COVALENT METROLOGY

Welcome

COVALENT METROLOGY

3D TOMOGRAPHY USING THE DUALBEAM (SEM-FIB): IMAGING, EDS AND EBSD

JOINT SPEAKERS:

Rick Passey
Product Marketing Engineer,
SEM and Small DualBeam,
Thermo Fisher Scientific

and

Naima Hilli, PhD
Director, SEM & FIB,
Covalent Metrology

Jan 28, 2021 | 11am PDT

ThermoFisher SCIENTIFIC

COVALENT METROLOGY



COVALENT METROLOGY

Silicon Valley-based analytical labs and platform delivering quality data and expert analysis for advanced materials and device innovation



Comprehensive Solutions Stack

40+ cutting-edge instruments, offering 100+ Techniques

Analytical Services

Advanced Modeling

Method Development

Temp. Staffing Solutions



Affordable and Fast

Fast Turnaround Times, No Expedite Fees

Volume Savings

Instant Access to Data and Reports in Secure Portal



Flexible Business Model

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Training and Certification on Instrumentation

Co-op and Tool-Share Opportunities

Laboratory Audits

Rigaku · J.A. Woollam · ThermoFisher SCIENTIFIC · PVA TePla · Anton Paar · Asylum Research an Oxford Instruments company

Rich Network of Partnerships

Partner to World's Leading Instrument Manufacturers and Labs

Expanding Instrumentation, Lab Connections and Learning



Who We Are, Who We Serve

500 Clients + 40-60 New Clients/ Quarter

40 People, 13 PhDs

Cutting-edge Analytical Capabilities

Lab Locations: Sunnyvale, Santa Clara

Covalent Technical Groups and Organization

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PCBA, Semiconductor, and Electronic Device Metrology & Failure Analysis

- DPA / Mechanical Cross-section
- Dye & Pry Test
- Hot Spot Detection
- IR Imaging / Emission Microscopy
- Root-Cause Failure Analysis
- EBIC / OBIC failure analysis

+ many more advanced methods!

Electron / Scanning Probe Imaging & Microscopy

- SEM (+ EDS)
- FIB-SEM (+ EDS)
- S/TEM (+ EDS / + EELS)
- AFM & Advanced AFM Modes
- Scanning Acoustic Microscopy (SAM)

Optical Microscopy & Spectroscopy

- Laser Scanning Confocal
- White Light Interferometry
- Chromatic Aberration
- Digital Optical Microscopy
- Spectral Ellipsometry
- UV-Vis-NIR Spectroscopy

X-Ray Characterization

- X-Ray Diffraction
- X-Ray Reflectometry
- Micron-spot ED-XRF
- WDXRF
- Micro-CT
- 2D X-ray Inspection & X-ray Radiography

Chemical Analysis

- ICP-MS & LA-ICP-MS
- GC-MS
- FTIR
- Raman
- NMR (1D or 2D; solid-state and solution-state)
- EPMA

Nanoparticle Analysis

- Dynamic Light Scattering (DLS)
- Laser Diffraction Particle Size Analysis (PSA)
- Particle Zeta Potential

Material Property Characterization

- DSC
- TGA
- DMA & TMA
- Rheometry
- Surface Zeta Potential
- Nanomechanical Analysis
- Tensile-Test
- Taber Test

Surface Analysis

- X-ray Photoelectron Spectroscopy (XPS)
- Ion Scattering Spectroscopy (ISS)
- Ultraviolet Photoelectron Spectroscopy (UPS)
- ToF-SIMS (Static-SIMS)
- Dynamic-SIMS

Covalent Instrument Partners

ThermoFisher SCIENTIFIC

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- **Thermo Scientific ™ Talos ™ F200 G2 (Scanning) Transmission Electron Microscope (S/TEM)** – delivers fast, precise, quantitative imaging and elemental composition analysis on an angstrom scale with powerful electron optics and a high-sensitivity detector array
- **Thermo Scientific Helios 5 ™ DualBeam ™ Microscope** – incorporates the most advanced electron optics, detectors, and software technologies available for materials science to deliver unprecedented imaging performance with sub-nm resolution
- **Thermo Scientific ™ Nexsa ™ X-ray Photoelectron Spectrometer (XPS)** – produces high-throughput, high-quality results across XPS, ISS, UPS, REELS, and Raman spectroscopy with fully automated multi-technique workflows that allow true correlative analysis

Other Partners

Rigaku

PVA TePla

J.A. Woollam

KEYENCE

OXFORD INSTRUMENTS

ASYLUM RESEARCH

Rick Passey

Product Marketing Engineer, SEM and Small DualBeam, Thermo Fisher Scientific

Rick Passey is an SEM/DualBeam applications expert who has been with Thermo Fisher Scientific (formerly FEI) for 12 years. His experience covers a wide range of microscopes and techniques, from environmental SEMs to the plasma FIB, 3D EDS/EBSD characterization to advanced TEM sample preparation. Prior to working with FEI, Rick spent 13 years as a Process Engineer with Hewlett Packard, leading an SEM/FIB laboratory, supporting materials characterization and failure analysis of inkjet and related technologies.



Naima Hilli, PhD

Director, Scanning Electron Microscopy,
Covalent Metrology



Naima Hilli is the Director of SEM/FIB group, focusing on providing advanced Scanning Electron Microscopy (SEM) and Focused Ion Beam (FIB) services to Covalent's customers by using state-of-the art capabilities and expertise. She has 15+ years of experience in the microstructural characterization of materials by SEM, FIB, TEM and 3D reconstruction. Before joining Covalent Metrology, Naima was the SEM/FIB facility manager at Northwestern University, where she was overseeing the advanced SEM and specimen facility, providing training and consulting services to clients from both academia and industry on new material development, characterization, and failure analysis of various materials and devices including, alloys, polymers, ceramics, semiconductors, composites, and nanomaterials.

Naima holds a PhD in Materials Science and Engineering from the School of Mines (France), a master degree in Polymer Science and a BS in Materials science from the University of Lyon (France).

Introduction to 3D Tomography with DualBeam Technology

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What will be covered over the next 45 minutes?

- DualBeam Technology
  - Scanning Electron Microscopy (SEM)
  - Focused Ion Beam (FIB) Microscopy
- 3D tomography
  - Why is it used?
  - How is it done?
  - Considerations for the best results
  - Reconstruction examples

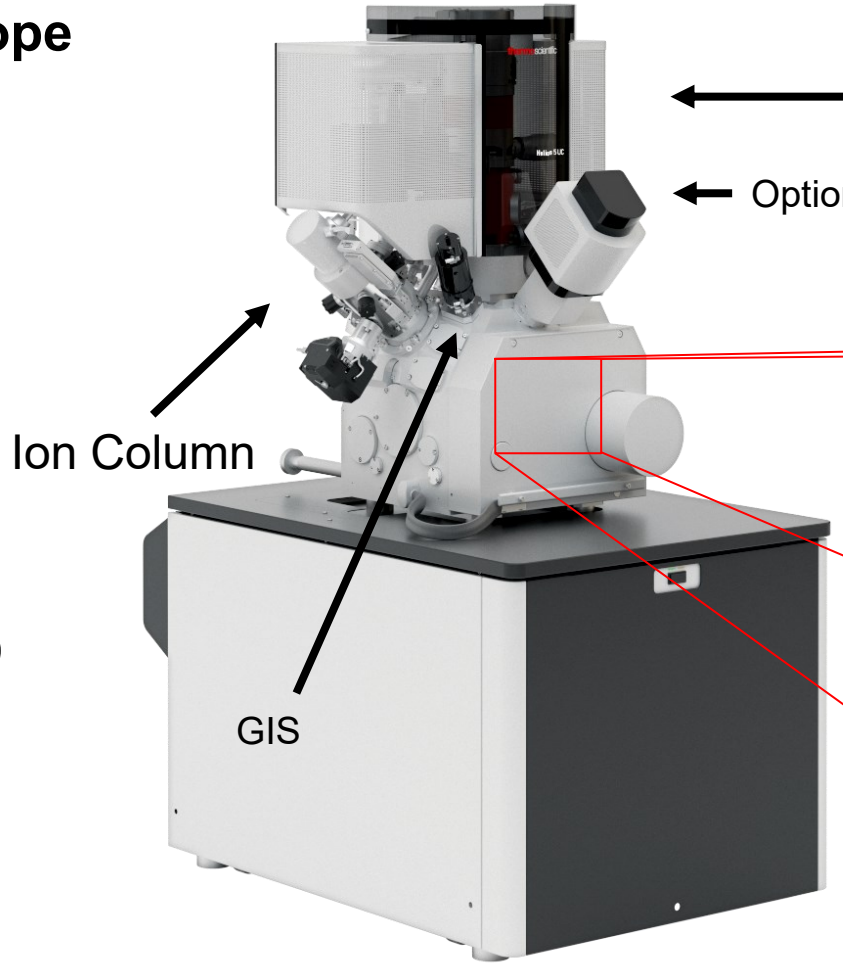
- Questions and answers

DualBeam Fundamentals

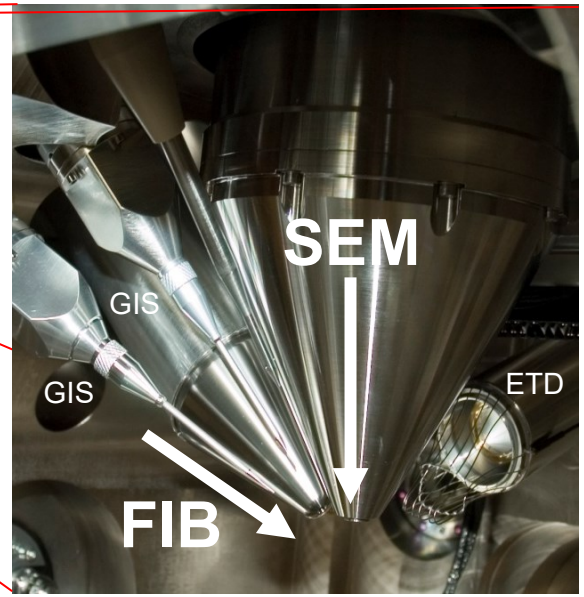
A DualBeam is defined by two primary components…

Scanning Electron Microscope
and
Focused Ion Beam

Other critical components…
Sample chamber
Stage
Vacuum system
Gas Injection Systems (GIS)
Manipulator

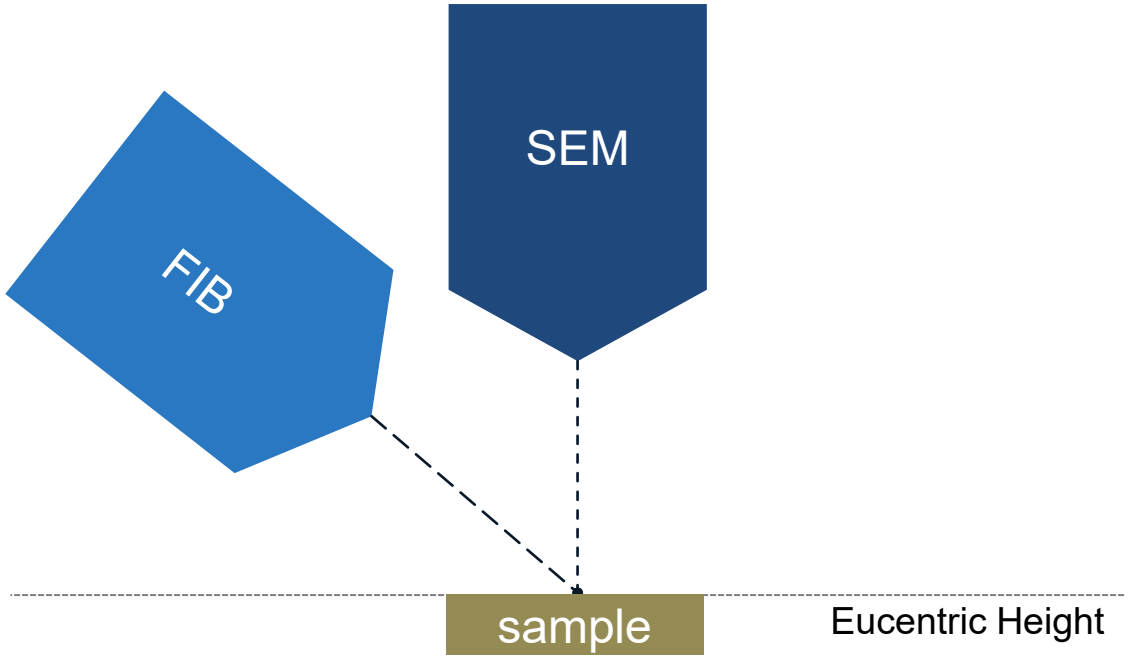


Bottoms of the columns



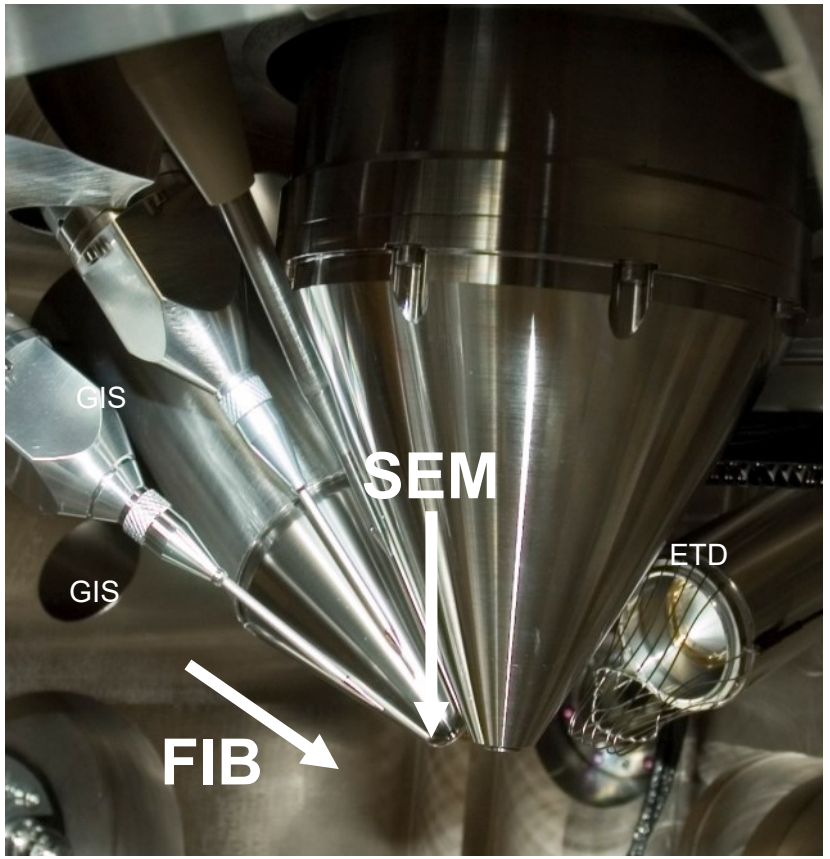
Inside the chamber

DualBeam Fundamentals



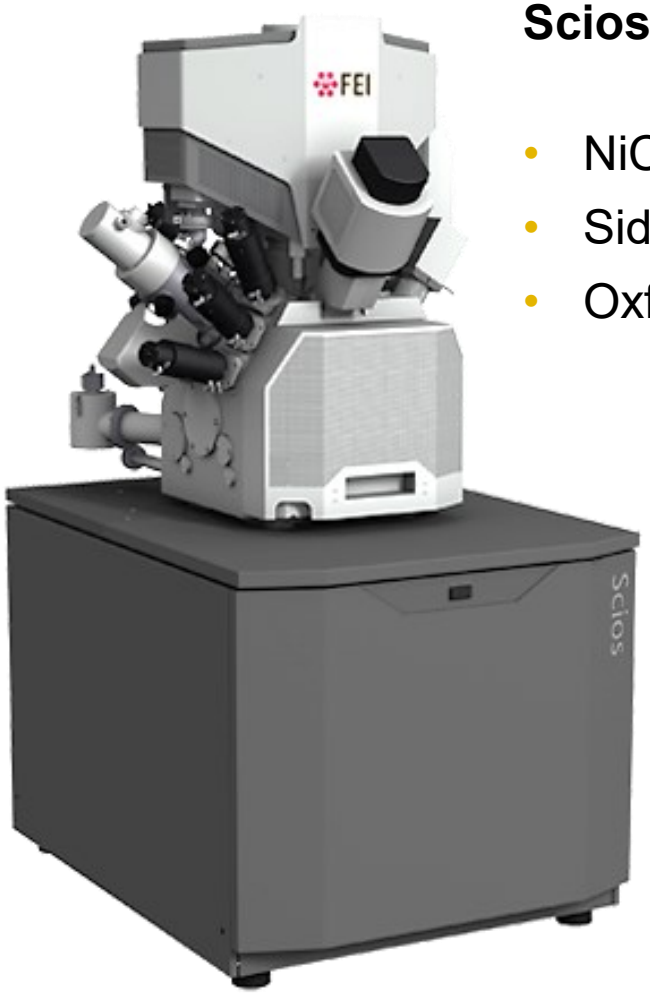
In general…

- The SEM is used for imaging
- The FIB is used for sample modification (milling)
- Sample tilt is used to optimize imaging and milling



Inside the chamber

Covalent DualBeams



Scios

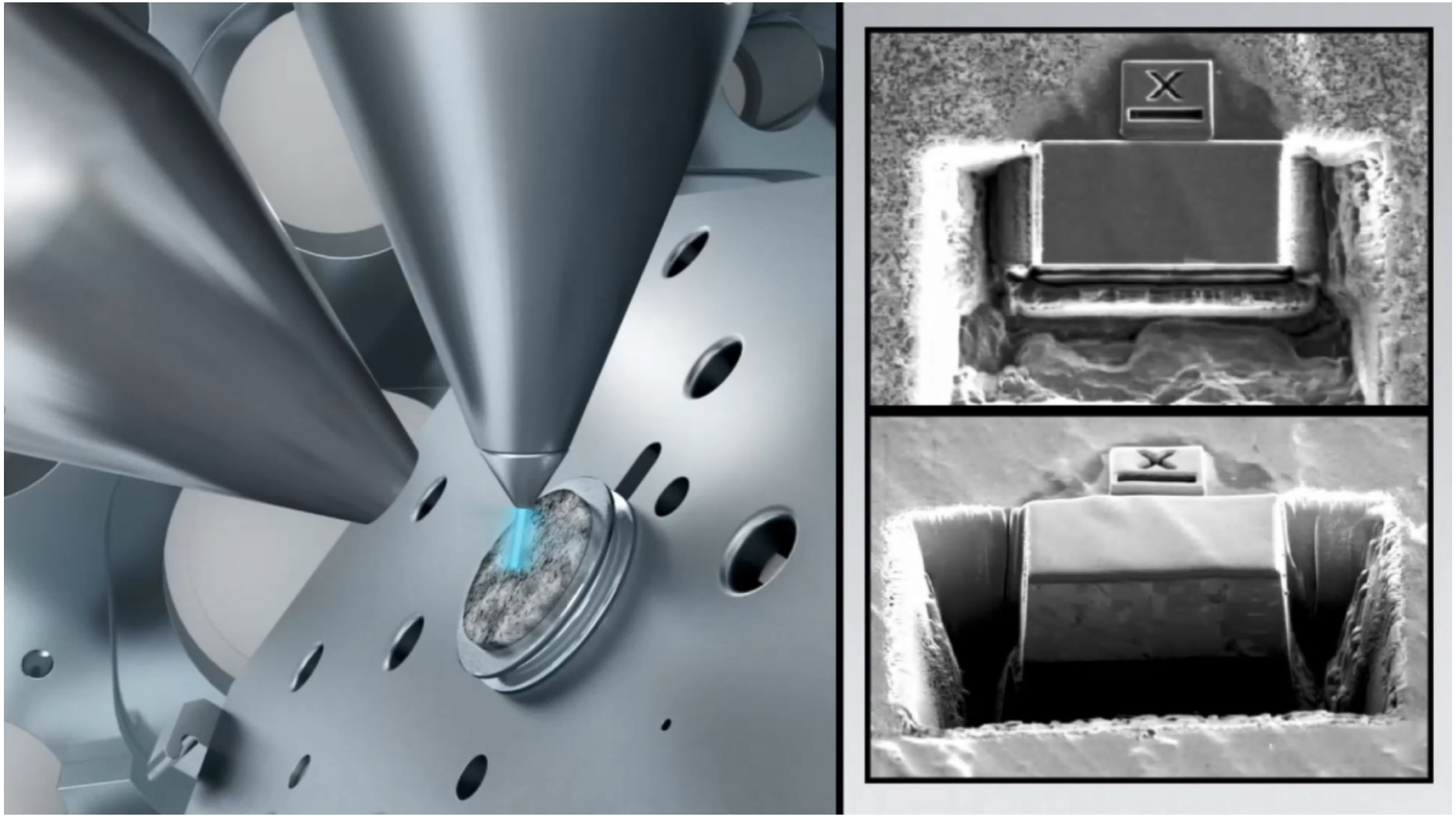
- NiCol e-column
- Sidewinder FIB
- Oxford 150mm^2 detector

Helios 5 UC

- Elstar UC+ e-column
- Tomahawk HT FIB
- Oxford 100mm^2 detector



DualBeam Fundamentals



- Ion-beam perspective: top-down relative to the beam
- Electron-beam perspective: a tilted view of the cross-sectioned face relative to the beam

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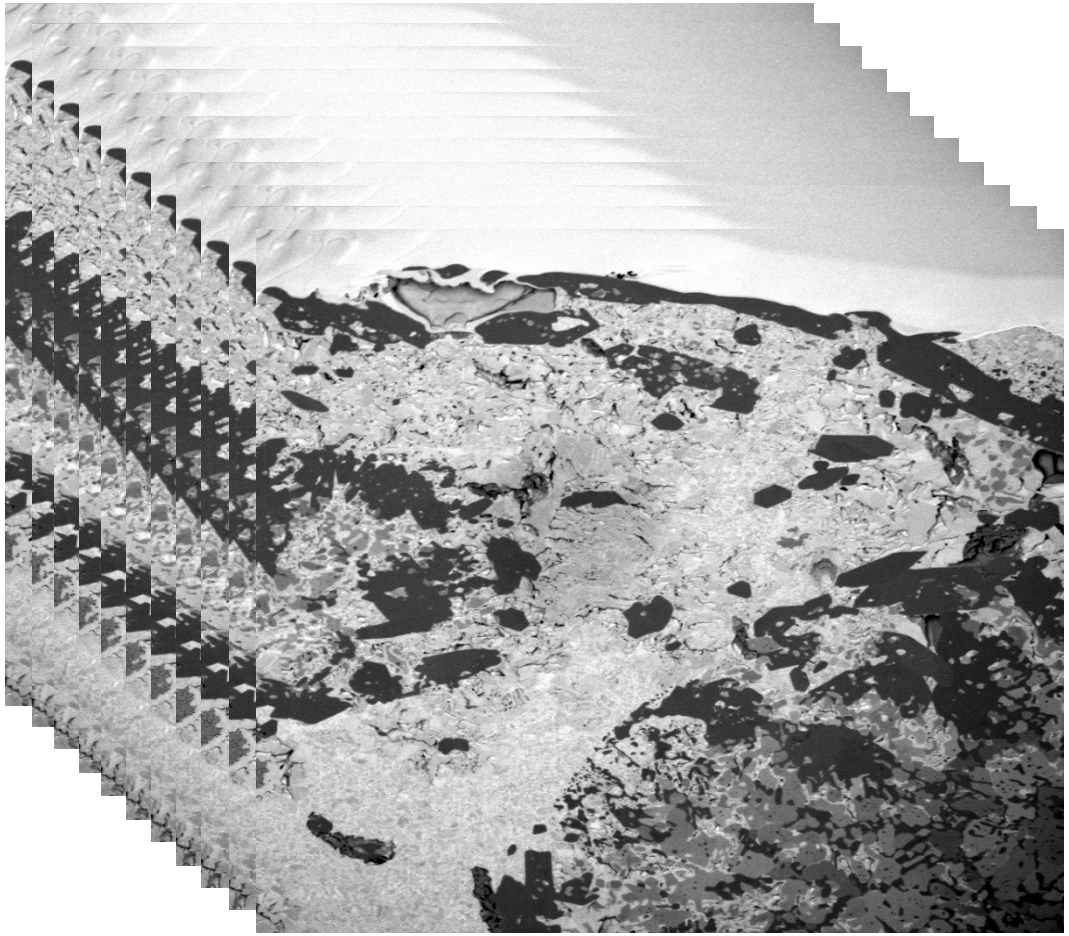
Summary

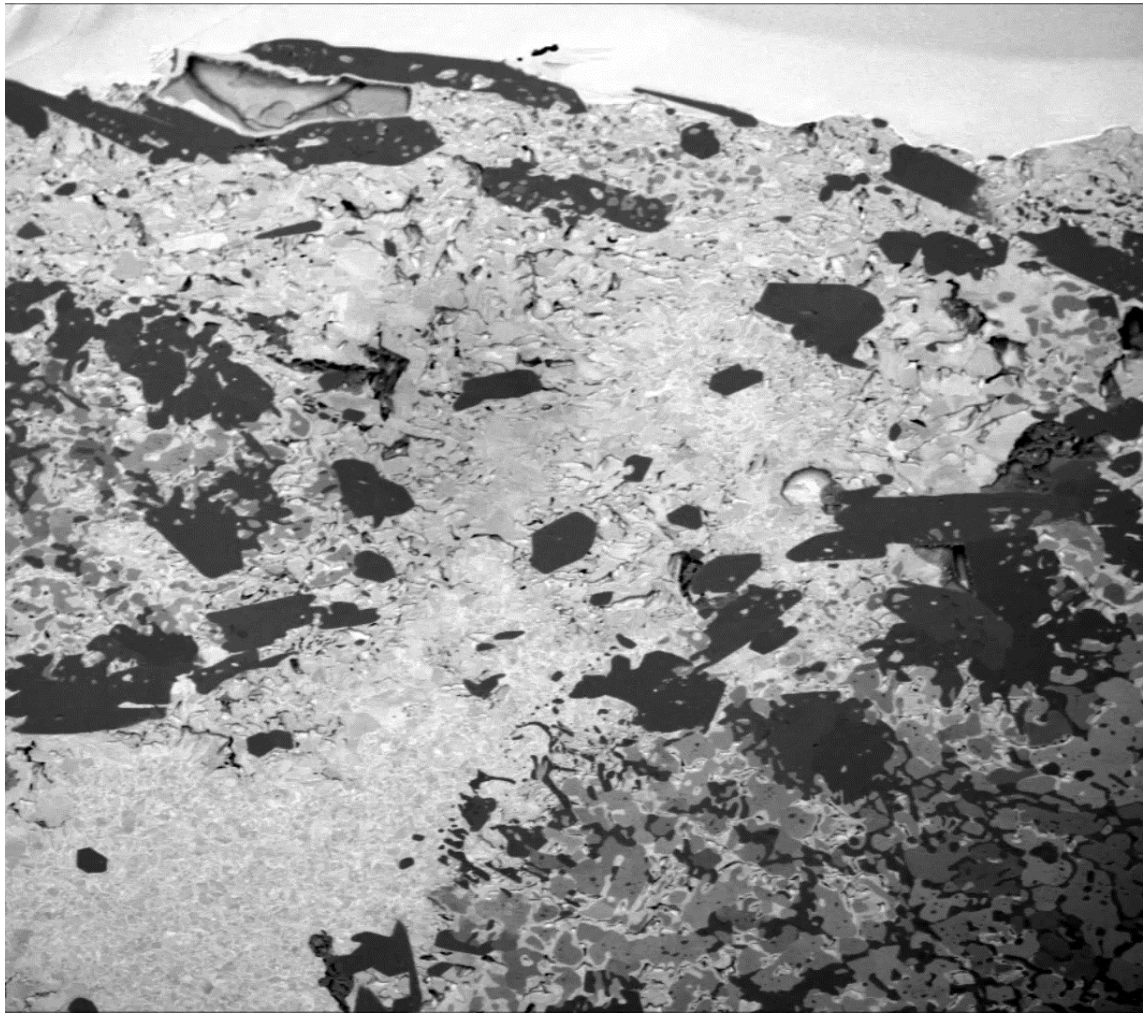
- The DualBeam combines an SEM and an FIB into one powerful platform for high resolution imaging and precise, site specific milling
- DualBeams can have different ion sources: gallium, Xe^+ plasma, or multi-ion source plasma (Xe^+, Ar^+, O^+, or N^+)
- The DualBeam can do more than just make a site-specific cross-section – TEM/APT tomography sample prep, 3D tomography, and lithography are all common applications
- The choice of ion source depends on application: gallium for the best sample prep, plasma for the fastest large volume milling
- Site specific milling can include protective capping materials and etch enhancement using gas injection

- There are many other applications for the DualBeam!

What is a 3D tomography?

Slice, collect data, slice, collect data , rinse and repeat 10s to 100s of times… reconstruct





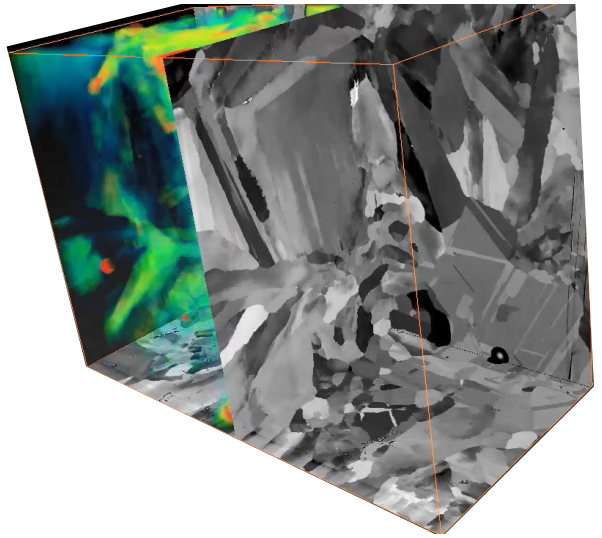
movie

What is 3D tomography?

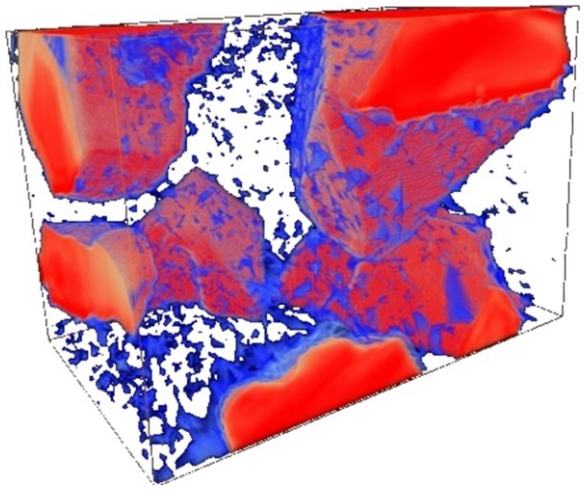
The source of the data can be:

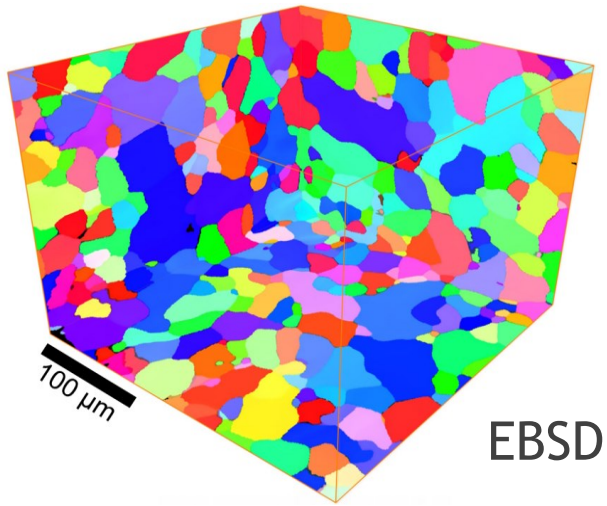
- SEM or FIB images
- EDS x-ray maps
- EBSD maps
  - IPF, unique grains, strain, etc.
- Or project can be a combination of all three!

SE/BSE/SI Images



EDS maps





EBSD maps

What is 3D tomography?

- All data is processed into a “stack” – a time sequenced compilation of each slice
- Stacked data is processed using high-end reconstruction/visualization software
  - Avizo for material science
  - Amira for life science
  - FIJI (ImageJ)
  - Other 3rd party options
- Processing includes several key steps
  - Aligning
  - Filtering
  - Segmentation
  - Data extraction
  - Animation

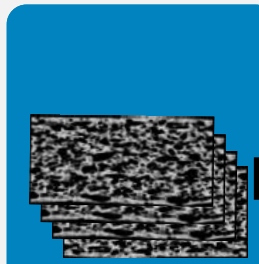
What is 3D tomography?

The 3D data collection and processing workflow

Data Acquisition

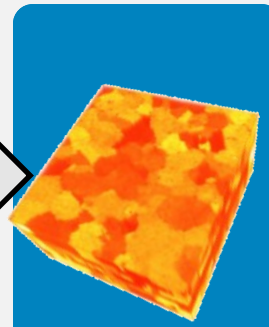


Image Processing



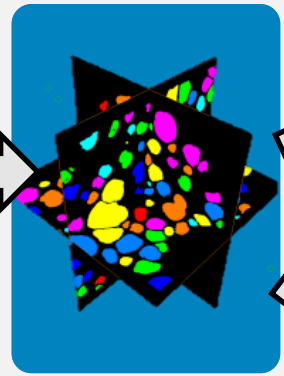
- Alignment
- Curtaining removal
- De-noising
- Shading correction

3D Exploration



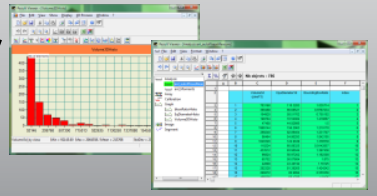
- Volume rendering
- Slicing
- Color mapping

Segmentation

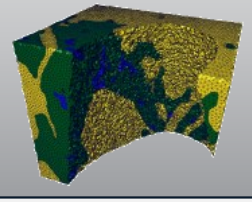


- Watershed segmentation
- Multi-phase segmentation
- Masking

Quantification, Simulation & Analysis

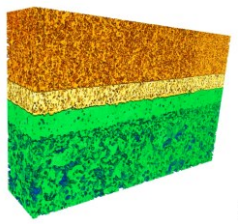


3D Model Reconstruction

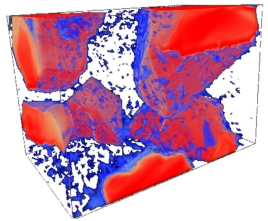


Multi-scale, multi-modal 3D characterization

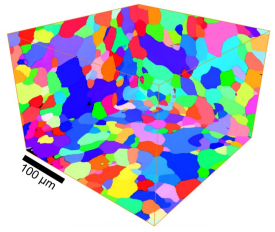
SE/BSE Images



EDS maps

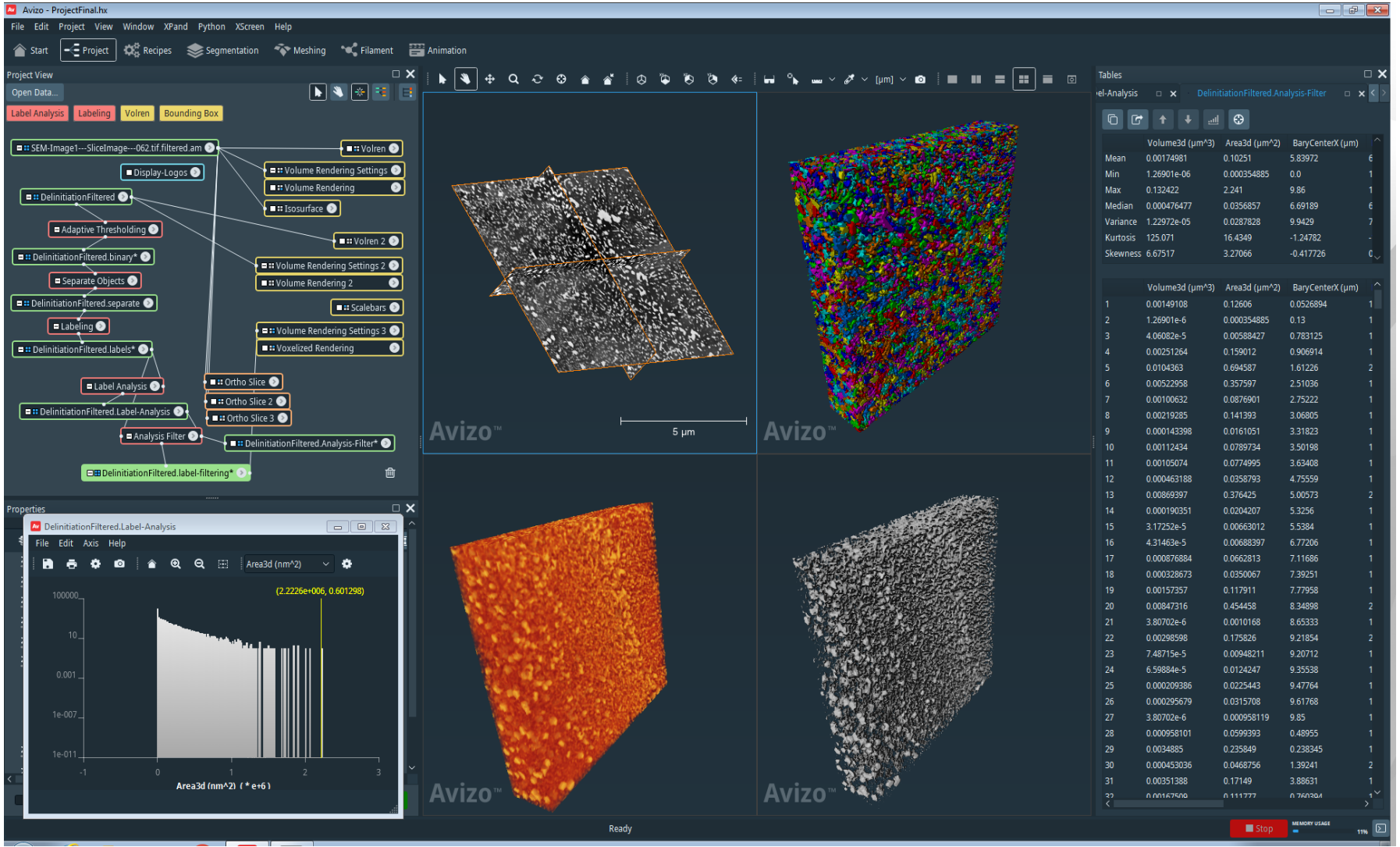


EBSD maps



What is 3D tomography?

Data reconstruction with Avizo 3D Reconstruction Software



- Top left: 3 ortho slices
- Top right: every instance of bright phase identified (> 30k in volume)
- Bottom left: Volren
- Bottom Right: Volume Rendering
- Right col: tabulation of every phase instance with individual measurements
- Bottom Left: Histogram of precipitate volumes

Considerations for collecting the best data

The key to achieving the best data starts with understanding the pixel–beam resolution relationship

Pixels are measured in the x and y axis

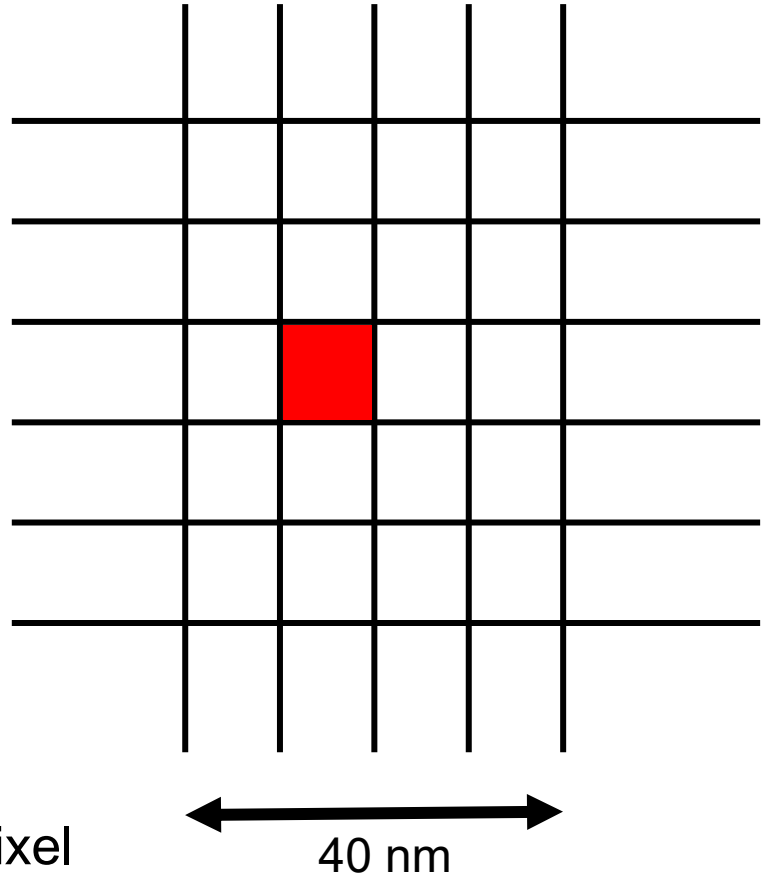
The digital resolution of the image determines the number of pixels, e.g. 1024 x 768, 4096 x 3072, etc.

The pixel resolution is set within the microscope user interface – it is selected from a list of options

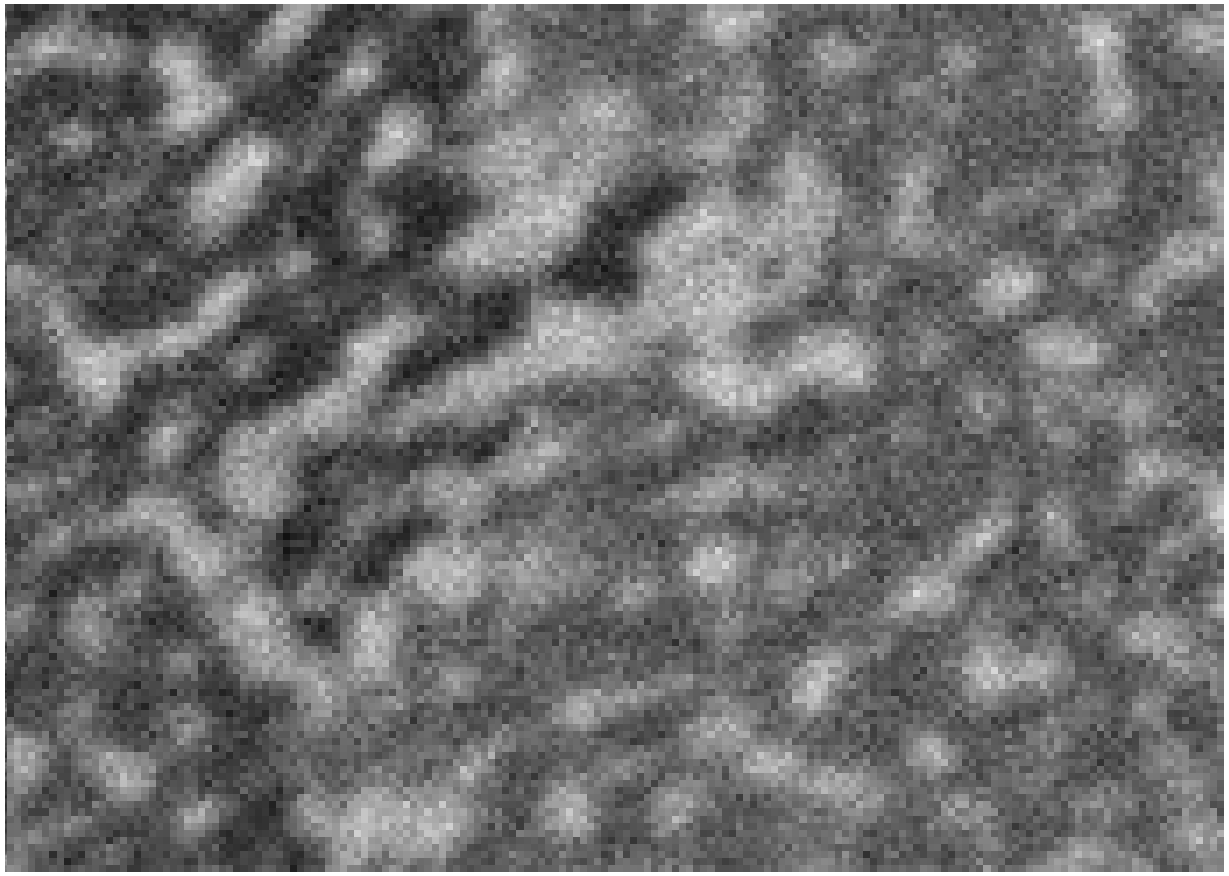
The pixel resolution is determined by the image width divided by the number of pixels

1024 pixels/10.24 µm image width = 10 nm pixel resolution

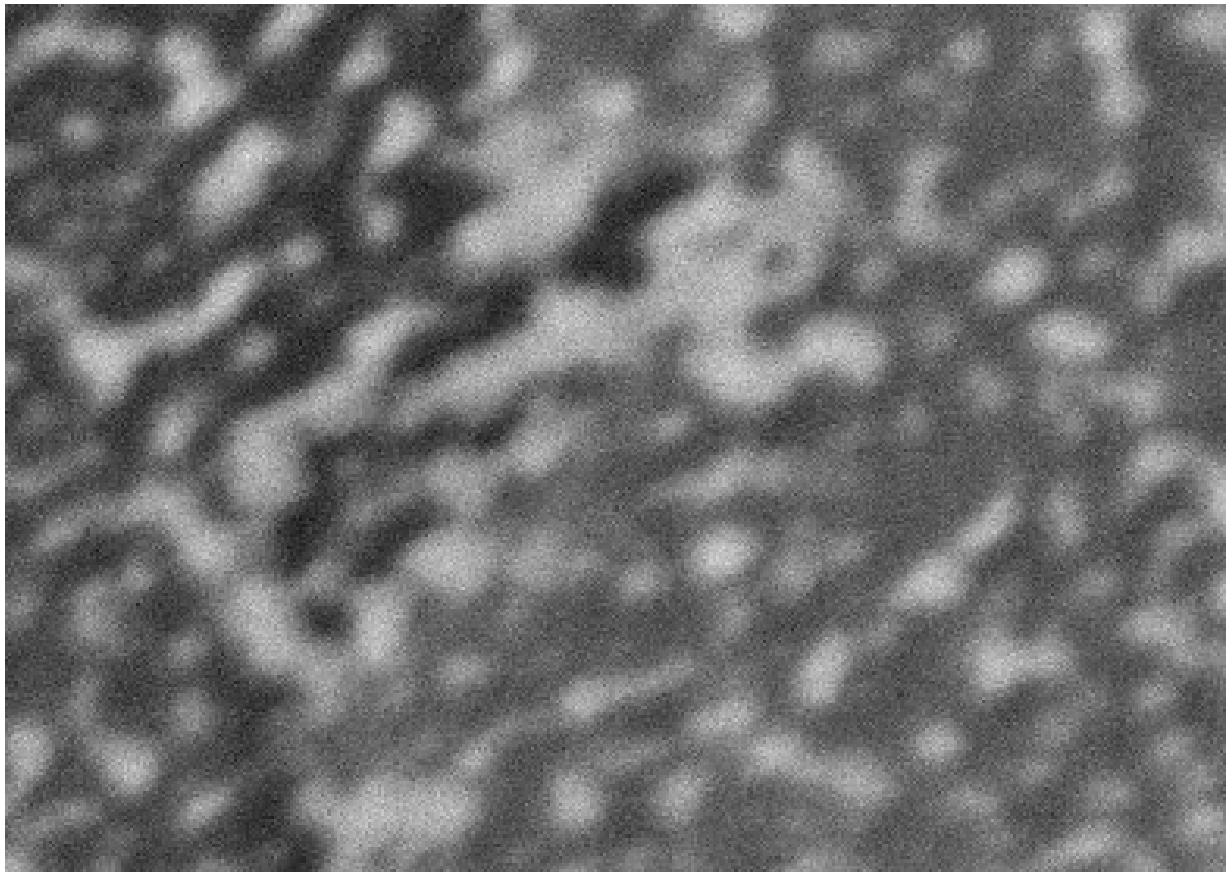
The probe-size of the electron beam should be smaller than the pixel



Considerations for collecting the best data



1536 x 1094, 14.5 nm pixel

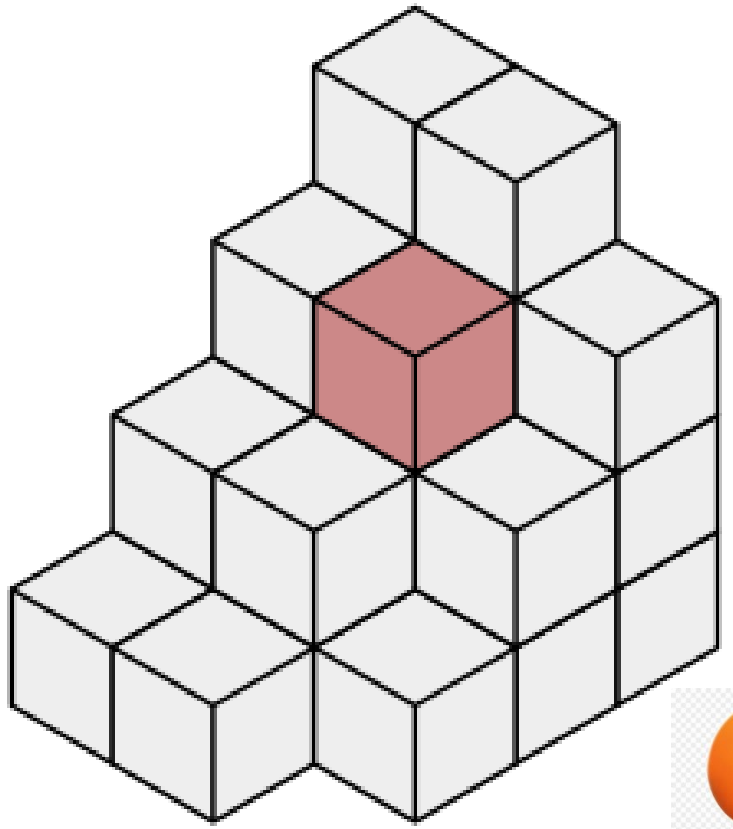


6144 x 4376, 5.8 nm pixels

Considerations for collecting the best data

The key to achieving the best data starts with understanding the “voxel” – the 3D version of the pixel

- Voxels are measured in the x, y, and z-axis
- The “z-axis” resolution is the chosen slice thickness
- What slice to use is dependent on specific factors:
  - The slice thickness should be close the x/y-axis resolution
  - The slice thickness will determine the resolution of the 3D reconstruction
    - There needs to be 5-7 pixels (or more) in a feature to delineate it as “real” vs noise





Considerations for collecting the best data



- How do you choose a voxel size?

    1. What is the smallest feature you need to resolve?

    2. Divide the size of the feature by 5 (or more)
        - More pixels in the feature = more data to reconstruct = higher “final” resolution
        - Too many pixels = oversampling = wasted time
        - EDS and EBSD have other factors to consider when choosing the voxel size

- If the slice thickness is larger than the smallest features, features will be milled away and not appear in the reconstruction (or only be partially visible)

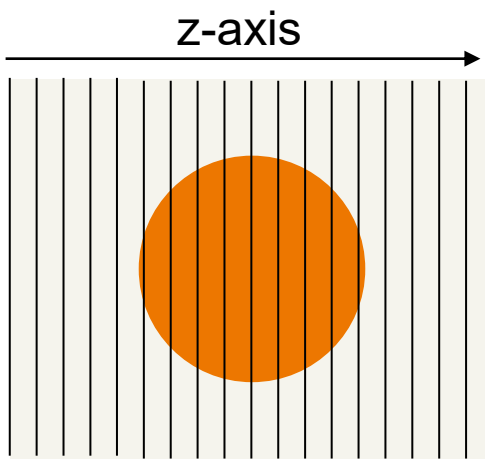
- If the slice thickness does not (roughly) match the x-y pixel resolution, the reconstruction will look distorted in the x-z and y-z orientation – rectangles vs squares

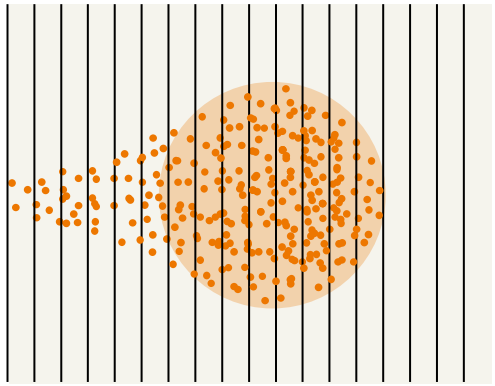
What is 3D tomography?

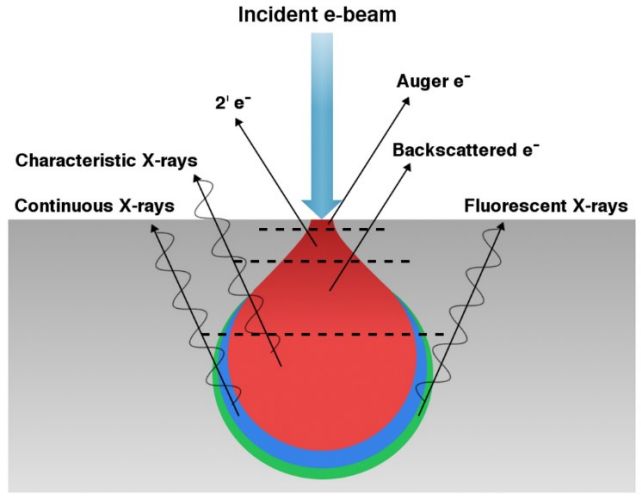
Considerations for collecting the best data

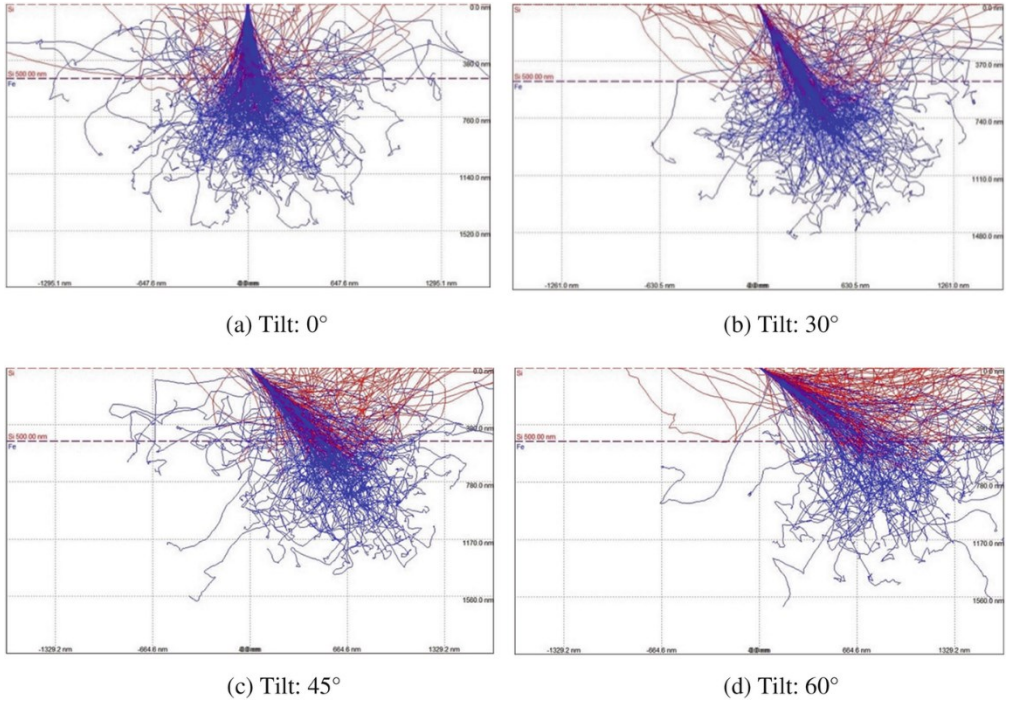
3D EDS has several factors that affect the data:

- EDS software provides limited x-y pixel resolutions
- Higher beam currents limit spatial resolution (beam diameter, at the surface and inside the sample)
- Mapping speed
- Interaction volume – x-rays come from deeper in the sample than electrons (high keV vs low keV)









(a) Tilt: 0°

(b) Tilt: 30°

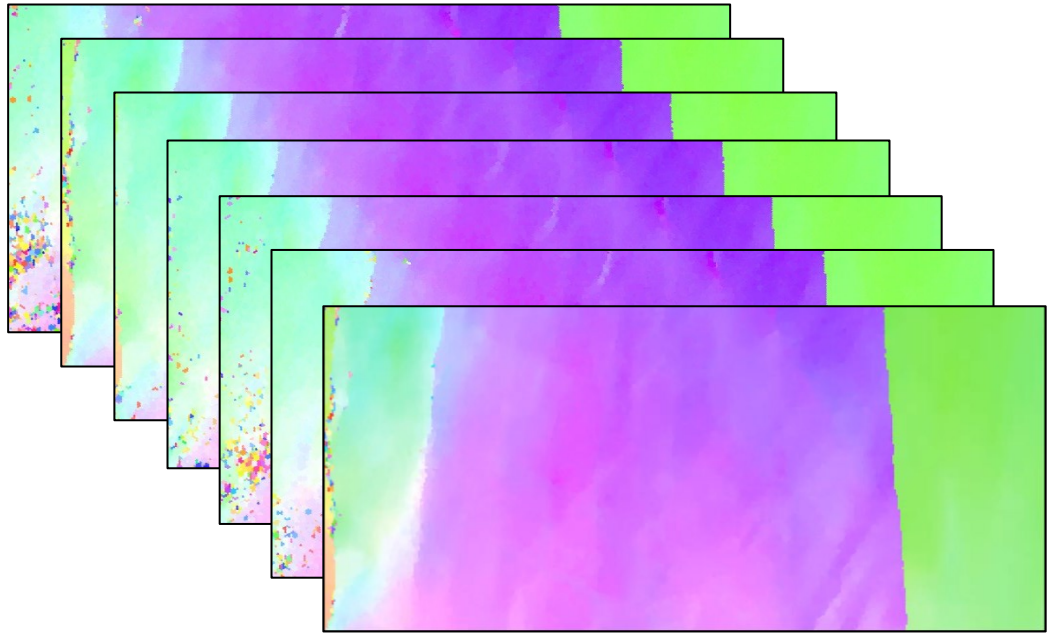
(c) Tilt: 45°

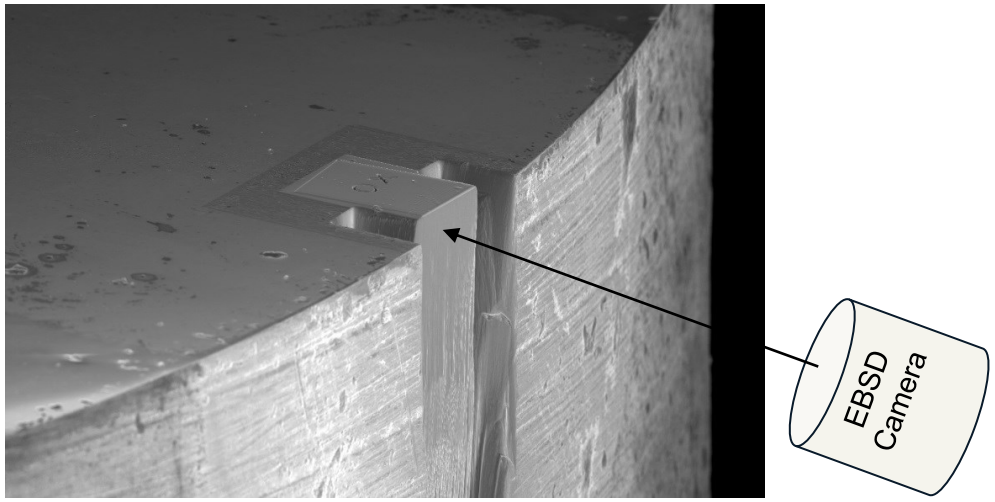
(d) Tilt: 60°

Considerations for collecting the best data

3D EBSD has several factors that affect the data:

- Sample geometry
- EBSD “step size” equals x-y pixel resolution
- Higher beam currents limit spatial resolution (beam diameter)
- Feature size: grain size, grain boundaries, dislocations, strain, etc.
- Mapping speed

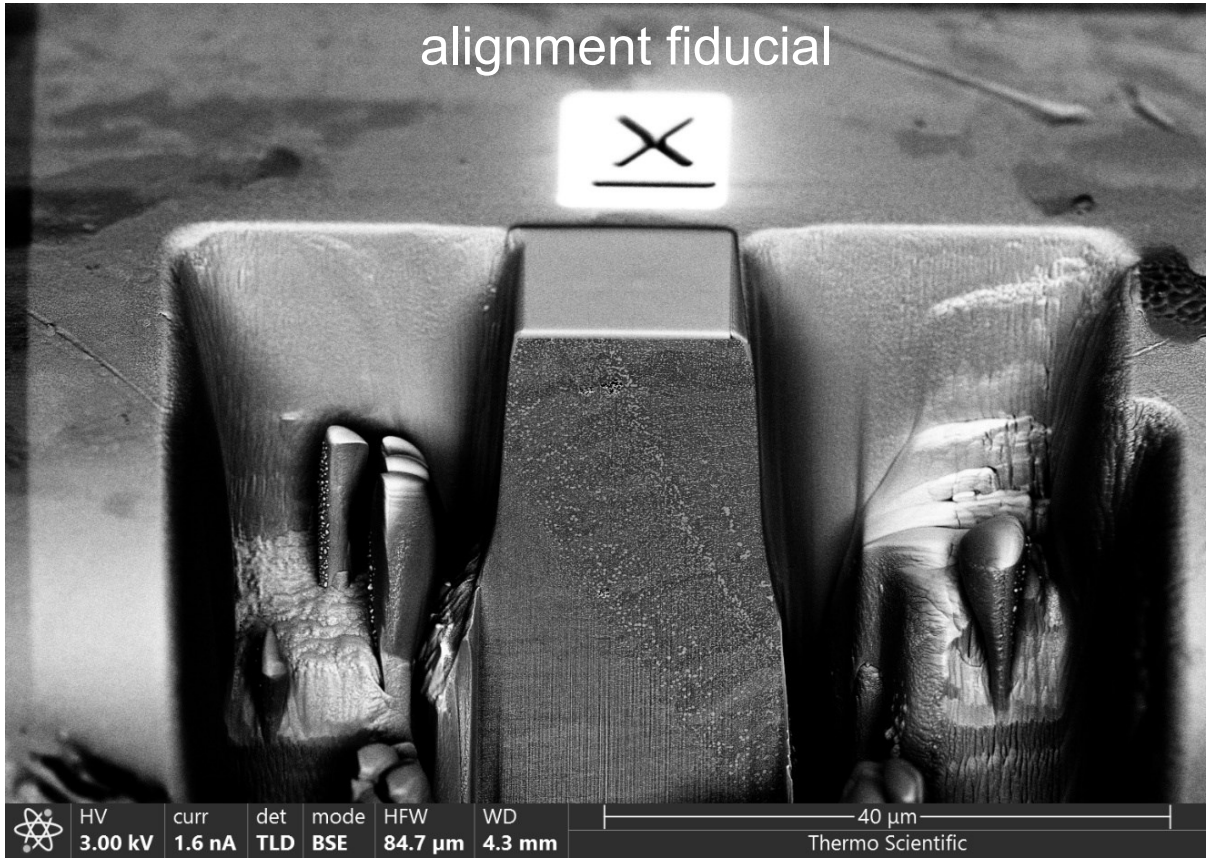


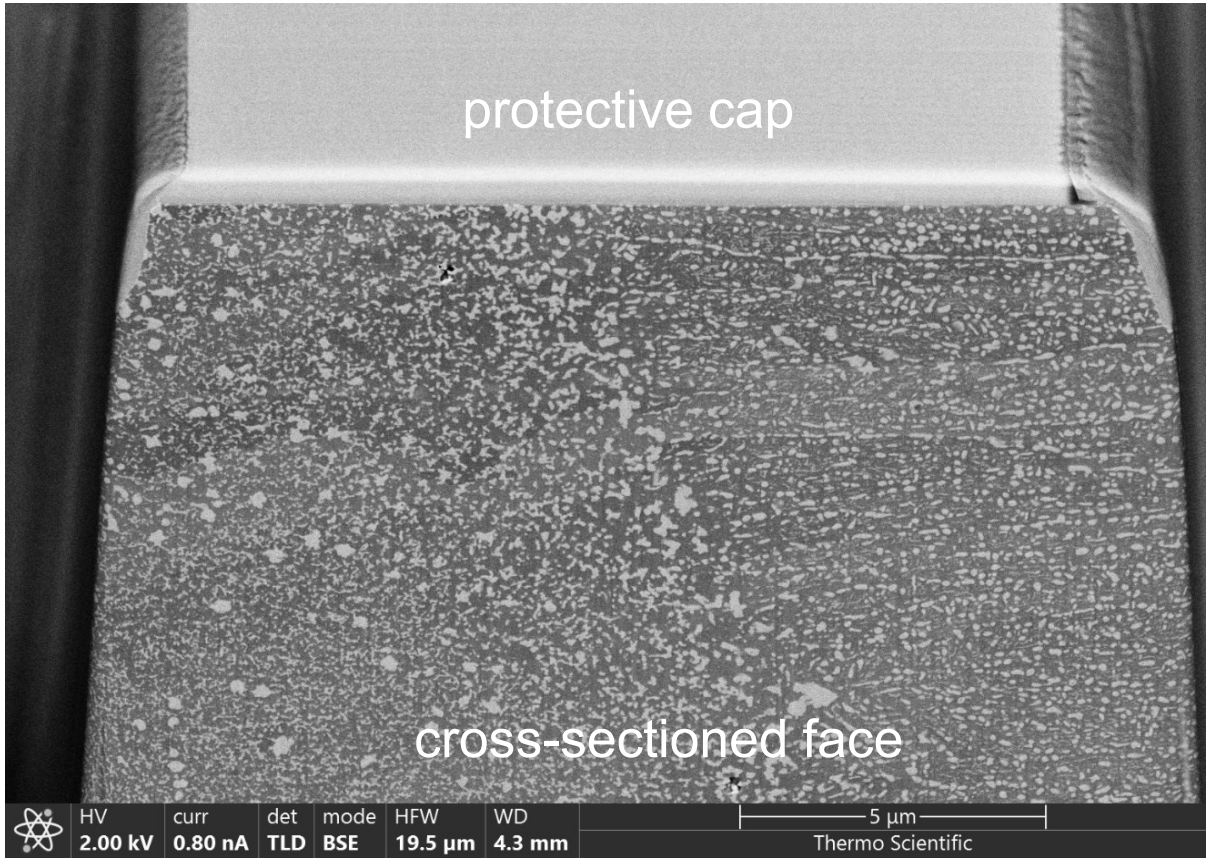


Pt grains
20 keV, 7 nA
100 µm horizontal field width
1 µm EBSD step size
5 min/IPF map
1 µm slice thickness
100 maps

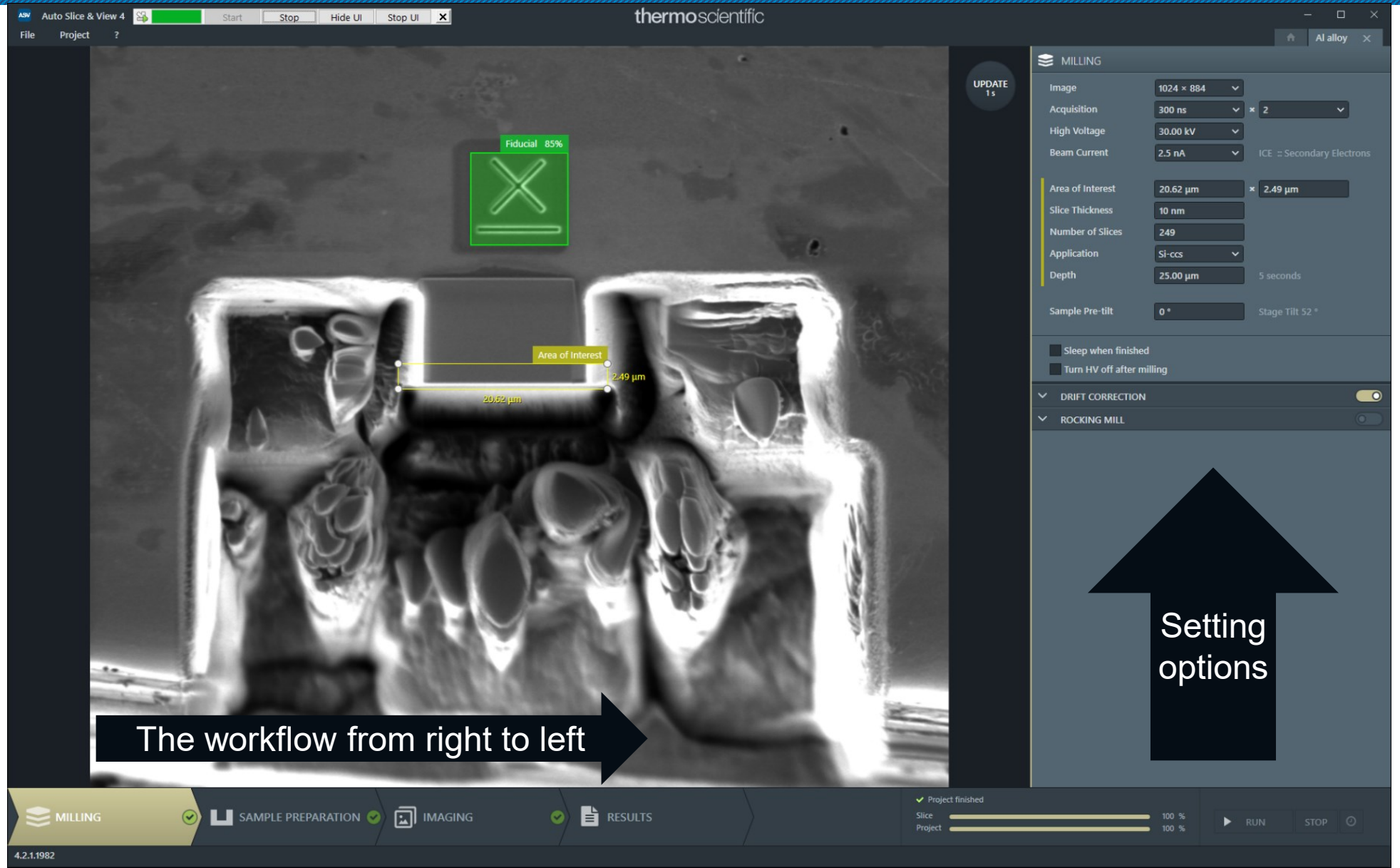
What is 3D tomography?

Fully Prepared Auto Slice and View Site





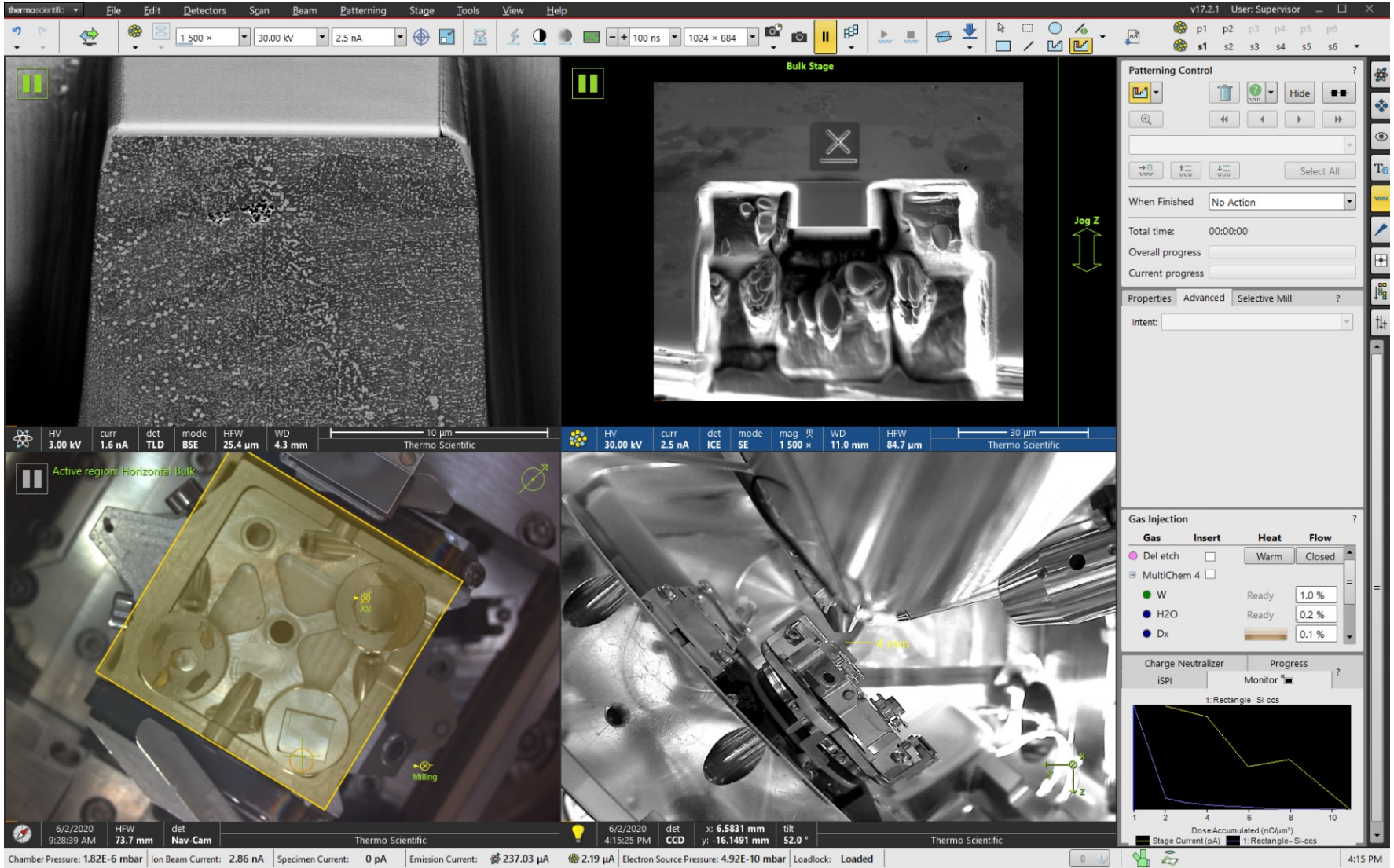
Auto Slice and View 4



Auto Slice and View 4

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xT microscope UI view of fully prepared Auto Slice and View site



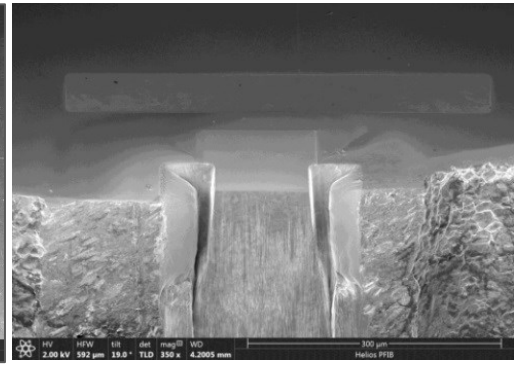
Auto Slice & View Methods

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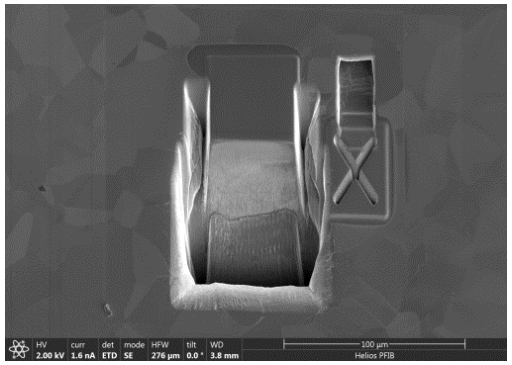
1. Default – In bulk



2. Default – At edge



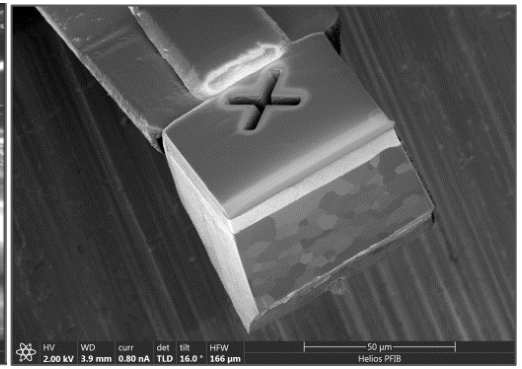
3. Zero Degree Tilt

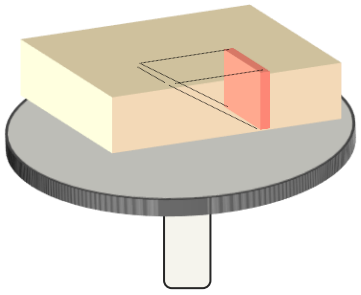


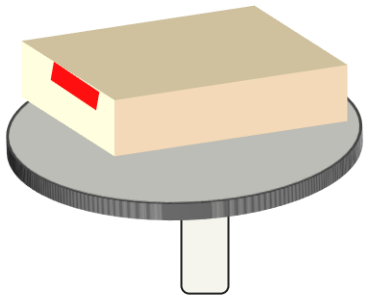
4. Pre-Tilted (edge)

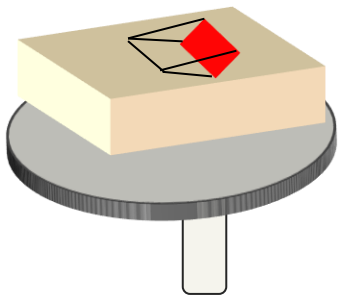


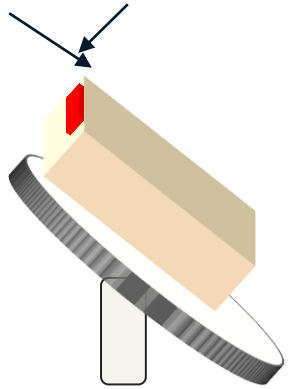
5. Chunk Liftout

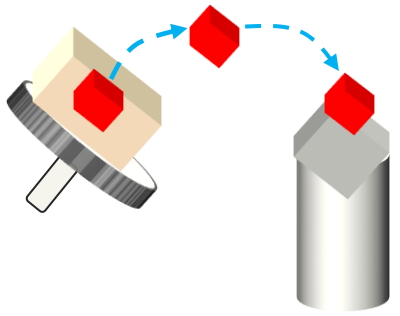








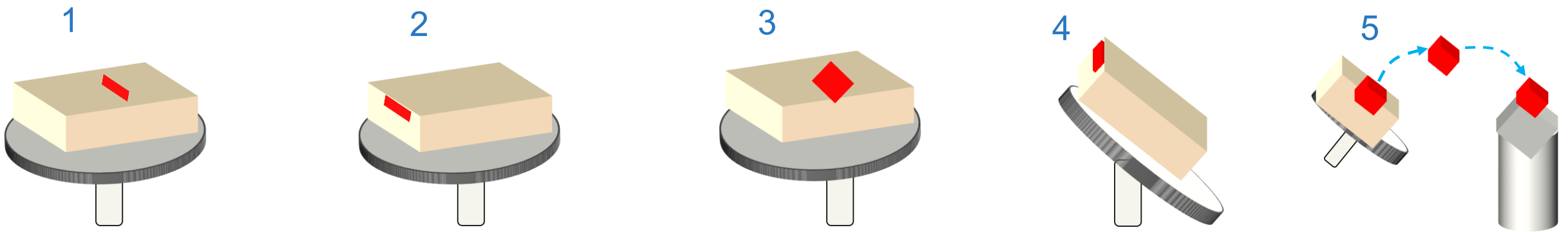




Comparison of Auto Slice & View Methods

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Mounting/Milling Method	Speed	Resolution	SEM signal Access	EDS Signal Access	EBSD Signal Access	Normal to X-sec SEM View	CBS Imaging
1. Default - In Bulk	Fast	Med	Blocking Potential	Blocking Potential	Not Possible	N	N
2. Default - At Edge	Fastest	High	Open	Open	Not Possible	N	Possibly
3. Zero degree	Fast	High	Open	Open	Not Possible	Y	Y
4. Pre-tilted - At Edge	Fastest	Ultra	Open	Open	Open	Y	Y
5. Chunk Lift-Out (pre-tilted)	Time cost	Ultra	Wide Open	Wide Open	Wide Open	Y	Y



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Case Studies & Examples

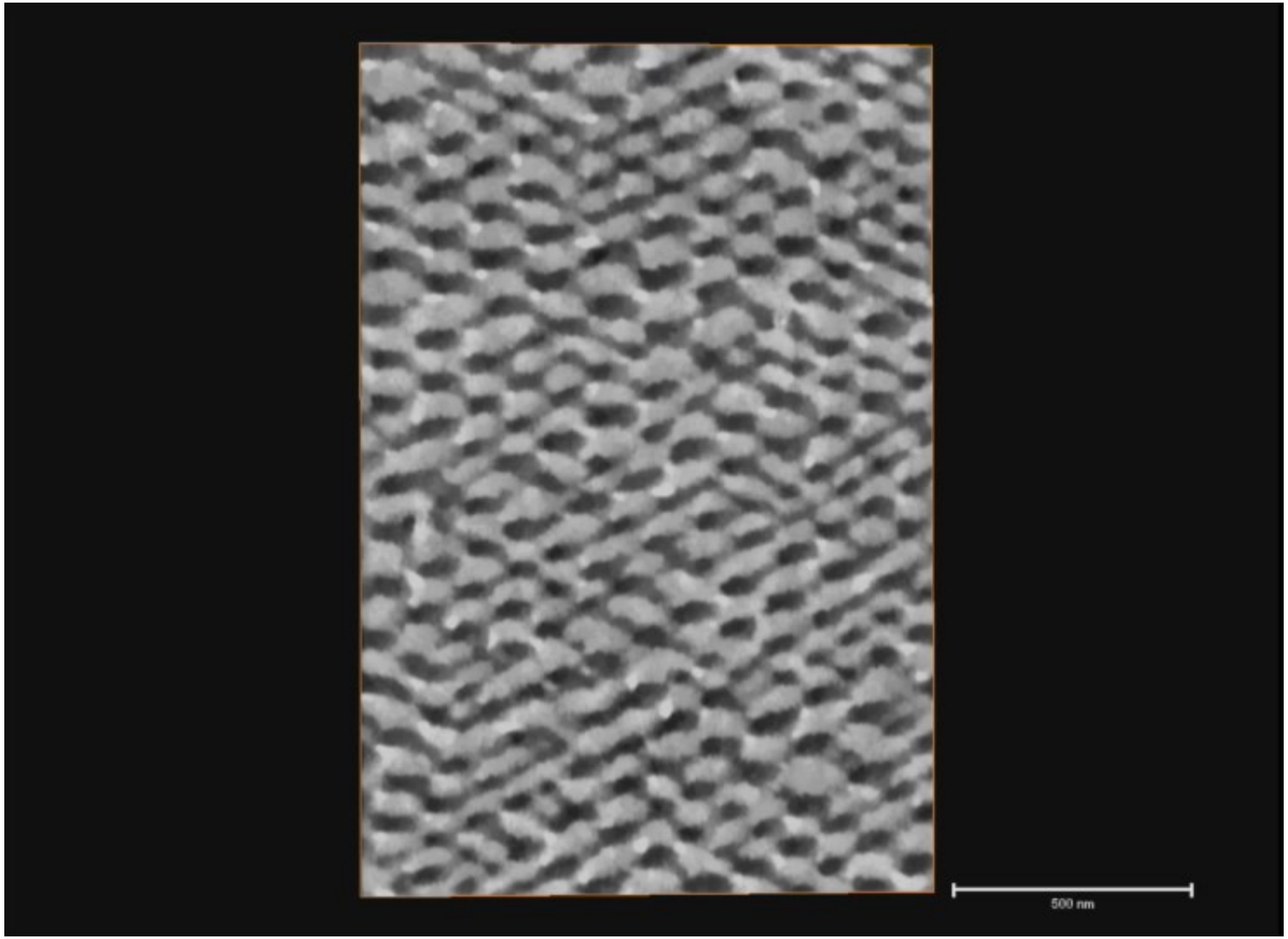
Example of 3D tomography - Imaging



- Boron nitride with cobalt particles: histogram segmentation

movie

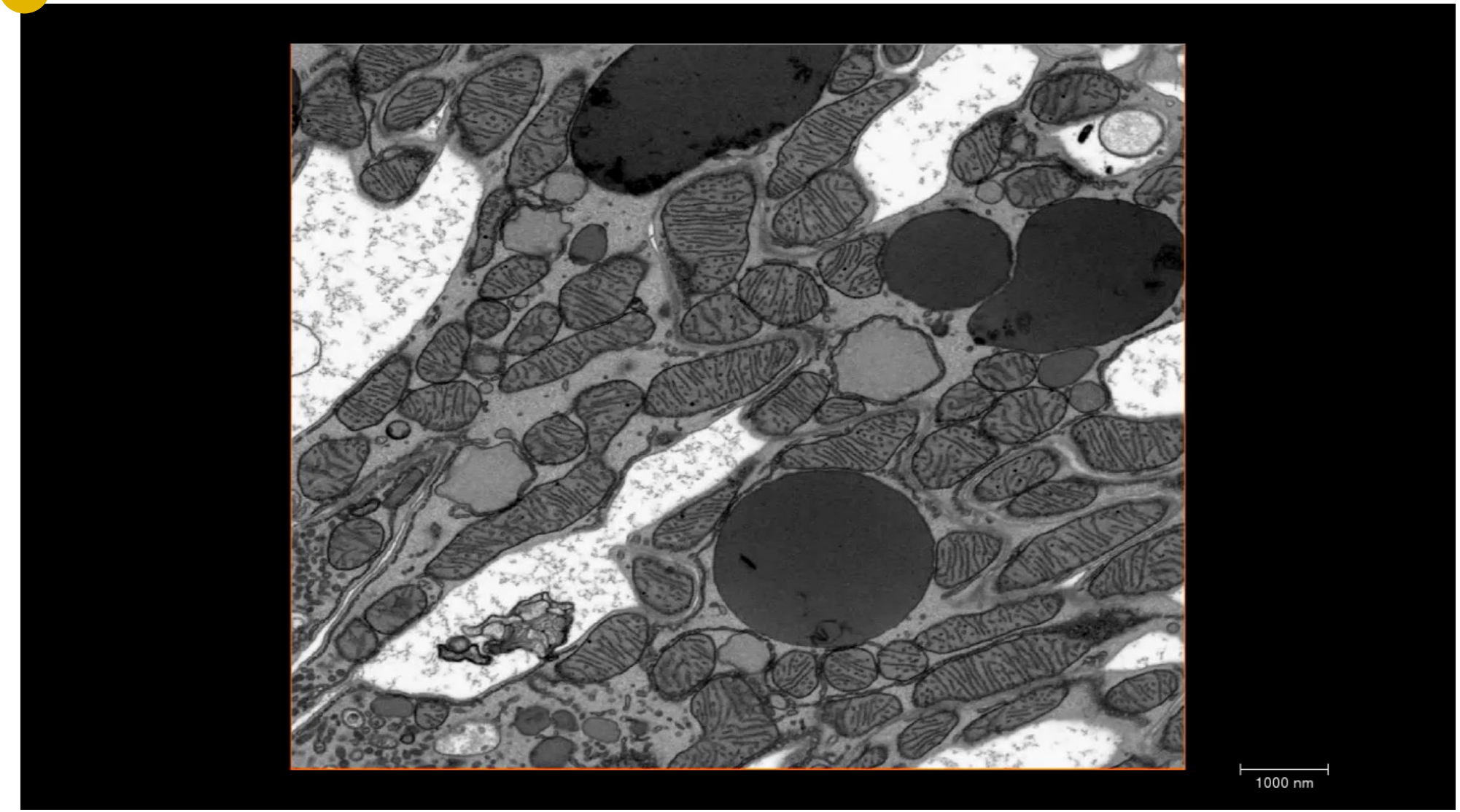
Example of 3D tomography - Imaging



movie

- Alnico 9: Histogram segmentation

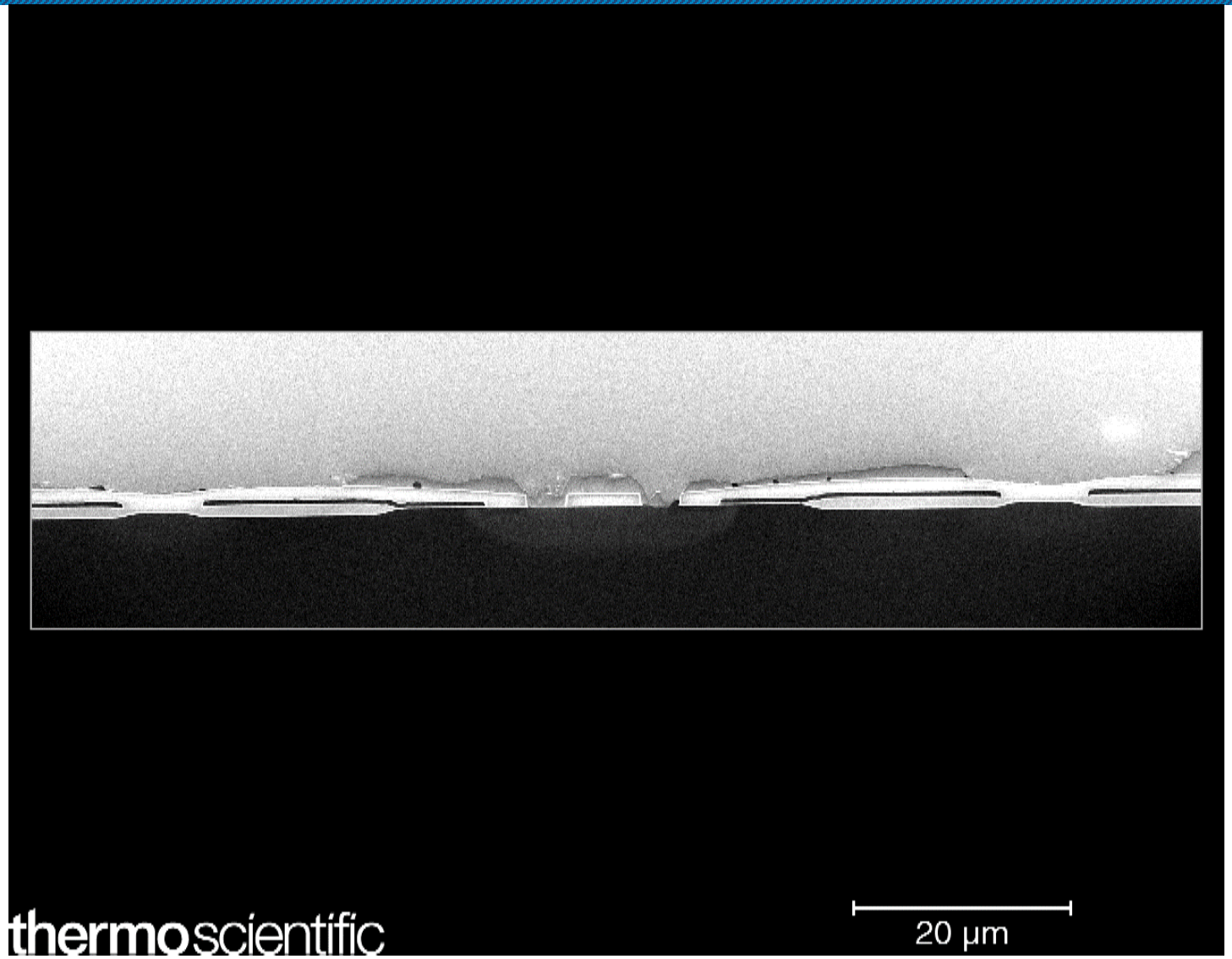
Example of 3D tomography - Imaging



movie

- Life science example: Volume rendering

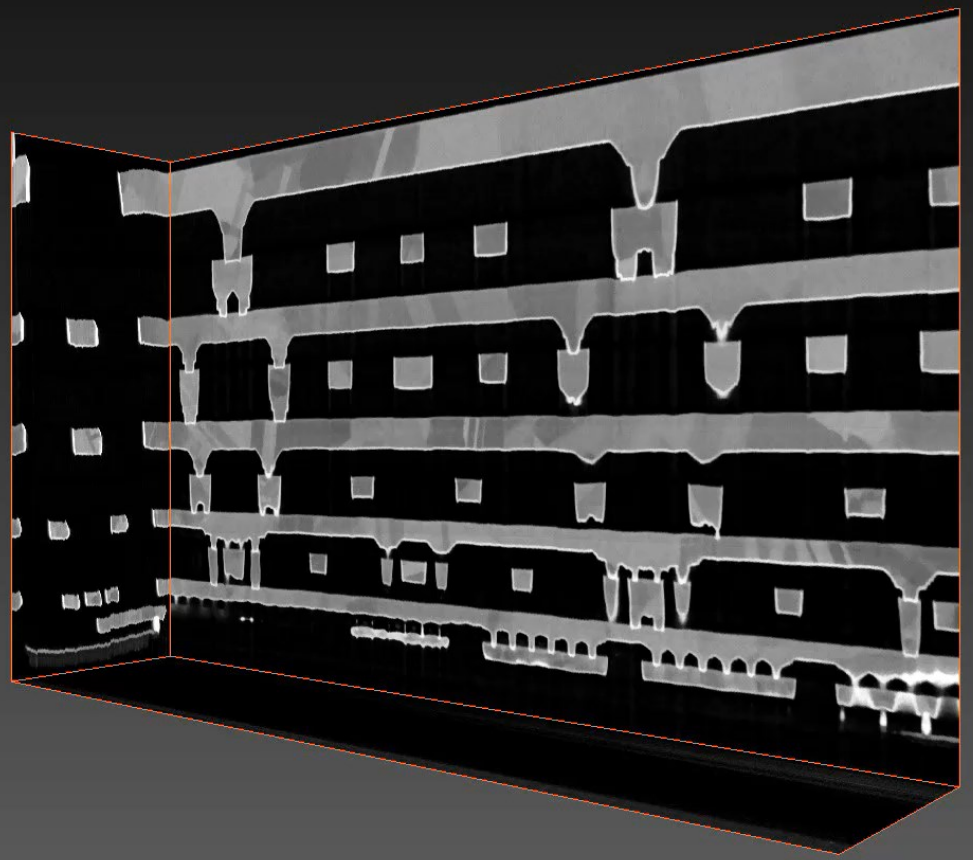
Example of 3D tomography - Imaging



movie

- Semiconductor example: SiC junction

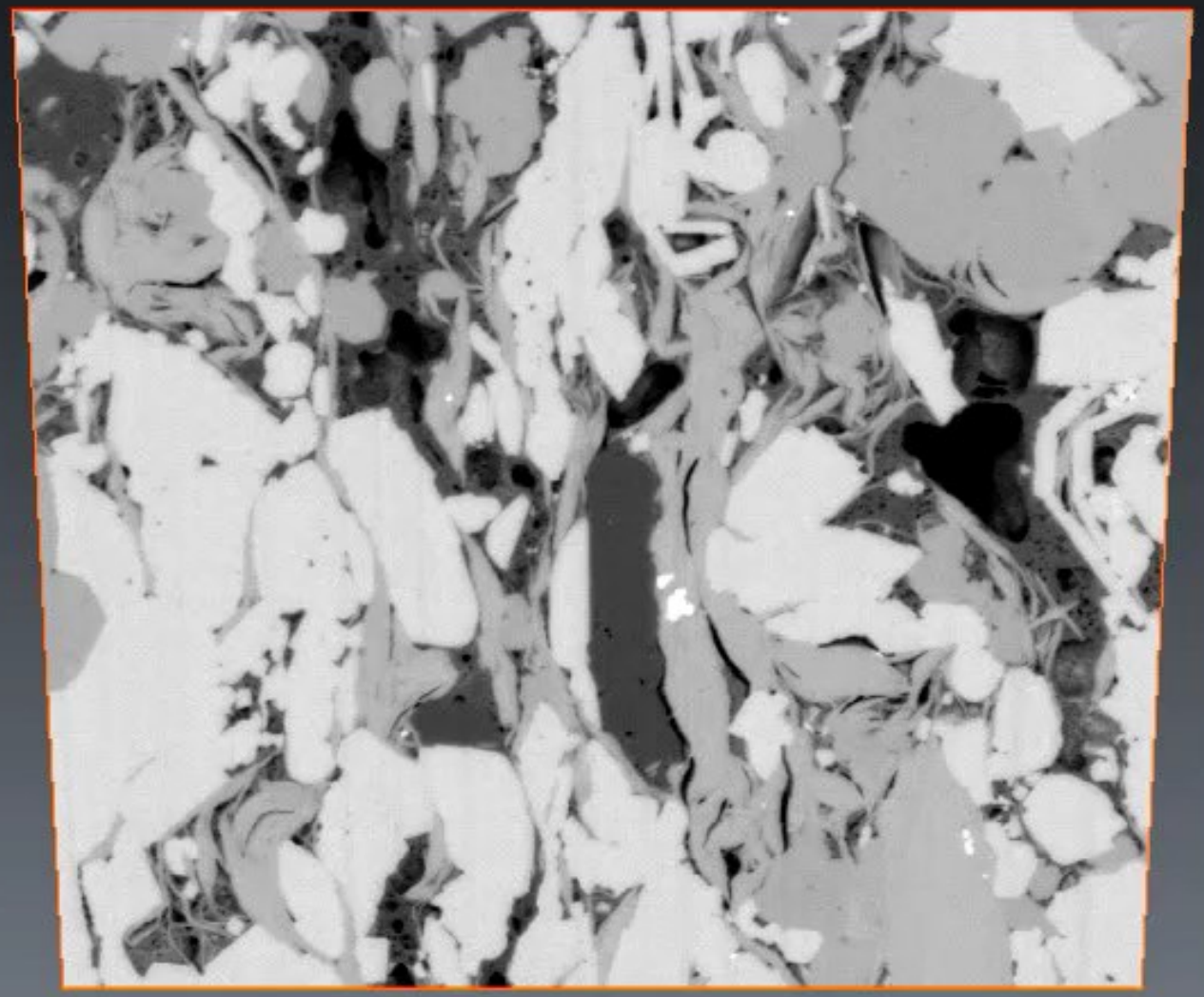
Example of 3D tomography - Imaging



movie

- Semiconductor example: 45 nm fly-through

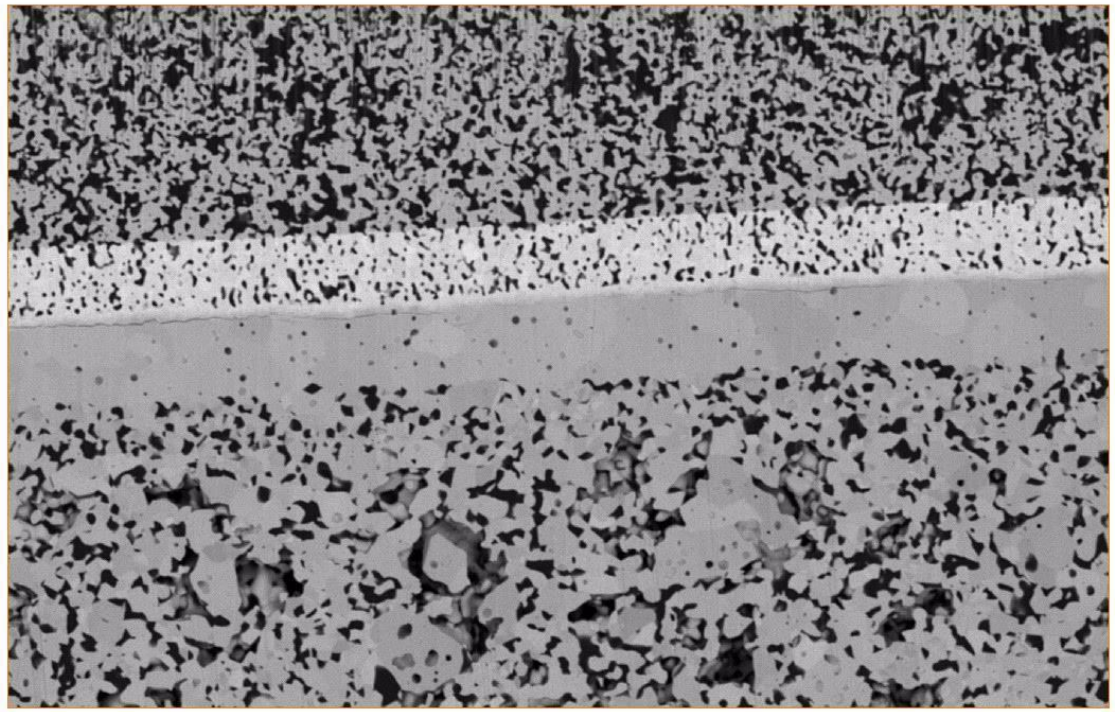
Example of 3D Tomography – Imaging



- Shale

movie

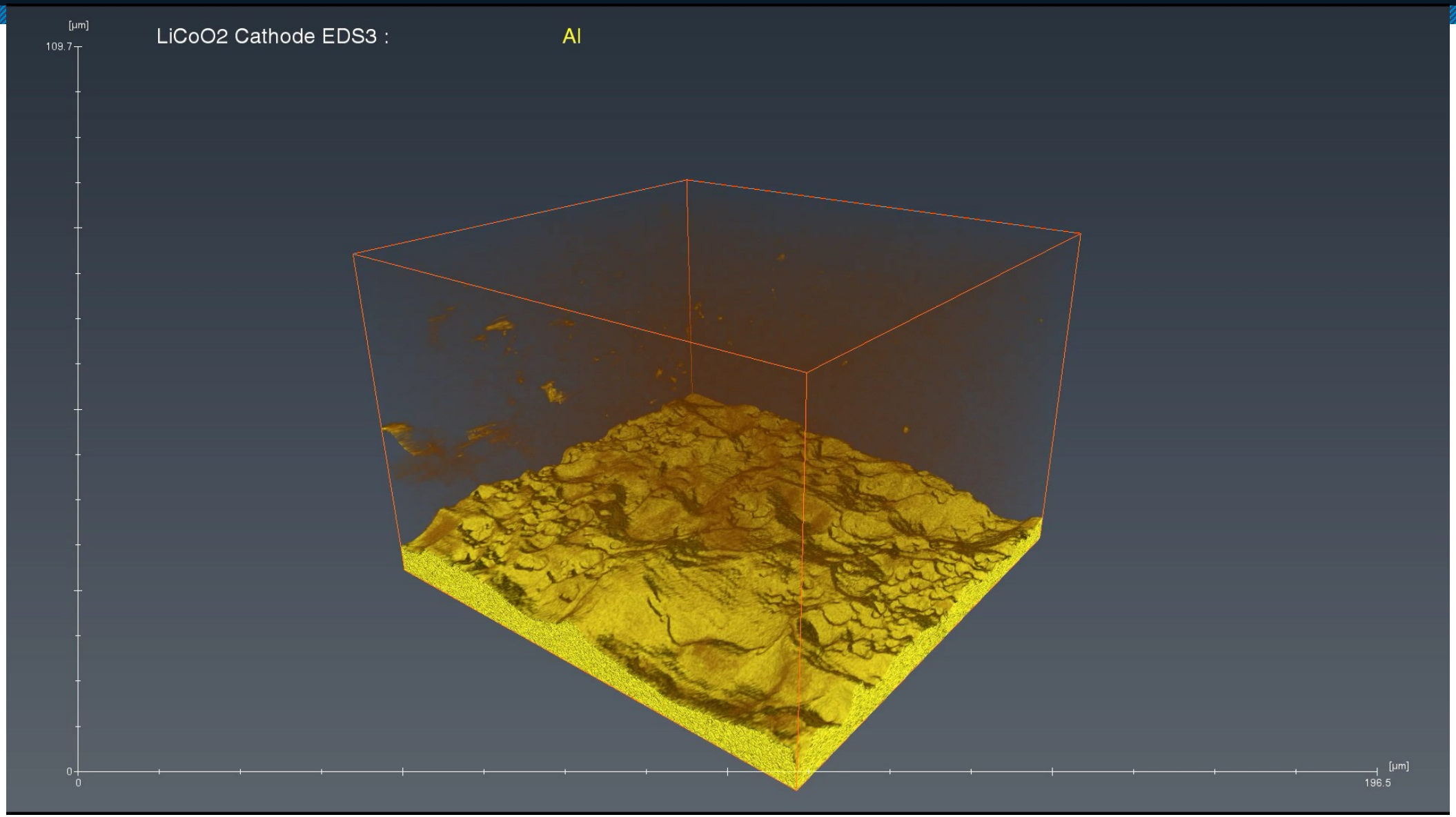
Example of 3D Tomography – Imaging



movie

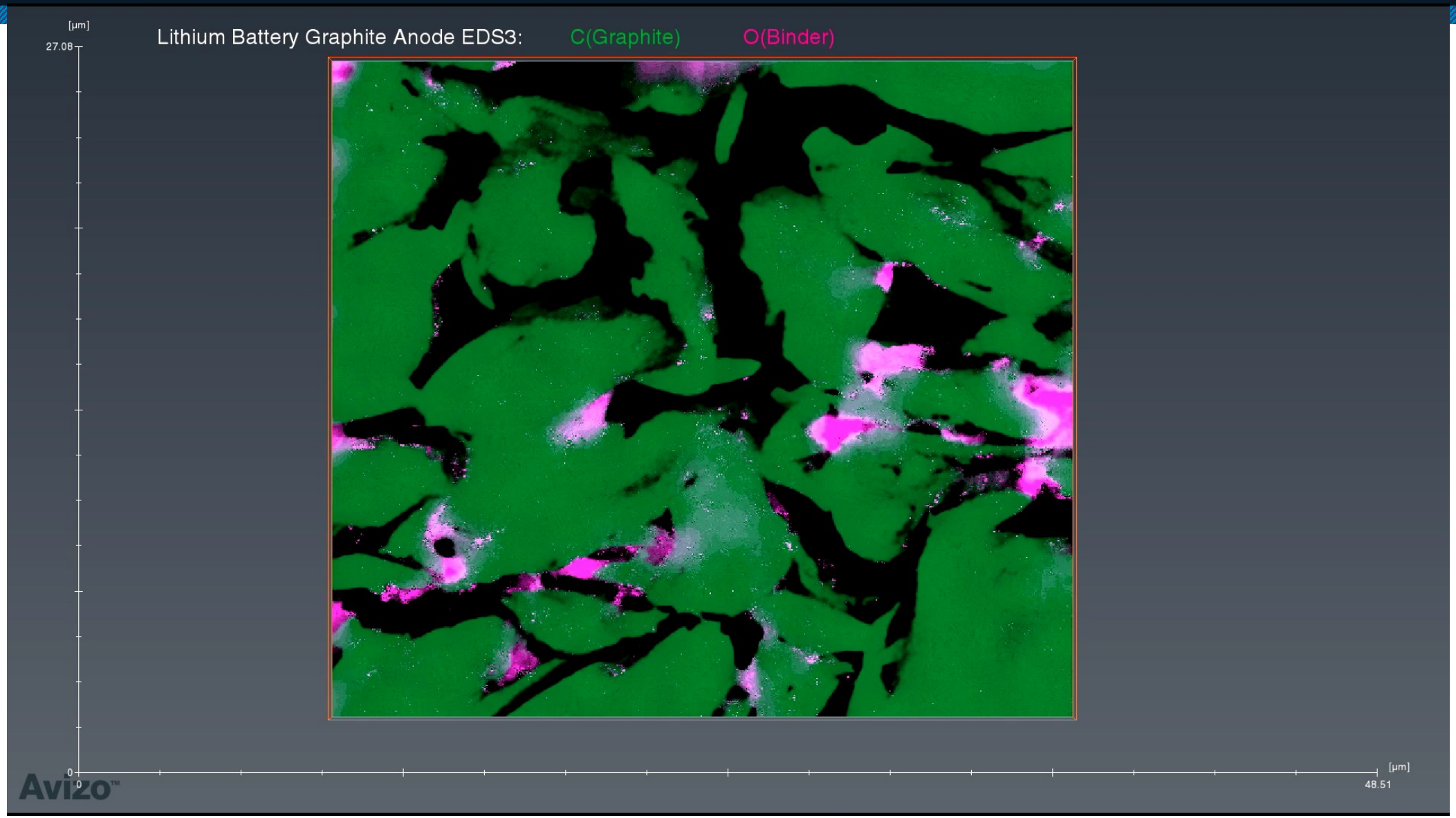
- Battery
- Cathode/ Anode Layers

Example of 3D tomography – EDS x-ray maps



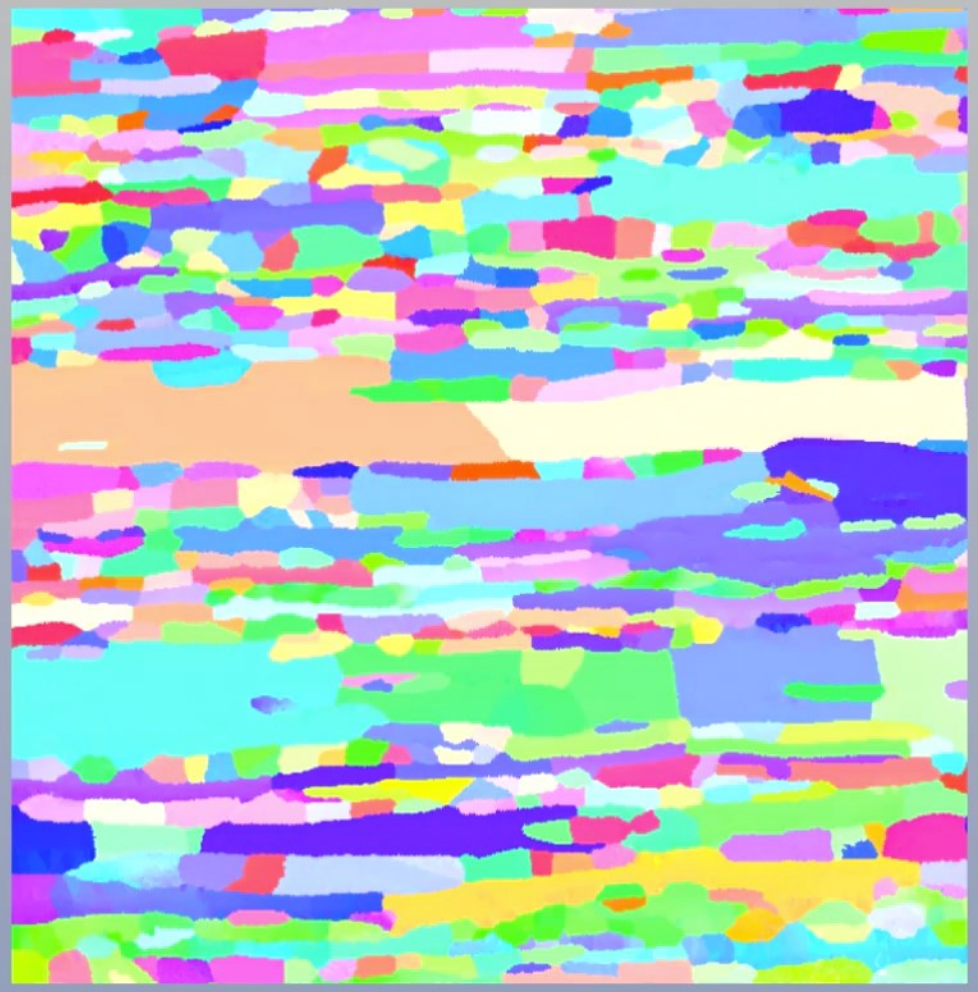
movie

Example of 3D tomography – 3 keV EDS x-ray maps



movie

Example of 3D tomography – EBSD



movie

- Austenitic and ferritic steel

Summary

- 3D tomography using the “slice and view” methodology can provide high-resolution three-dimensional volumes
- The data volumes can be interrogated in several different ways, from simple visualizations to revealing valuable statistical information about the sample
- Imaging, EDS, and EBSD can all be used, individually or combined into a single run

Coming Up…

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Stay Tuned! The next episode will be announced soon at our Website:

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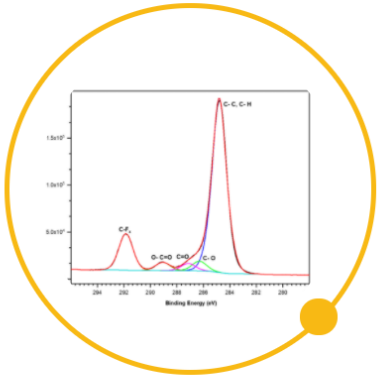
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Q & A Session

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Thank you