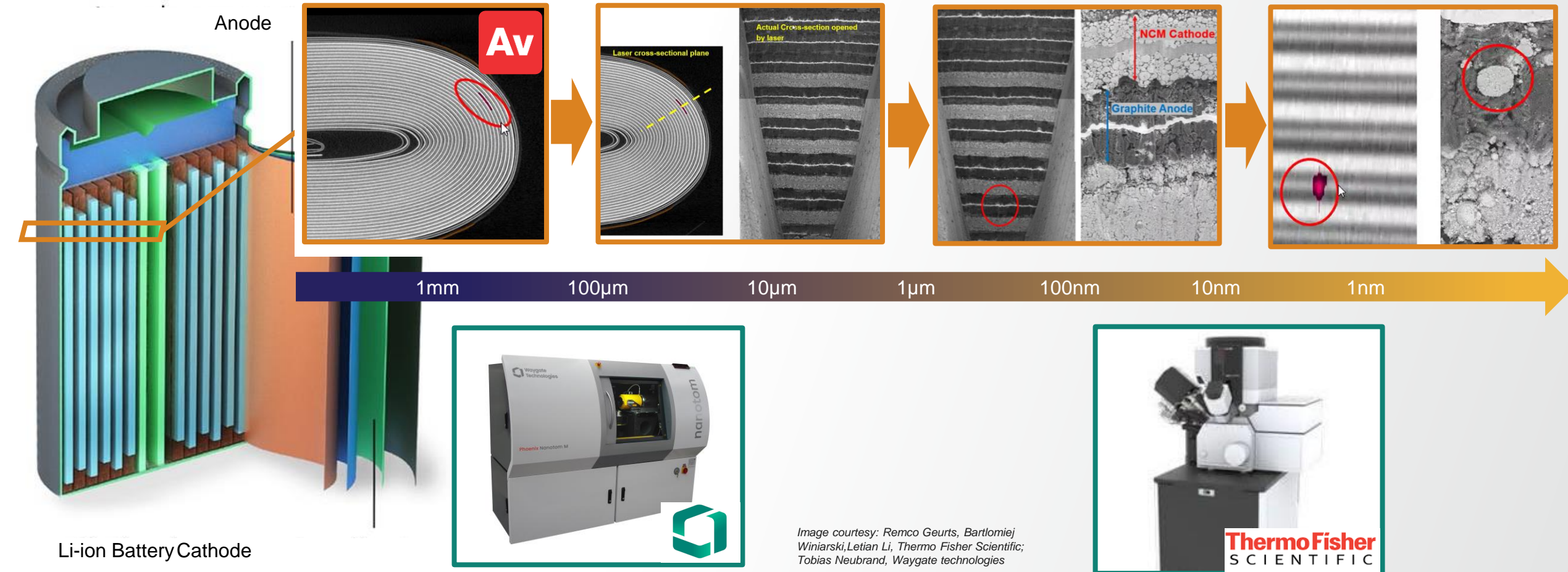
20 μm

CMC

The CMC is seen to coat the Si particles in the graphite matrix

# Failure analysis: impurity detection in pouch cell

## Production FA: CT to EM for impurity analysis



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# Summary

- EM provides wealth of signals
- 3D FIB analysis correlates structure with property
- CT and EM are complementary techniques that WANT each other! 😊

# Thank you

