Thermo Fisher SCIENTIFIC

Webinar Series in TEM: Sample Preparation

Non-Biological Samples

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Customer Success Manager

Material and Structural Analysis - APAC

DIREKTORAT RISET & PENGEMBANGAN

LabTEM HIGH RESOLUTION

multi

The world leader in serving science

TEM Sample Preparation – Non-Biological Sample

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Outline

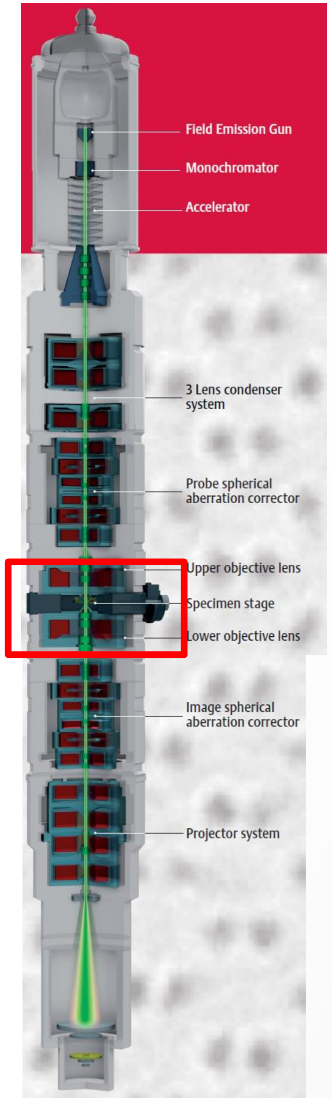
- Introduction
- Sample Preparation
  - Conventional Techniques
  - Focused Ion Beam Techniques
    - Ga ion
    - Plasma
    - Plasma + Laser
- Artifacts caused by bad sample preparations

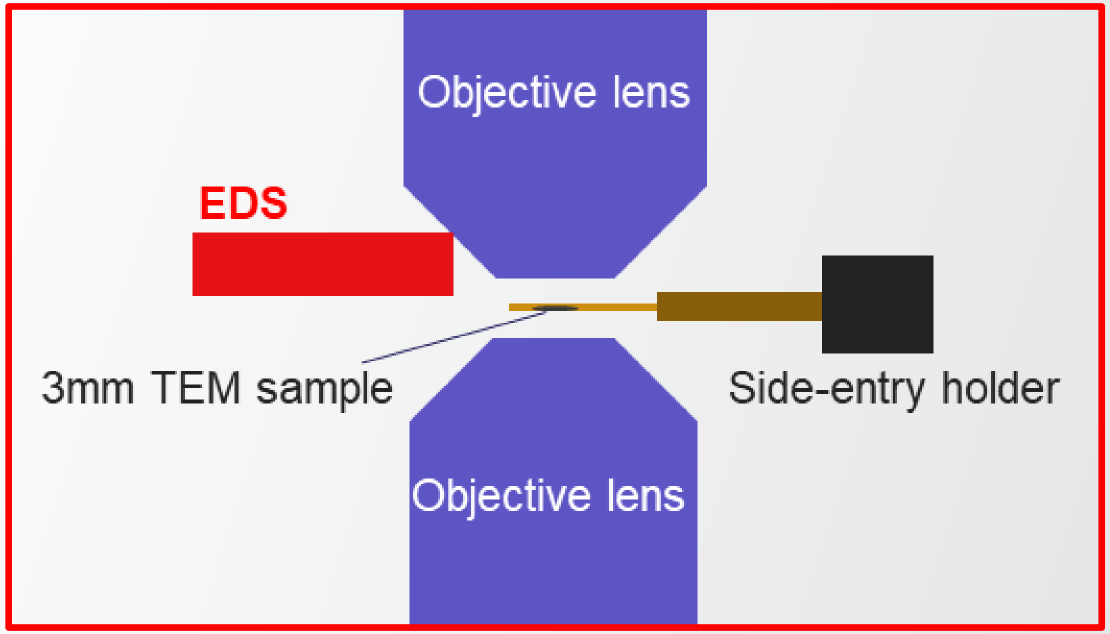
Helios 5 CX

mohamadriza.iskandar@thermofisher.com | 16-March-2023

Sample Position in TEM

Side Entry Holder





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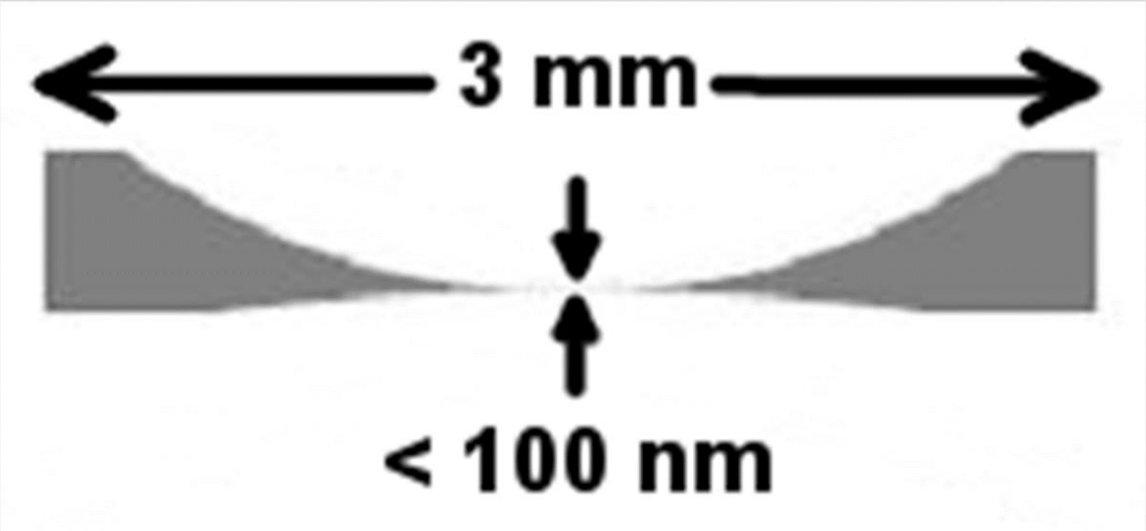
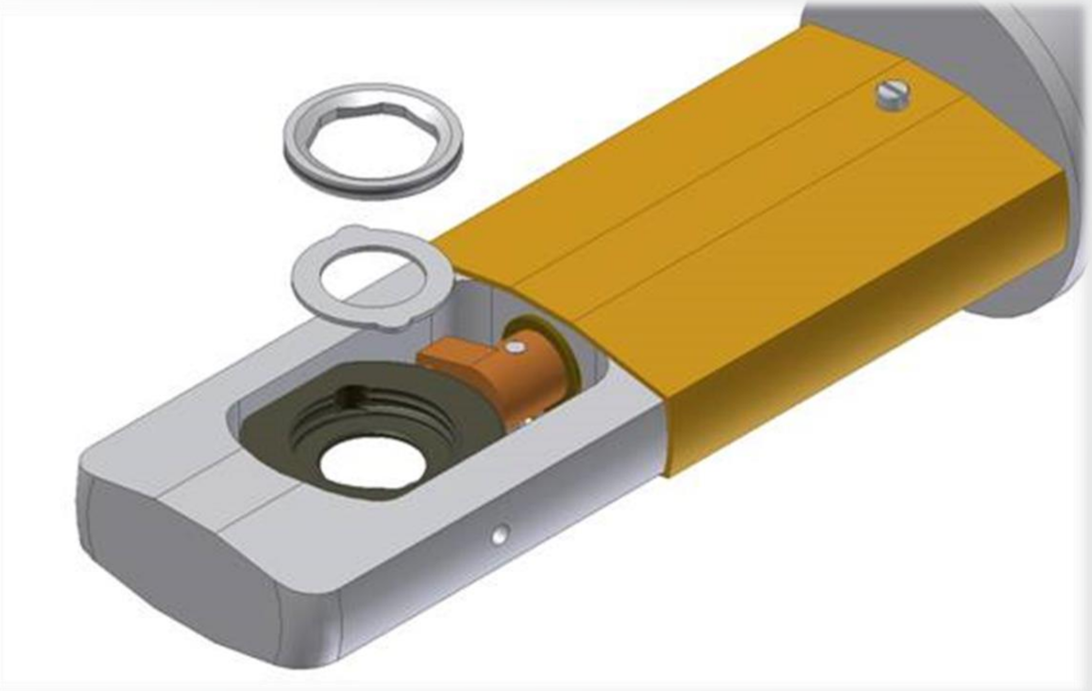
TEM Sample Requirements

Electron Transparent!

Specimen‘s requirements:

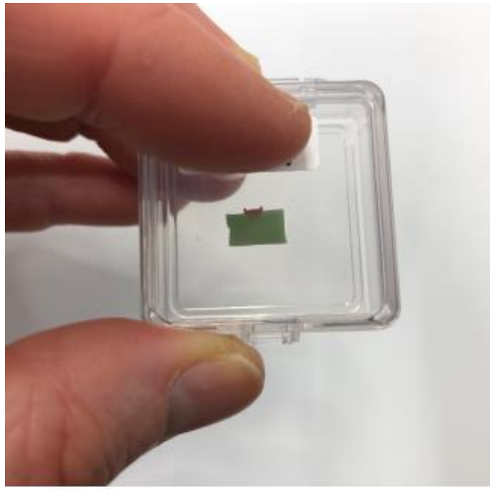
- Fit into TEM holder cup (3 mm disc)
- Electron transparent (< 100 nm)





TEM Sample

Shape and Size of various TEM Grid

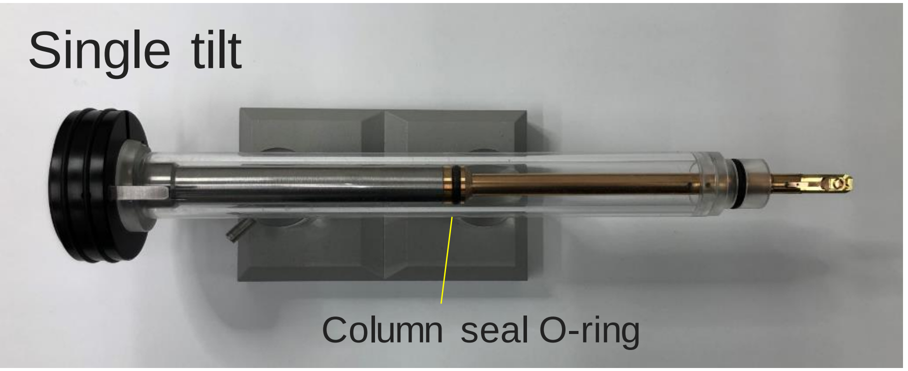


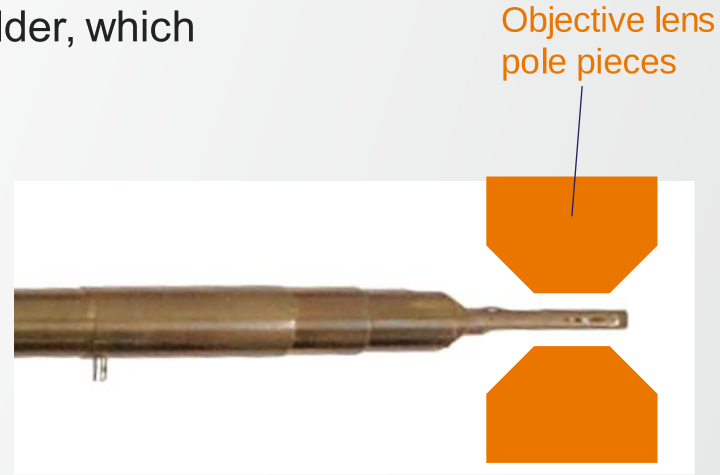


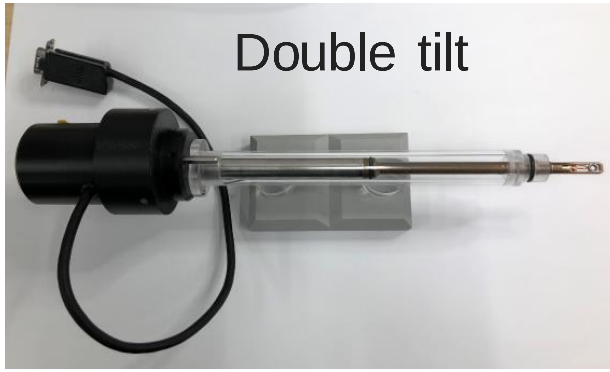
Sample Holders

A Single and Double Tilt TEM Holder

On a Materials Science TEM, the sample is normally inserted into the narrow gap between the objective lens pole pieces (i.e., into the middle of the objective lens) using a “side entry” holder, which looks like this:







Thermo Fisher TEM Holder

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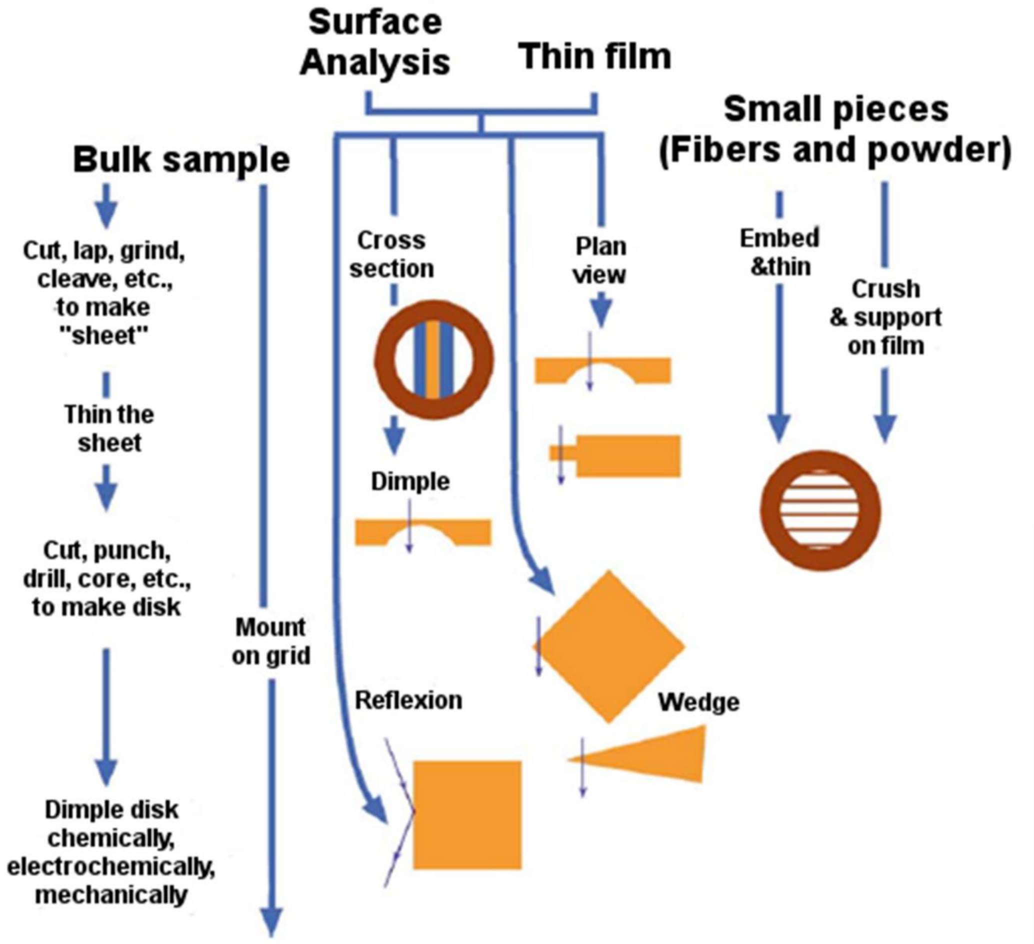
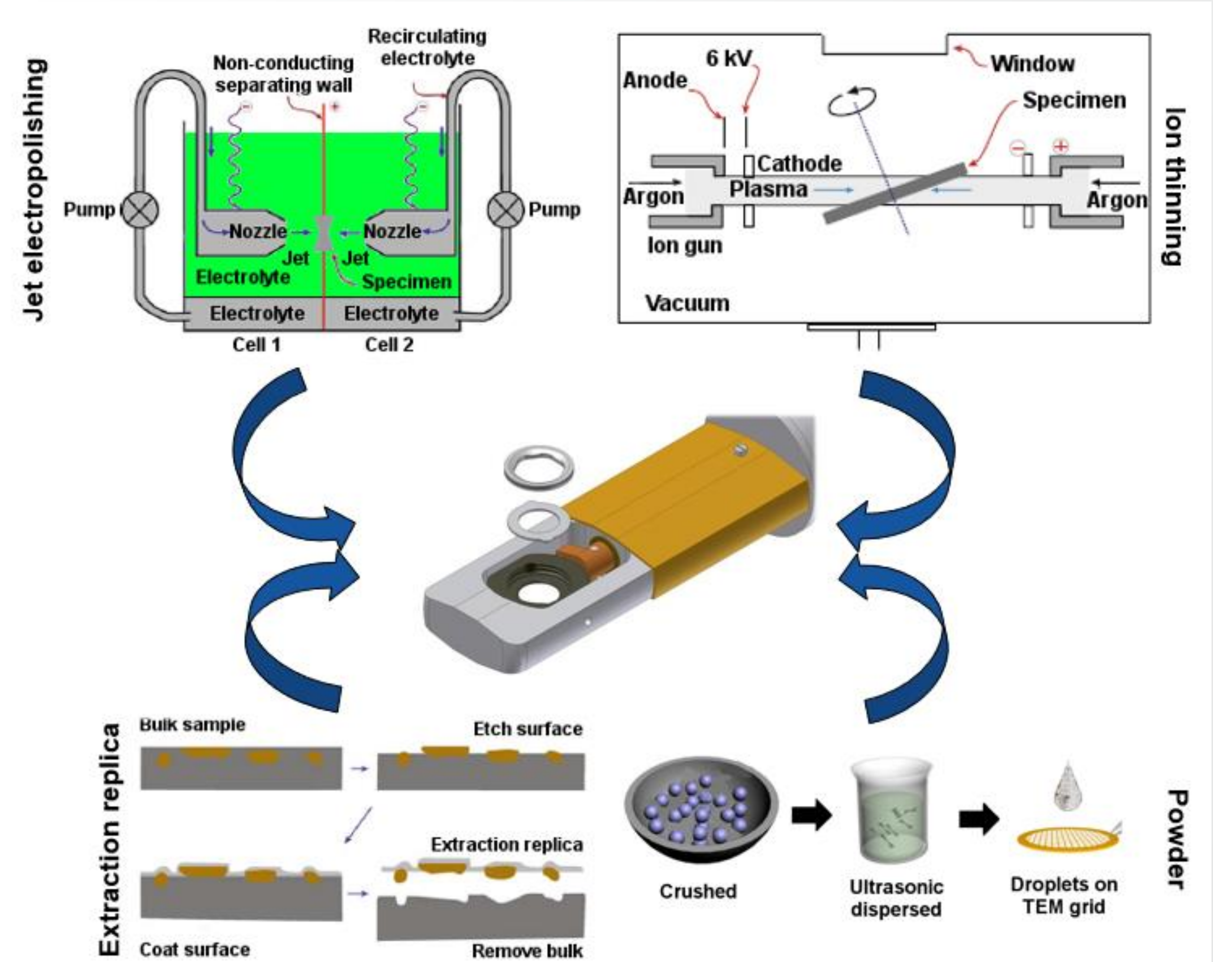
Scios 2

Non-Biological Sample Preparation

Conventional Techniques

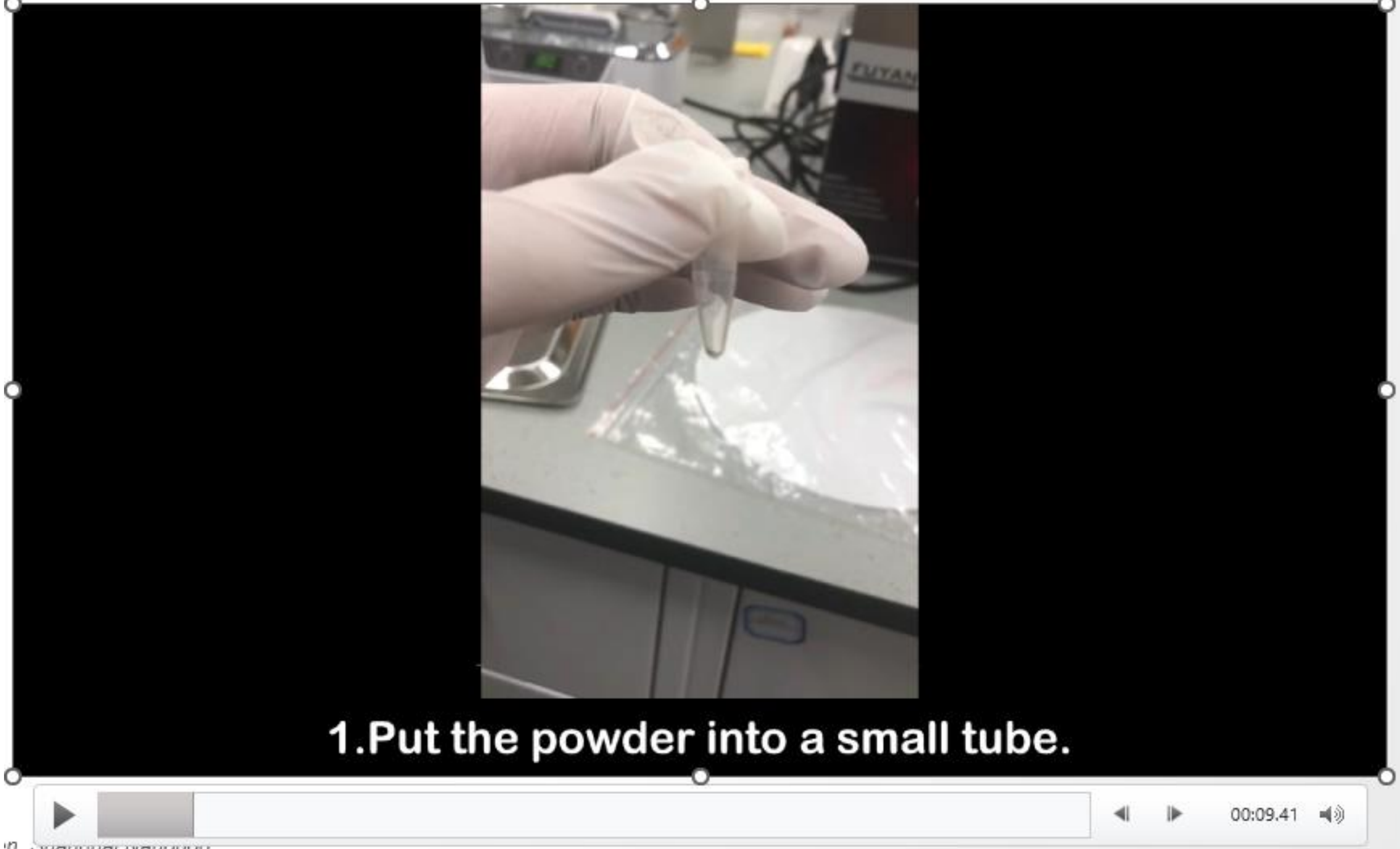




David B. Williams and C. Barry Carter, Transmission Electron Microscopy, A Textbook for Materials Science, Springer Science Business Media, LLC 1996, 2009
http://bsa.bionanomaterials.ch/images/step_tem.png
http://www.jdcsh.com/Upfile/86563302-61e8-477a-a30a-17f614769cd1.jpg

Non-Biological Sample Preparation

Conventional Technique: Powder Sample Preparation



Movie Courtesy Fangren Shen, Shanghai Nanoport

Non-Biological Sample Preparation

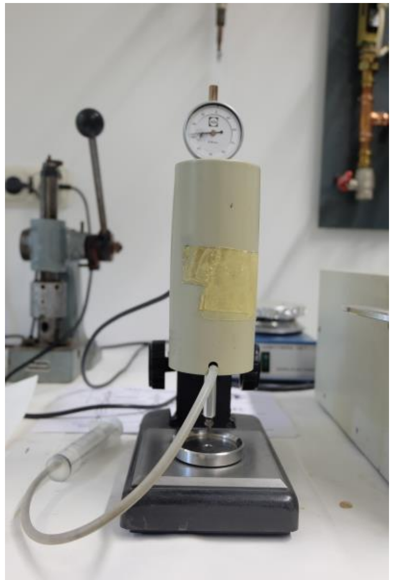
Powder Sample Preparation

- Procedure:
  - Drop small amount powder into glass tube or microcentrifuge
  - Fill the tube with ethanol or distilled water
  - Sonicate the solution in an ultrasonic bath
  - Using a pipette, drop a small amount of the solution (≈1 µl) on the TEM grid
  - Heat the grid, so the ethanol or other solution is evaporated

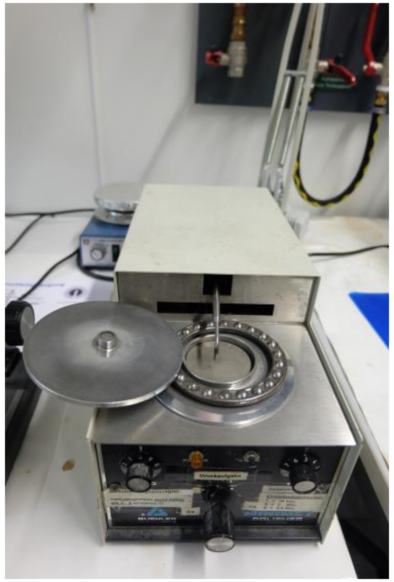
- Tips:
  - Use only a small amount of the sample (a tip on the spatula or 1 mg is already enough)
  - Typically, two minutes of sonification is enough, as ultrasonic might alter the sample properties
  - Be careful with impurity due to other elements from tweezer, filter paper, or even tube (use only new tube)
  - Prepare more than one grid, with a number of droplets variations
  - For samples that are air-sensitive, heat the grid in the vacuum condition

Non-Biological Sample Preparation

Conventional Technique: Ion Milling for Bulk sample

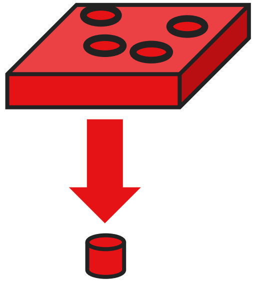


Ultrasonic Cutter



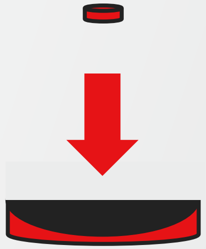
Disc Polisher

Dimple Grinder



≈ 0.5 hour

≈ 1 hour



≈ 3 hours

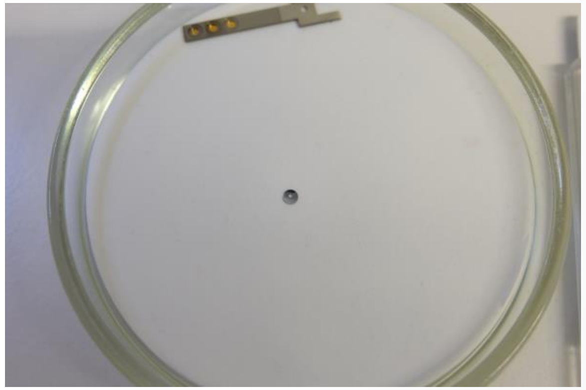
Photographs courtesy Dr.-Ing. Fadli Rohman
Research Center for Advanced Materials - BRIN

Non-Biological Sample Preparation

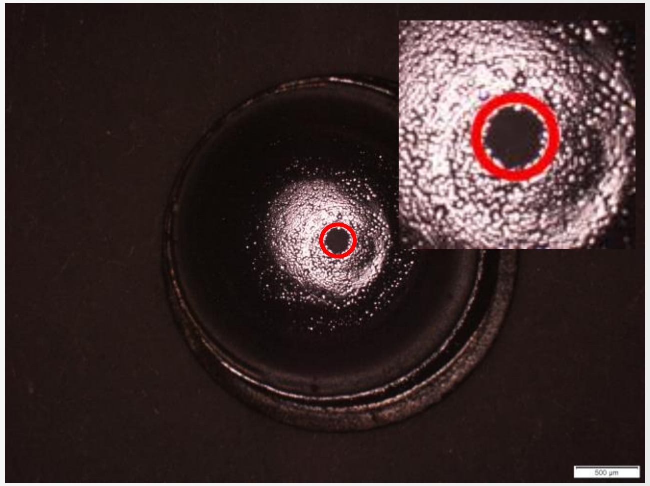
Thermo Fisher SCIENTIFIC

Conventional Technique: Ion Milling for Bulk sample

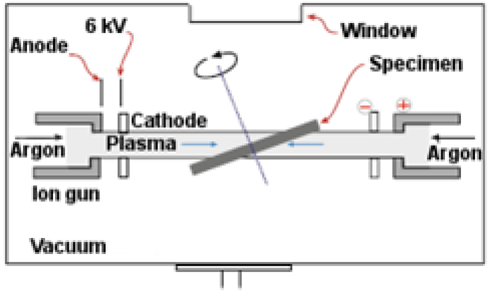
Precision Ion Polishing System

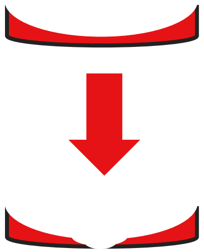


A 3 mm disc with a hole in the center

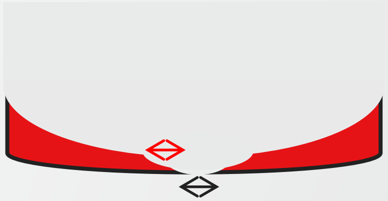


Investigate area inside the red circle





≈ 8 hours - 3 days



≈ 2 – 7 days / sample

Successful rate 30-40%

Photographs courtesy Dr.-Ing. Fadli Rohman
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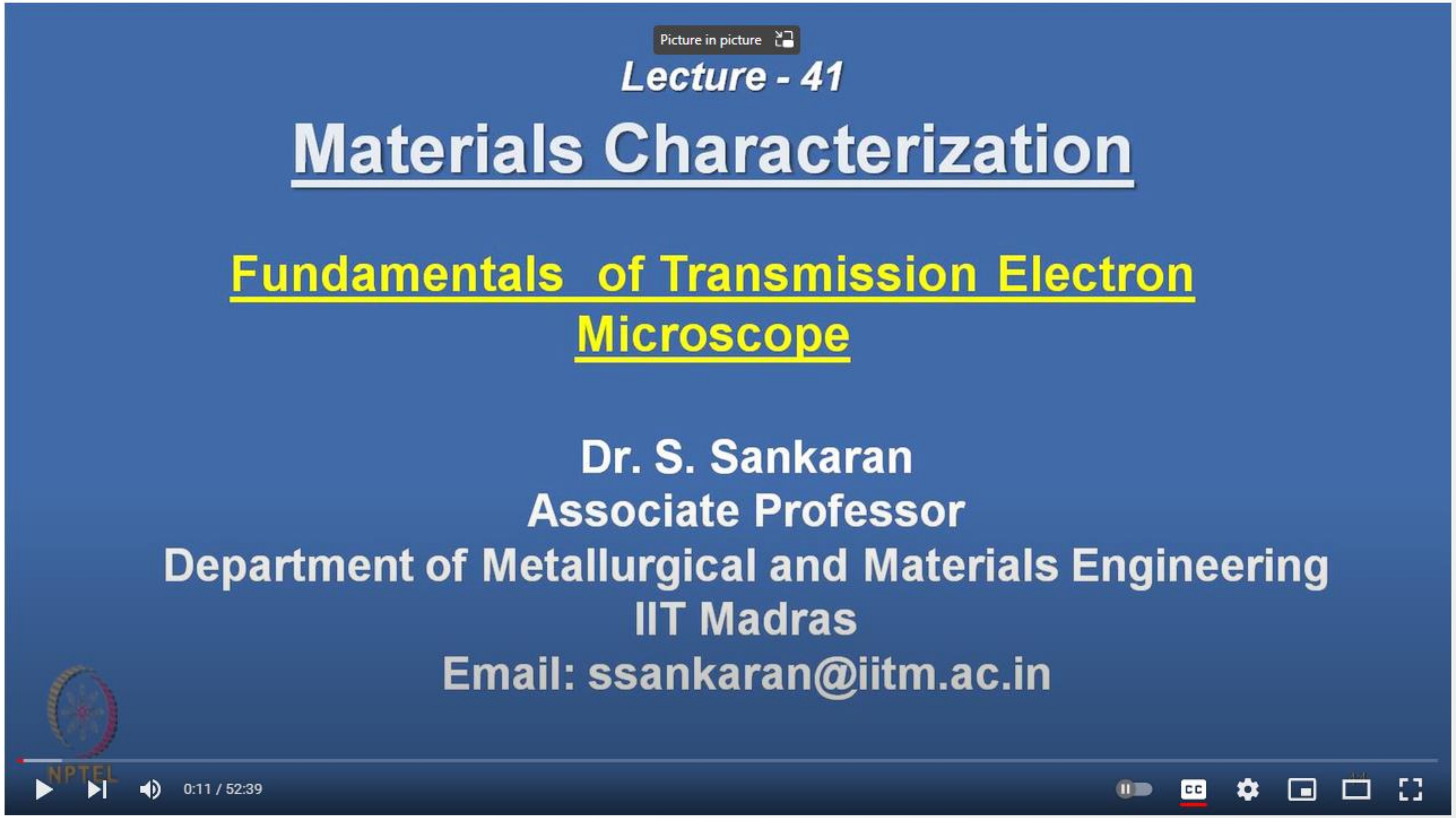
Non-Biological Sample Preparation

Conventional Technique: Ion Milling for Bulk sample



https://www.youtube.com/watch?v=1Ia6bbUtQMs
https://www.youtube.com/watch?v=62eT9hr4rUI

Non-Biological Sample Preparation

Conventional Technique: Ion Milling for Bulk sample

- Procedure:
  - A bulk sample is ground up to 5 mm thickness
  - Cut to 3 mm diameter disc using an ultrasonic cutter
  - Thinning the disc up to 1 mm using a disc polisher
    - Polish both sides to make sure the disc has the same thickness
  - Make the center of the disc further thinner using a dimple grinder
    - Grinding the disc on both sides too.
  - Create an electron transparent area using Precision Ion Polisher System (PIPS)

- Tips:
  - Create significant discs (at least 10 disc/sample) as the success rate is low (more discs are needed for inexperienced users)
  - Use appropriate mixture for epoxy glue (1:10 for resin and hardener)
  - Critical steps:
    - Sample dropped off during disc polishing
      - Make sure the sample is glued perfectly
    - Sample breaks during dimple grinder
      - Pay attention to the initial sample thickness
      - Be careful when switching the sample side
    - Be cautious of material redeposition during PIPS
      - Ion beam angles depend on materials and initial thickness
      - Initial polishing step 8-10° and down to 3° for final thinning
      - Check the surface condition and stop the polishing when the redeposition appears

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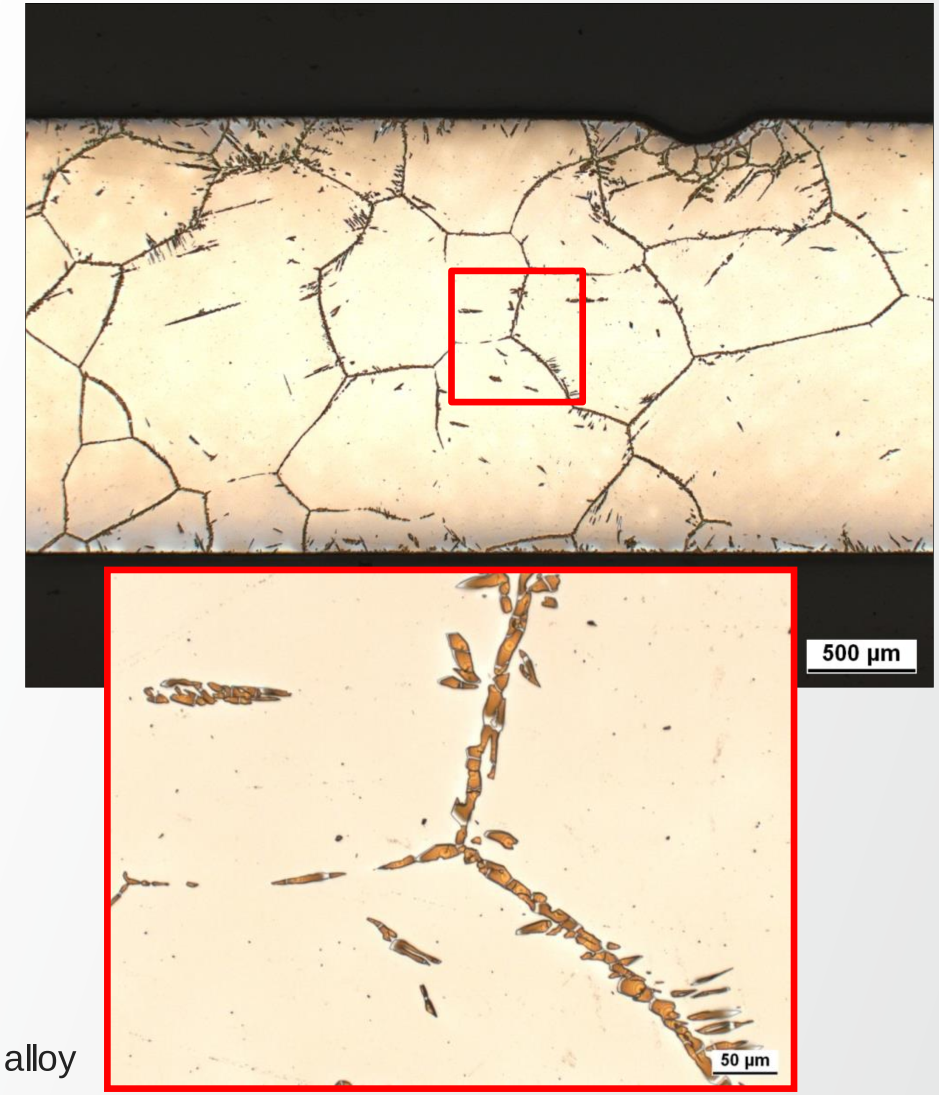
Helios G4 PFIB UXe

 mohamadriza.iskandar@thermofisher.com

Non-Biological Sample Preparation

Focused Ion Beam Technique

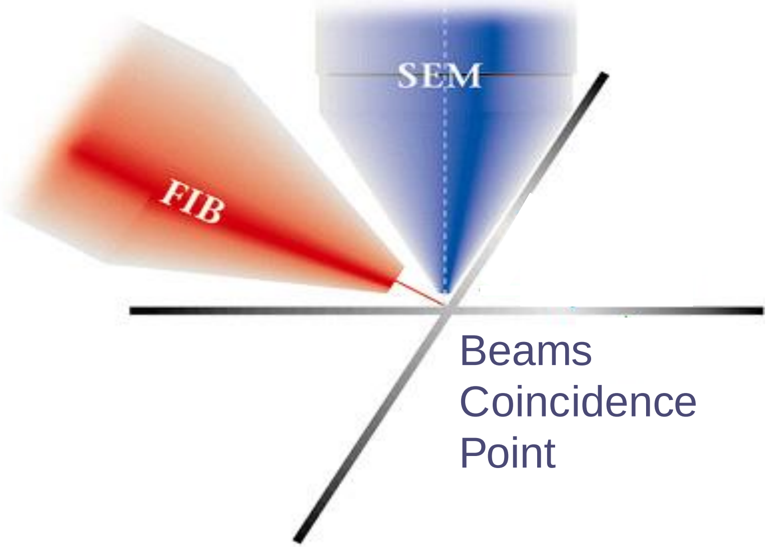
- Target grain boundaries and end-point on features with nanometer precision
- Examine Interfaces between hard and soft materials
- Target specific orientations in crystalline materials
- Prepare multiple samples and keep bulk samples intact for repeat analysis

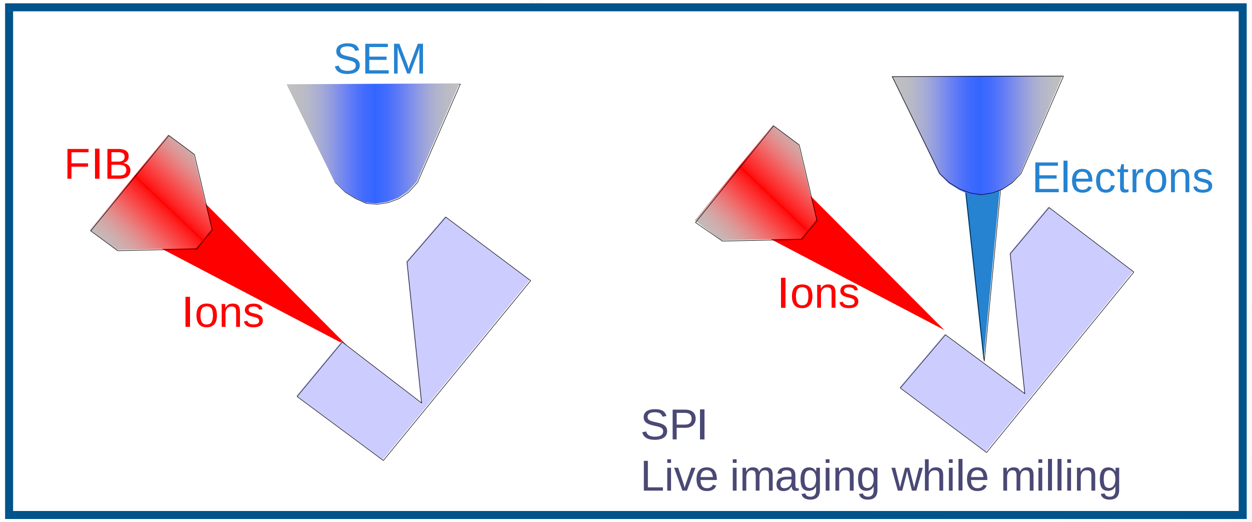


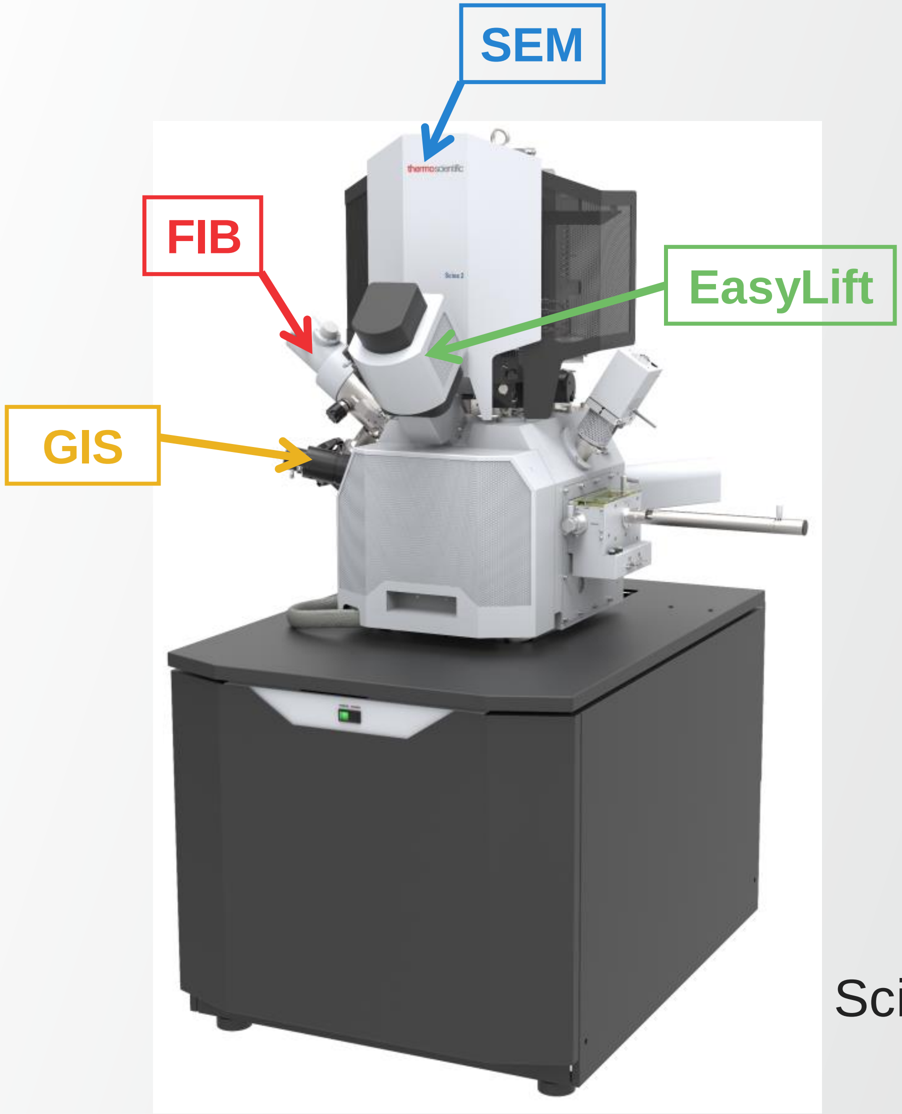
Sigma phase formation and Fe-Cr based alloy

Focused Ion Beam Technique

Principle of a DualBeam™



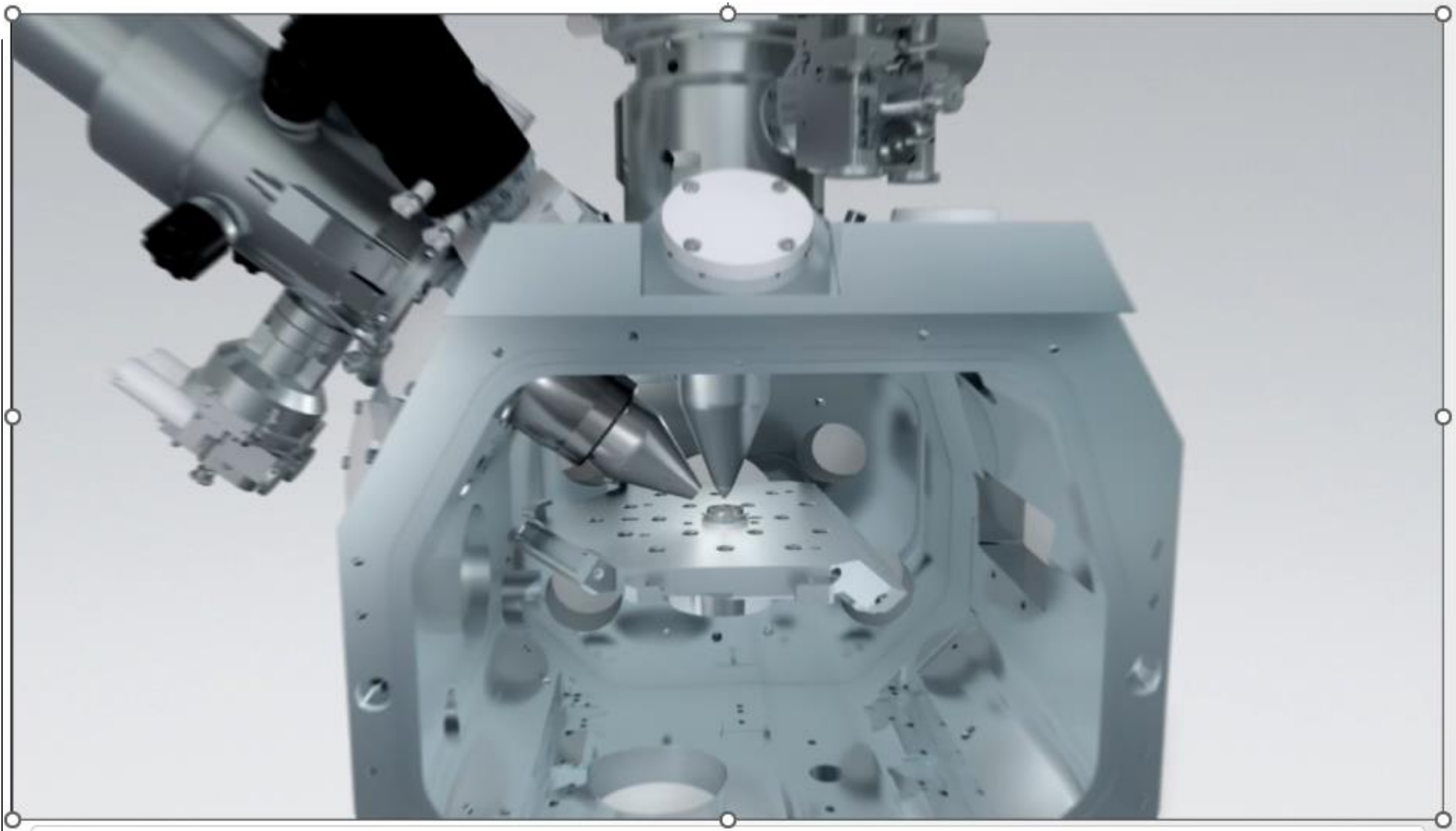




Scios 2

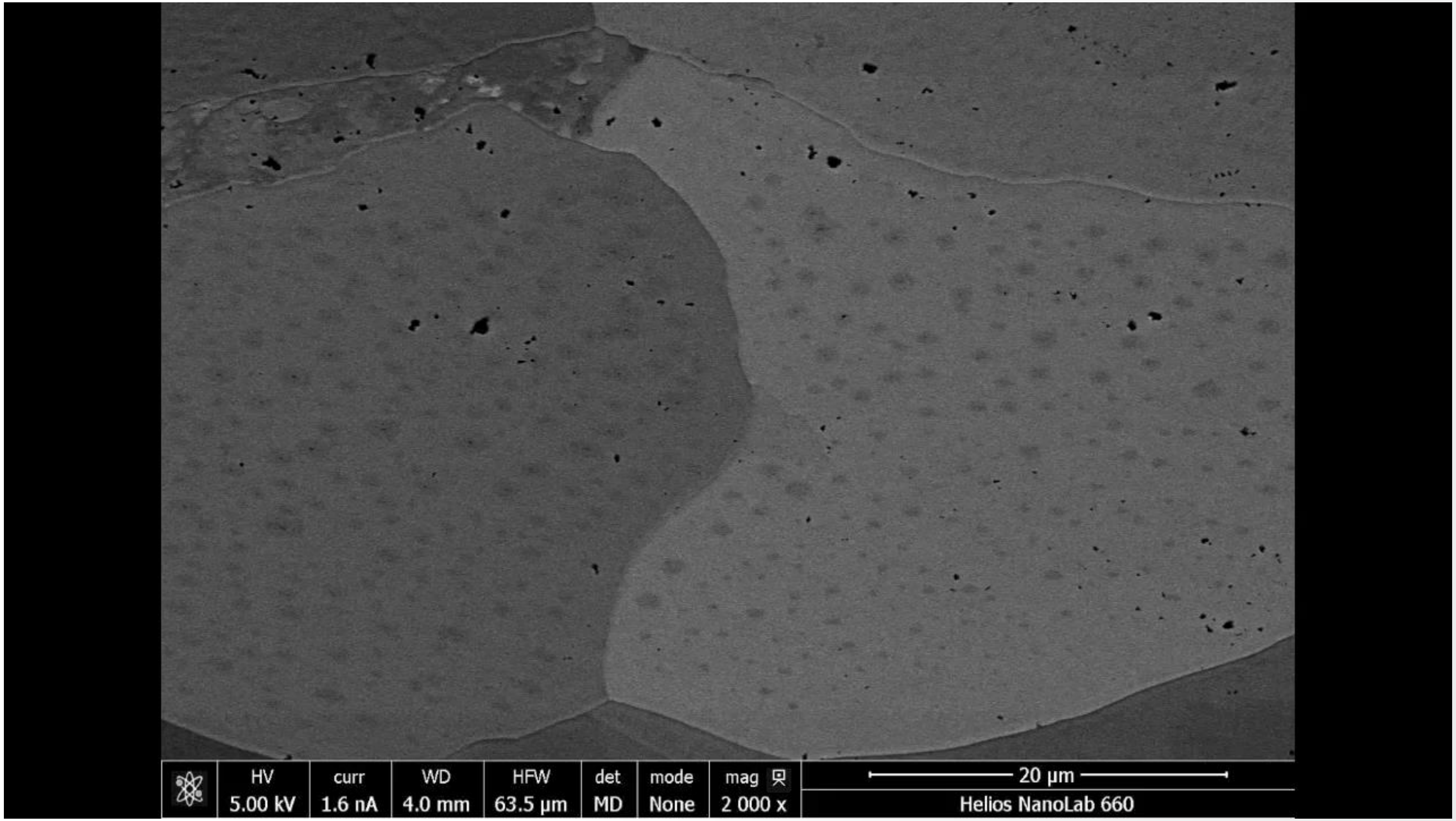
Focused Ion Beam Technique

How does Dual Beam FIB Work?



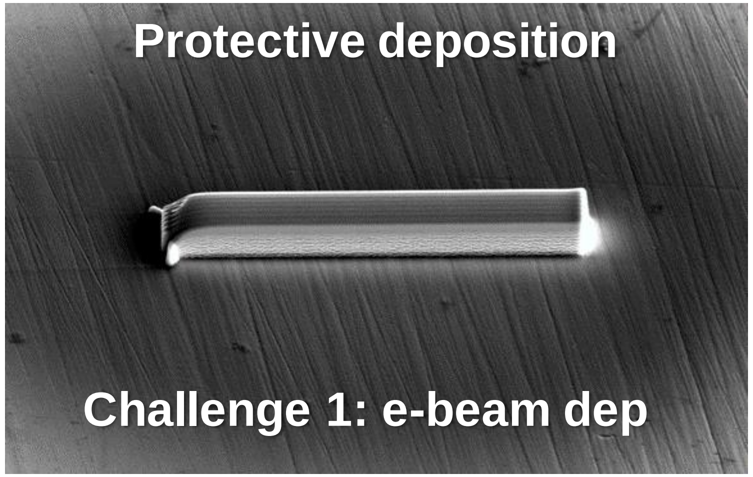
Focused Ion Beam Technique

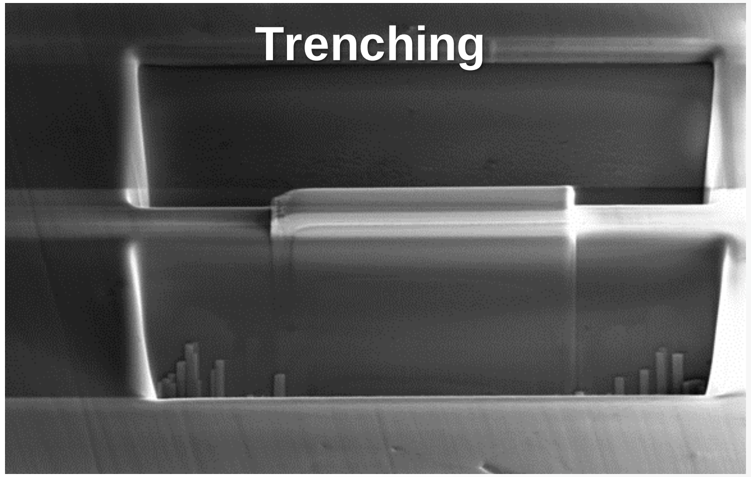
S/TEM Sample preparation workflow

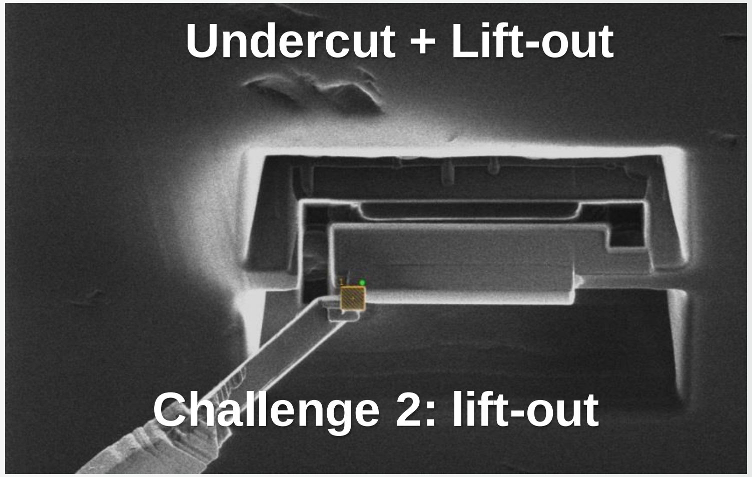


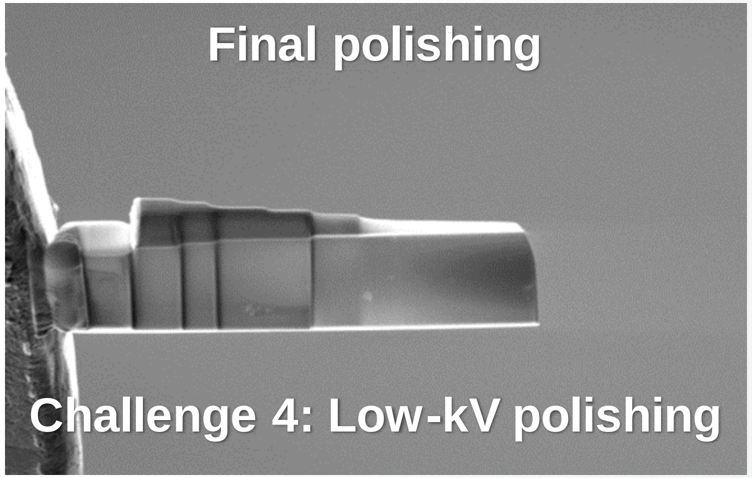
Focused Ion Beam Technique

S/TEM Sample preparation workflow – Challenges







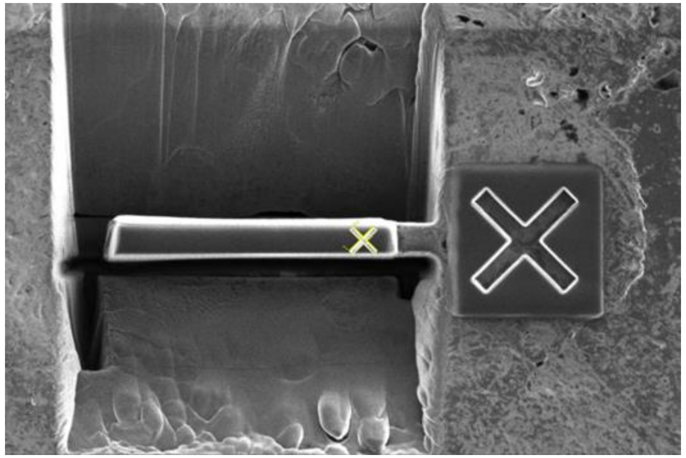


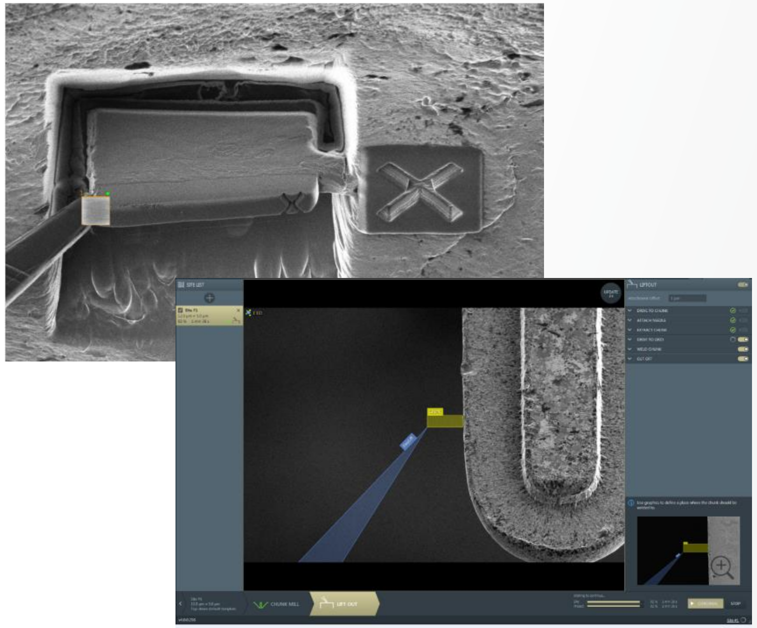
1. E-beam deposition
2. Lift-out
3. Final thinning
4. Low-kV polishing
5. Expert level operator is needed

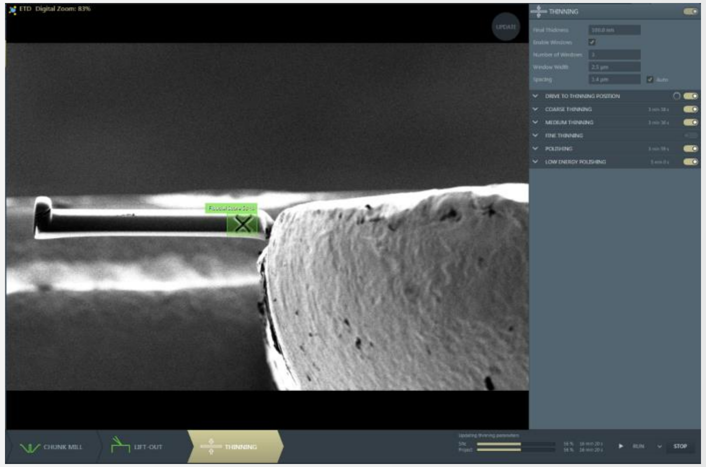
AutoTEM 5 offers complete, fully automated in-situ lift-out preparation process

Focused Ion Beam Technique

AutoTEM 4 – Complete *in situ* Lift-out Workflow







- Automatic
- Automatic with manual recovery
- Guided

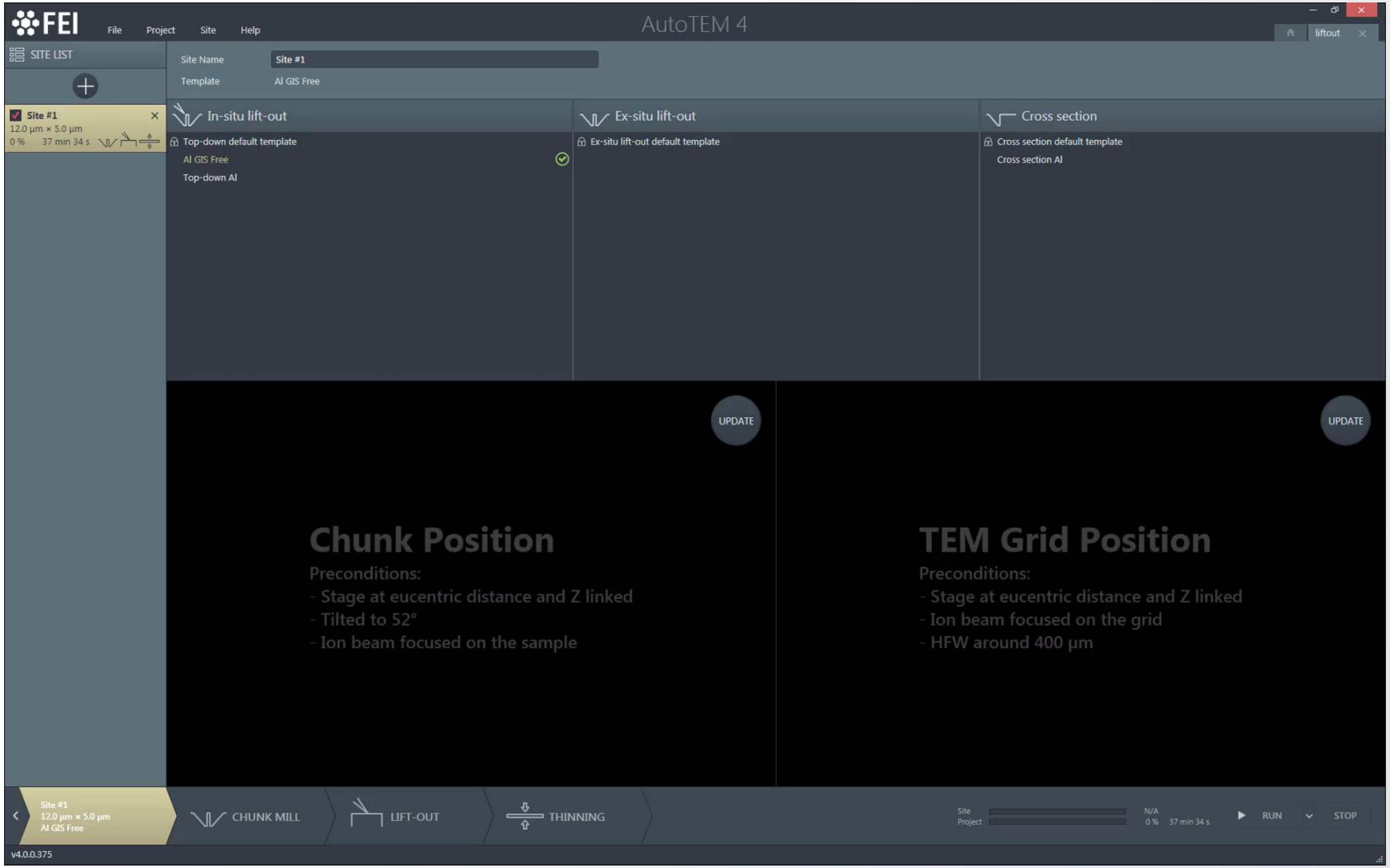
- Guided

- Automatic
- Automatic with manual recovery
- Guided

Focused Ion Beam Technique

AutoTEM 4 – Run in 3 clicks

- Select template
- Define position in the bulk
- Define grid position
- RUN

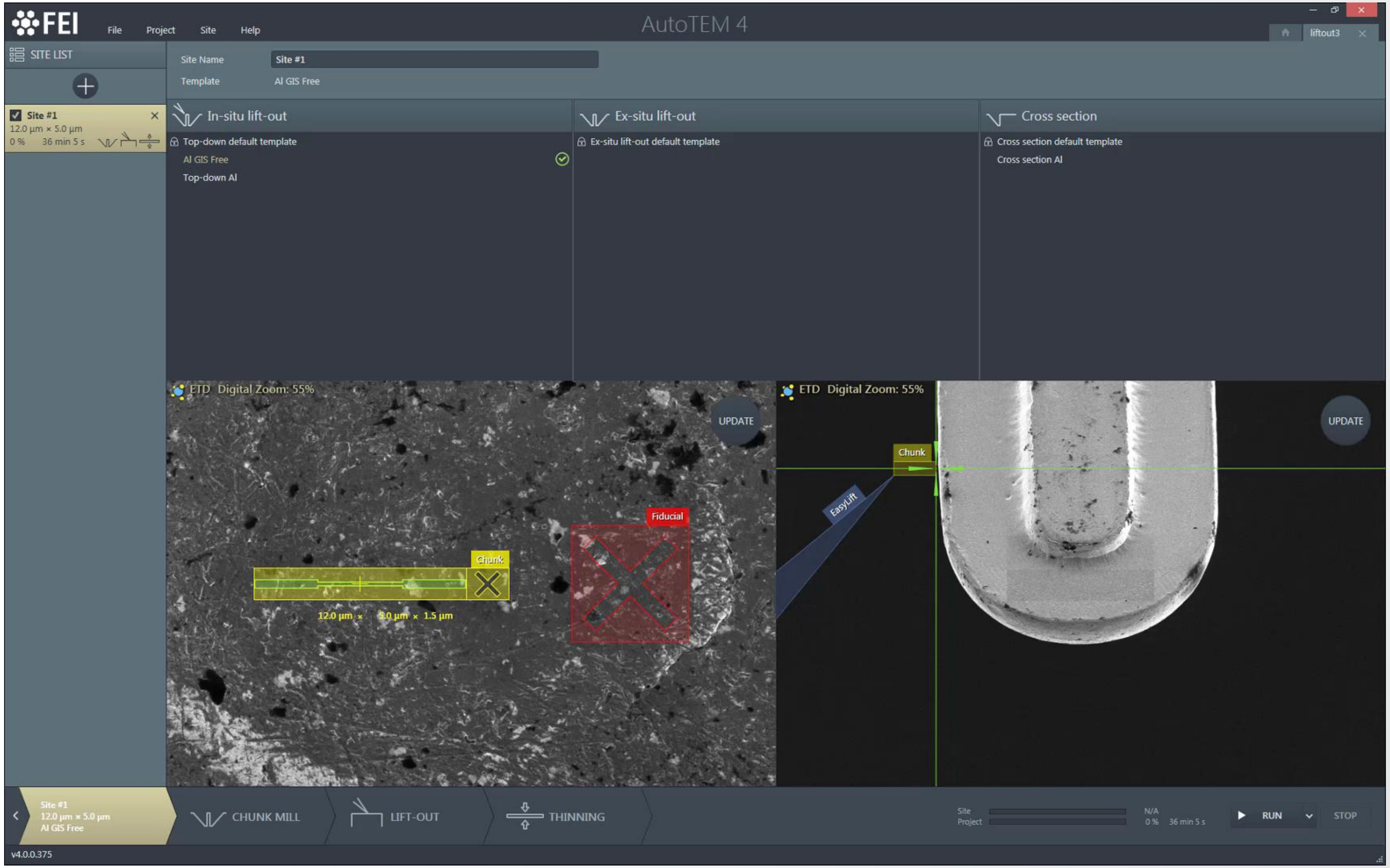


Focused Ion Beam Technique

AutoTEM 4 – Chunk mill

Chunk mill

- CREATE FIDUCIAL
- GROUND
- PROTECTIVE LAYER
- ROUGH MILLING
- MEDIUM MILLING
- CUTOUT
- CLEANUP
- CREATE THINNING FIDUCIAL

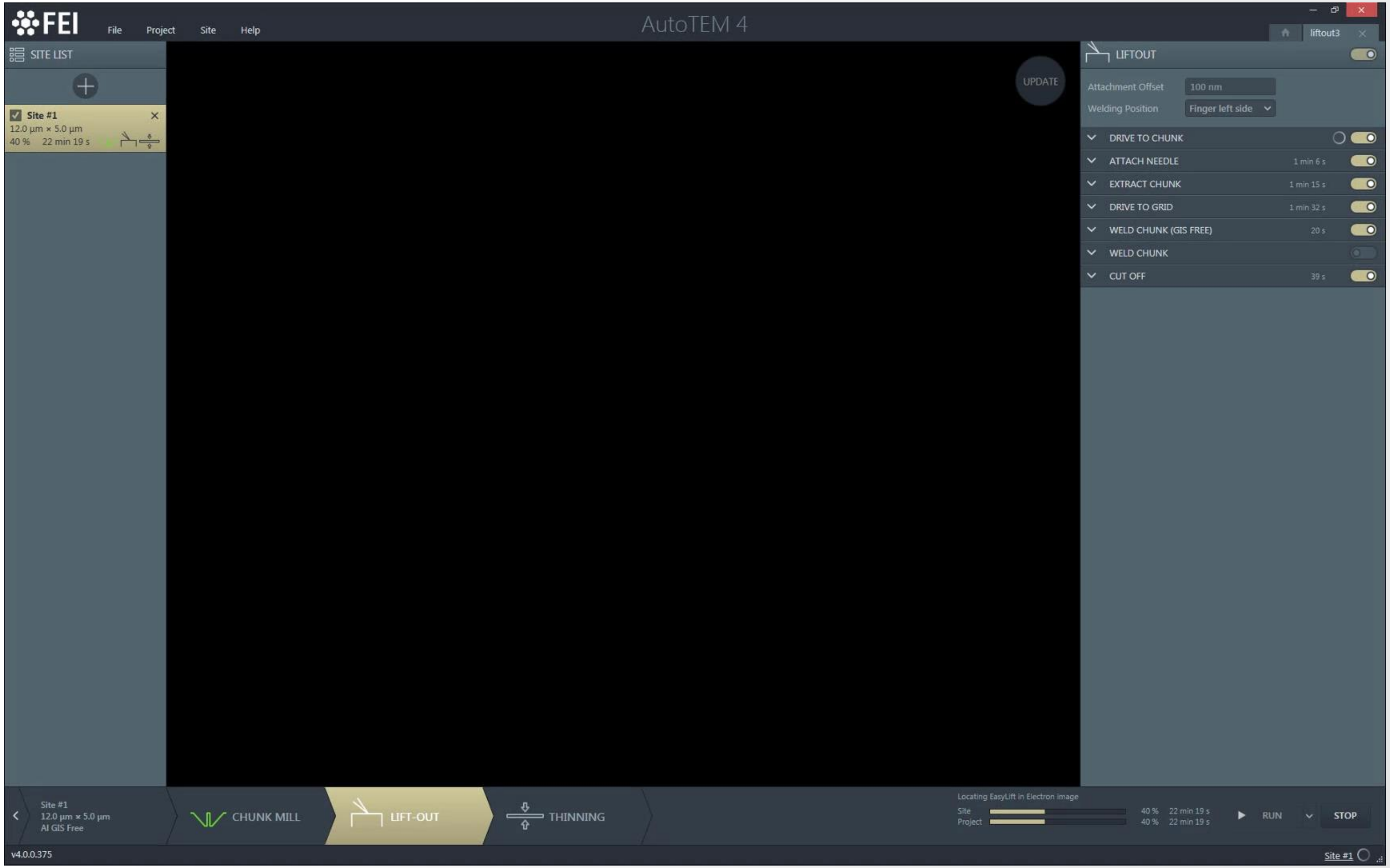


Focused Ion Beam Technique

AutoTEM 4 – Guided in-situ lift-out

Guided lift-out

- DRIVE TO CHUNK
- ATTACH NEEDLE
- EXTRACT CHUNK
- DRIVE TO GRID
- WELD CHUNK
- WELD CHUNK (GIS FREE)
- CUT OFF

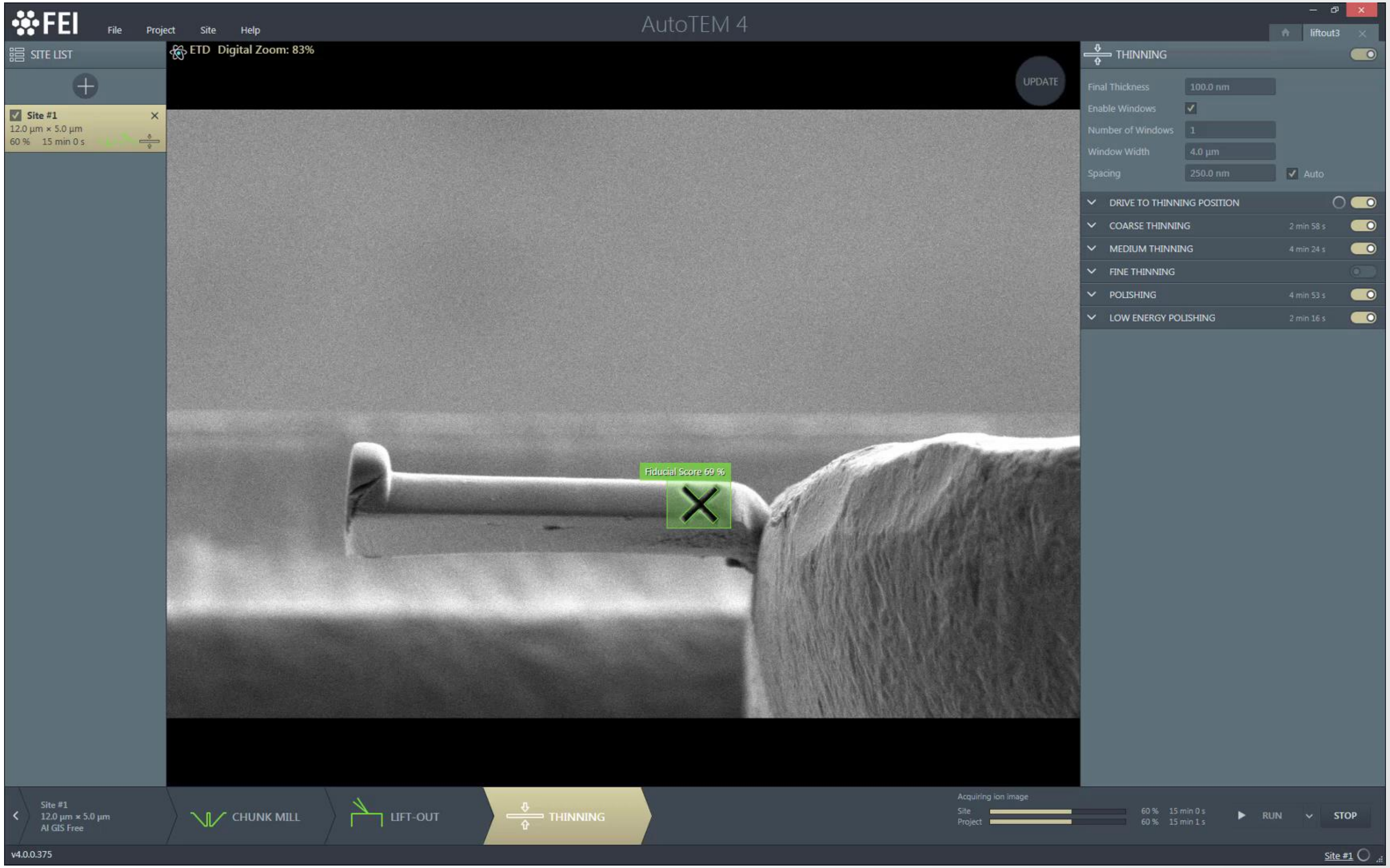


Focused Ion Beam Technique

AutoTEM 4 – Thinning

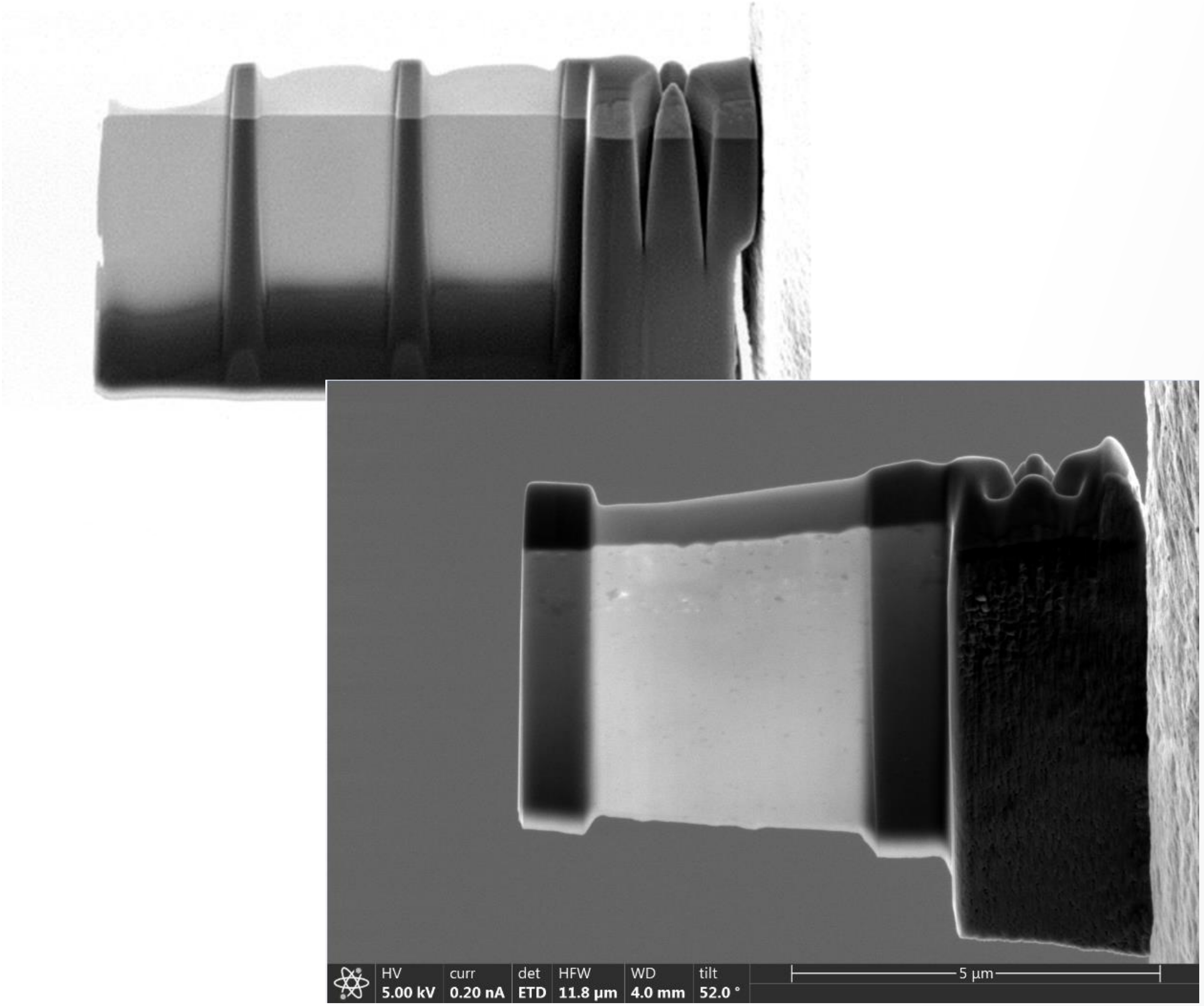
Thinning

- DRIVE TO THINNING POSITION
- COARSE THINNING
- MEDIUM THINNING
- FINE THINNING
- POLISHING
- LOW ENERGY POLISHING

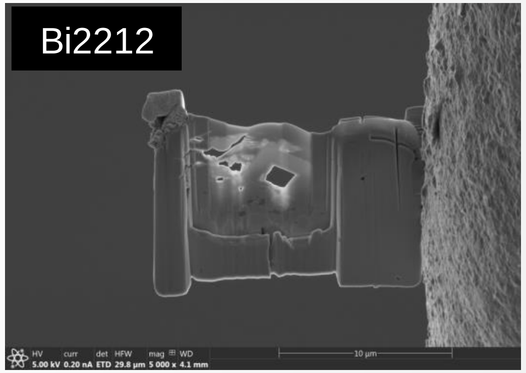


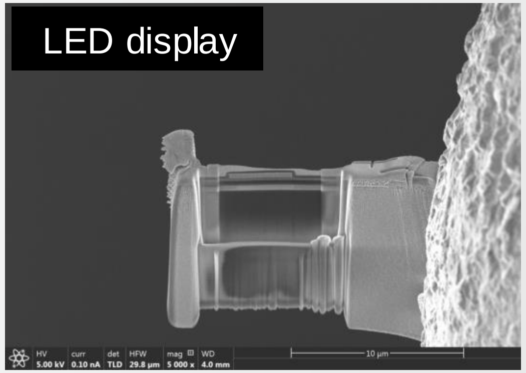
Focused Ion Beam Technique

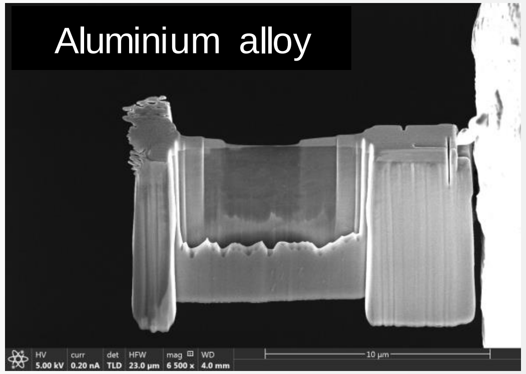
Robust, predictable results for a wide range of materials

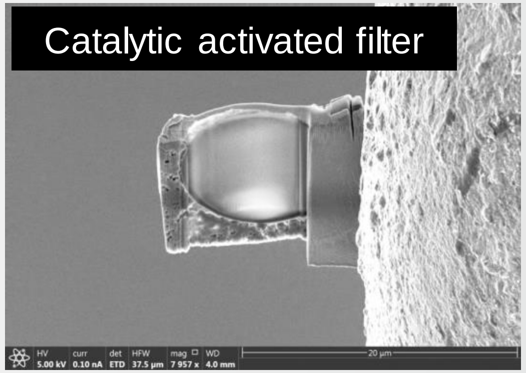


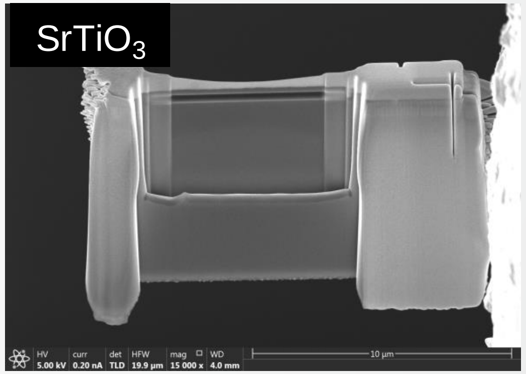
Courtesy: Remco Geurts, Thermo Fisher

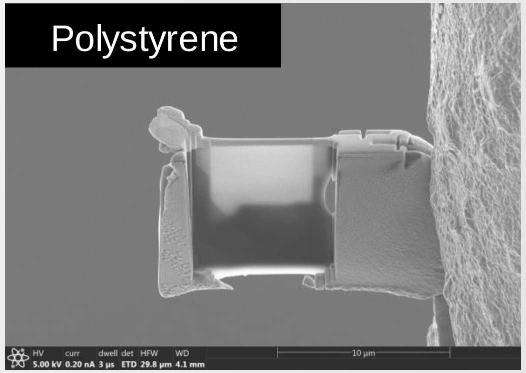






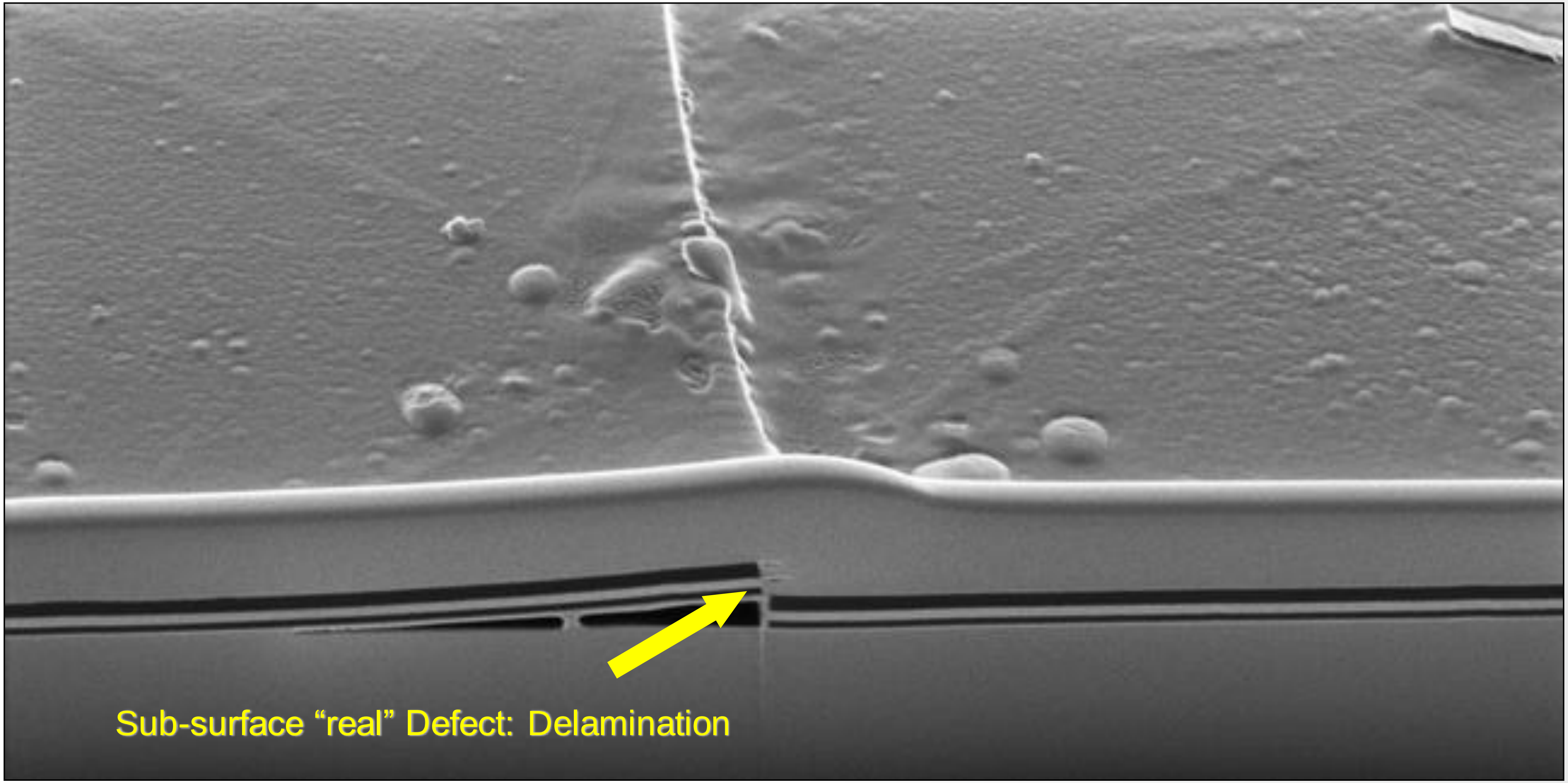






Focused Ion Beam Technique

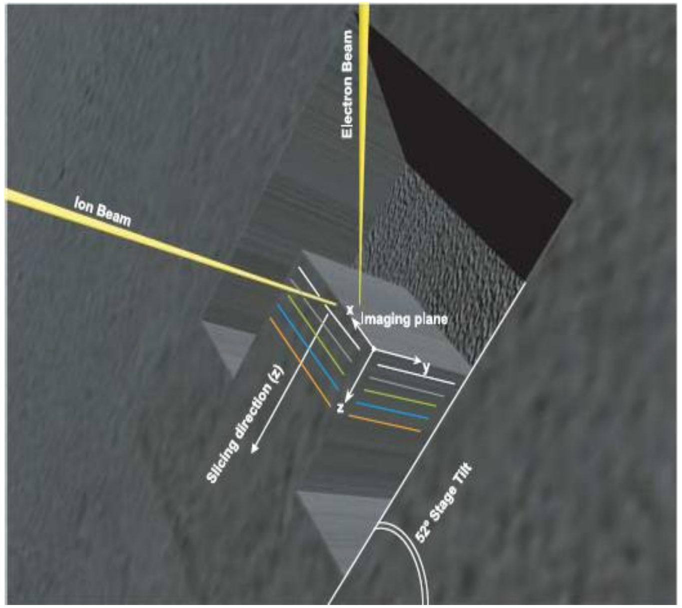
3D characterization by FIB

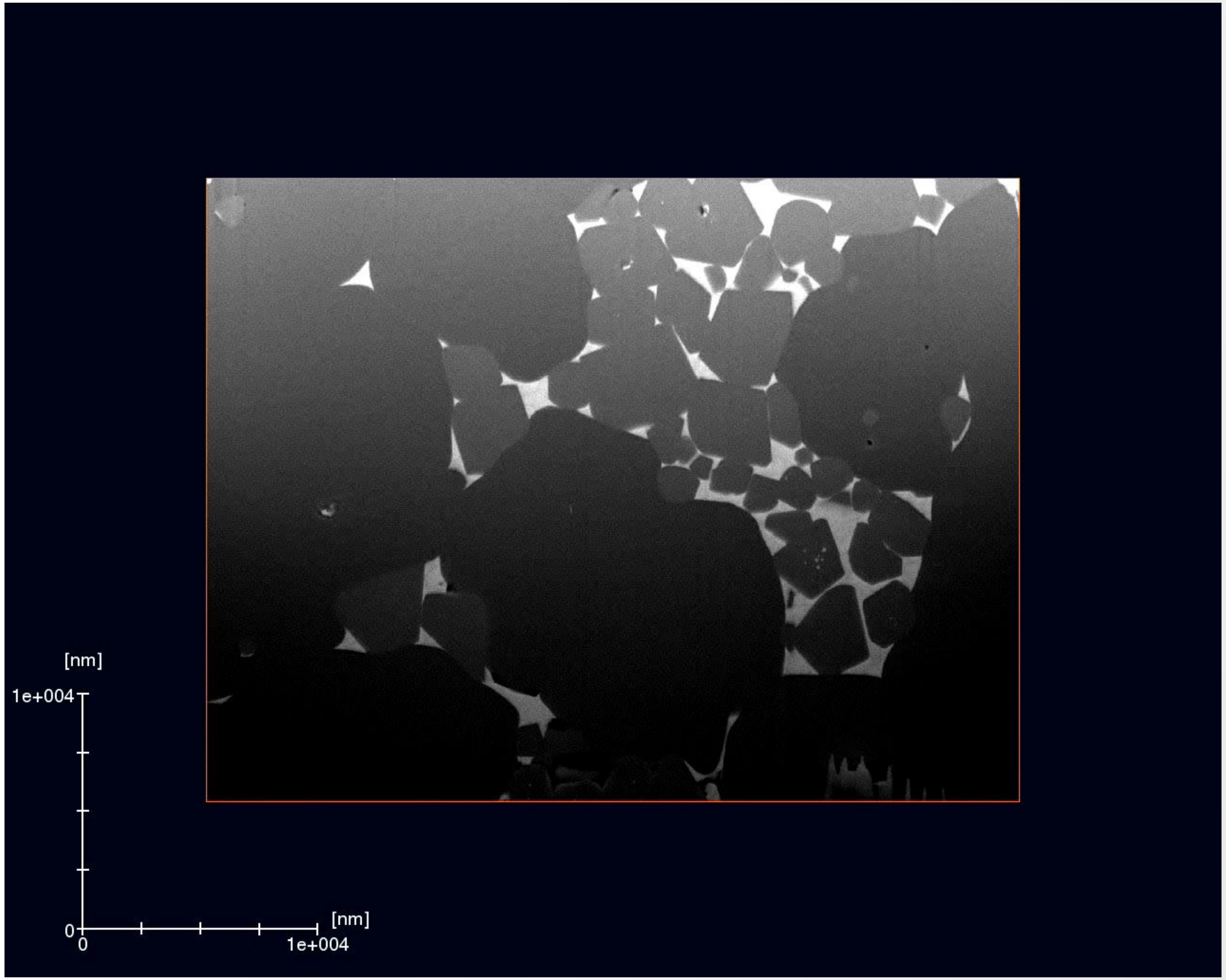


Sub-surface defect, anti-reflective coating on clear plastic motorcycle visor

Focused Ion Beam Technique

3D characterization by FIB



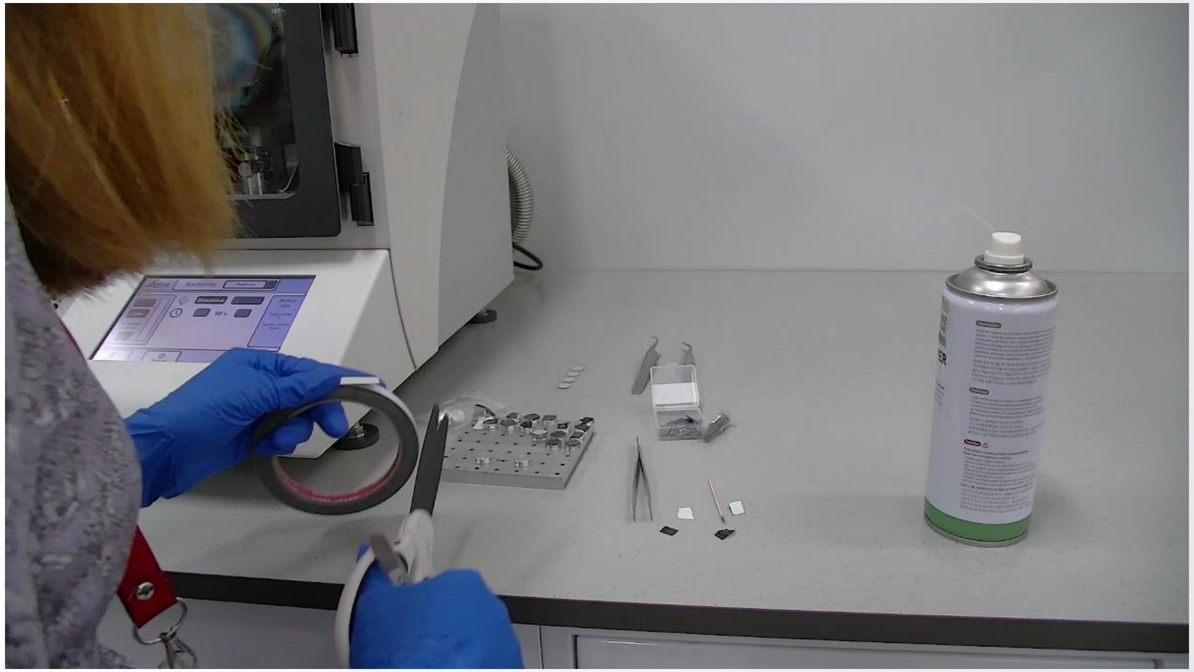


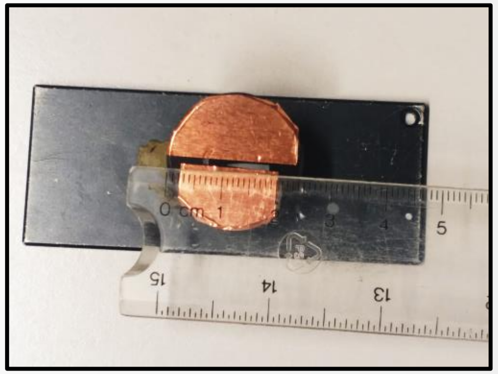
L. HOLZER, et al., Journal of Microscopy Vol. 216, Pt 1 October 2004, pp. 84–95

Focused Ion Beam Technique

Sample pre-conditioning before FIB preparation

- Pre-coated with gold, carbon, or Pt
  - Powder and surface-sensitive samples
  - Ga-ion is very destructive. It can remove 10-20 nm surface material in a single scan
  - Increase sample conductivity
  - As a reference for the final sample condition, i.e., the selected layer is destroyed by the Ga-ion
- Non-conductive sample
  - Can reduce image quality and introduce sample drift.
  - If coating strategy can't be performed, use a silver paste to glue the sample into the FIB sample holder and cover some parts of the sample with it.
  - Use copper tape to cover most of the sample to reduce the charging effect.





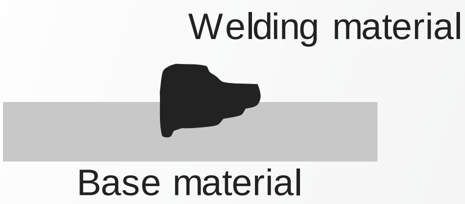
Movie Courtesy Wang Yi, Shanghai Nanoport

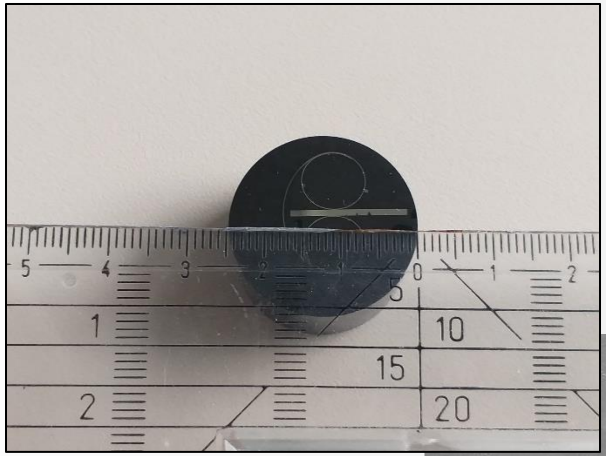
Photograph Fadli Rohman Research Center for Physics LIPI (BRIN) and GFE RWTH Aachen

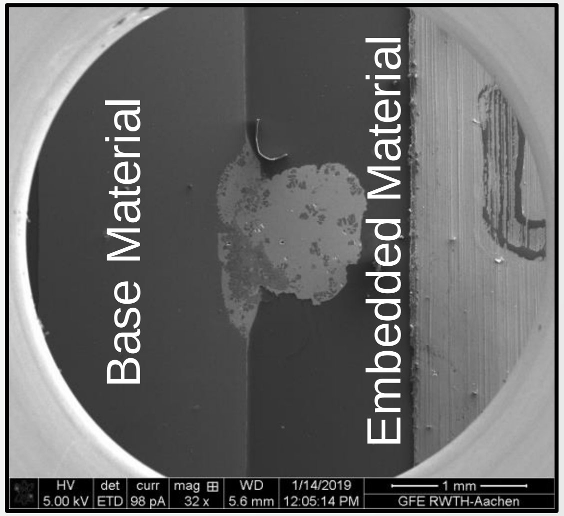
Focused Ion Beam Technique

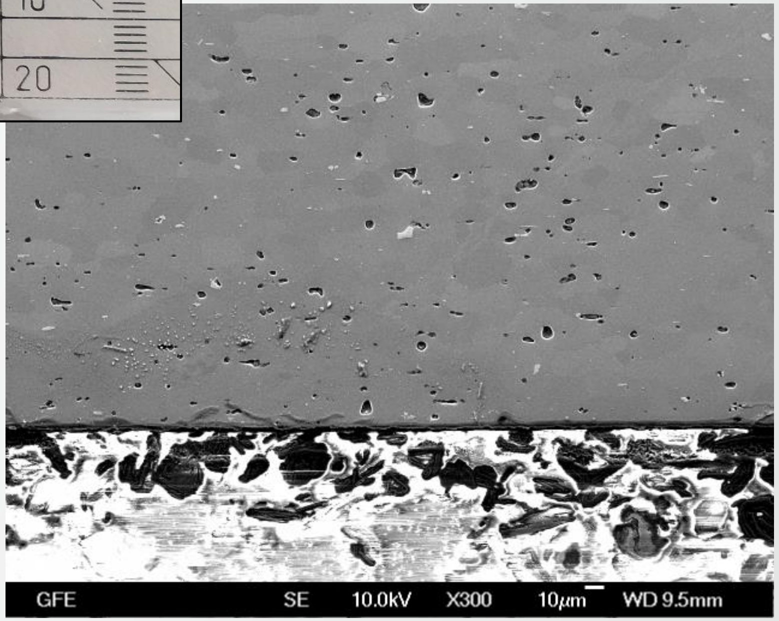
Sample pre-conditioning before FIB preparation

- Embedded samples
  - A very thick cross-section samples: soldering materials, oxide layer containing samples, or sample with precipitates distributions.
  - Larger investigation area
  - Eliminated FIB depth milling condition (4 µm)
- Type of embedded materials:
  - Epoxy if the area of interest is not located at the sample surface. The charging effect as epoxy is non-conductive.
  - Cu-C mixed based embedded
  - It doesn't introduce a charging effect
  - Be careful with redeposition of Cu-C particles during surface polishing









Non-Biological Sample Preparation

Focused Ion Beam Technique

- Preparation prior FIB
  - Coated the sample surface with carbon, gold, or platinum
  - For a specific area, investigate the sample using EDS or EBSD
  - Make sure the sample's dimensions is allowed by the FIB chamber
  - Make sure the sample is clean, so it won't affect the vacuum condition
  - Non-bulk samples can be prepared as embedded samples before being prepared by FIB

- Tips:
  - FIB is a local and specific preparation technique
    - Make sure to know what and where the lamella need to be prepared
    - A prior investigation is necessary
  - Choose the protection layer wisely
    - EDS overlapped signals with elements containing sample
    - Contrast differences, i.e., W/Pt can not be used for a sample containing a lower atomic number
  - Beware of artifacts due to:
    - Choose of tilt angle
    - HT for cleaning step
    - It all depends on the sample's materials
    - Small precipitates due to Ga ion redeposition

TEM Sample Preparation – Non-Biological Sample

Thermo Fisher SCIENTIFIC

Outline

- Introduction
- Sample Preparation
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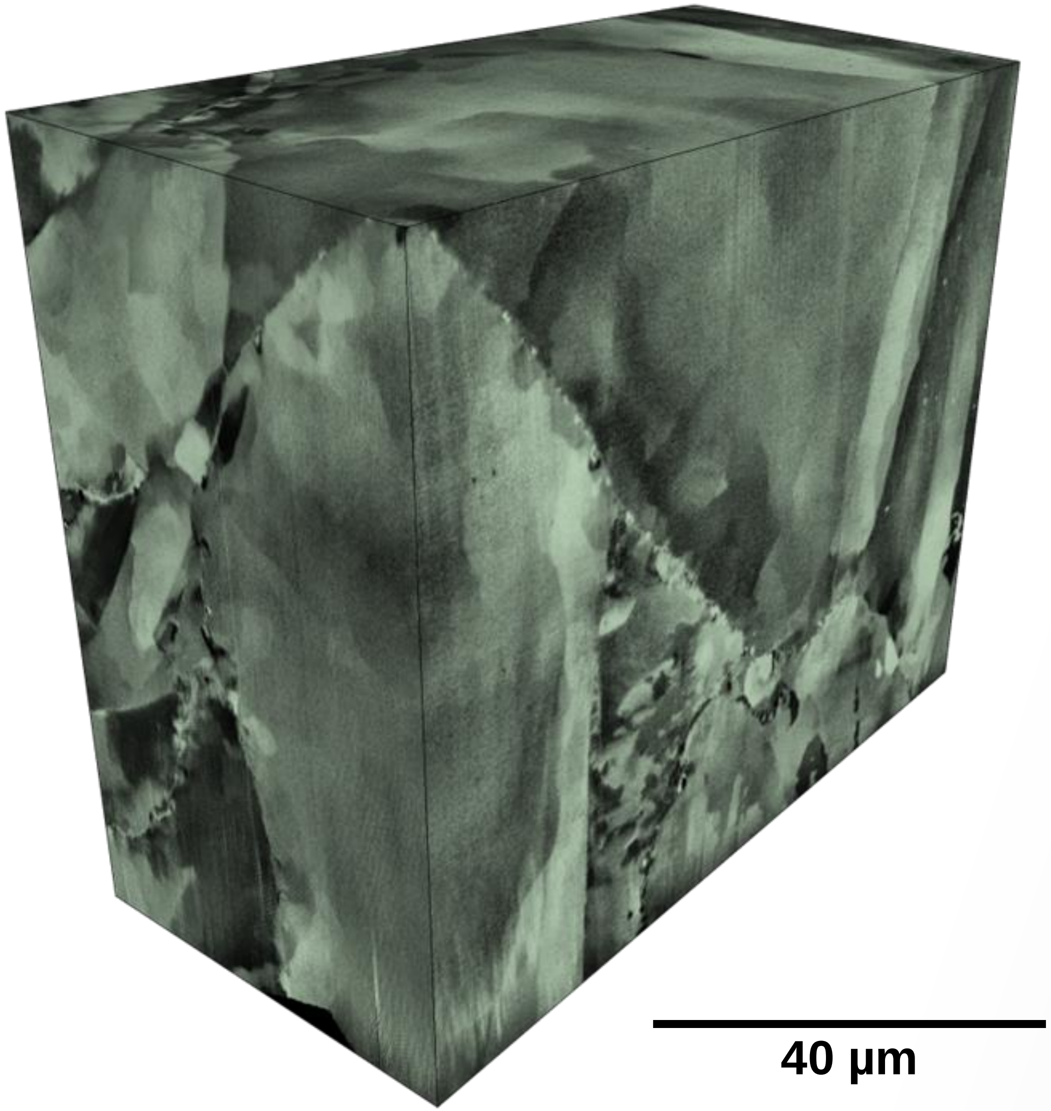
    - Plasma + Laser
- Artifacts caused by bad sample preparations

Helios G4 PFIB UXe

Plasma Focus Ion Beam (FIB)

Helios G4 PFIB – 100x more characterization without compromise

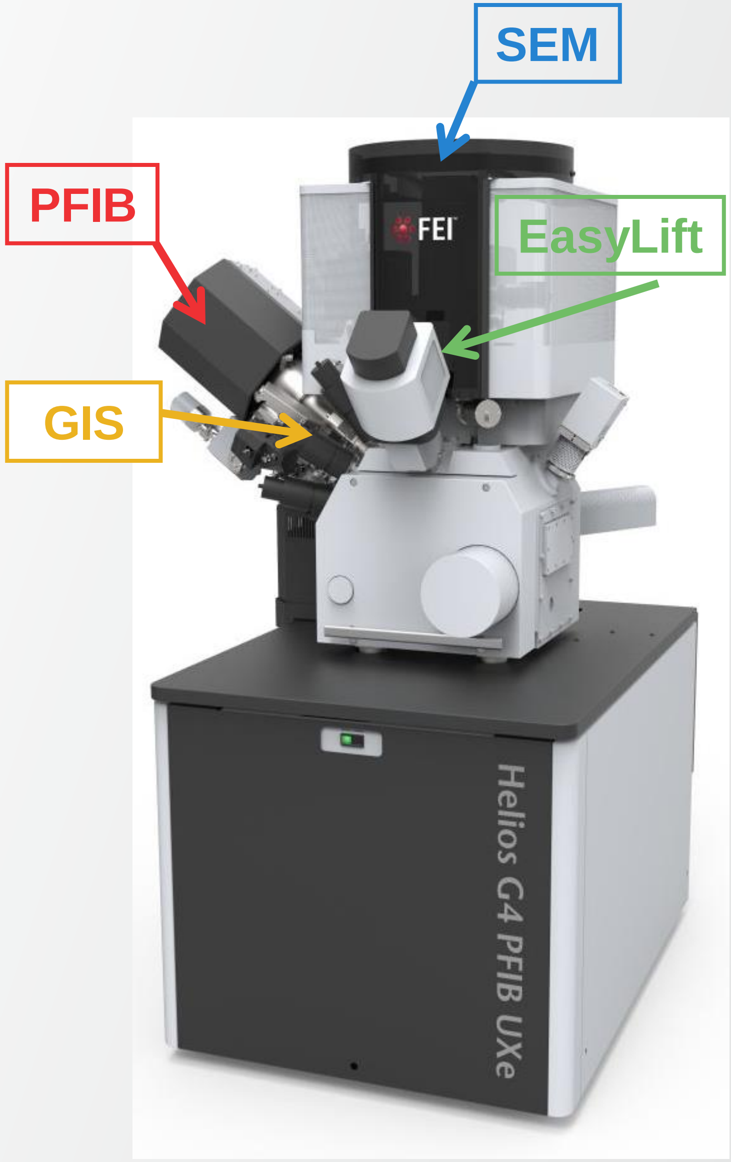


Xe+ PFIB

40 µm

Ga+ FIB

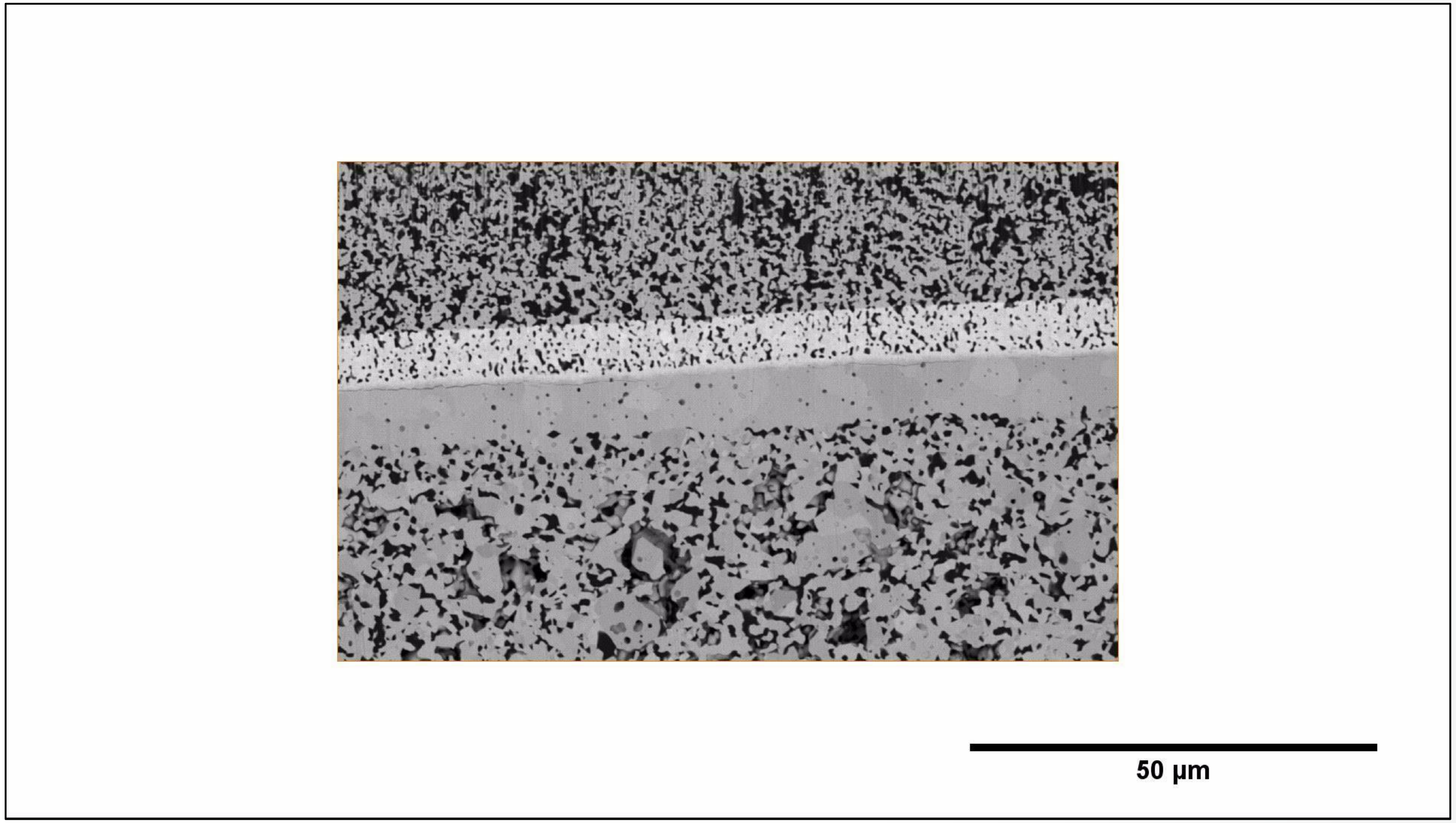
40 µm



Plasma Focus Ion Beam (FIB)

Helios G4 PFIB – High throughput large volume 3D characterization

Solid Oxide Fuel Cell: 3D rendering from anode to the cathode



- Reconstruction of the entire fuel cell structure
- The pore space is segmented and a pore network is build
- Balls and sticks represent here pores and pore throats respectively

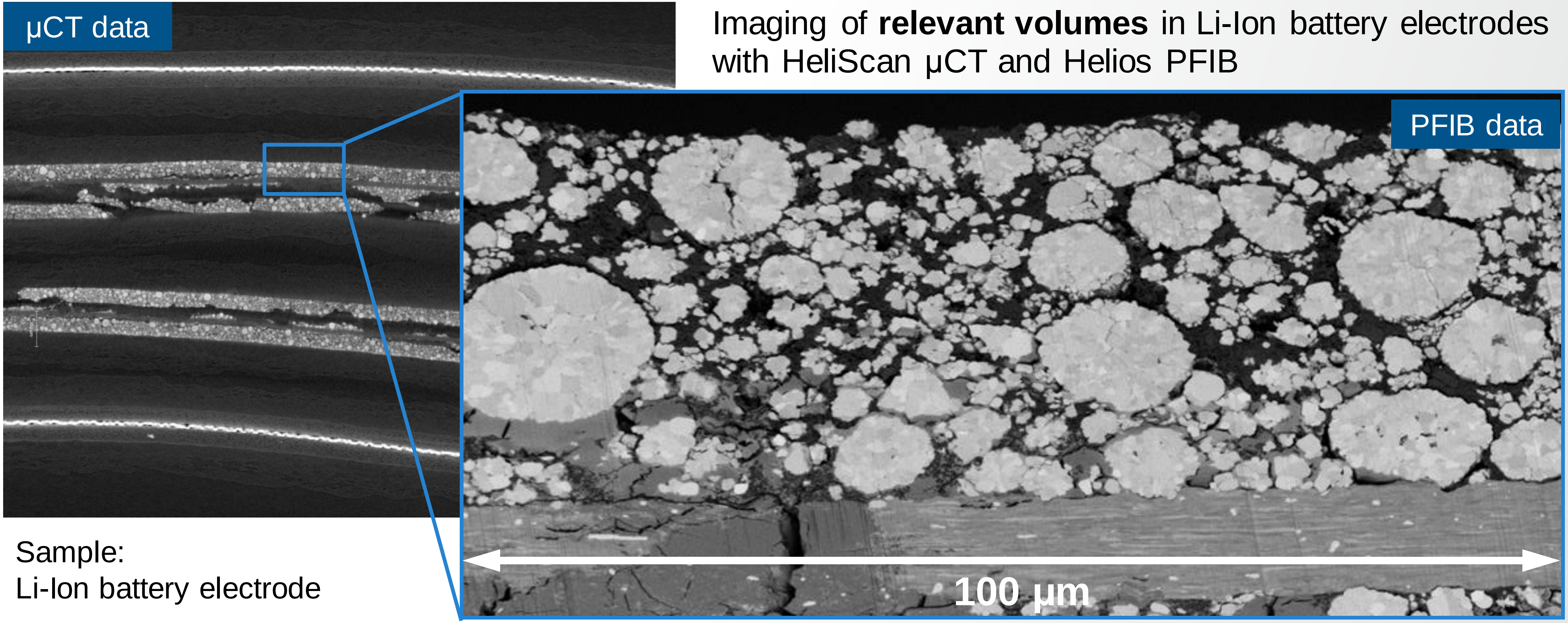
Sample courtesy: Jochen Joos, KIT

Data acquired: Remco Geurts, Thermo Fisher

Plasma Focus Ion Beam (FIB)

Helios G4 PFIB – Correlative workflow (μCT-Dual Beam)

Imaging of **relevant volumes** in Li-Ion battery electrodes with HeliScan μCT and Helios PFIB



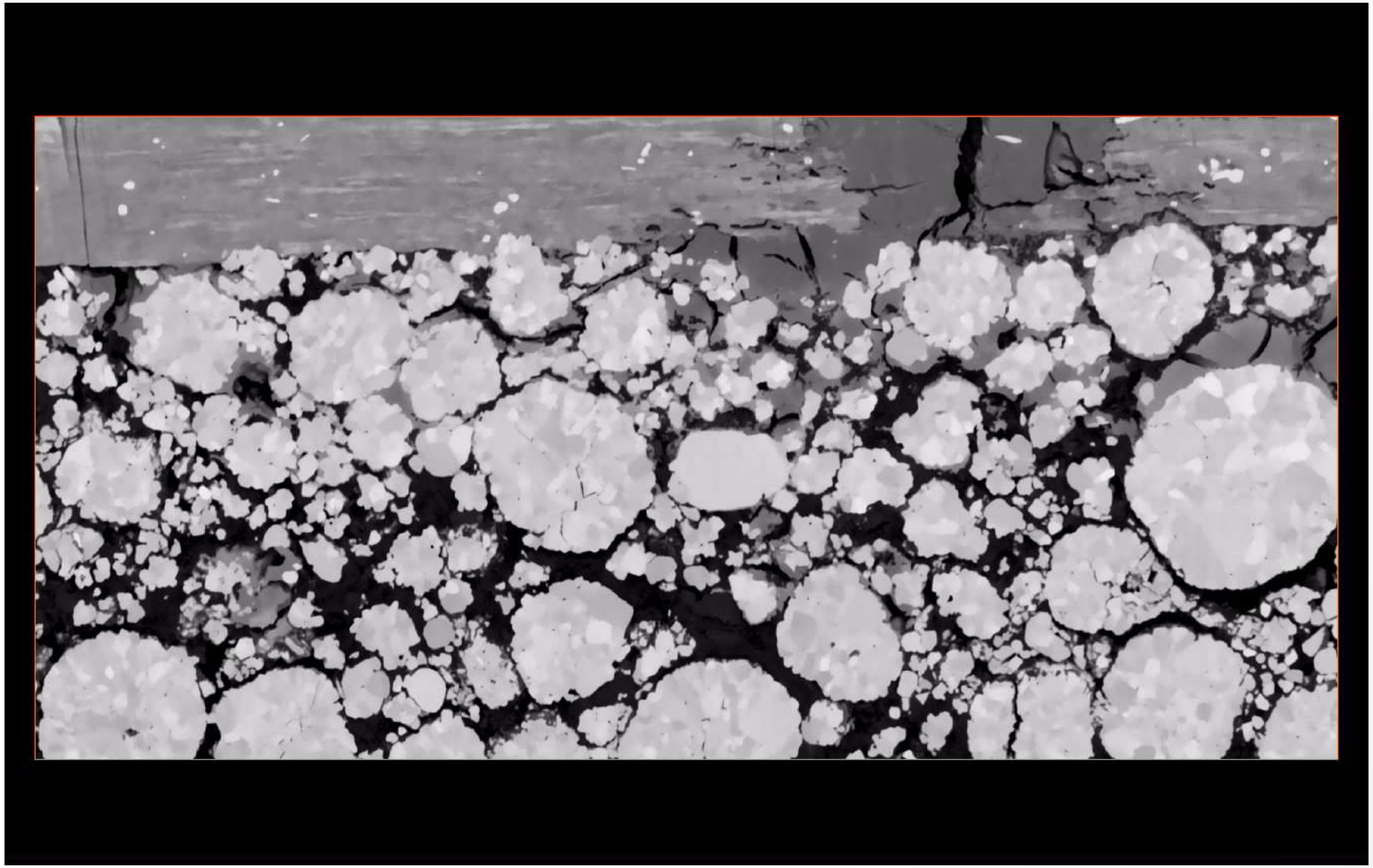
Sample:
Li-Ion battery electrode

Sample Courtesy: Johannes Schmalstieg (ISEA), Fabian Frie (ISEA), Joachim Mayer (GFE) RWTH, Aachen, Germany

Data acquired: Chengge Jiao, Thermo Fisher

Plasma Focus Ion Beam (FIB)

Helios G4 PFIB – Correlative workflow (µCT-Dual Beam)



Avizo:
- Reconstruct the data
- Build the pore network
- Link with performance

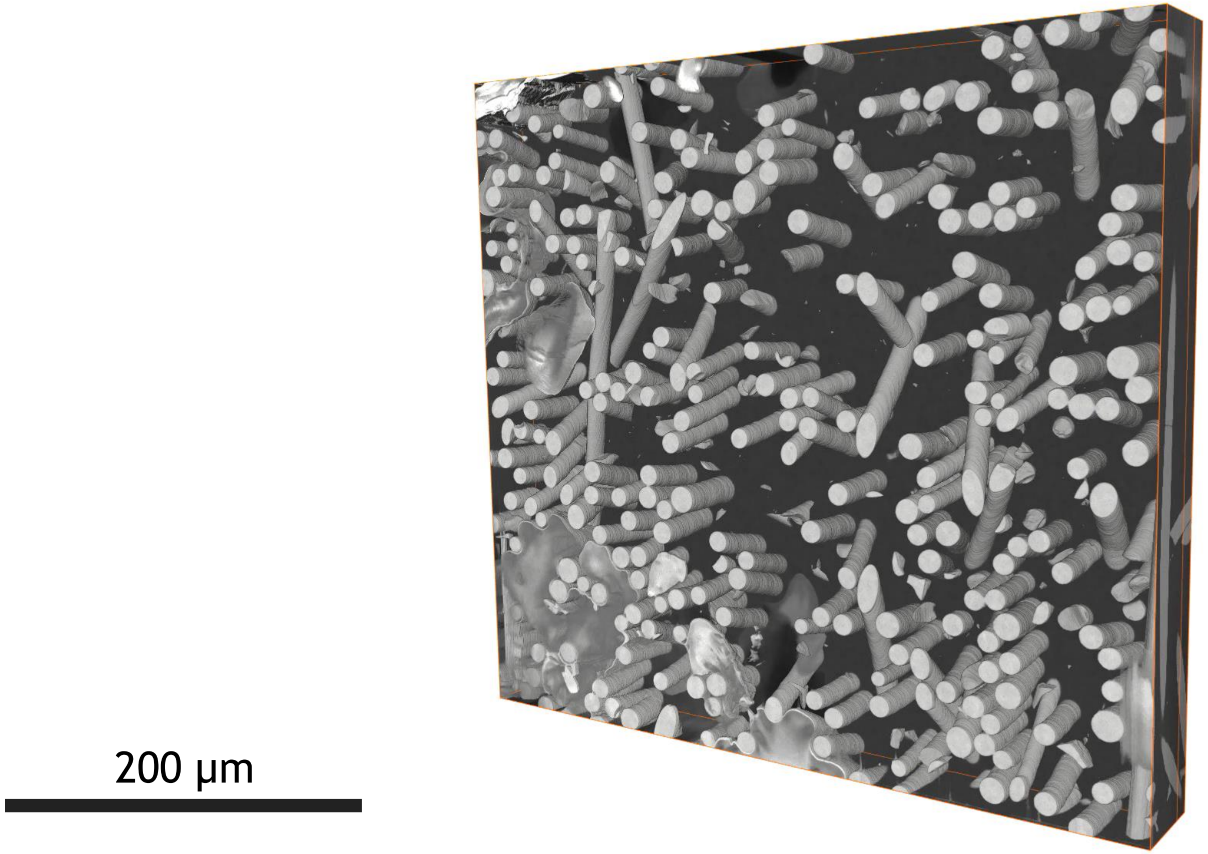
Sample:
Li-Ion battery electrode

Sample Courtesy: Johannes Schmalstieg (ISEA), Fabian Frie (ISEA), Joachim Mayer (GFE) RWTH, Aachen, Germany
Data acquired: Chengge Jiao, Thermo Fisher

Helios 5 Hydra

Thermo Fisher SCIENTIFIC

Highest quality large volume 3D characterization - **Oxygen ions for improved cut face quality in Carbon-based materials**



Sample: Car Oil Filter Casing
(polymer/glass fibre composite)
Data courtesy of Bart Winiarski, ThermoFisher

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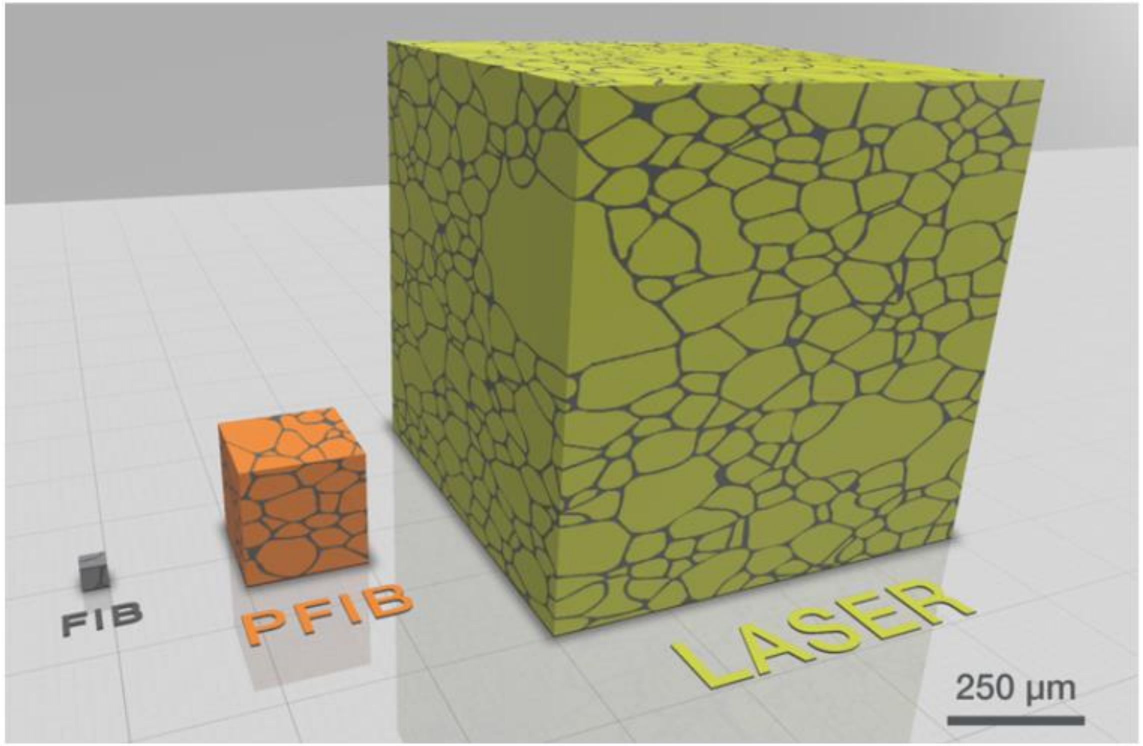
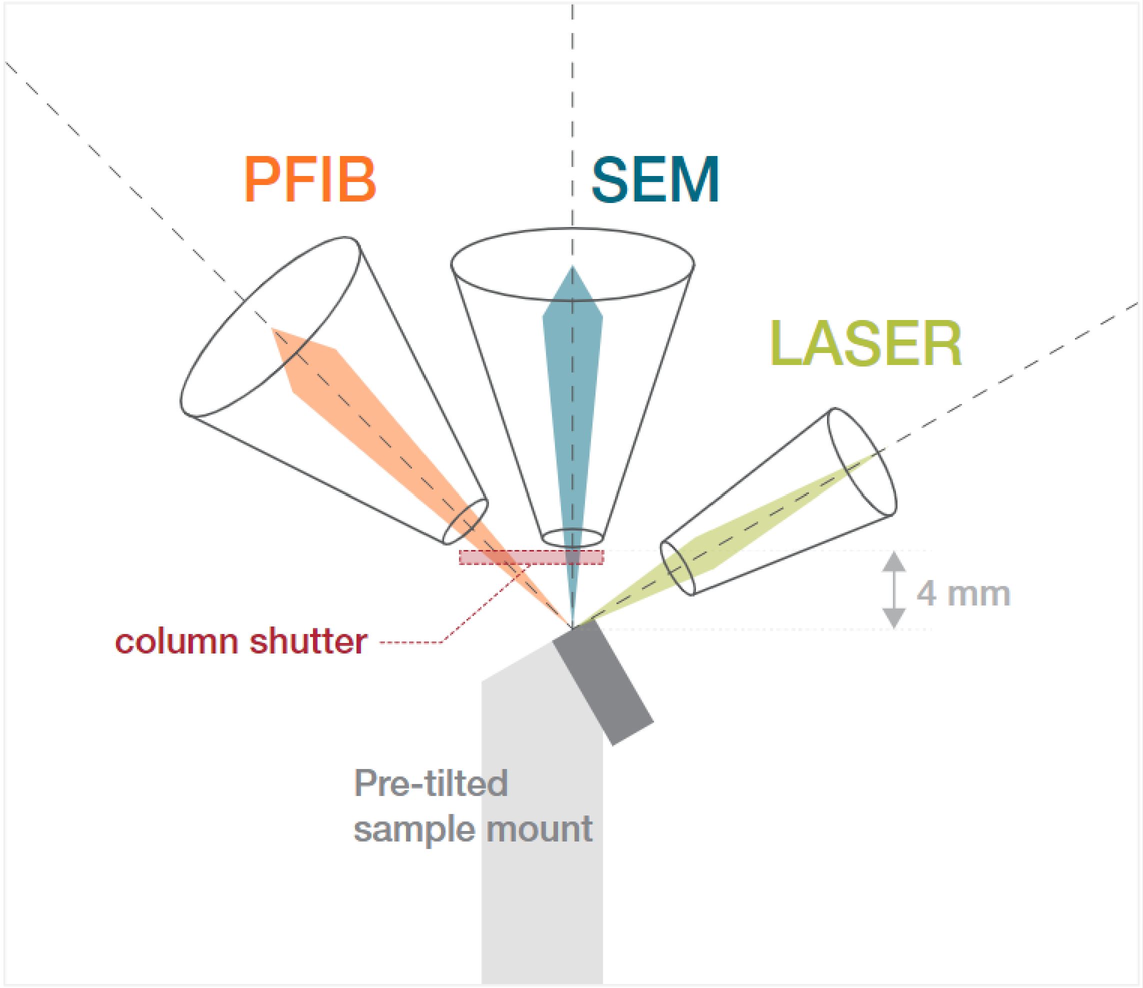
Helios 5 Laser PFIB

Helios 5 Laser Hydra System

Fast high-quality subsurface and 3D characterization at millimeter scale with nanometer resolution



Comparison of representative 3D volumes acquired within the same amount of time with FIB, Plasma FIB, and femtosecond laser.



Schematics of Helios 5 Laser Hydra. Three beams converging at a single coincident point allows fast switching between SEM imaging and laser processing, provides accurate and repeatable cut placement, increases throughput, and enables 3D characterization.

Helios 5 Laser Plasma FIB

Laser ablation for high-quality cross-sectioning of auto-body paints

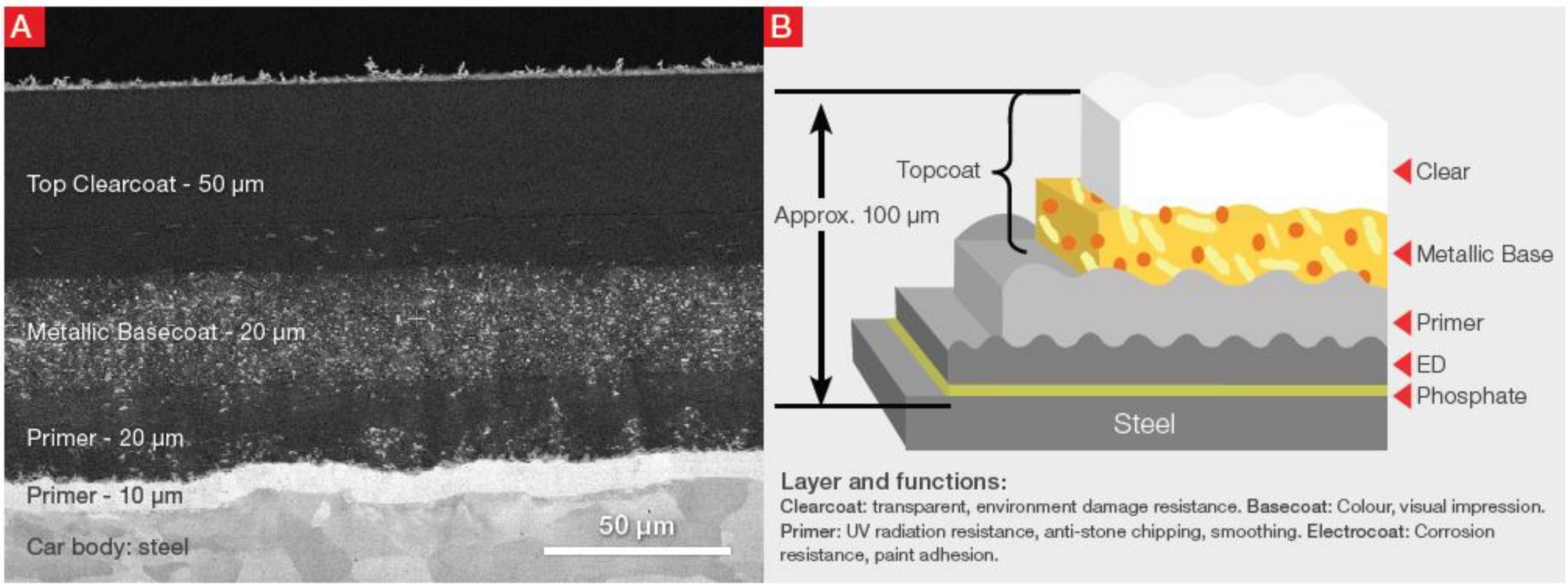
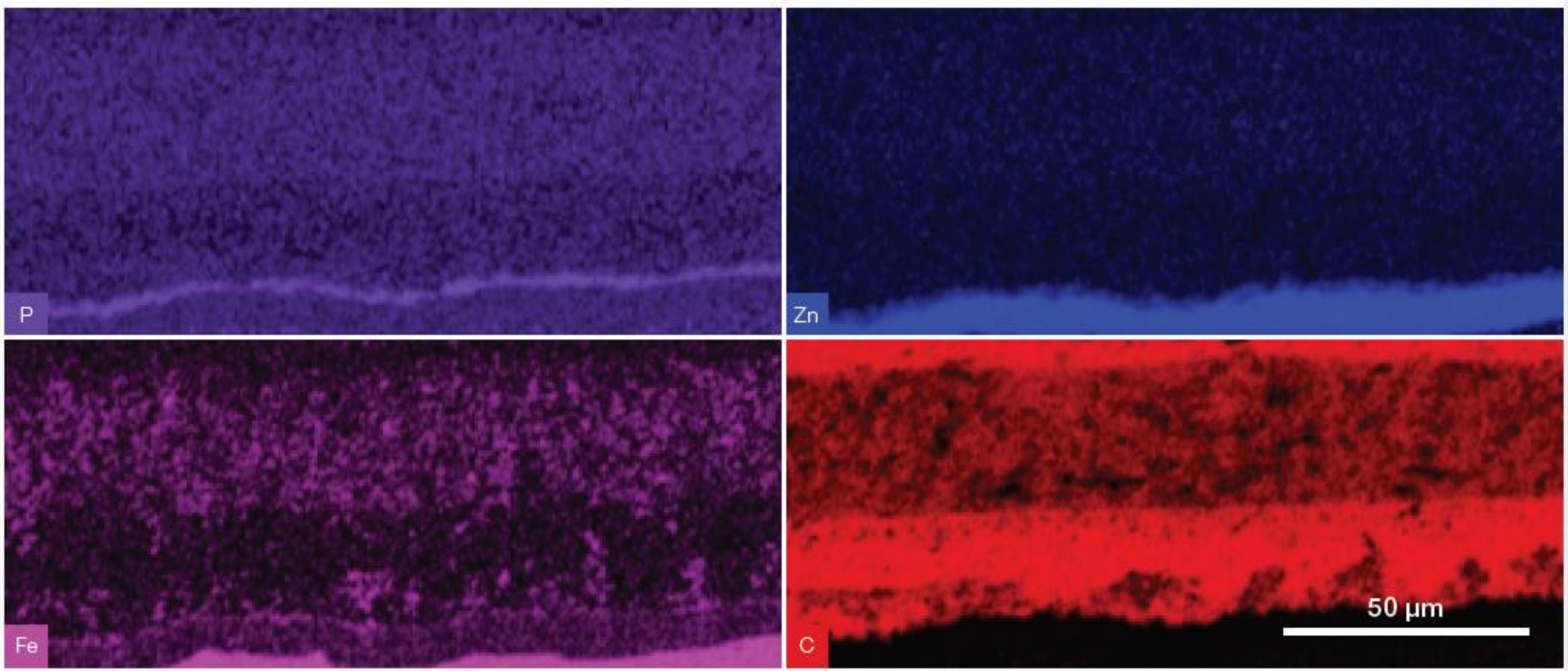


Figure 1. A) SEM backscattered electron image of an auto body coating cross-section. B) Typical four-layer automotive paint structure.

The schematic drawing is modified, with permission, from Akafuah, N, Poozesh, S, et al. Evolution of the Automotive Body Coating Process—A Review. Coatings, 6(2), 24. 2016. doi:10.3390/coatings6020024

Helios 5 Laser Plasma FIB

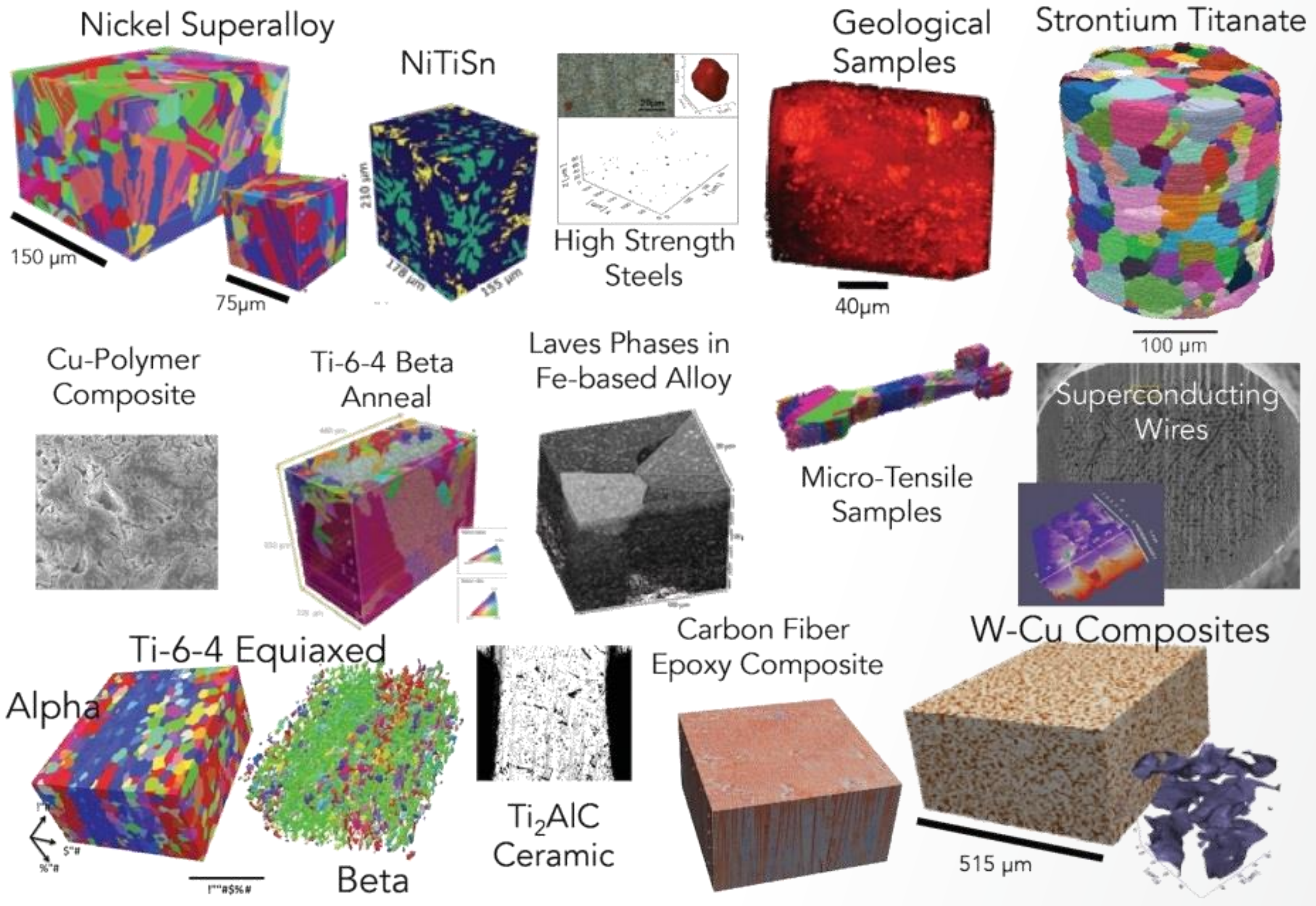
Laser ablation for high-quality cross-sectioning of auto-body paints



EDX elemental maps clearly correlate to the paint structure shown in the previous figure. Both cross-section preparation and imaging were performed using the Thermo Scientific Helios 5 Laser Plasma FIB. The cross-section was prepared using only the integrated in-chamber laser, without a PFIB clean up step. This demonstrates the high quality of femtosecond laser processing and enables the instrument to maintain high-throughput characterization.

Helios 5 Laser PFIB – Early adopters

Numerous examples and publications on metals, ceramics, composites, geoscience



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物質・材料研究機構
NIMS
National Institute for Materials Science

MANCHESTER 1824
The University of Manchester

HENRY ROYCE INSTITUTE

Data courtesy: UC Santa Barbara

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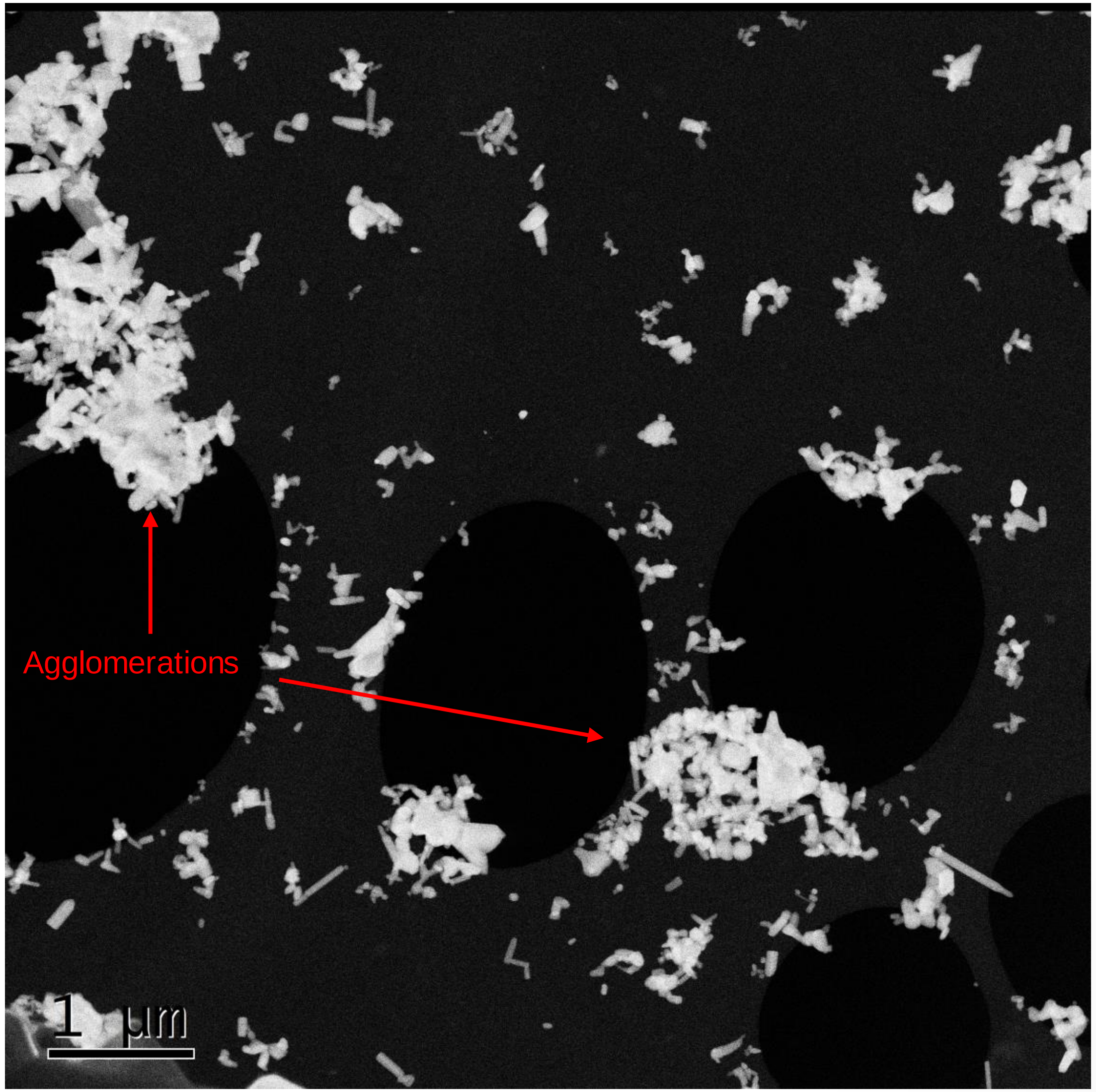
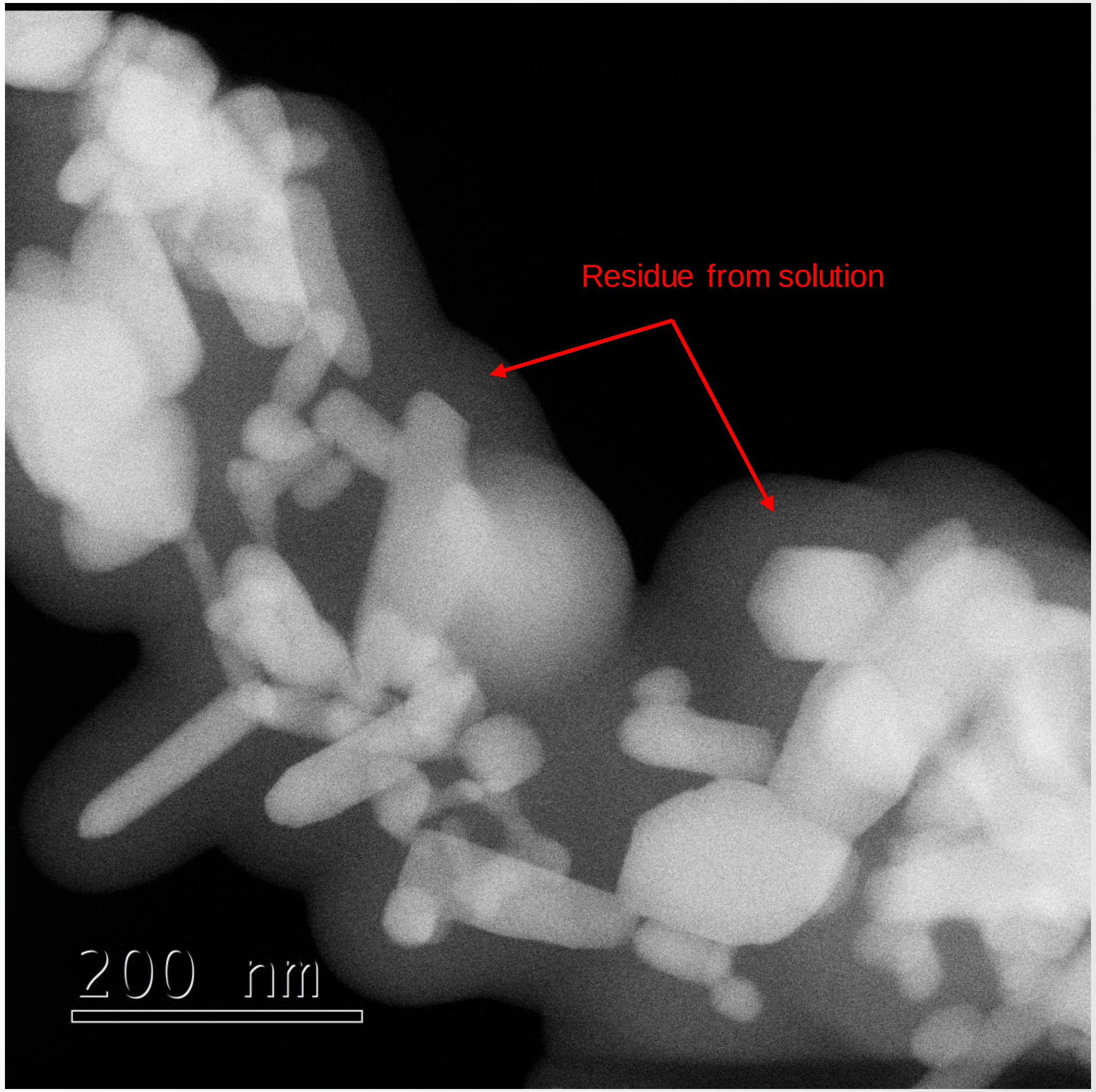
Talos F200X

Examples

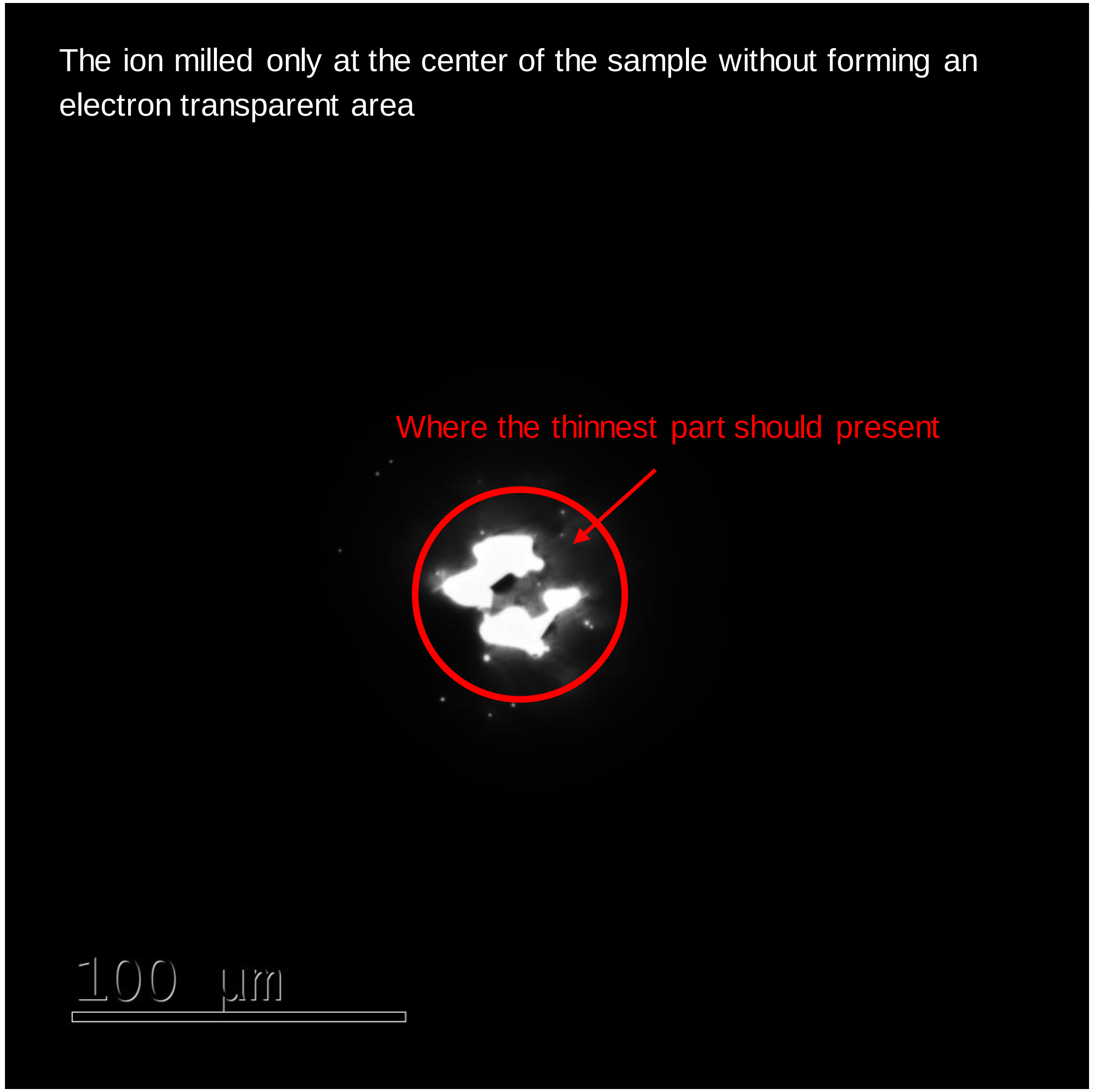
Powder Agglomerations and Residue from Solution

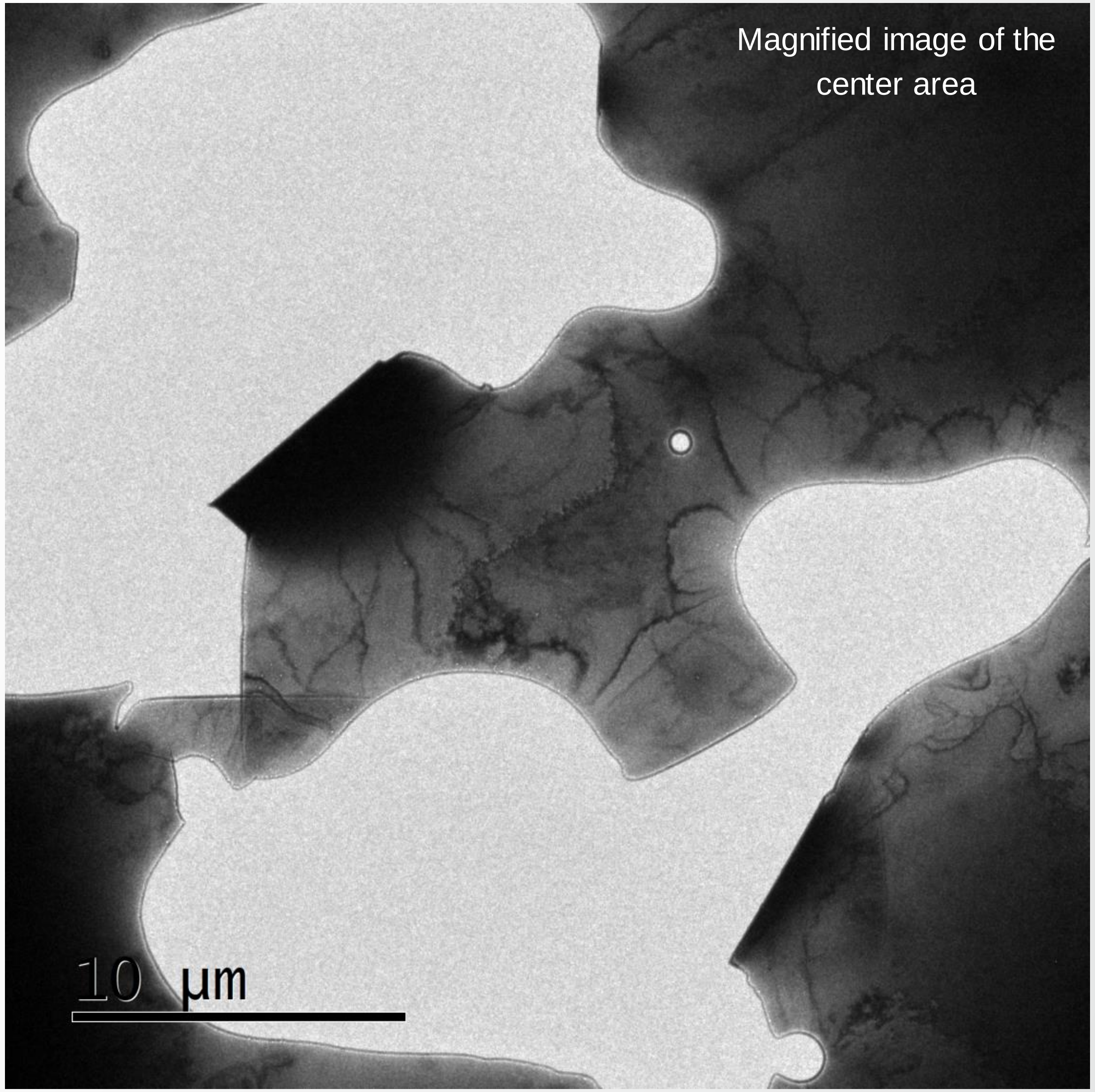




Examples

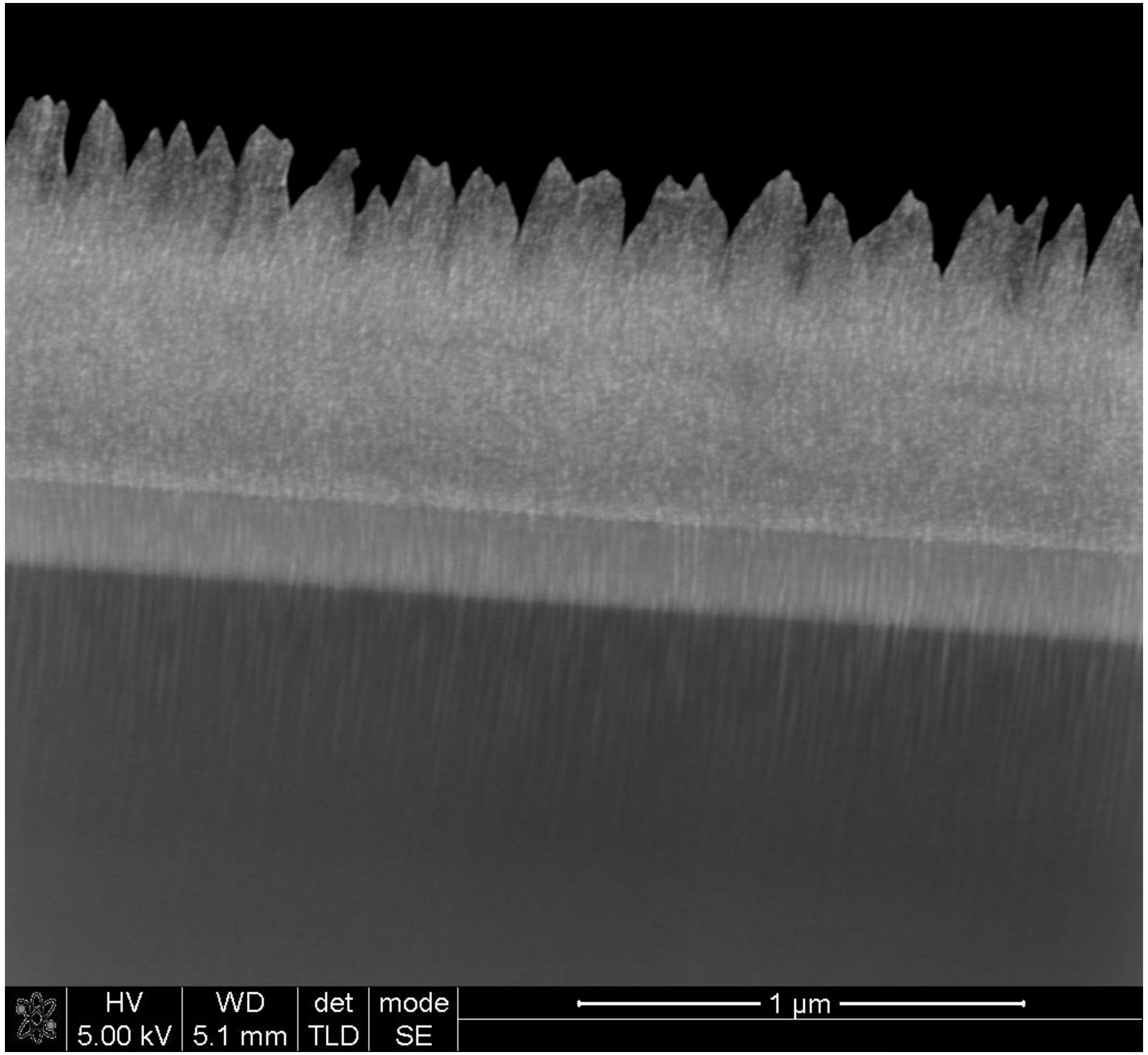
Ion Milling Sample





Examples

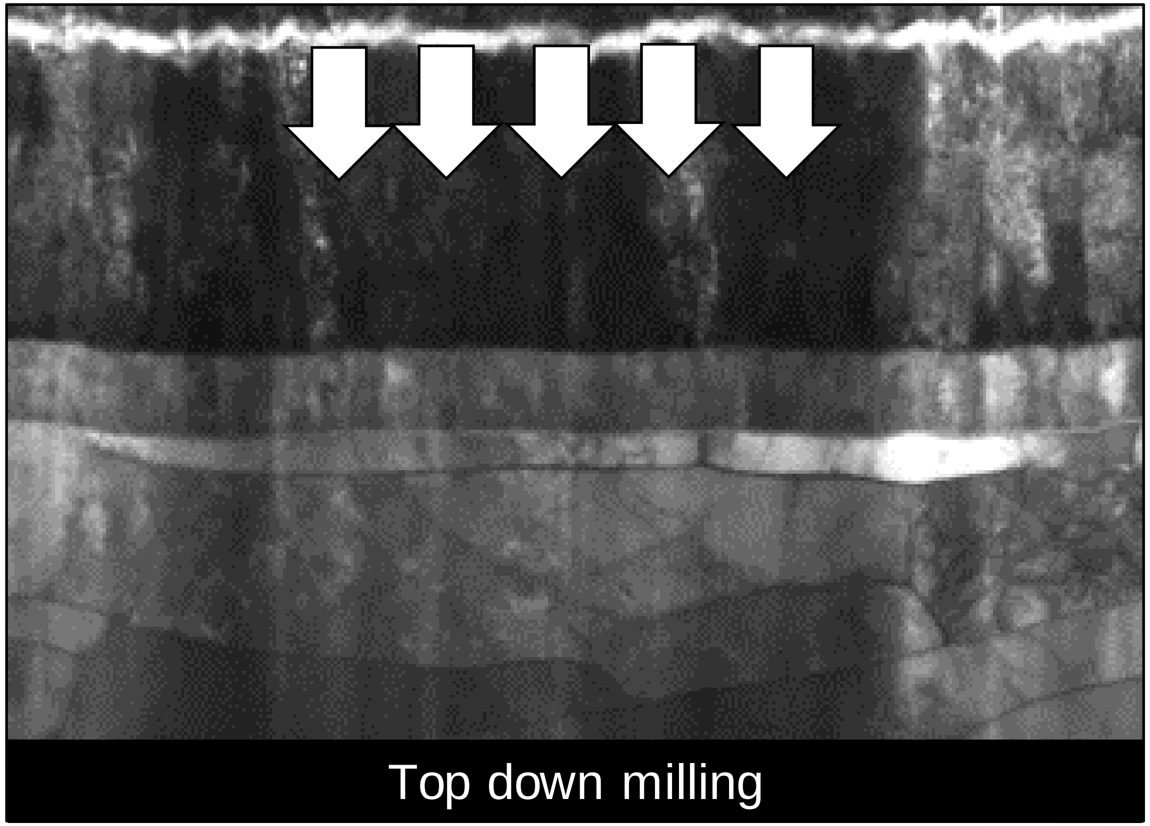
FIB Lamella, Curtaining Effect

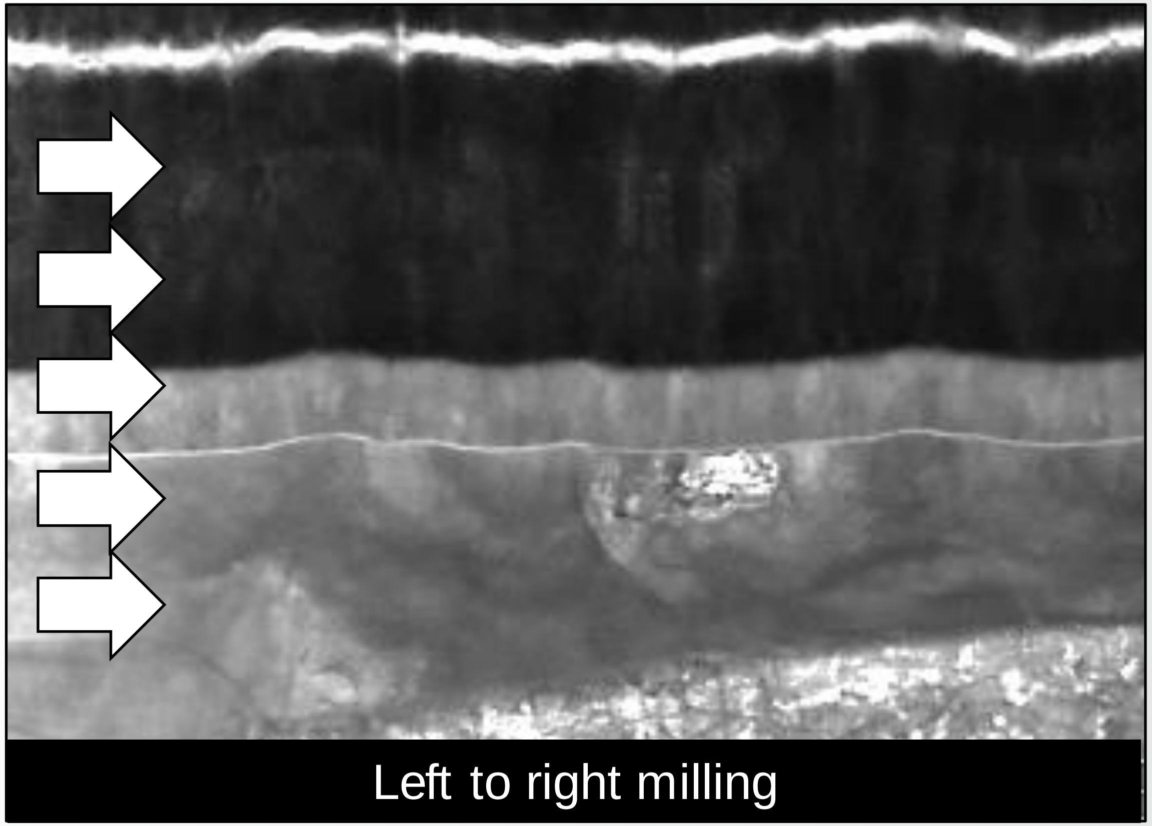


- As seen in the image to the left, curtaining artifacts induced by the granular Pt microstructure can obscure the features below and produce less than optimal images

Examples

FIB Lamella, Curtaining Effect: Change Milling Orientation: Easy Lift



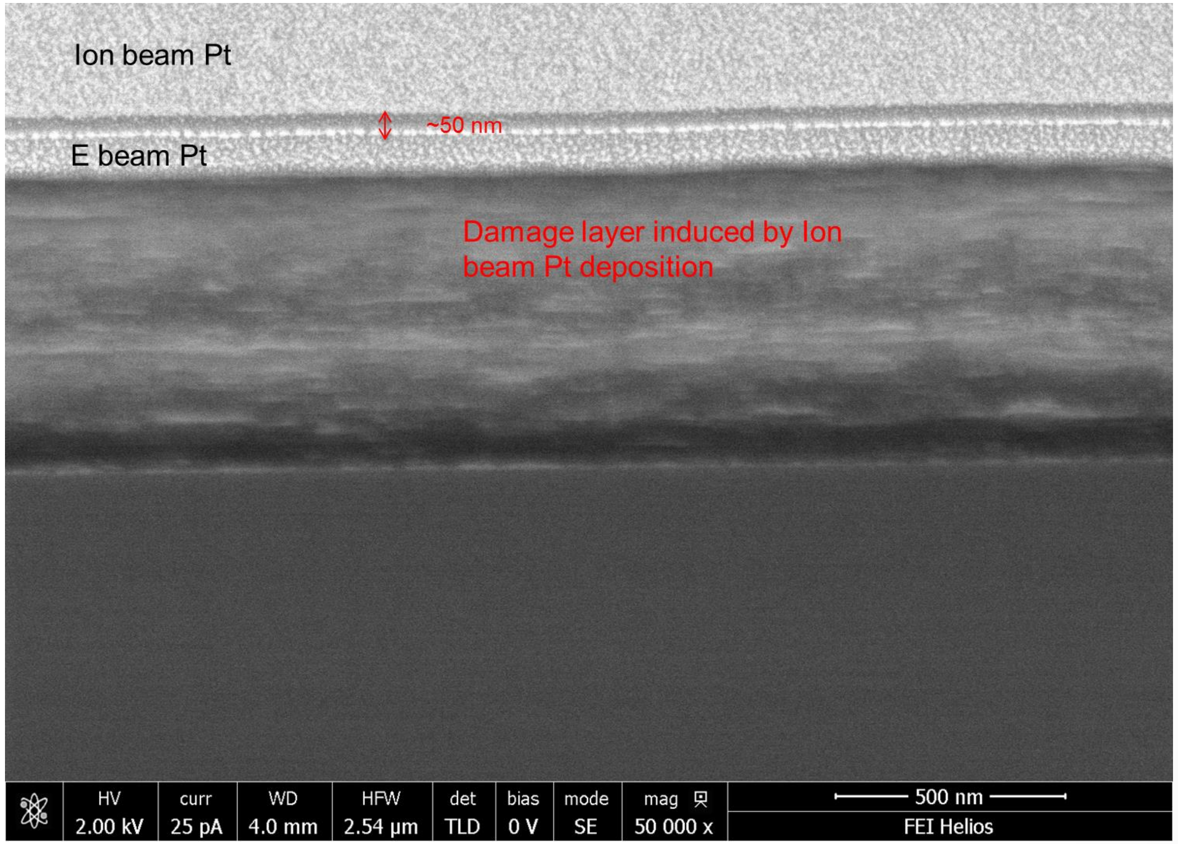


Change of orientation results in:

- More uniform lamella's without curtaining
- Avoiding smearing effects
- Preserve interfaces for measuring critical dimensions

Examples

Damage Layer Induced by Ion Beam

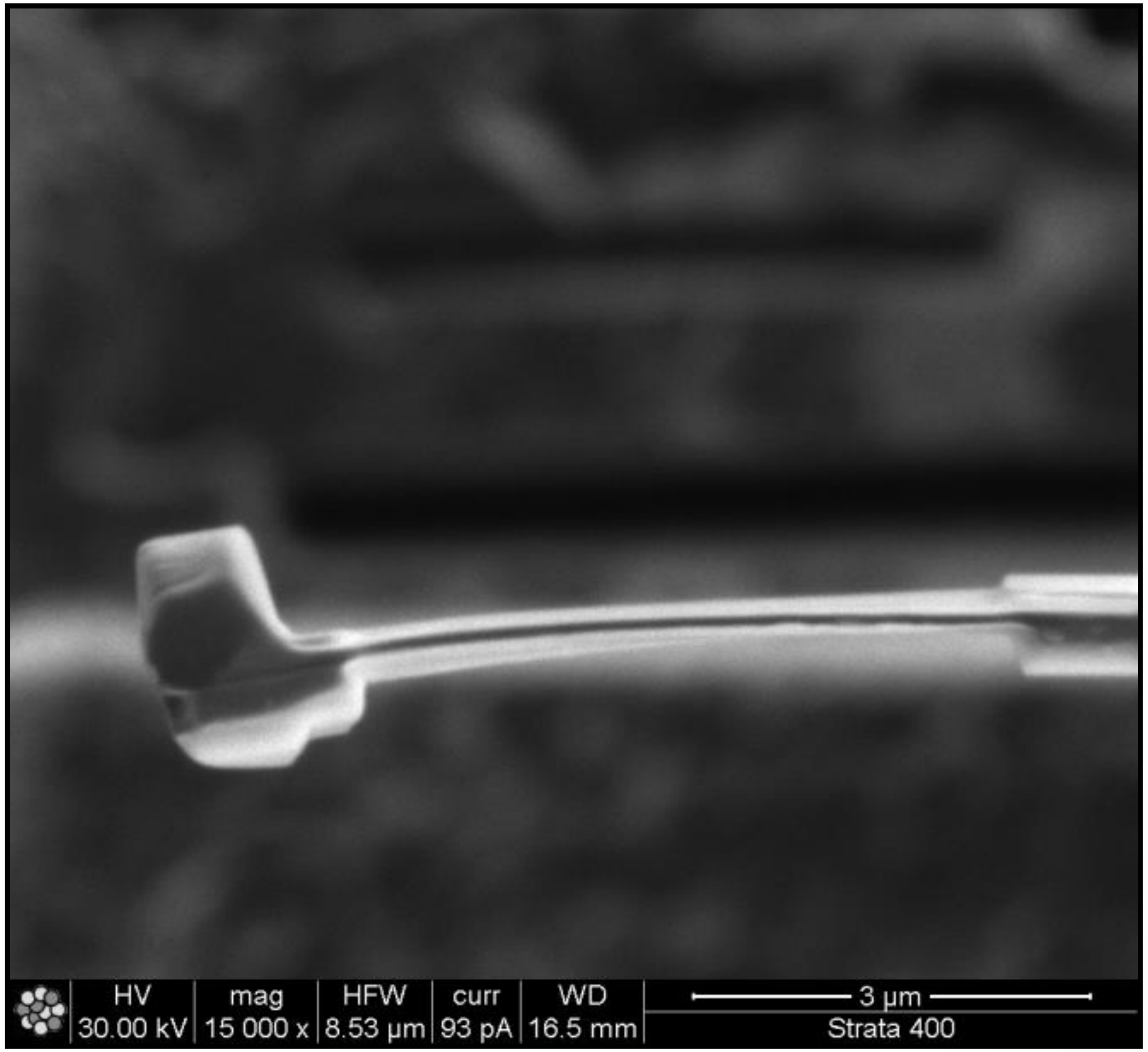


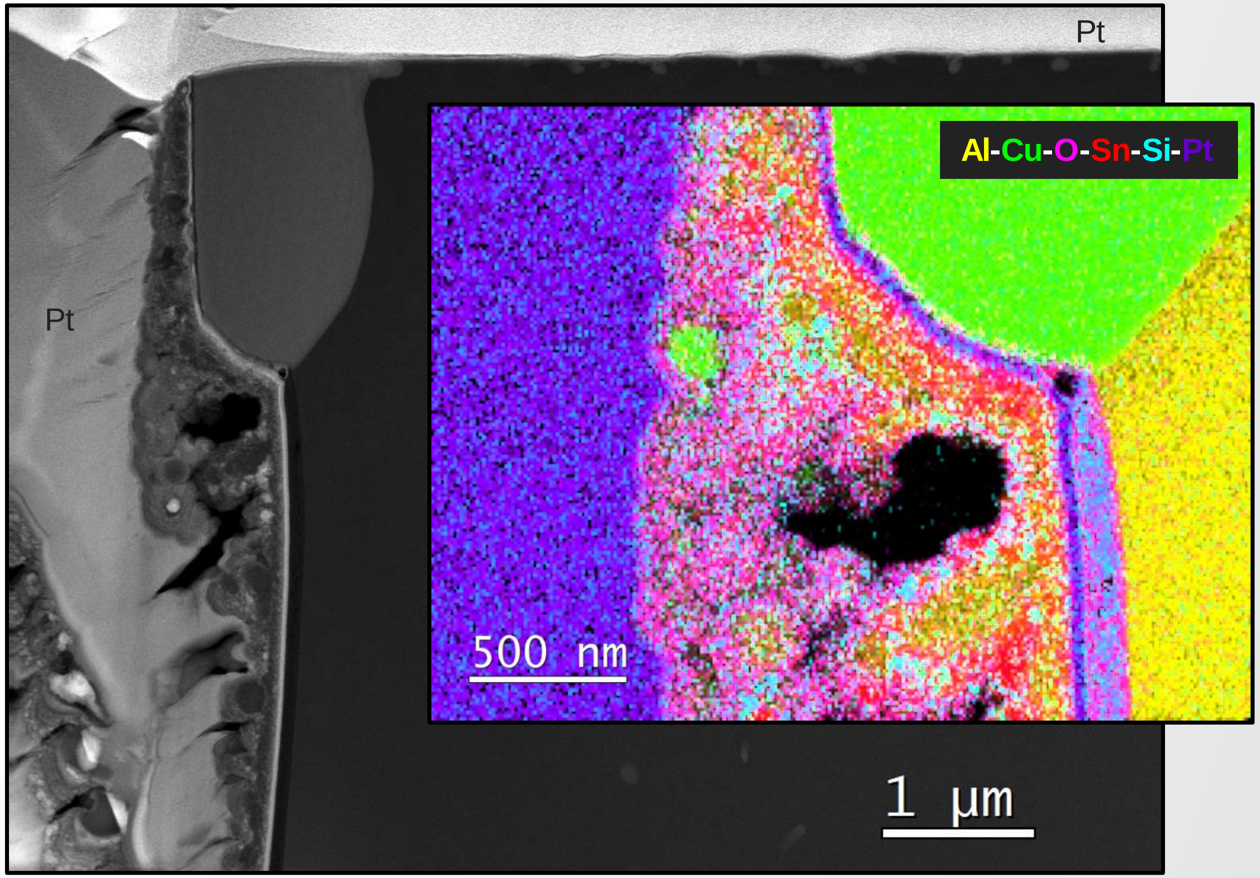
- Electron beam deposition is recommended when the top surface of the bulk sample is of interest
  - Deposition using the ion beam perpendicular to the sample surface will cause a layer of damage (~ 50nm depending on ion beam-material interactions) at the beginning of the deposition process

 mohamadriza.iskandar@thermofisher.com

Examples

Sample Bending and Pt inclusion

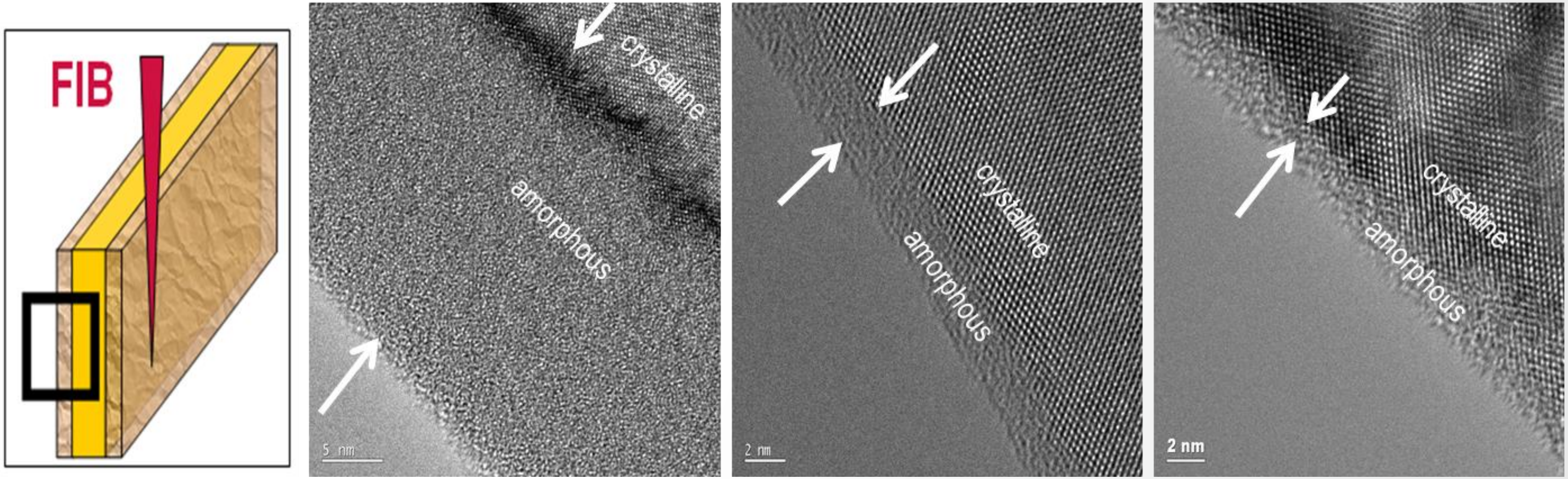




Examples

Amorphous Layer: Low kV Polishing

- ~ 20-25 nm of amorphous damage can be induced by the 30 kV ion milling on both sides of the lamella



30 kV
~ 21 nm each side

5 kV
~ 2 nm each side

2 kV
~ 0.5 – 1.5 nm

With Cs corrected TF20 – Giannuzzi et al. M&M 2005

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Thank you