COVALENT METROLOGY

Welcome

covalentmetrology.com

SAMPLE PREPARATION ORIENTATION AND ITS VALUE FOR TRANSMISSION ELECTRON MICROSCOPY (TEM)

SPEAKER:

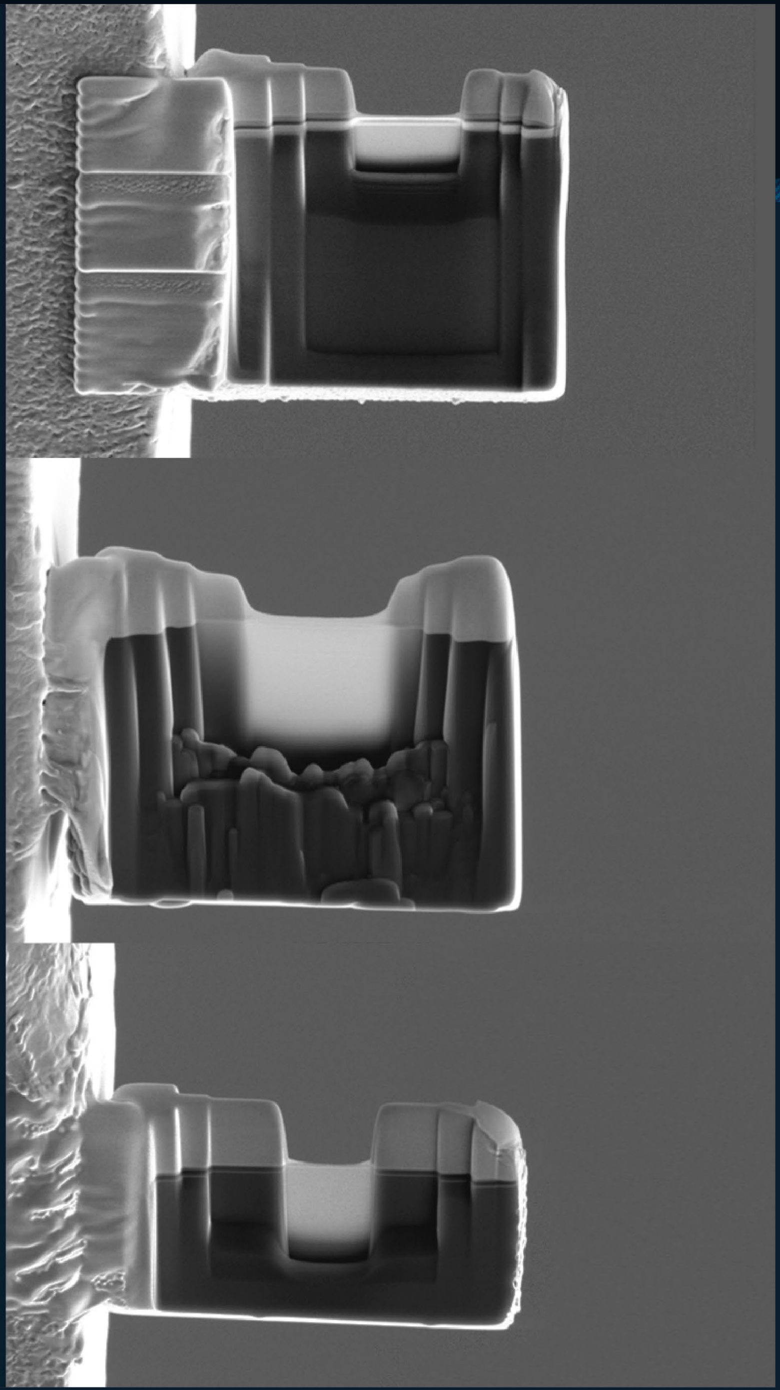
Ryan Dudschus

Senior Metrology Engineer,
Covalent Metrology

July 15, 2021 | 11AM PT

COVALENT ACADEMY

Episode 24



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

50+ cutting-edge instruments, offering 100+ Techniques

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Method Development

Temp. Staffing Solutions

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Training and Certification on Instrumentation

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Expanding Instrumentation, Lab Connections and Learning

Who We Are, Who We Serve

500 Clients + 40-60 New Clients/ Quarter

40 People, 13 PhDs

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Lab Locations: Sunnyvale, CA

Covalent Analytical Service Categories

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PCBA, Semiconductor, and Electronic Device Metrology & Failure Analysis

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

Electron Microscopy and Scanning Probe Microscopy

- AFM & Advanced AFM Modes (EFM, KPFM, MFM, PFM)
- Scanning Acoustic Microscopy (SAM)
- SEM (+ EDS)
- FIB-SEM (+ EDS)
- S/TEM (+ EDS / + EELS)
- **Nano-indent / Nano-scratch**

Optical Microscopy & Spectroscopy

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

X-Ray Characterization

- X-Ray Diffraction (XRD)
- X-Ray Reflectometry (XRR)
- Micron-spot ED-XRF
- WDXRF
- Micro-computed X-ray Tomography (Micro-CT)
- 2D X-ray Inspection & X-ray Radiography

Elemental / Chemical Composition Analysis

- EPMA
- **GD-OES**
- GC-MS
- **ICP-MS and LA-ICP-MS**
- **Raman Microscopy & Spectroscopy**
- NMR (1D or 2D; solid / liquid)

Particle Analysis

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

Material Property Characterization

- DSC
- DMA & TMA
- Rheometry
- TGA
- Surface Zeta Potential

Coming Soon:

- *Porometry / Porosity*
- *Gas Adsorption*
- *Gas Pycnometry*
- *Foam Density*
- *Tap Density*

Surface Spectroscopy Analysis

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

Ryan Dudschus, PhD

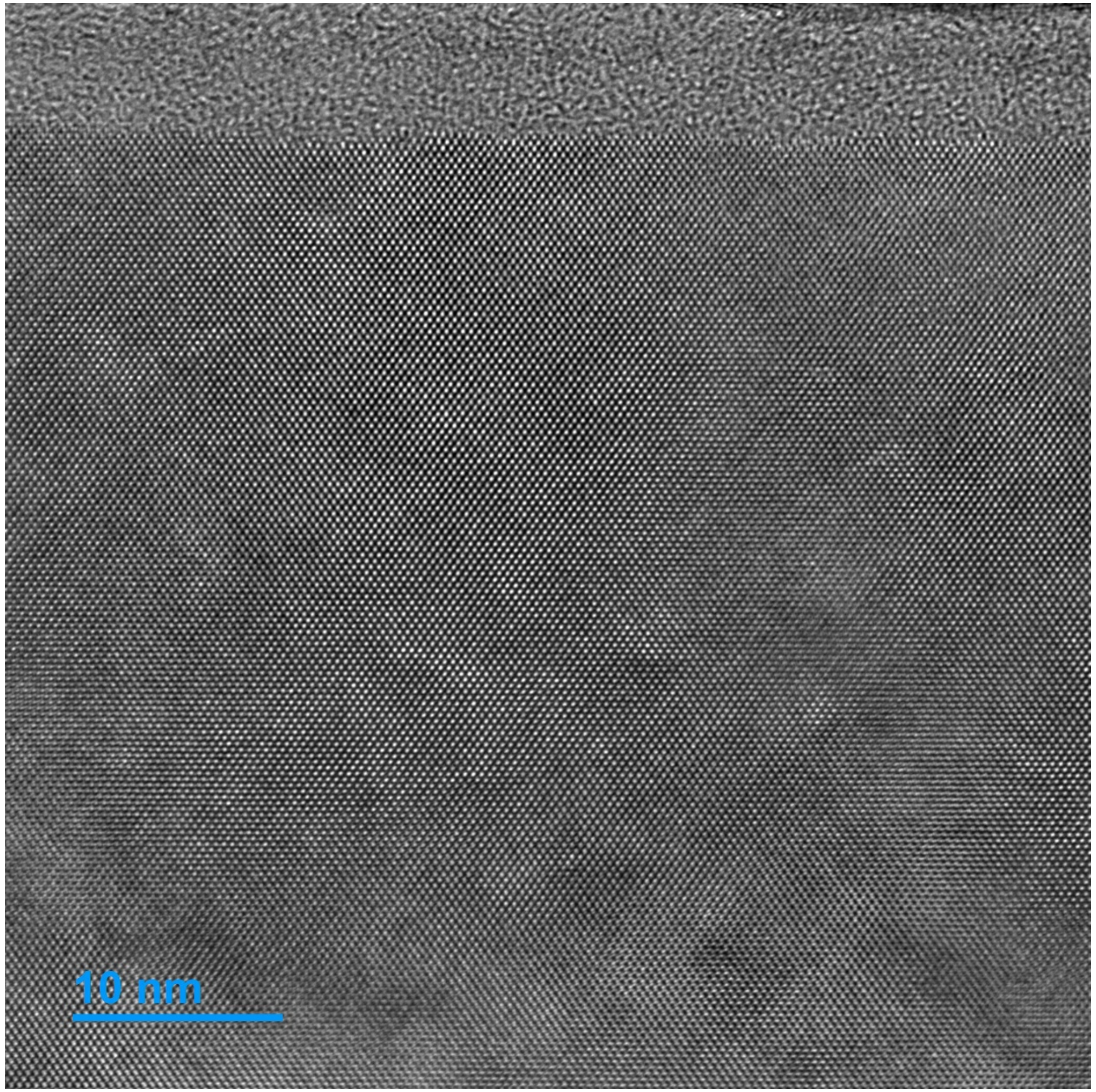
Senior Metrology Engineer, Lamella Preparation
Covalent Metrology

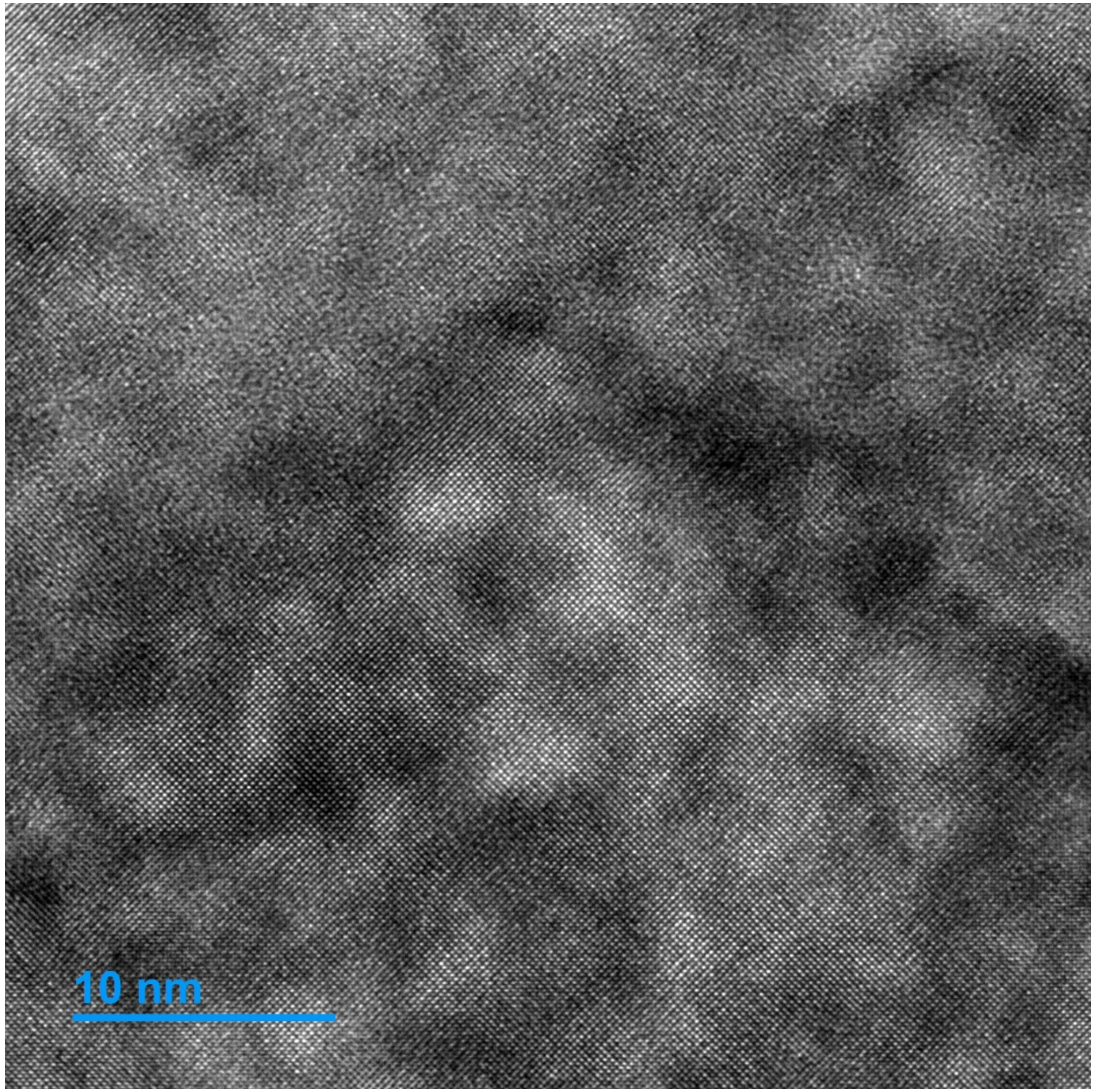
- Senior metrology engineer working primarily in Electron and Scanning Probe Microscopy groups
  - Using mainly SEM and AFM techniques
- He earned his B.S. and M.S. in Physics from UC Santa Cruz
  - Research focused in condensed matter
- Ryan's M.S. research used the Stanford Synchrotron Radiation Lightsource (SSRL) for Extended X-ray Absorption Fine Structure (EXAFS) data collection (instead of an SEM, XRD apparatus, or other standard measurement device)

Contents

Sample Prep. Correlation to Quality of TEM Image

Sample preparation accounts for ~90% of the TEM image quality…





Dualbeam Fundamentals for TEM Sample Preparation

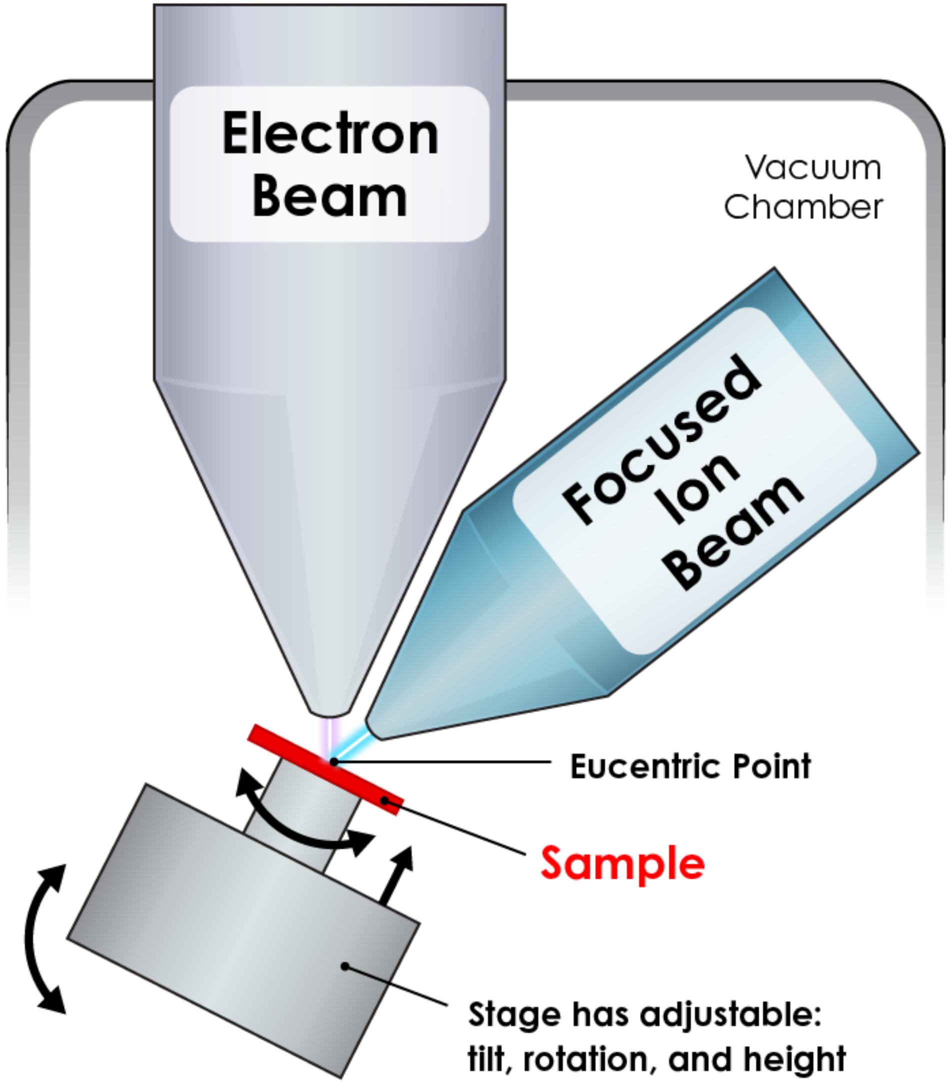
Basic DualBeam setup

SEM use cases:

- Imaging
- Depositions

FIB use cases:

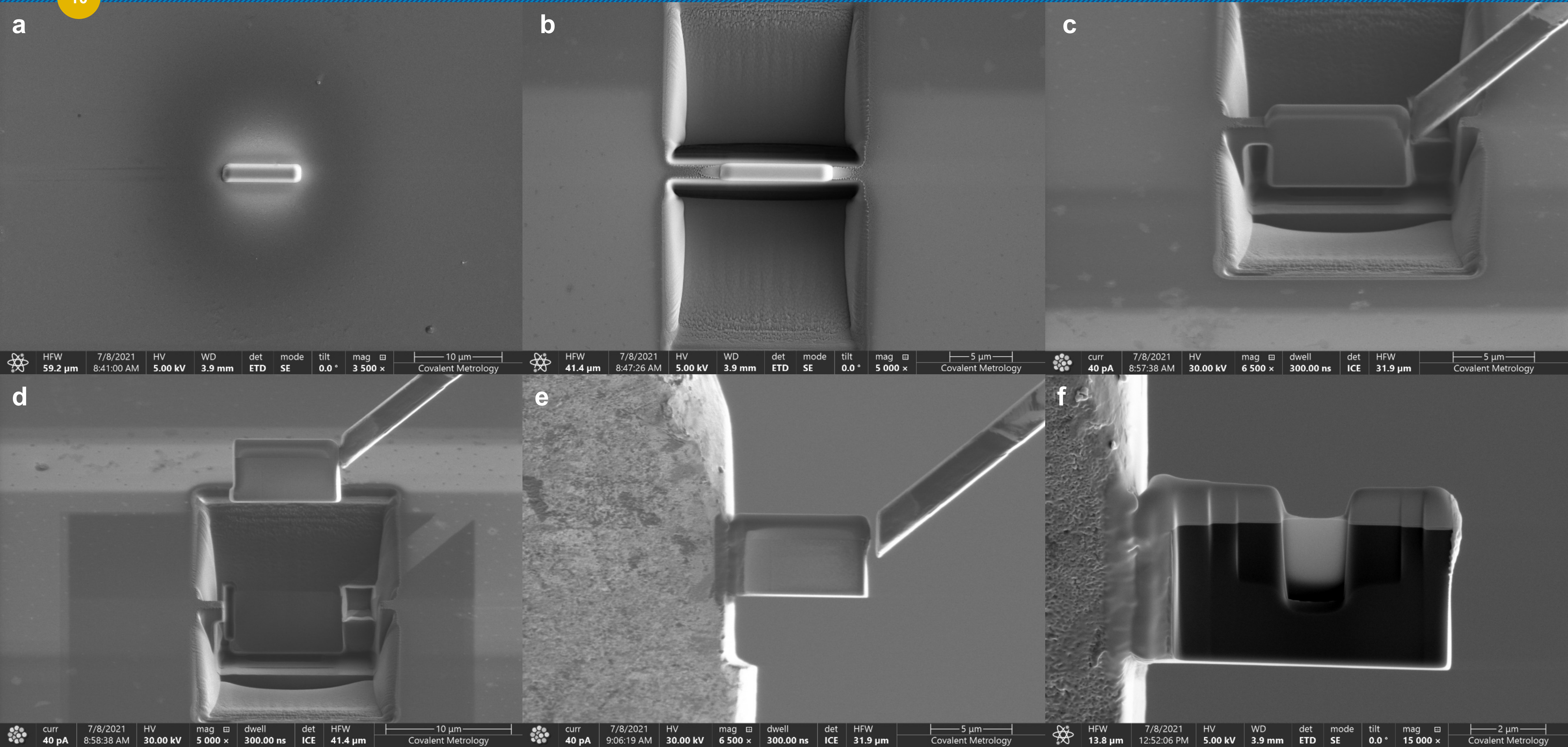
- Milling/Thinning
- Deposition



See Covalent Webinar Episode 05 – SEM/FIB Dual Beam Microscopy: A Powerful Combination for more information

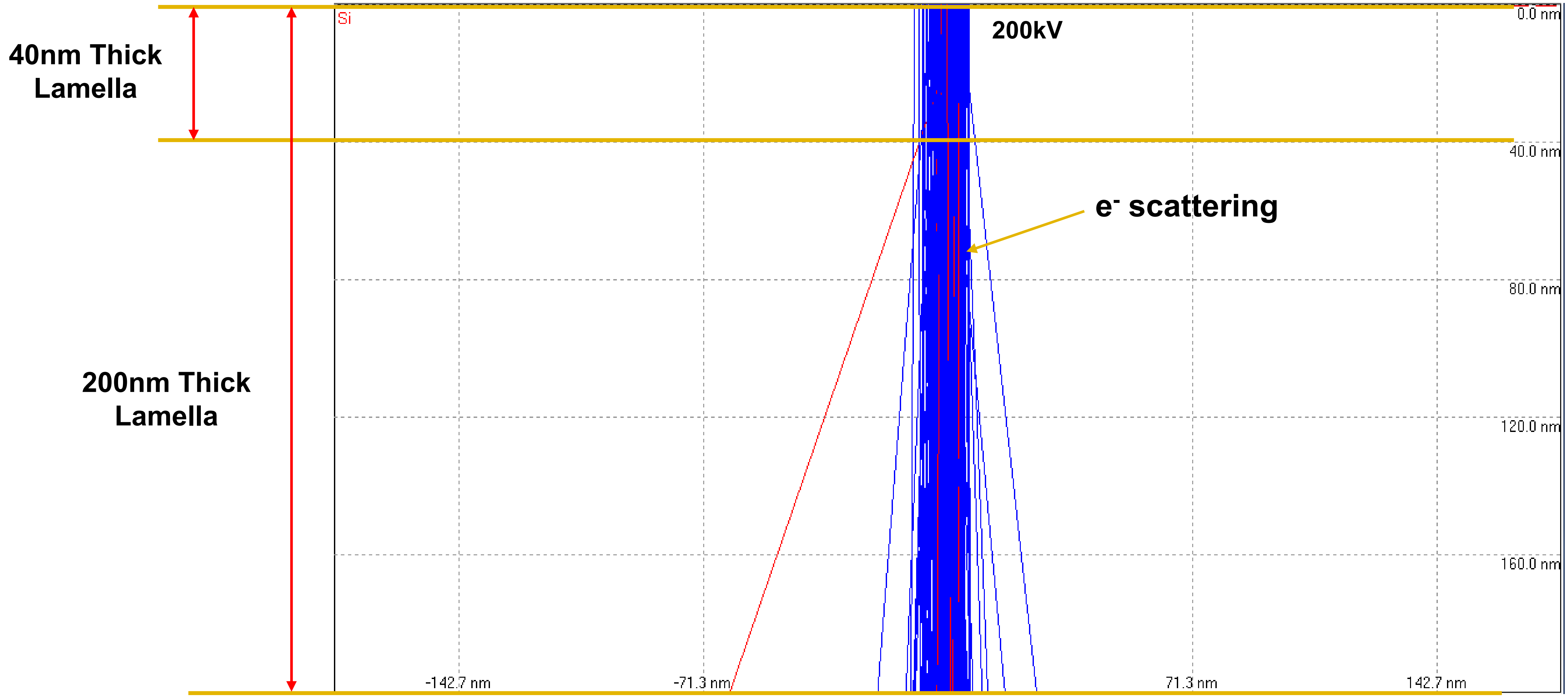
[1]

Lamella Preparation Overview – Step by Step



Electron Beam – Lamella Interaction

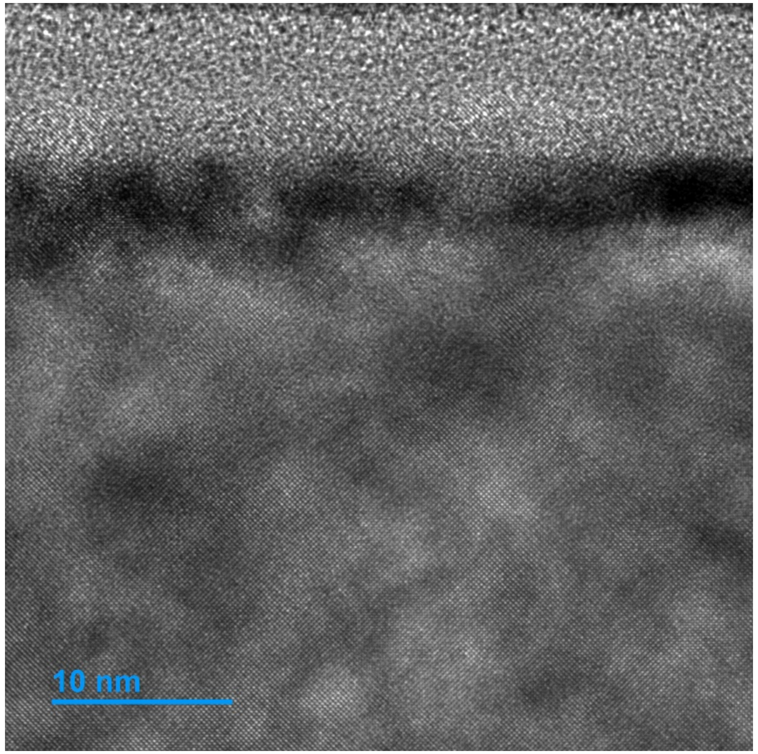
Thinner lamella provides in higher quality TEM imaging



Lamella Thickness for TEM Imaging

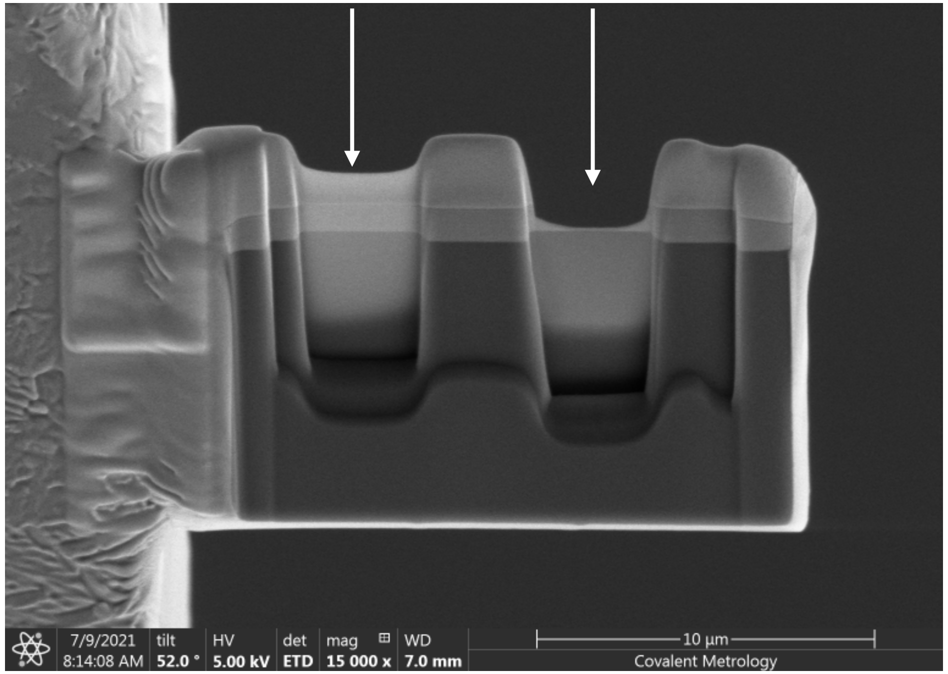
Physical example of TEM imaging between 100nm thick and 40nm thick lamella

~100 nm thick

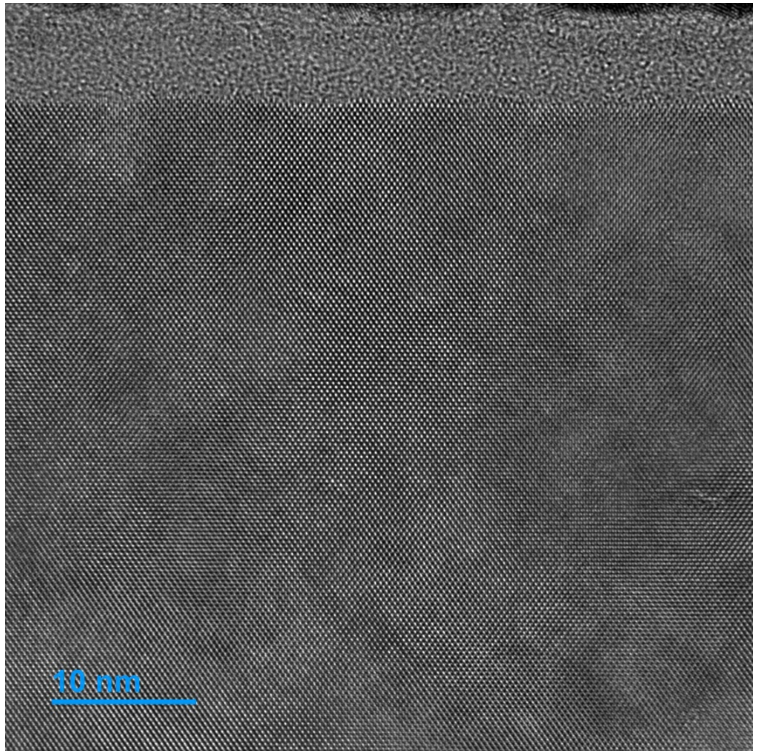


~100 nm thick

~40 nm thick



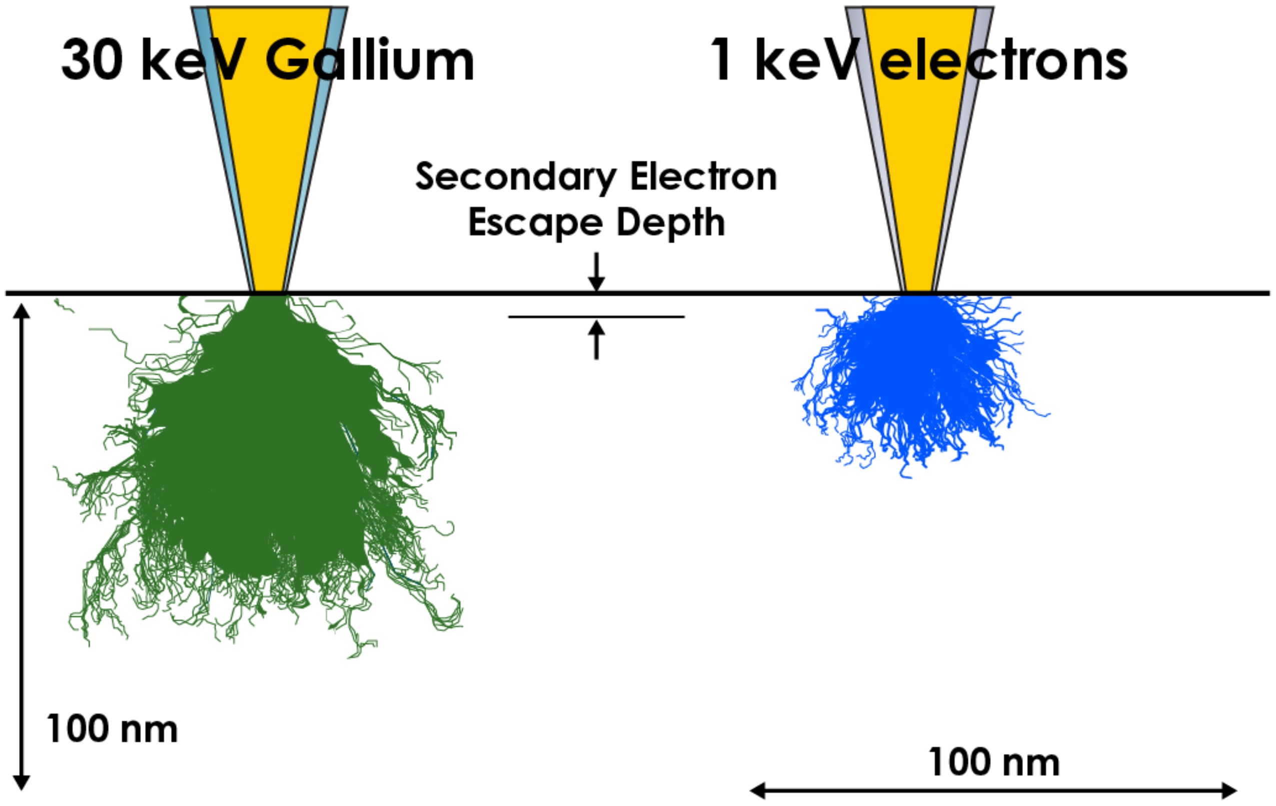
~40 nm thick



Note: the thinner the lamella, the more challenging further thinning becomes

FIB Sample Interaction

- Ga FIB has an interaction volume based on its lining energy.
- Ga ions may knock atoms off of lattice site.
- Ga ions may imbed themselves in the specimen



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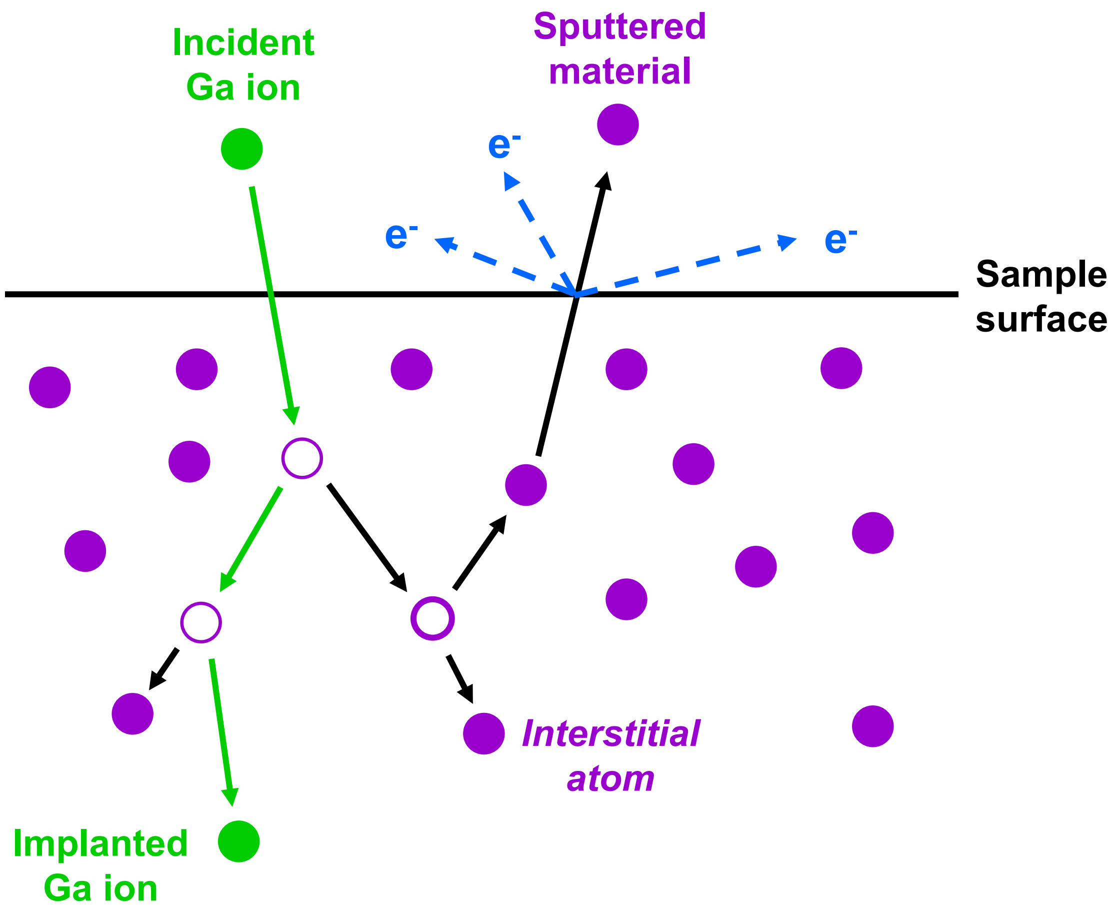
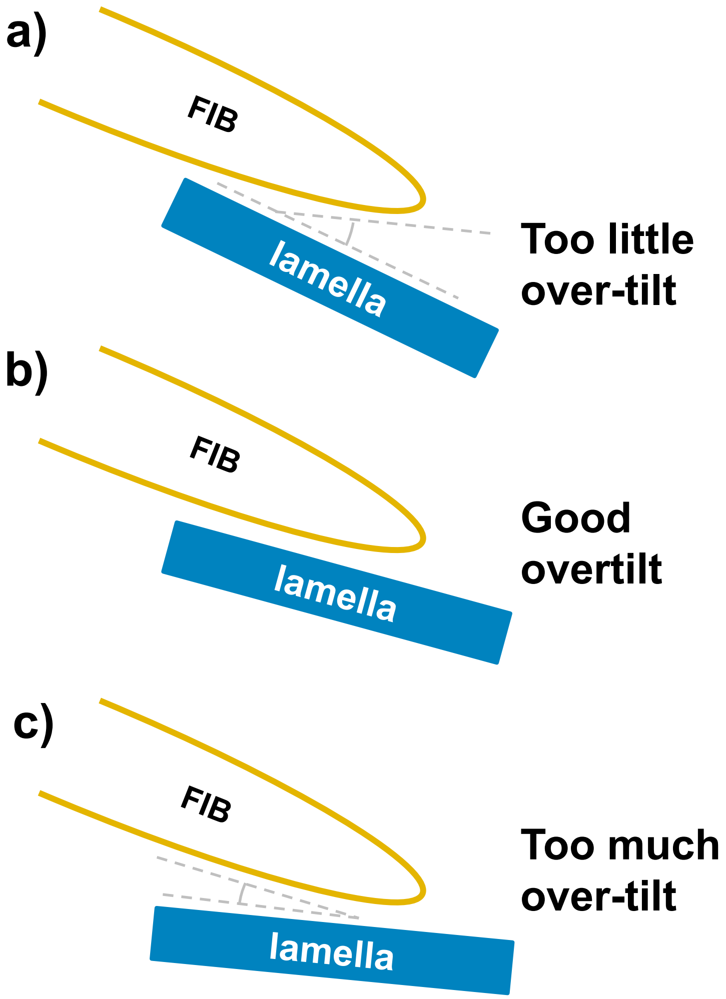
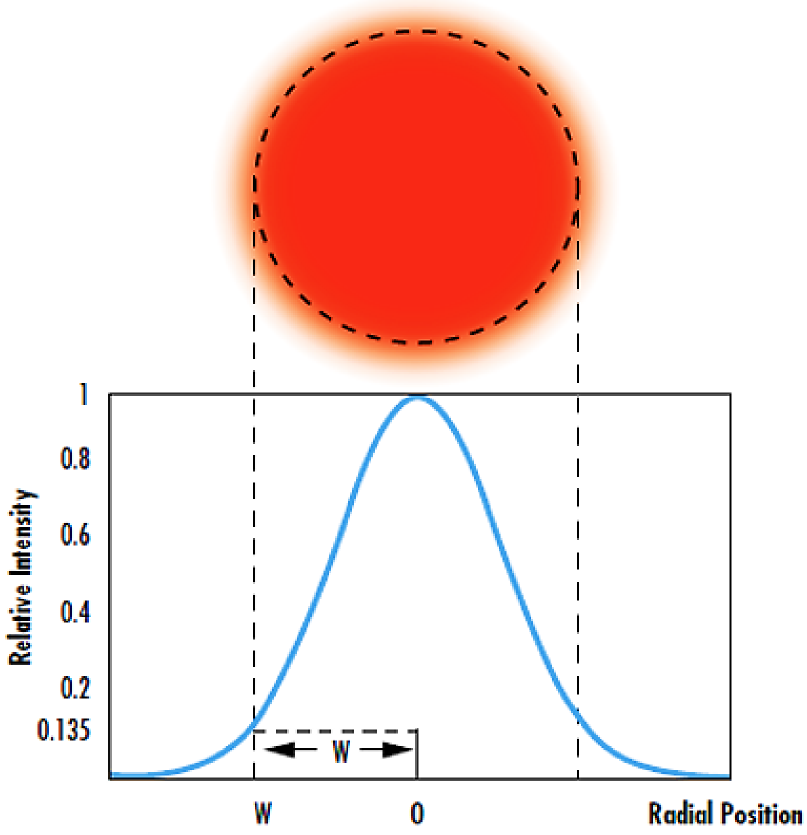


Milling Rates & Overtilt

- Ga FIB has **gaussian shape**
- Each material has own mill rate
- Over-tilt affects the shape of the lamella
- Lower currents for precise mills
- Higher currents for faster mills
- Short dwell time for uniform, accurate pattern shapes
- High dwell time for mill harder materials





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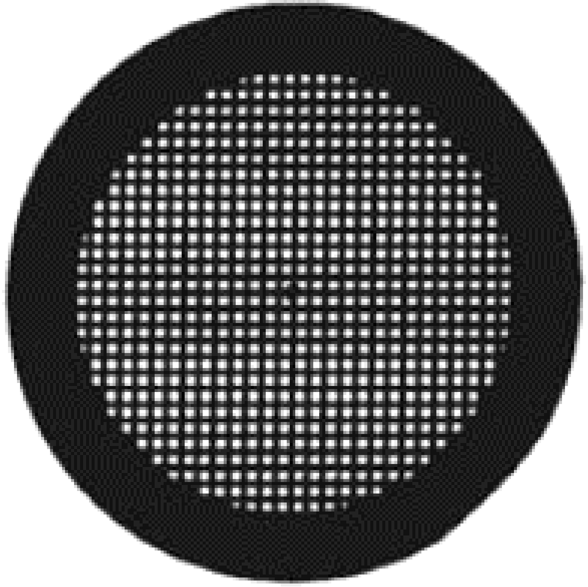
Grid Preparation and Types of Lamella Thinning

Lamella Grids

- Grid are chosen based on material composition of the specimen
- Grids could be made from various materials:
  Au, Be, C, Cu, Si, Mo, Ni

Ni – Mesh Grid

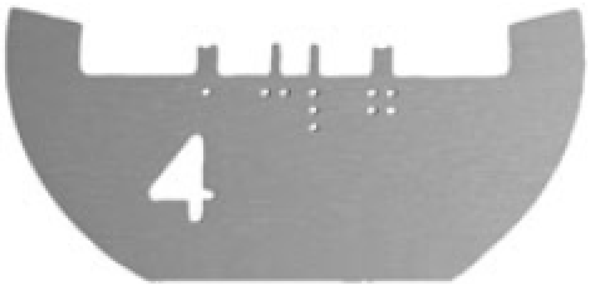


Narrow Posts

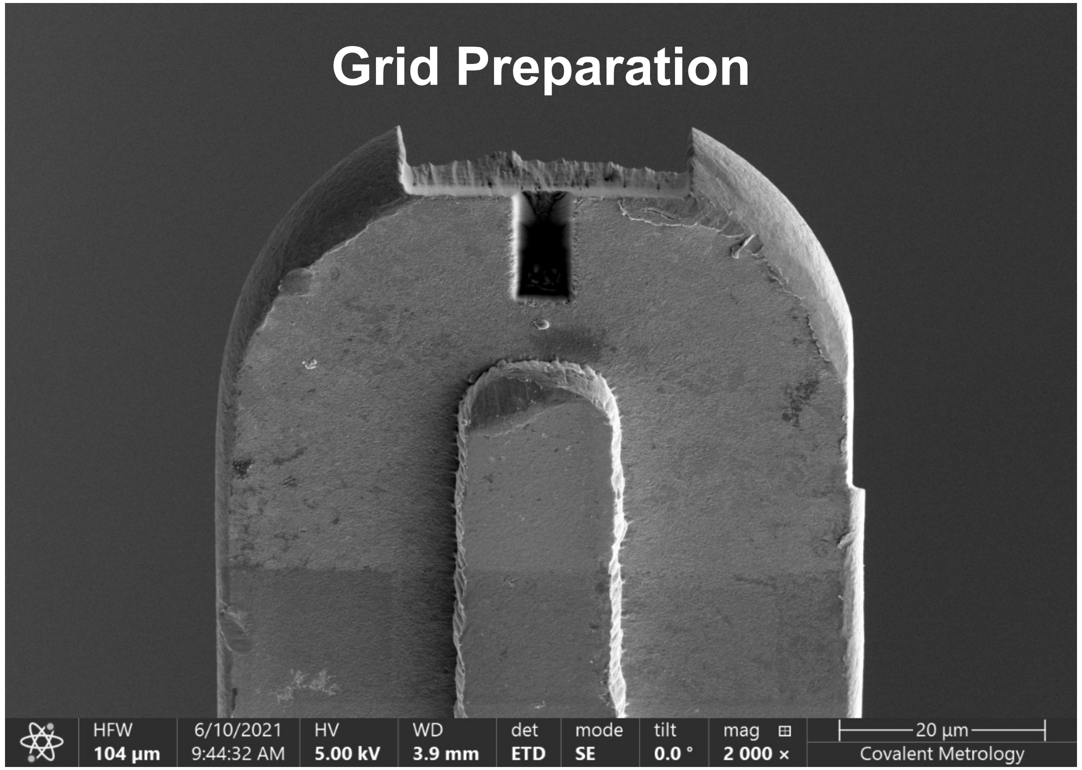
Cu

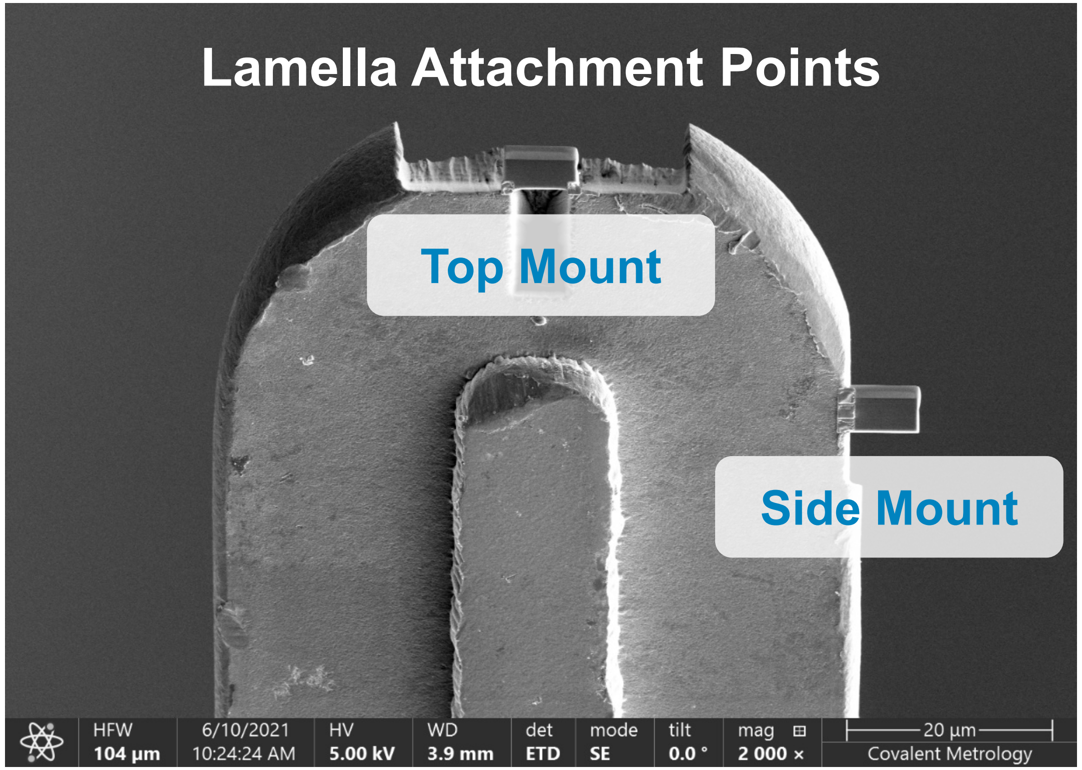
Wide Posts

Mo



Grid Preparation and Attachment Points



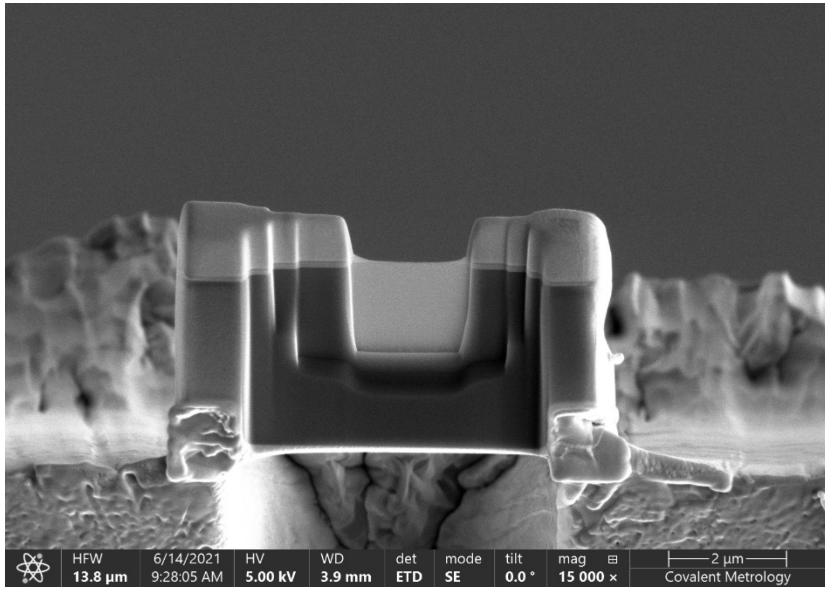


- Creates a clean surface for lamella attachment.
- Top mount "T-pit" prevents redeposition on lamella from grid.

- **Top Mount**
  - Two points of contact. Less prone to twisting (but curling still possible)
- **Side Mount**
  - Quick and more secure attachment; speeds up time to make a lamella
  - More prone to bending/twisting

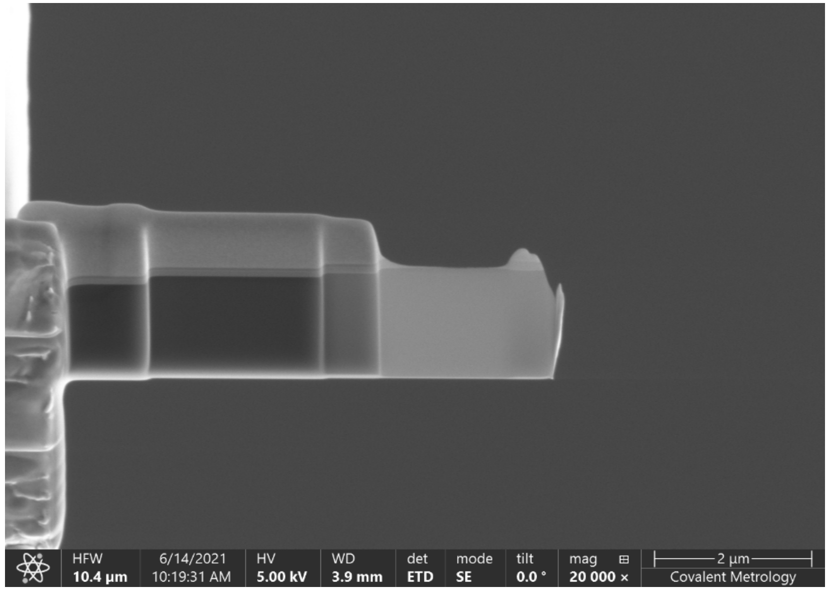
Basic Types of Thinning Lamella

Windowed



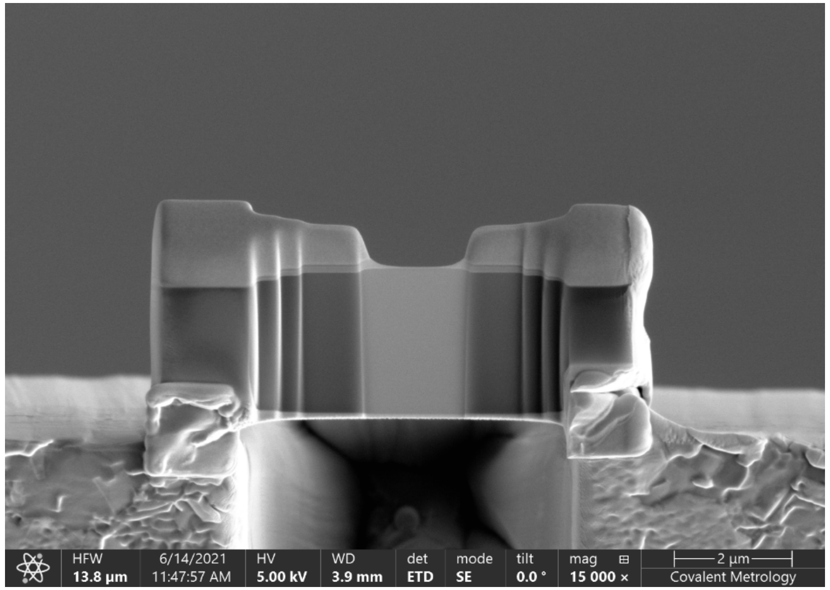
- Thick bottom helps prevent lamella bending.
- Lamella curling may still occur.

Pillar



- Good for near-surface layers/features.
- Pillar provides stabilization.
- Lamella bending and curling may still occur.

Through



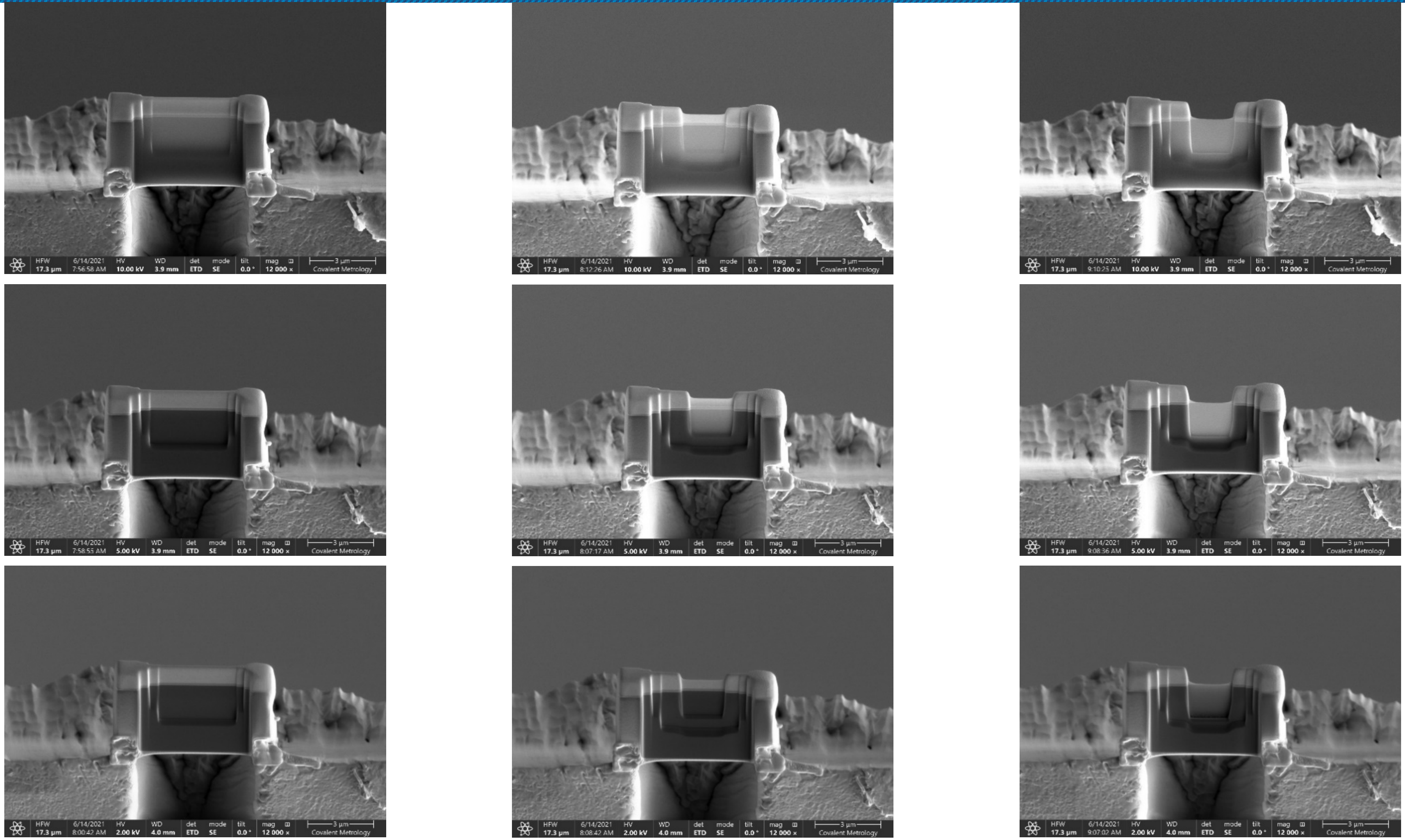
- Useful for large multi-layers/features.

Lamella – Electron Transparency

10kV

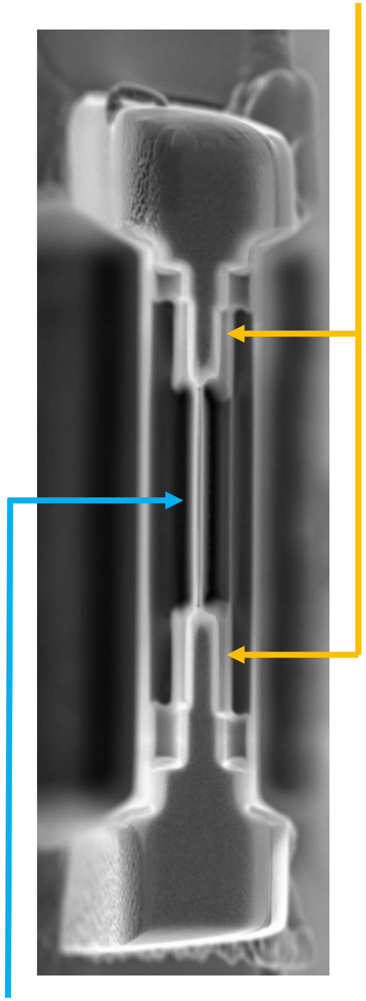
5kV

2kV



Decreasing Thickness →

10kV Transparency

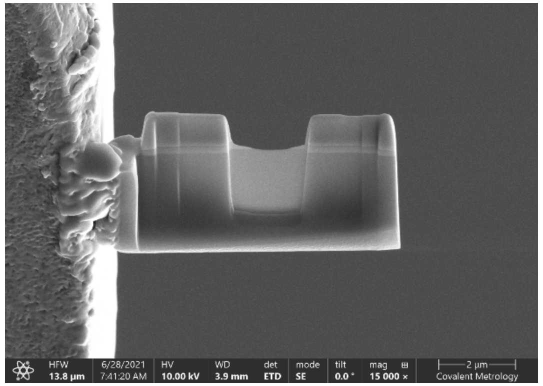


2kV Transparency

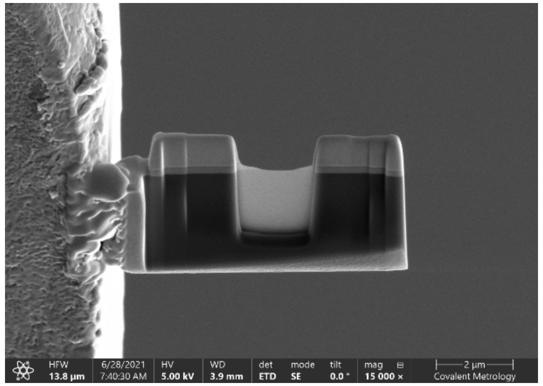
Note: electron transparency thickness is not the same for all materials

Lamella – Electron Transparency Thickness

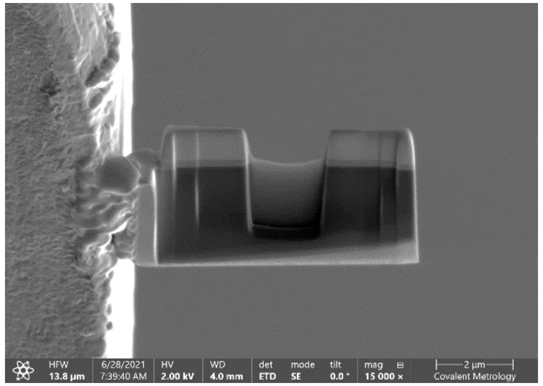
10kV

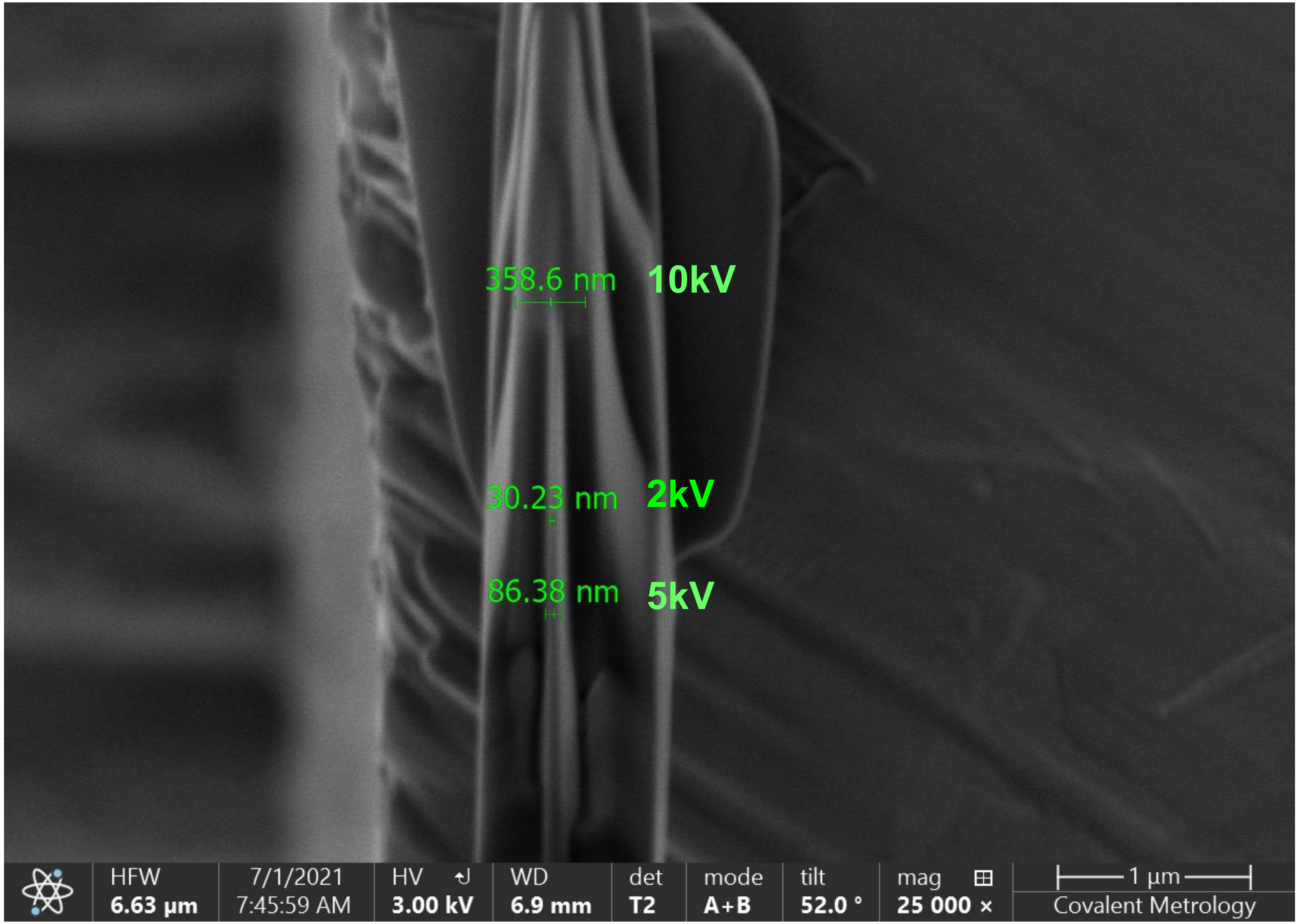


5kV



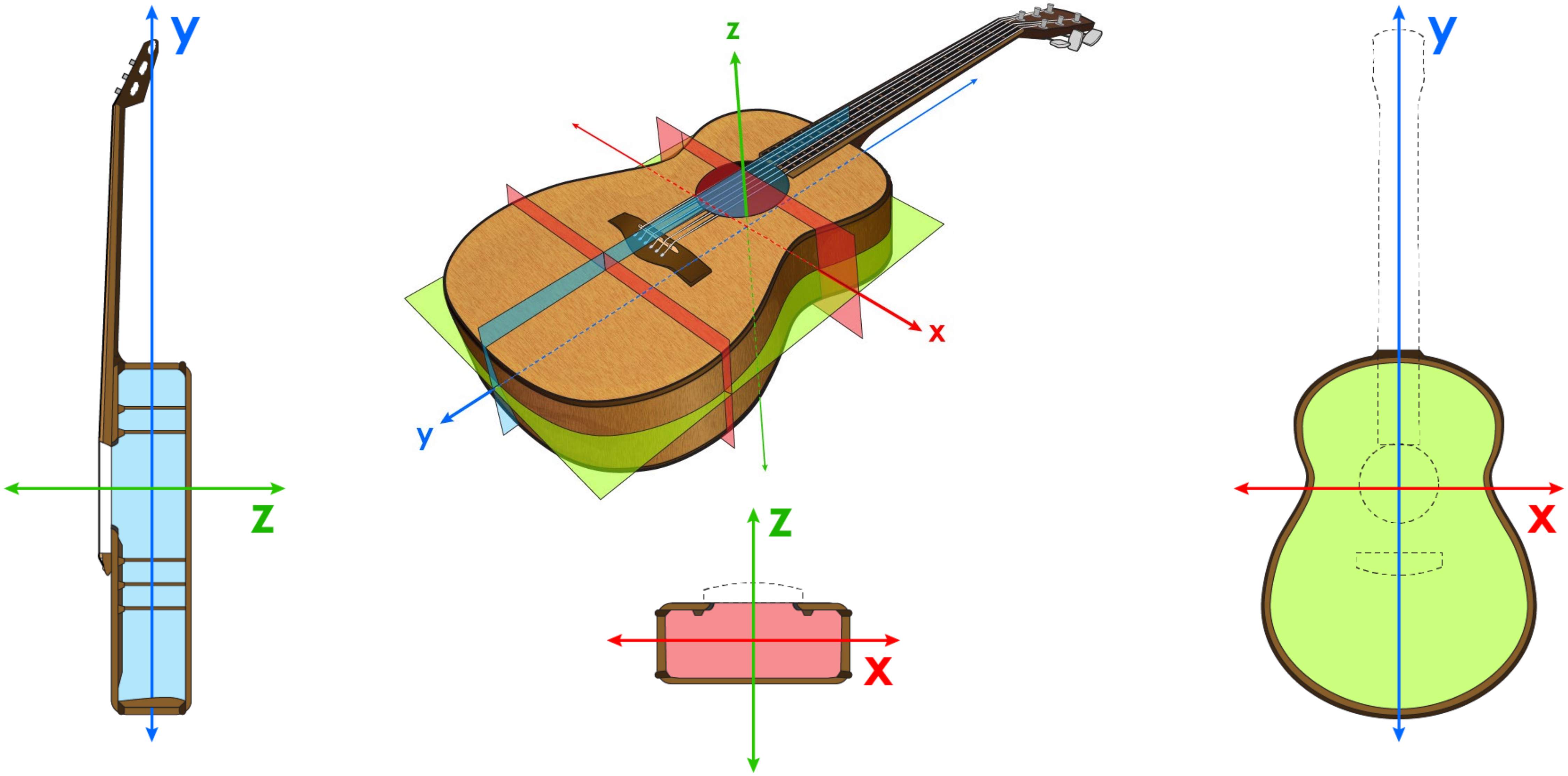
2kV



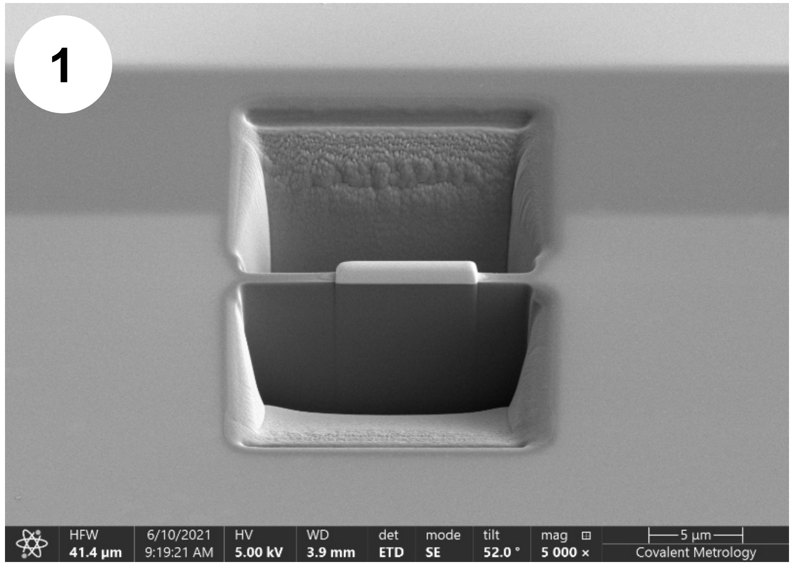


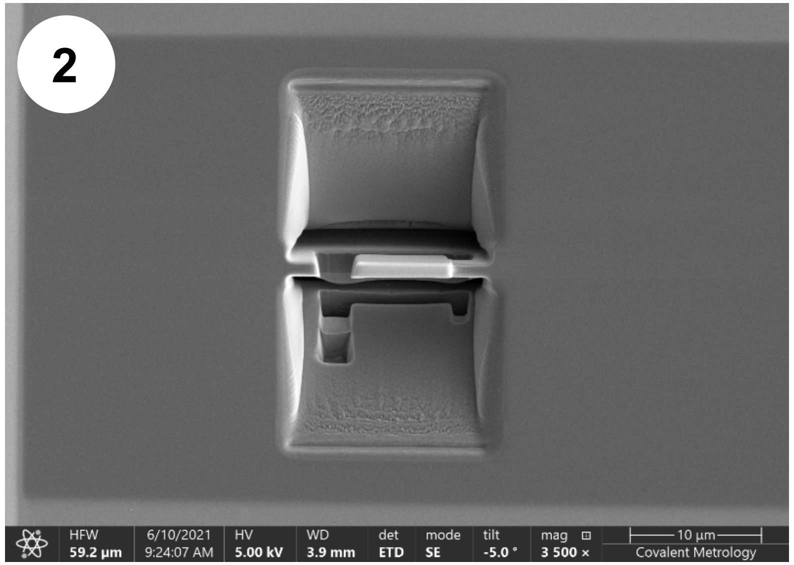
Sample Orientation and Variety of Lamella Lift-out

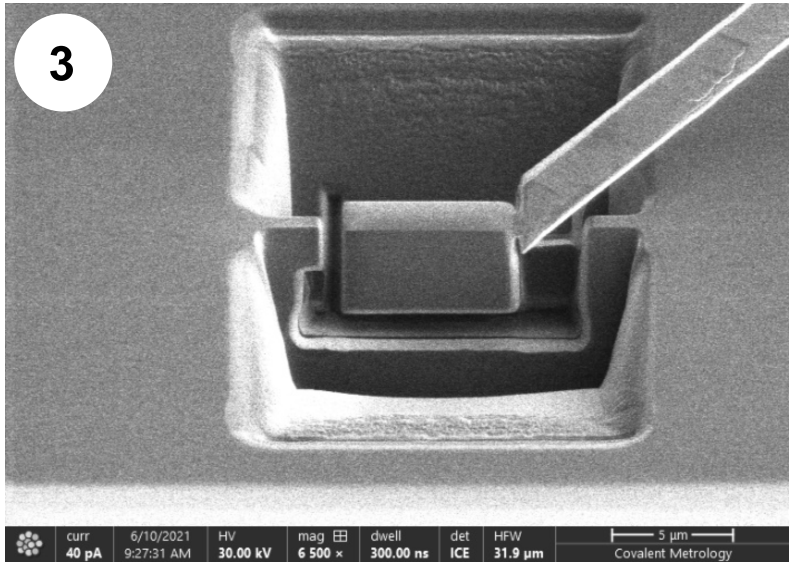
Sample Orientation Planning

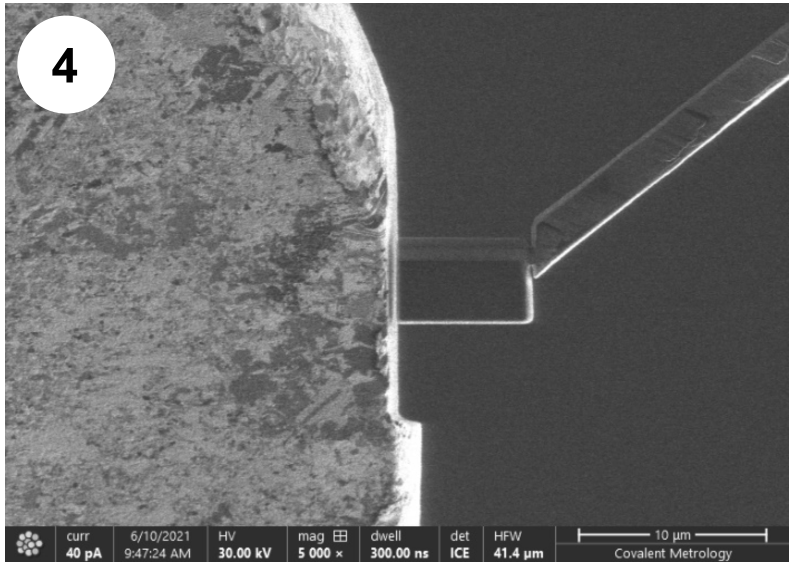


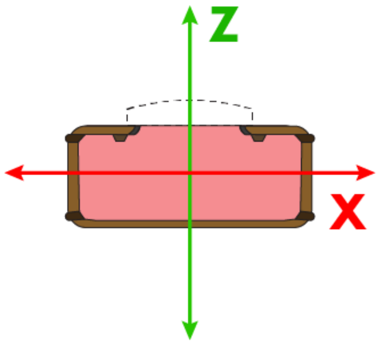
Lamella Lift-out: Top-Down – Process

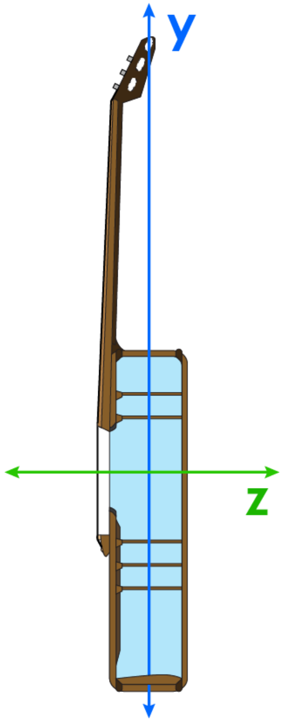




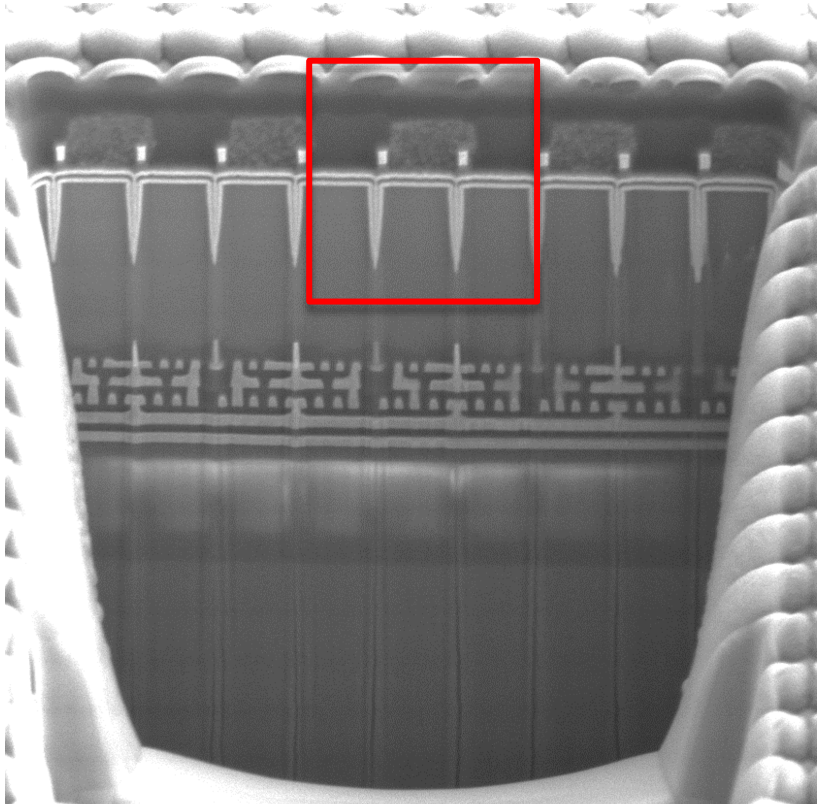




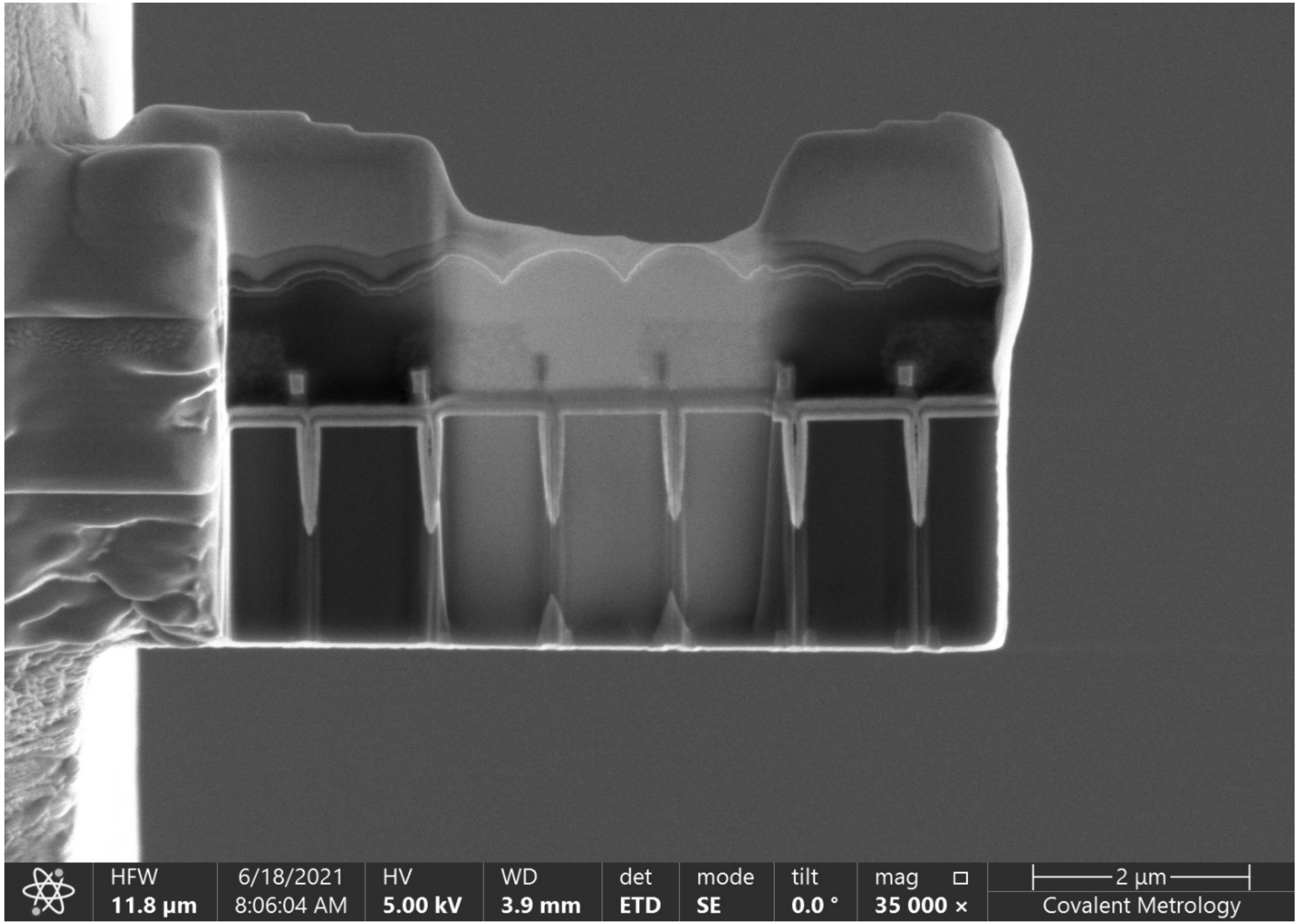




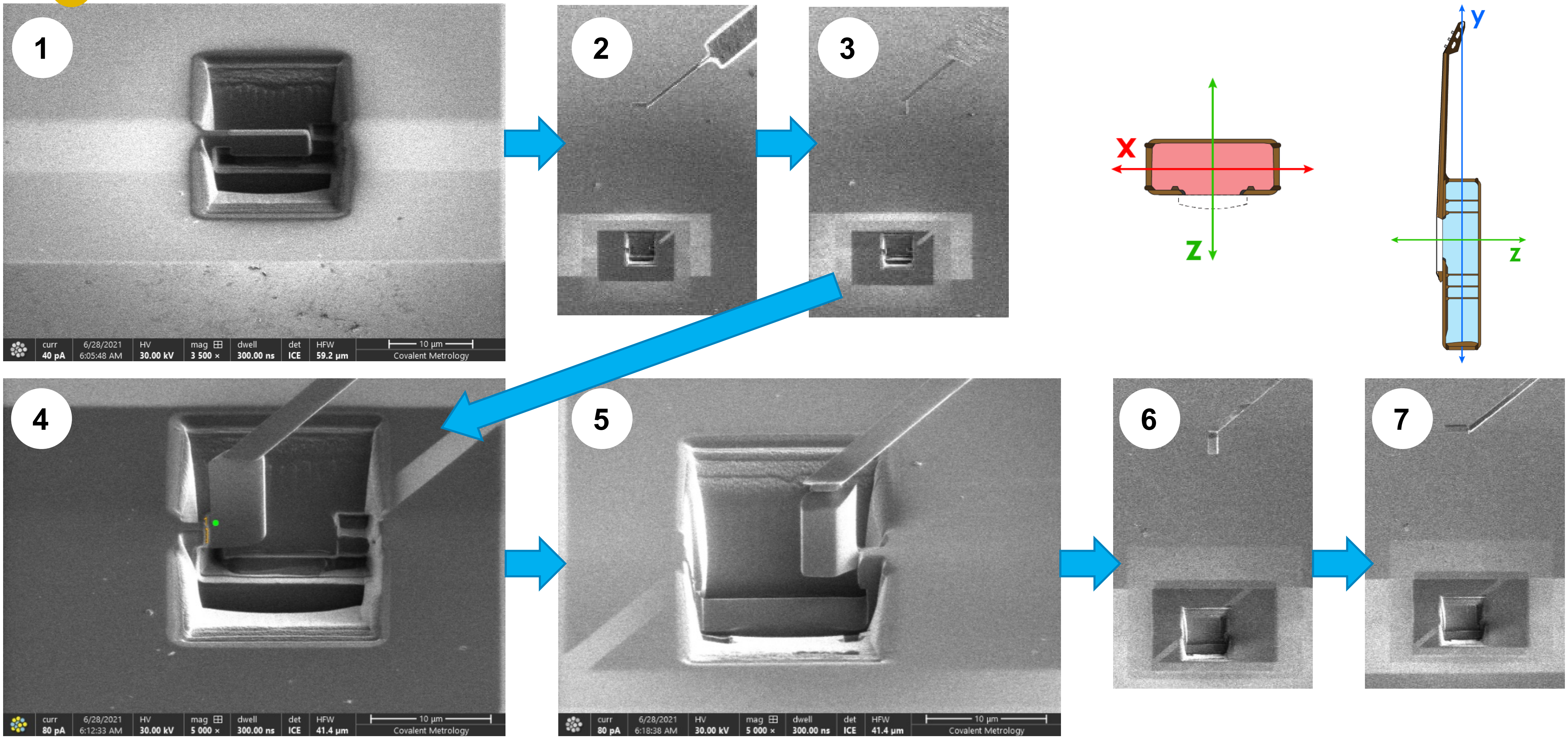
Lamella Lift-out: Top-Down



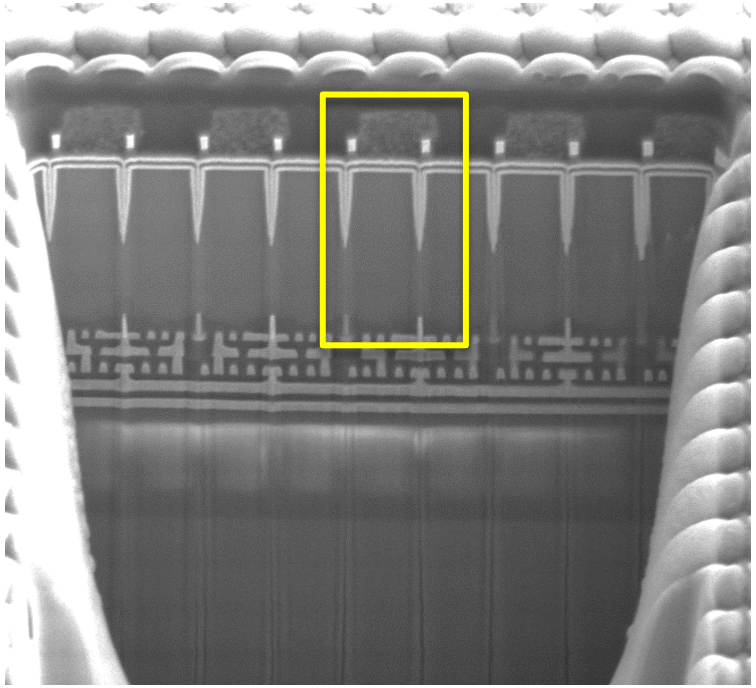
- Good for near-surface features
- Smooth top surfaces are ideal
- Most common prep technique



Lamella Lift-out: Inverted – Process

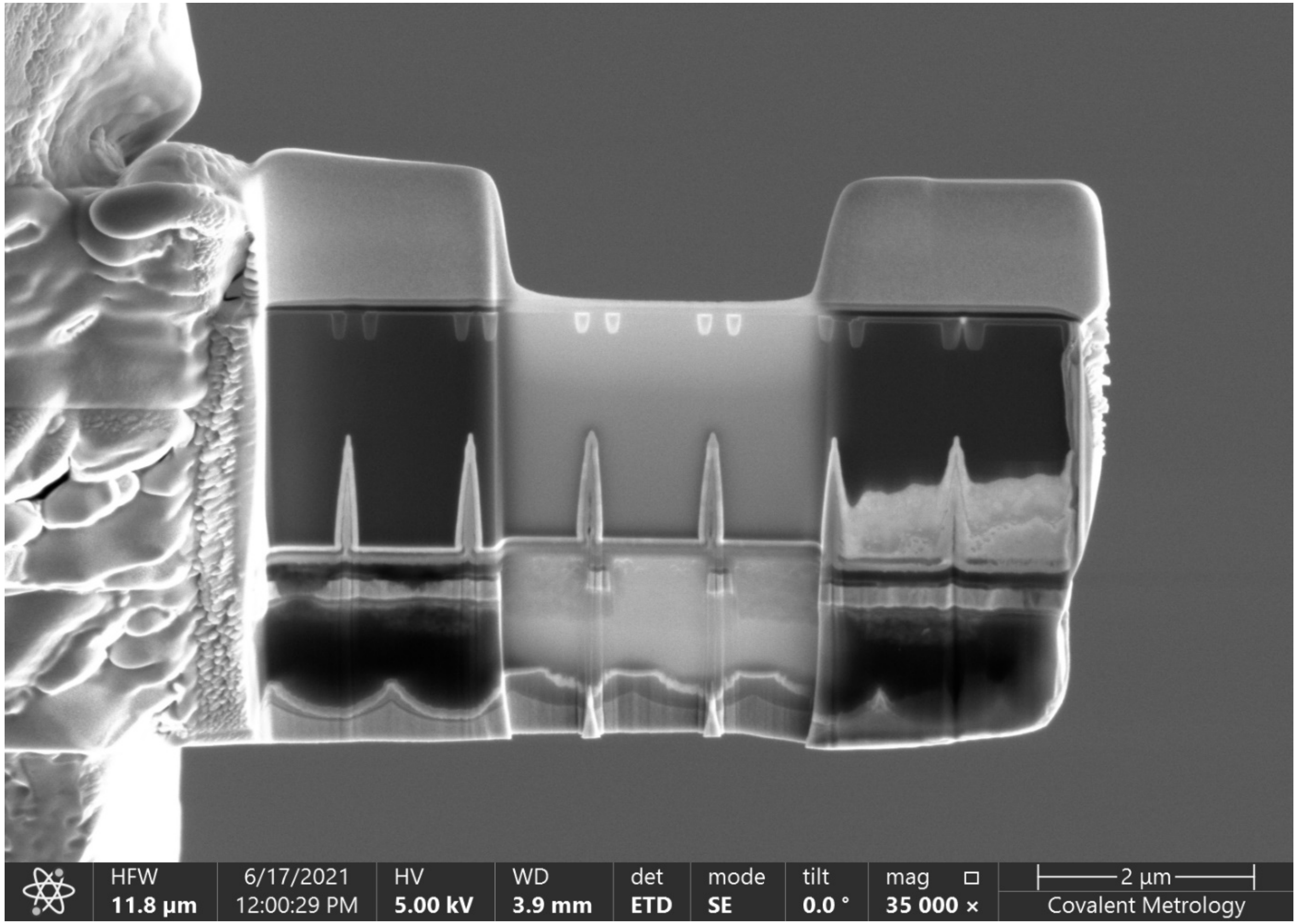


Lamella Lift-out: Inverted

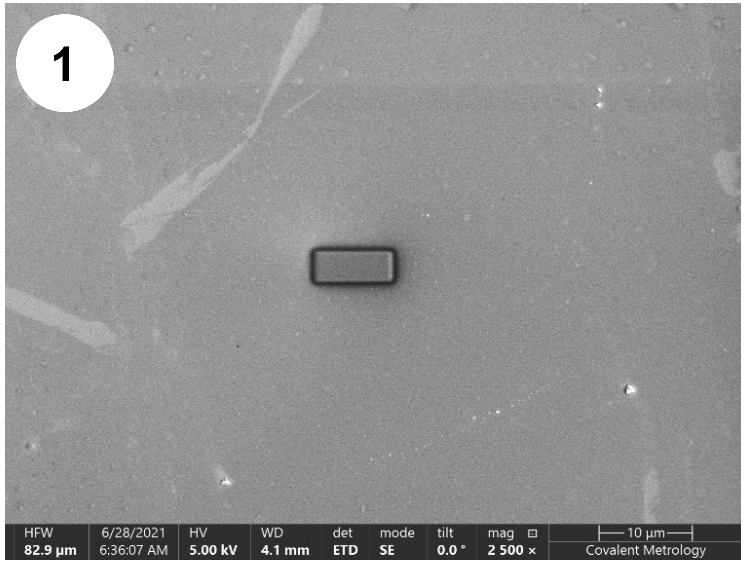


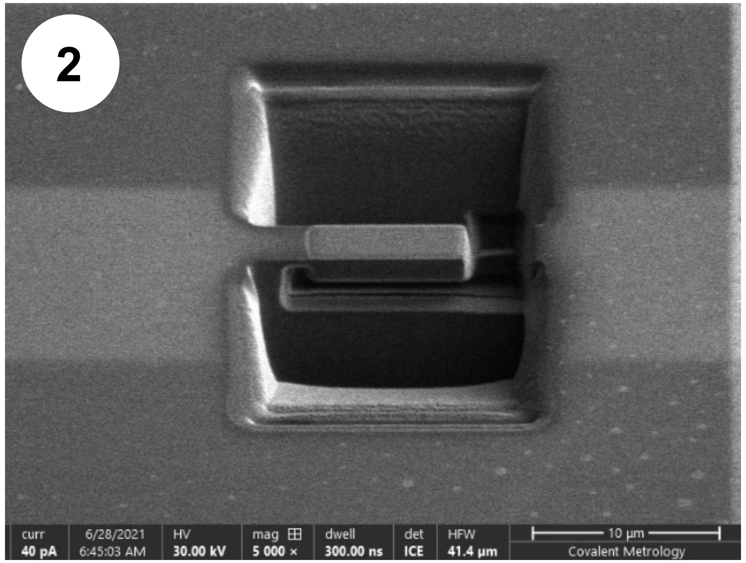
Used for:

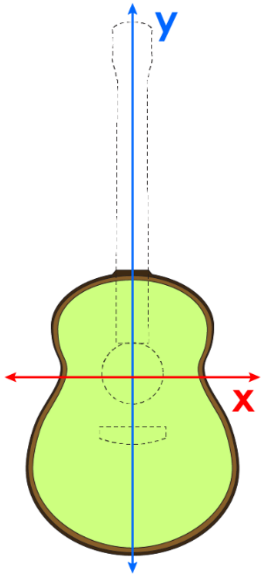
- Samples with a rigid/rough top surface
- Substantially harder material near lamella top to use lower overtilt angle and prevent milling through softer material before harder material is substantially thinner.

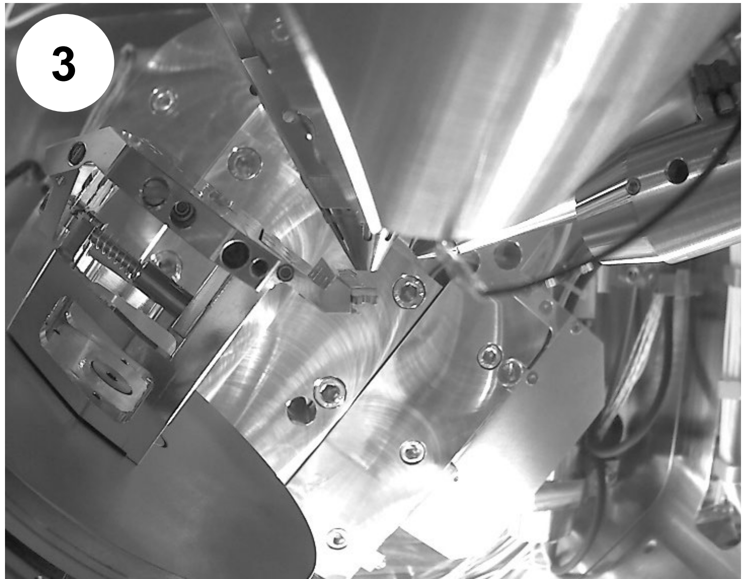


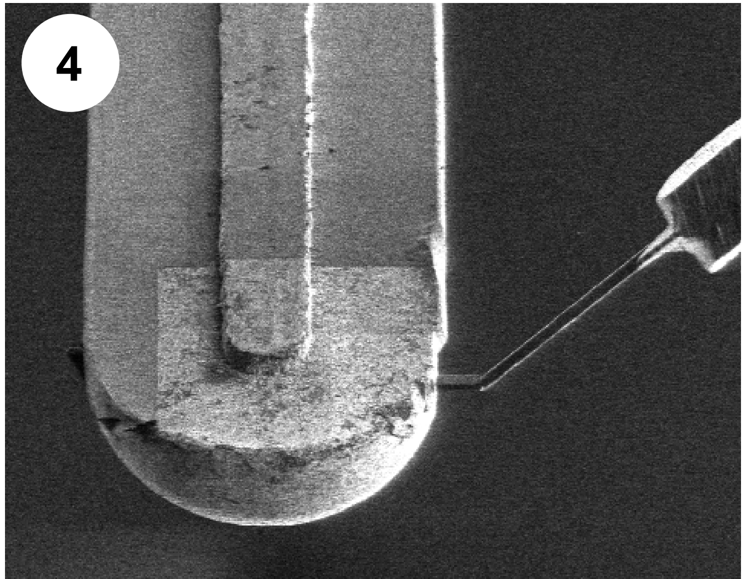
Lamella Lift-out: Planar – Process

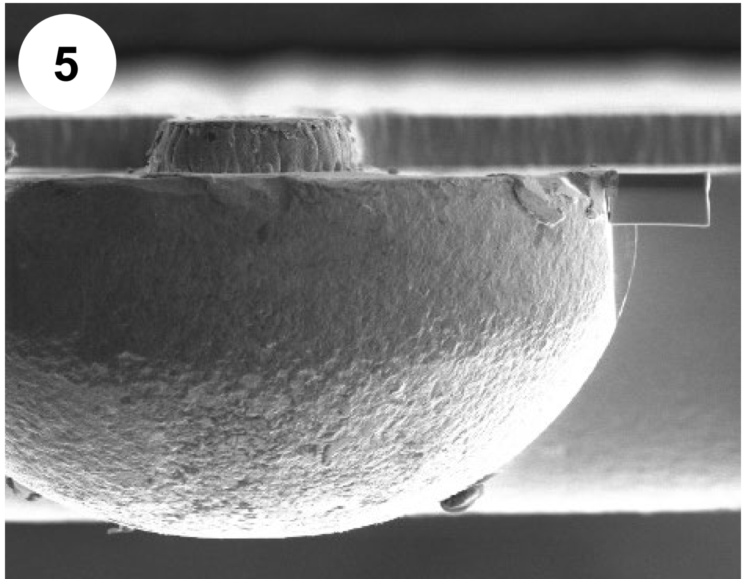




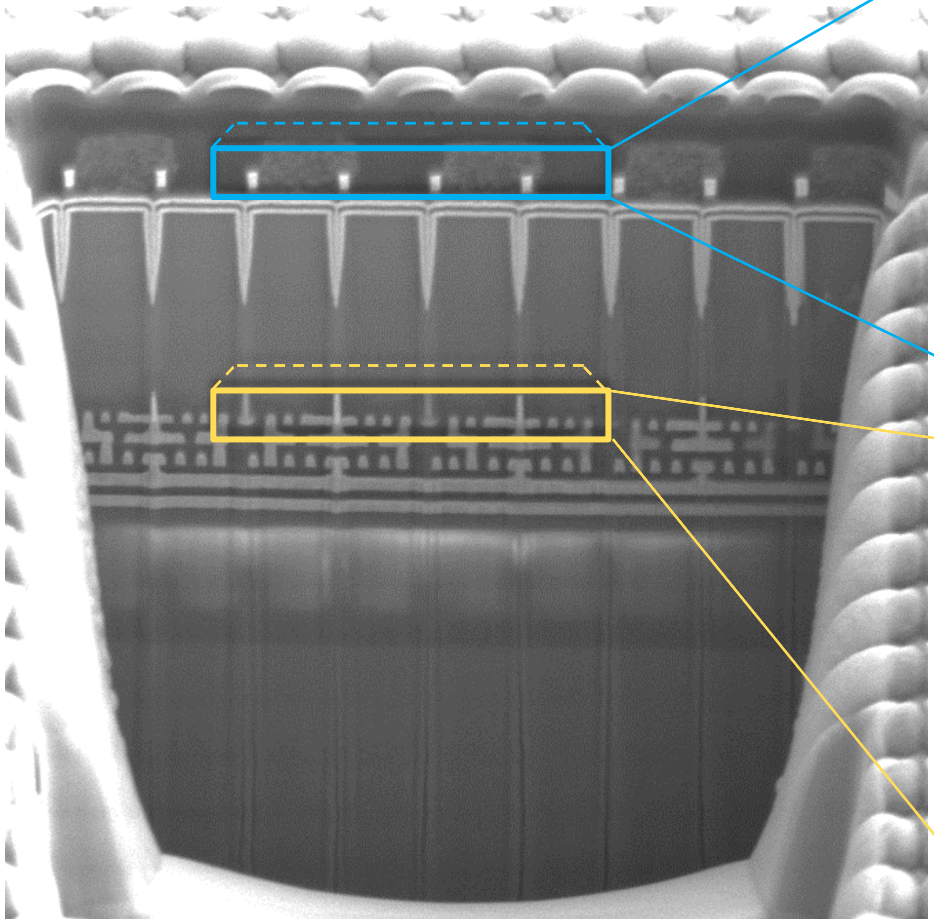


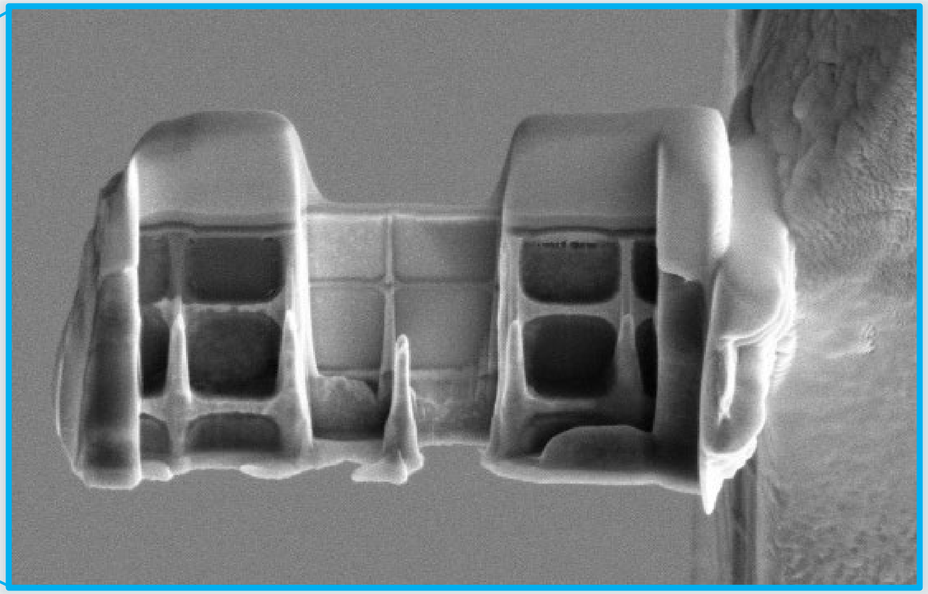


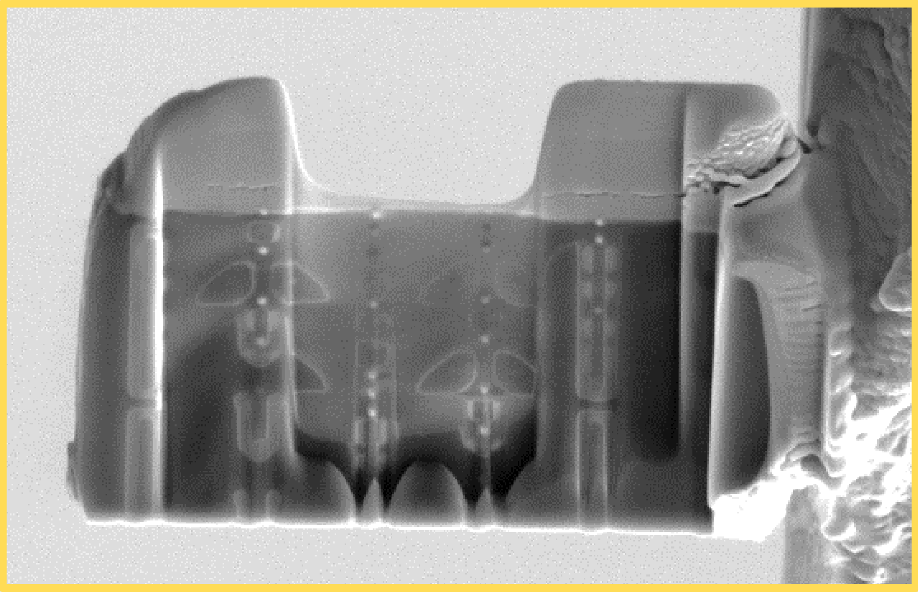


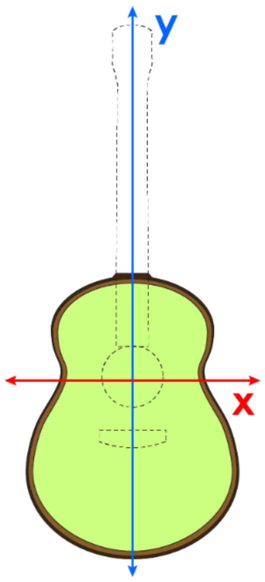


Lamella Lift-out: Planar



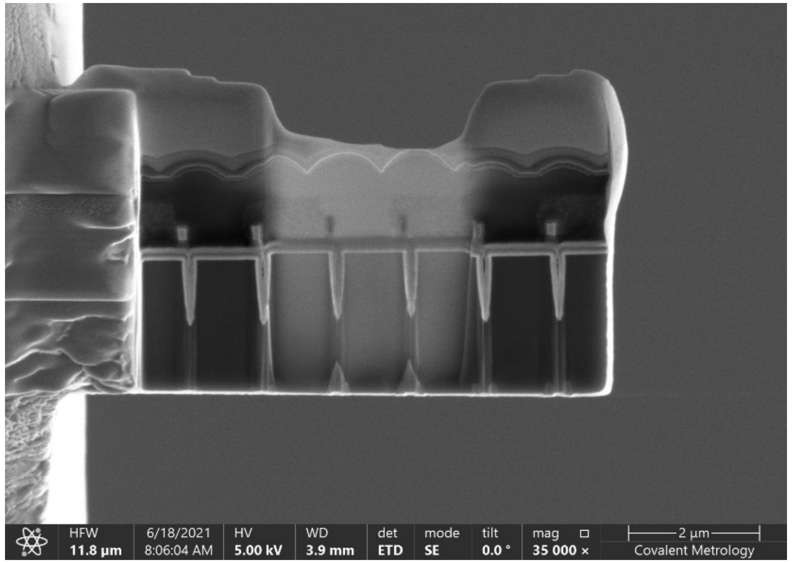






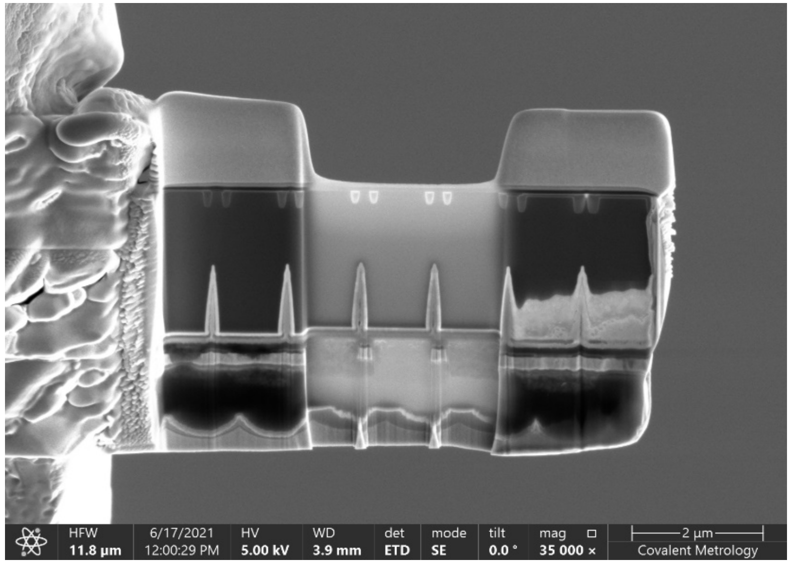
Variety of Lamella Lift-out – Recap

Top-Down



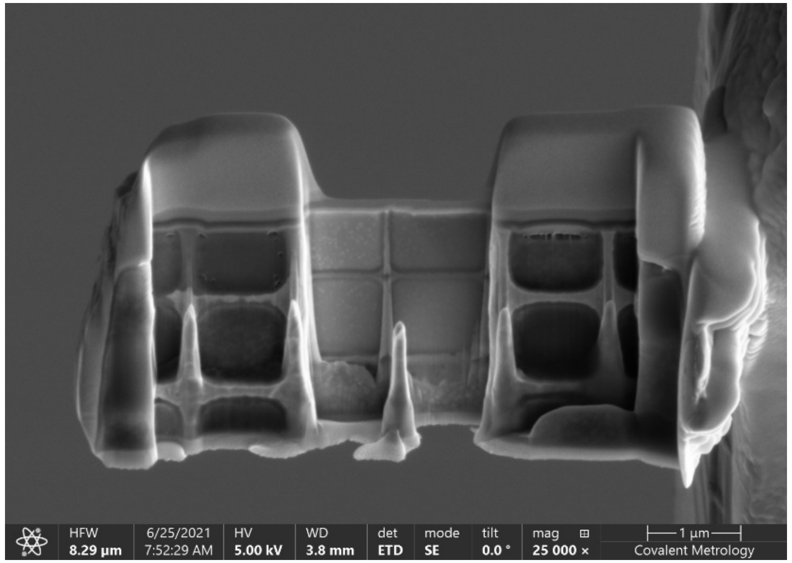
- Most common lamella type

Inverted



- Provide a uniform layer above feature to prevent curtaining
- Place lower mill rate material near top to use a lower overtilt angle while thinning.

Planar



- Examines layer/features on the x-y plane.
- Isolate individual layers for TEM/EDS analysis
- Provide another view on devices (planar cross-section)

Lamella Defects

Lamella Defects – Amorphous Damage

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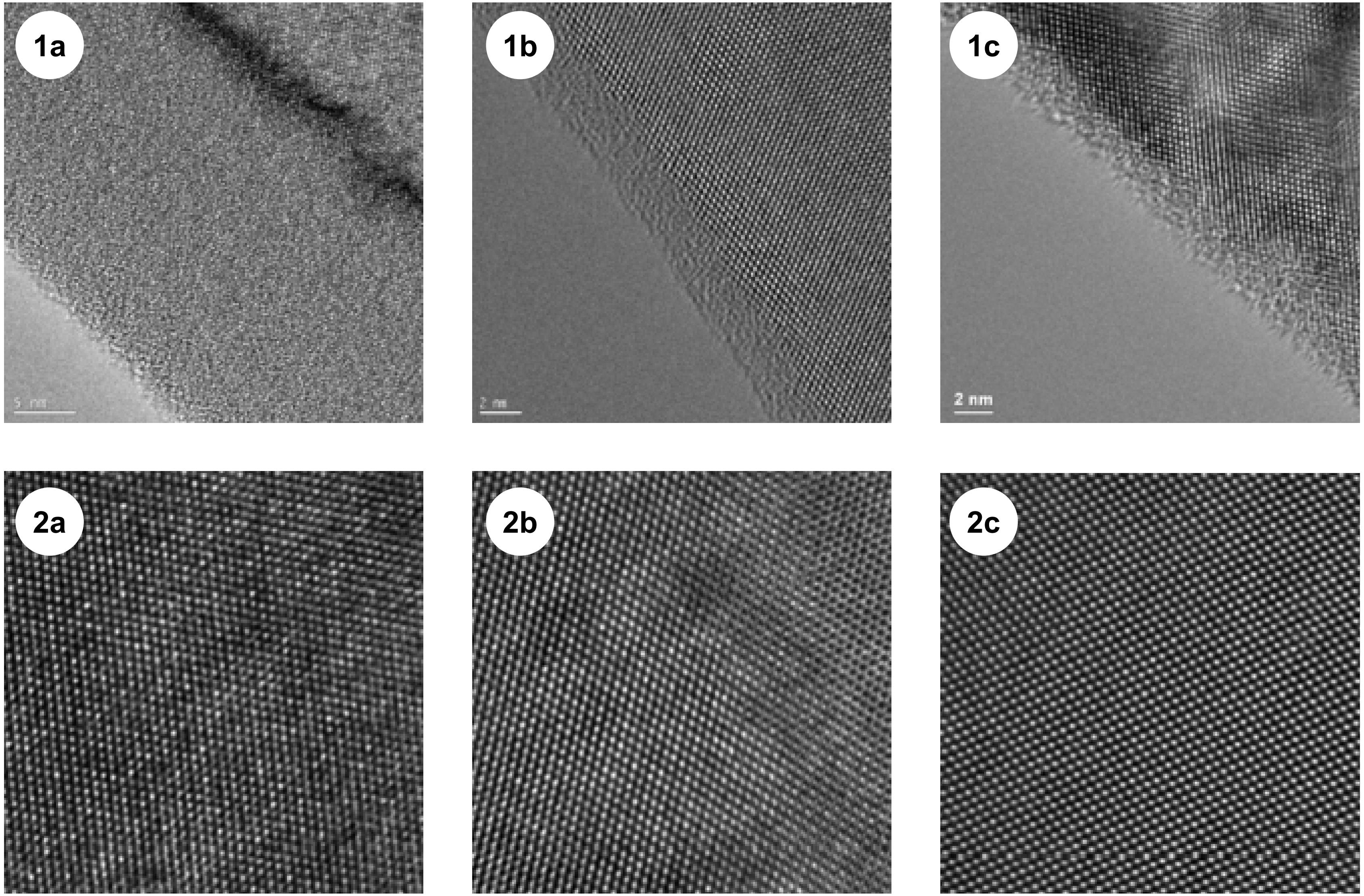
Amorphous damage in Si due to:

- 1a) 30 kV, ~ 21.5 nm
- 1b) 5 kV, ~ 6.6 nm
- 1c) 2 kV, ~ 3.1 nm

TEM imaged of Si polished with:

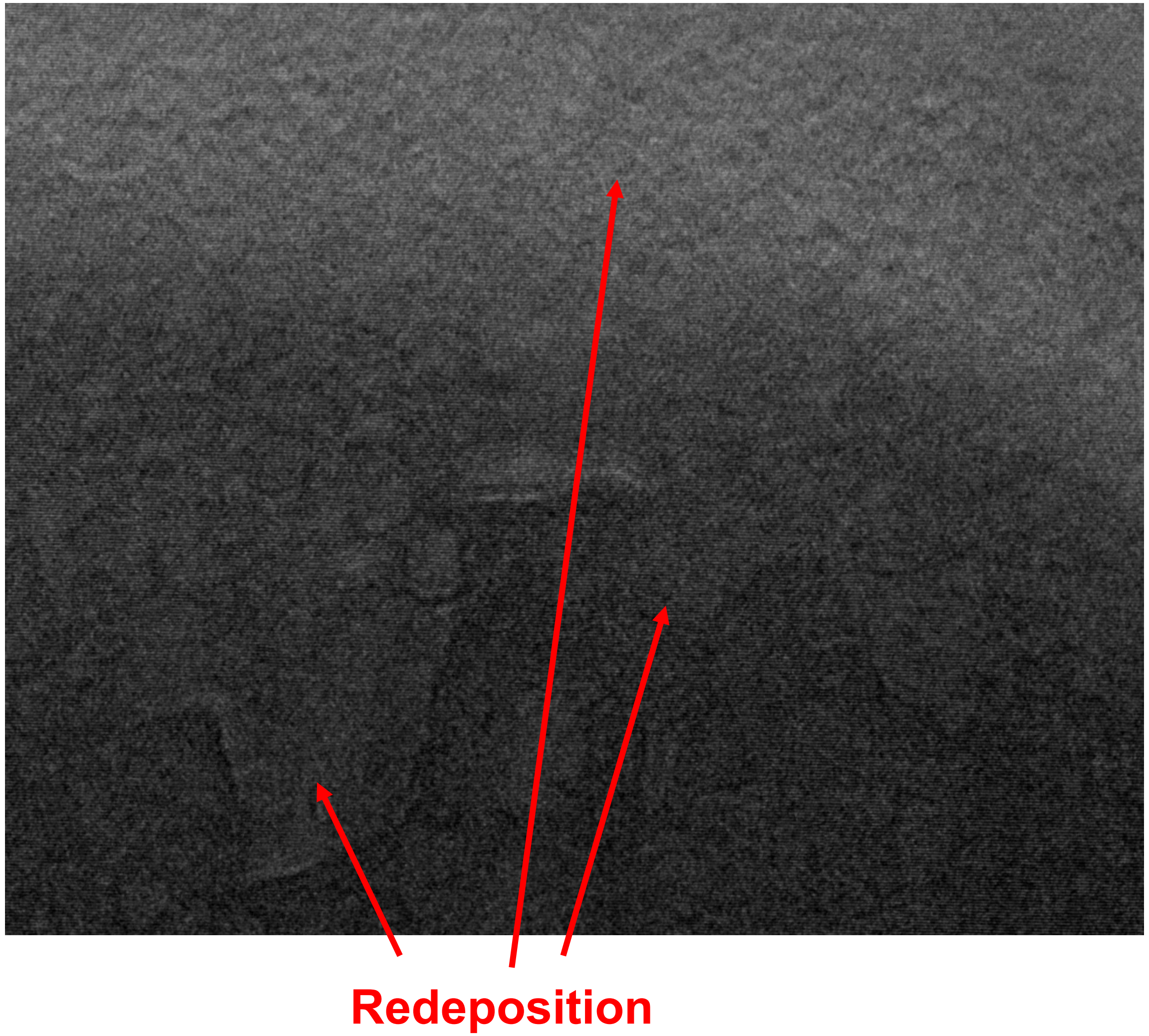
- 2a) 30 kV
- 2b) 5 kV
- 2c) 2 kV

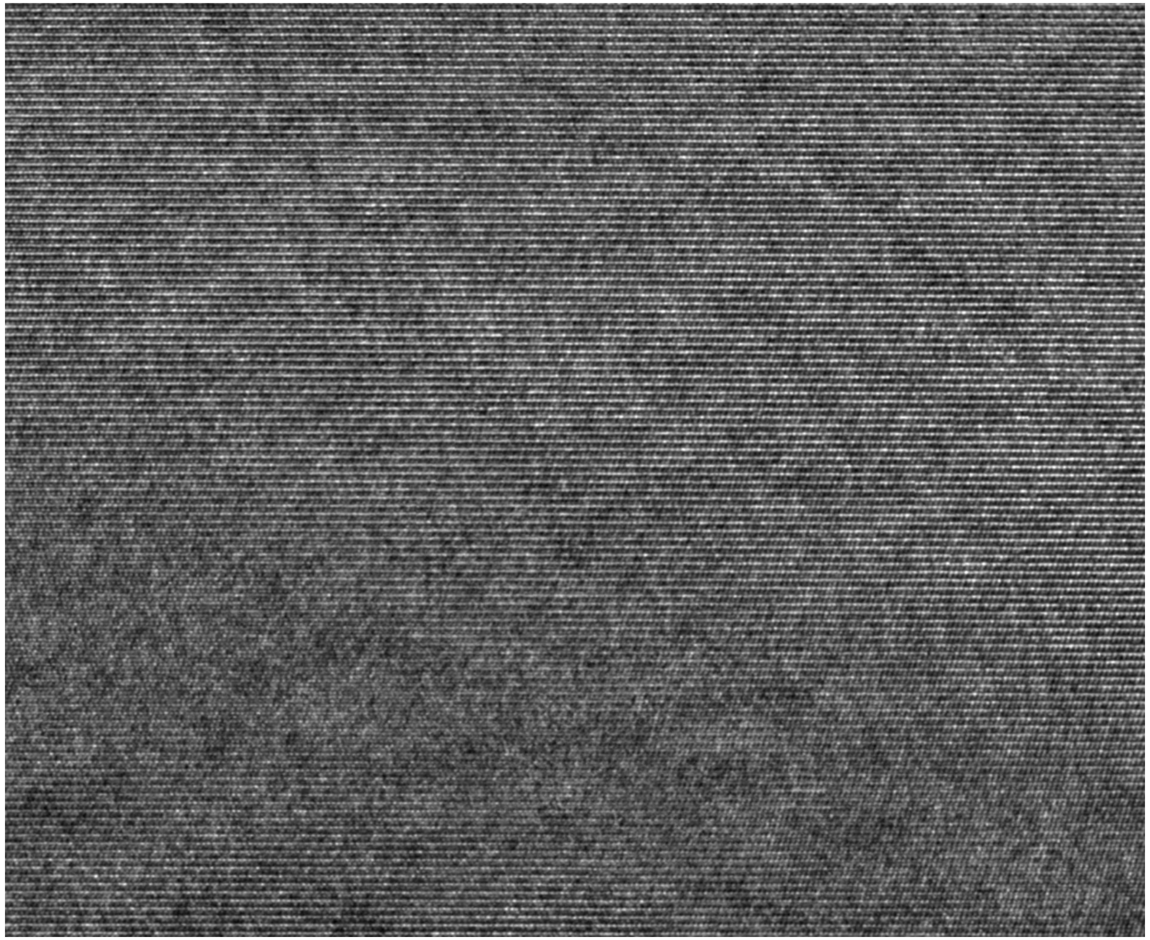


[3]

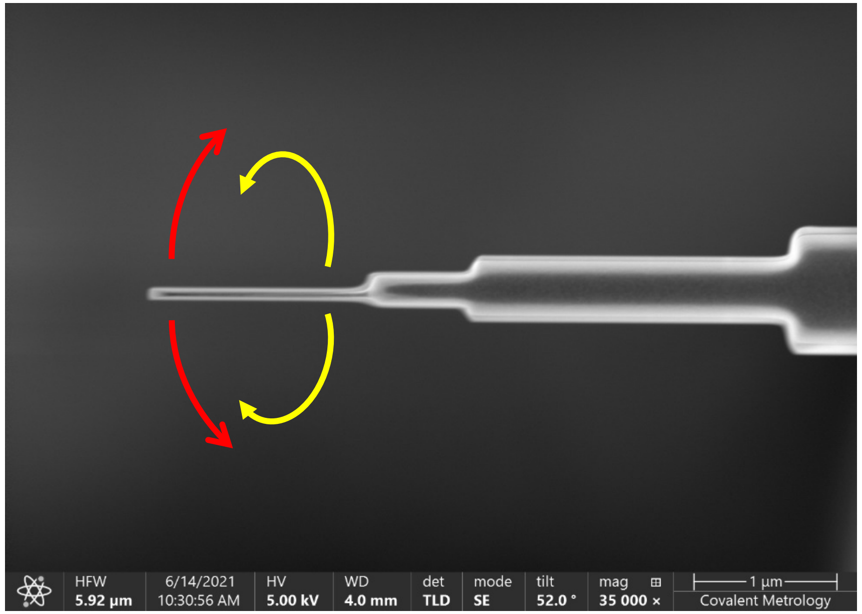
Thank you to Mark Najarian at ThermoFisher

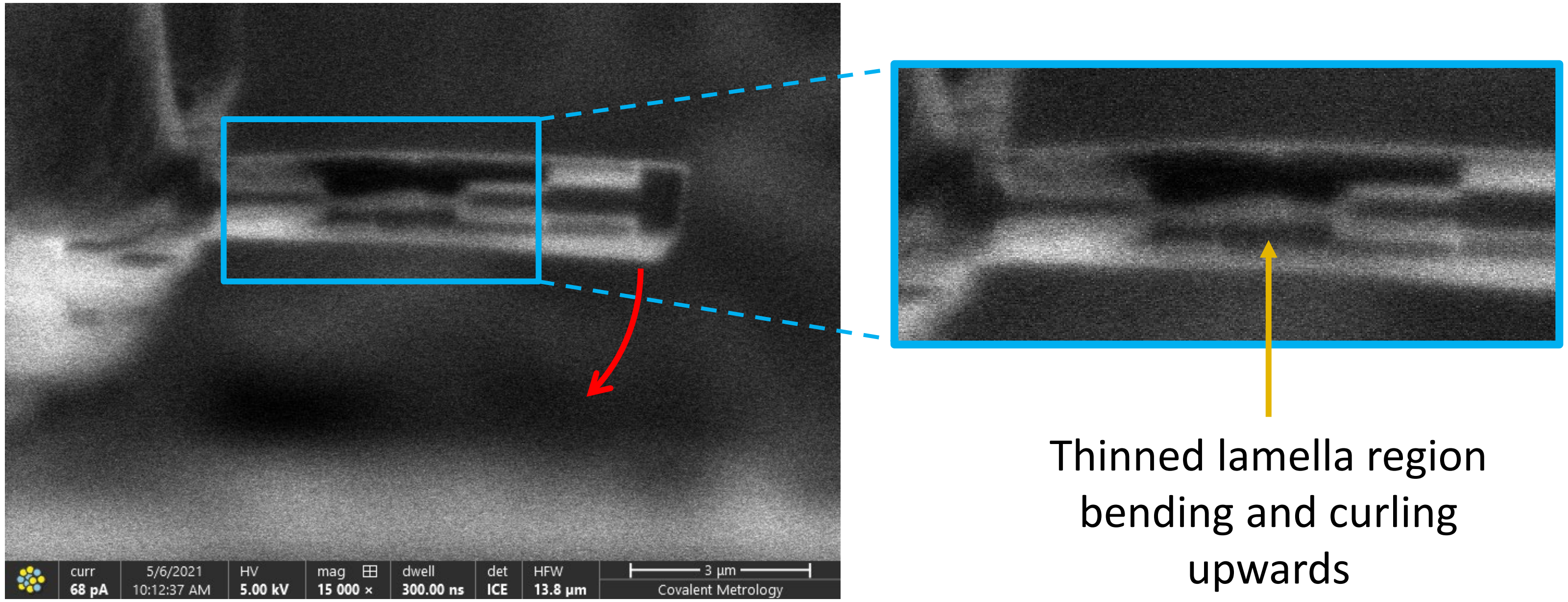
Reduction in TEM image quality due to variation in thickness





Lamella Defects – Bending/Curling



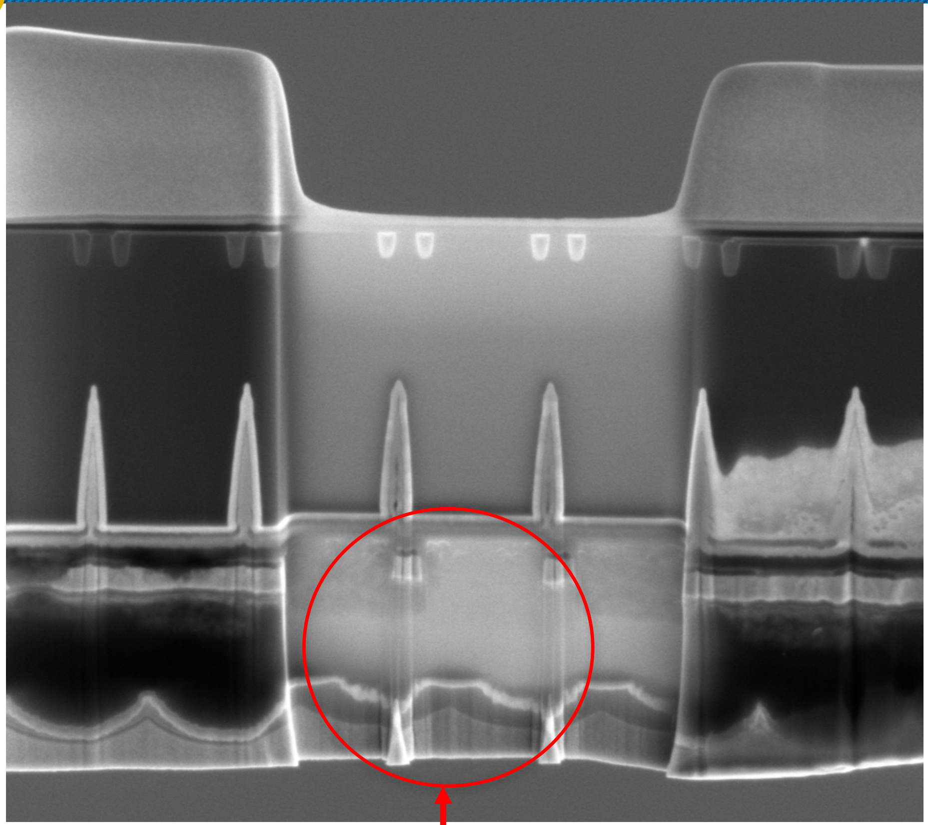


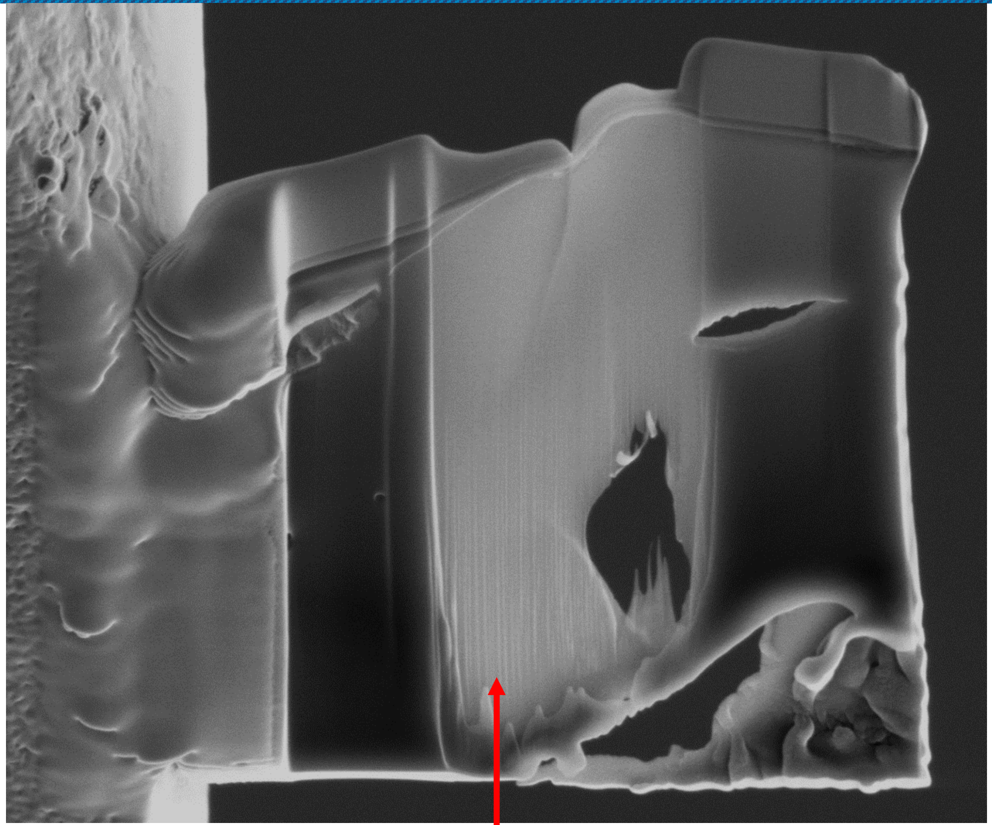
Thinned lamella region bending and curling upwards

- Lamella may bend at various thicknesses based on the sample material

- Deformations in feature/boundary interface can lead to inaccurate measurements

Lamella Defects – Curtaining



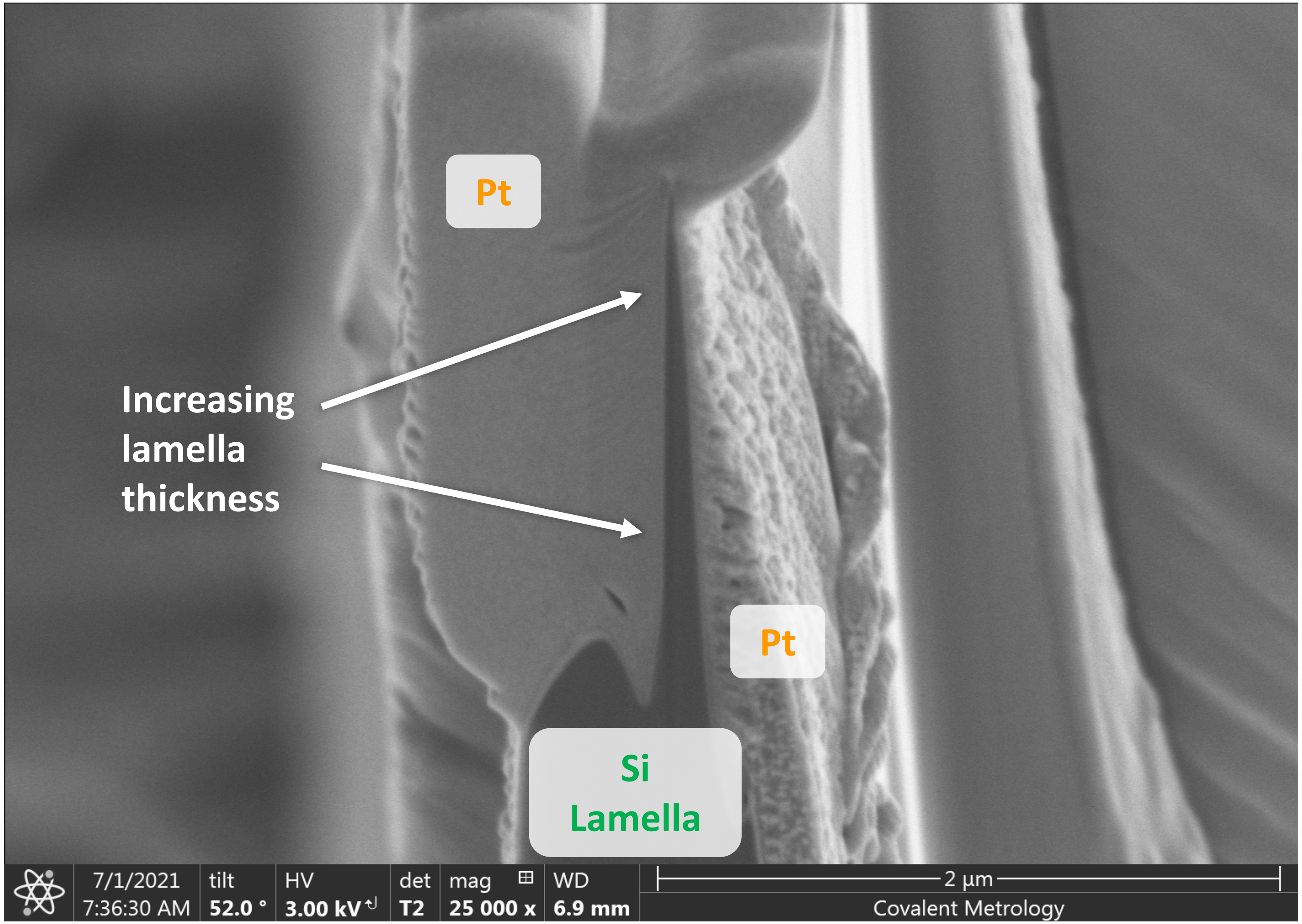


Curtaining occurs in:

- **Soft materials**
- Boundaries with a change in texture
- Grains
- **Devices/features**

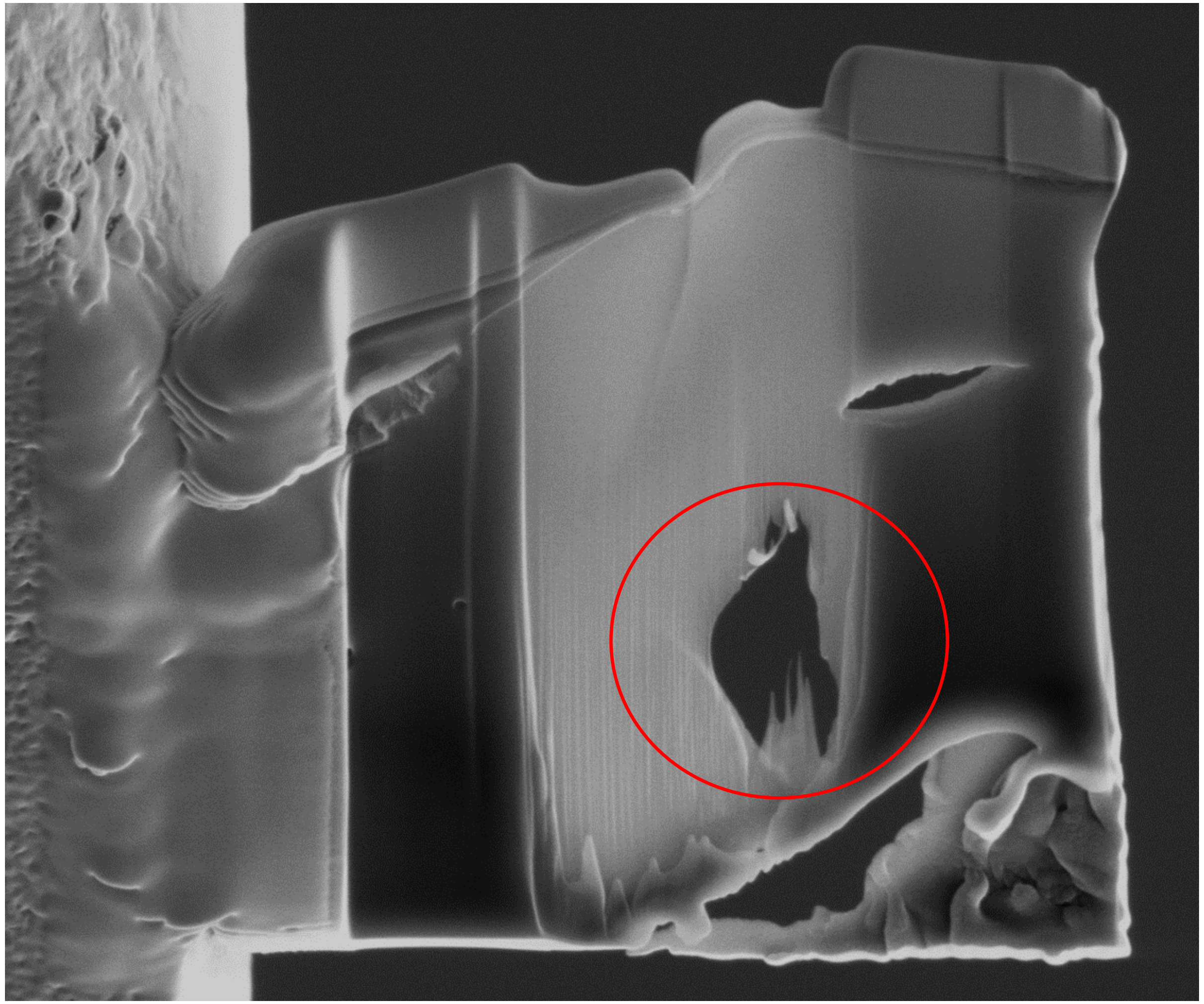
Lamella Defects – Wedging

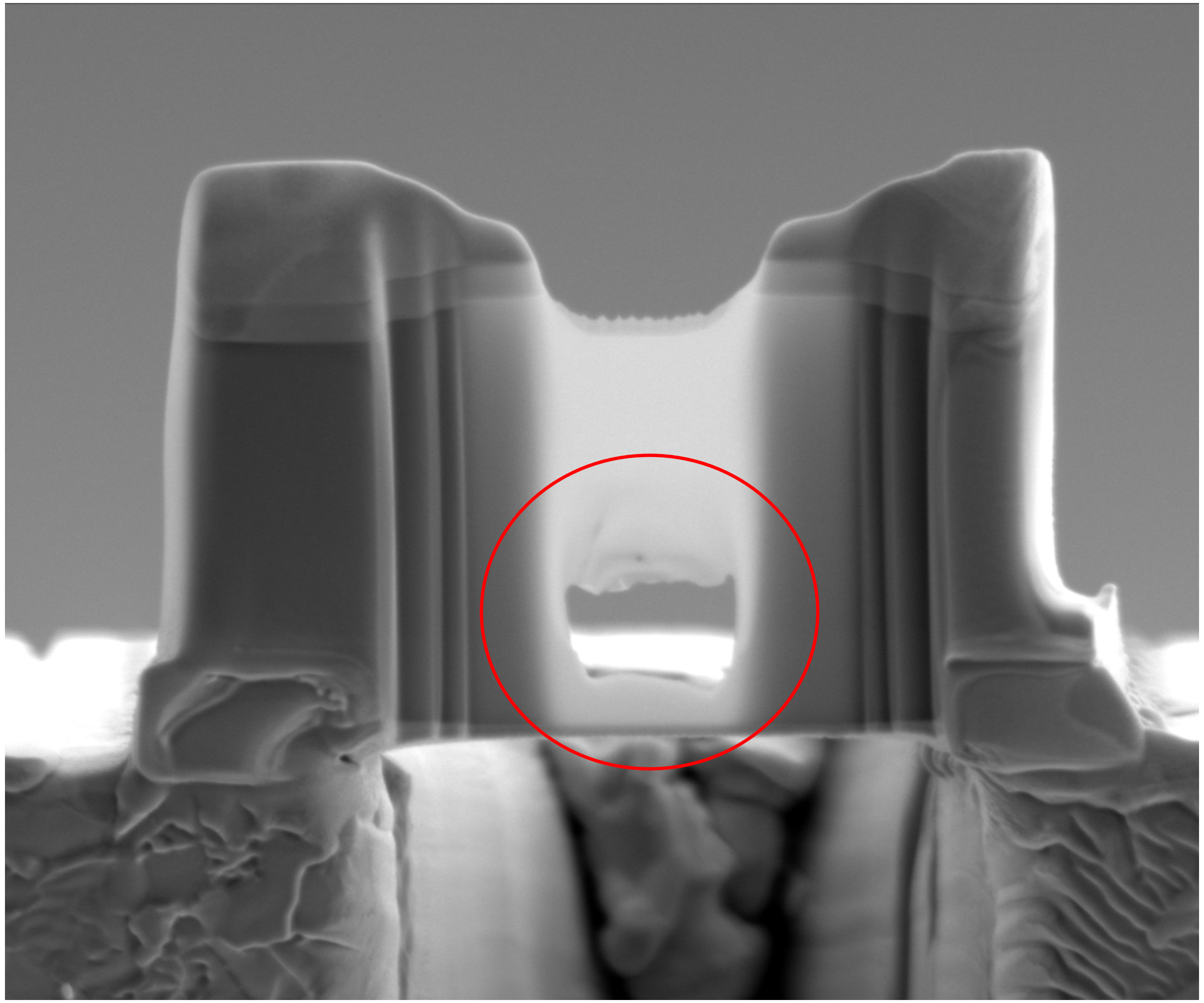
- Incorrect over-tilt angles – or specimens with materials that have different milling rates – result in "wedged" lamella
- Wedge lamella have the thickness either increase or decrease across the thinned window
- Results in loss of TEM image resolution across the thinned window



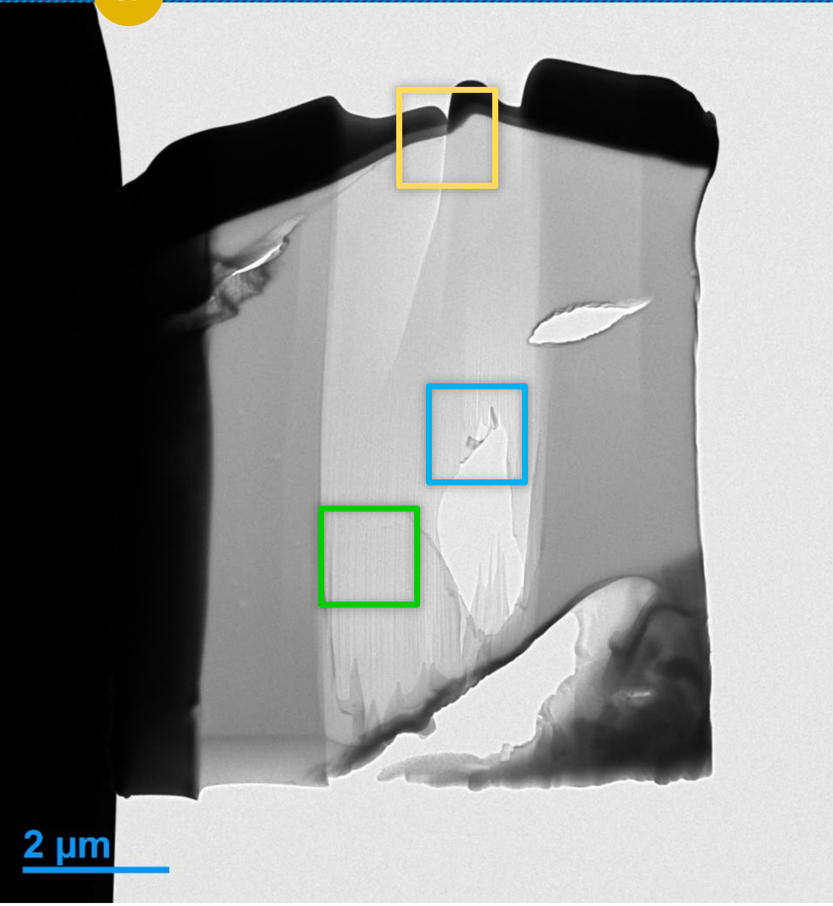
Lamella Defects – Holes

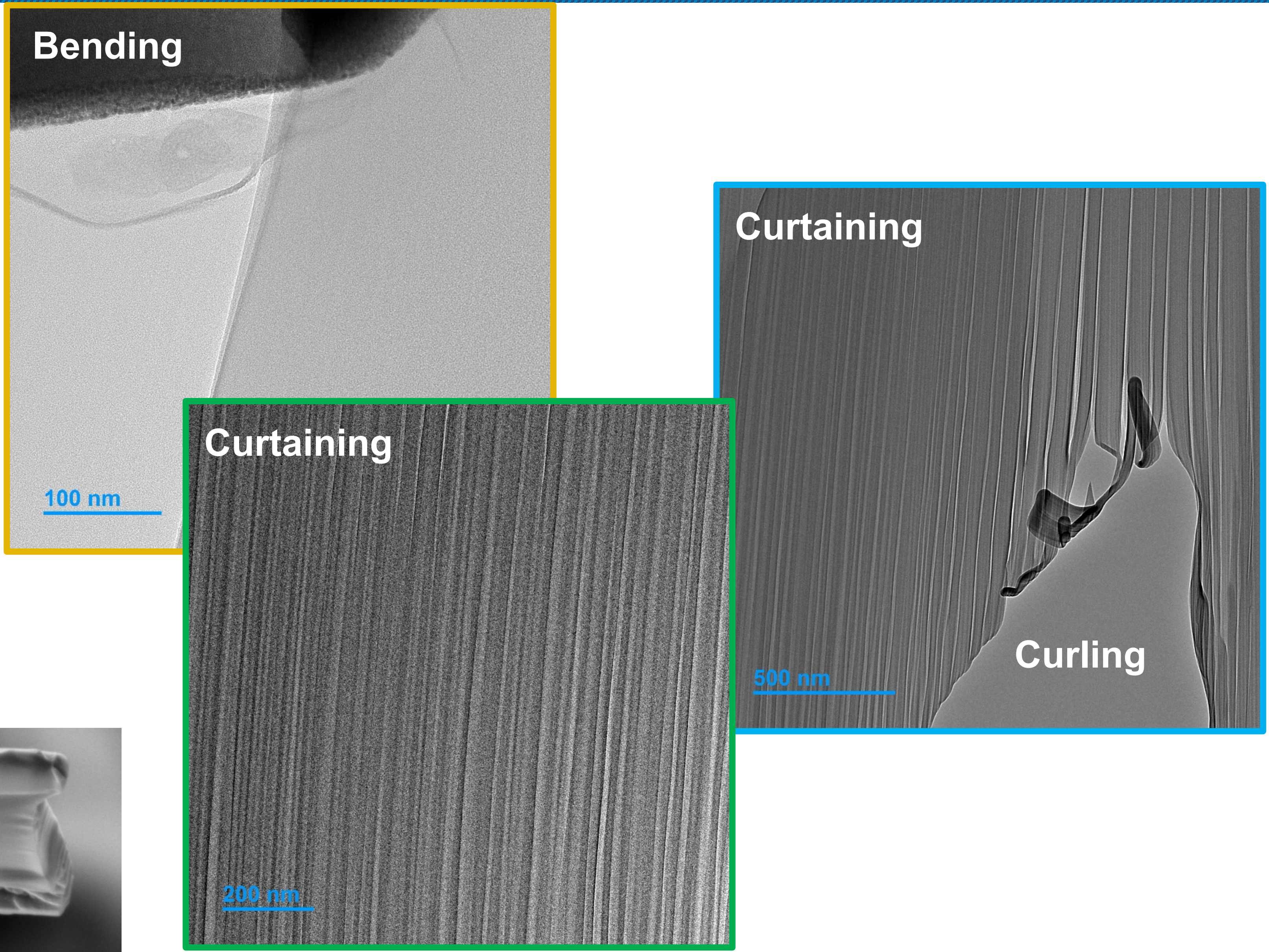
Holes may contribute to other lamella defects, such as bending/curling, as well as possibly destroying the feature of interest.

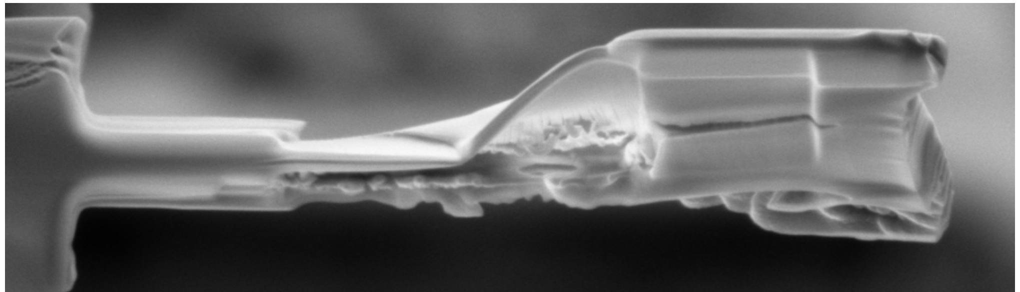




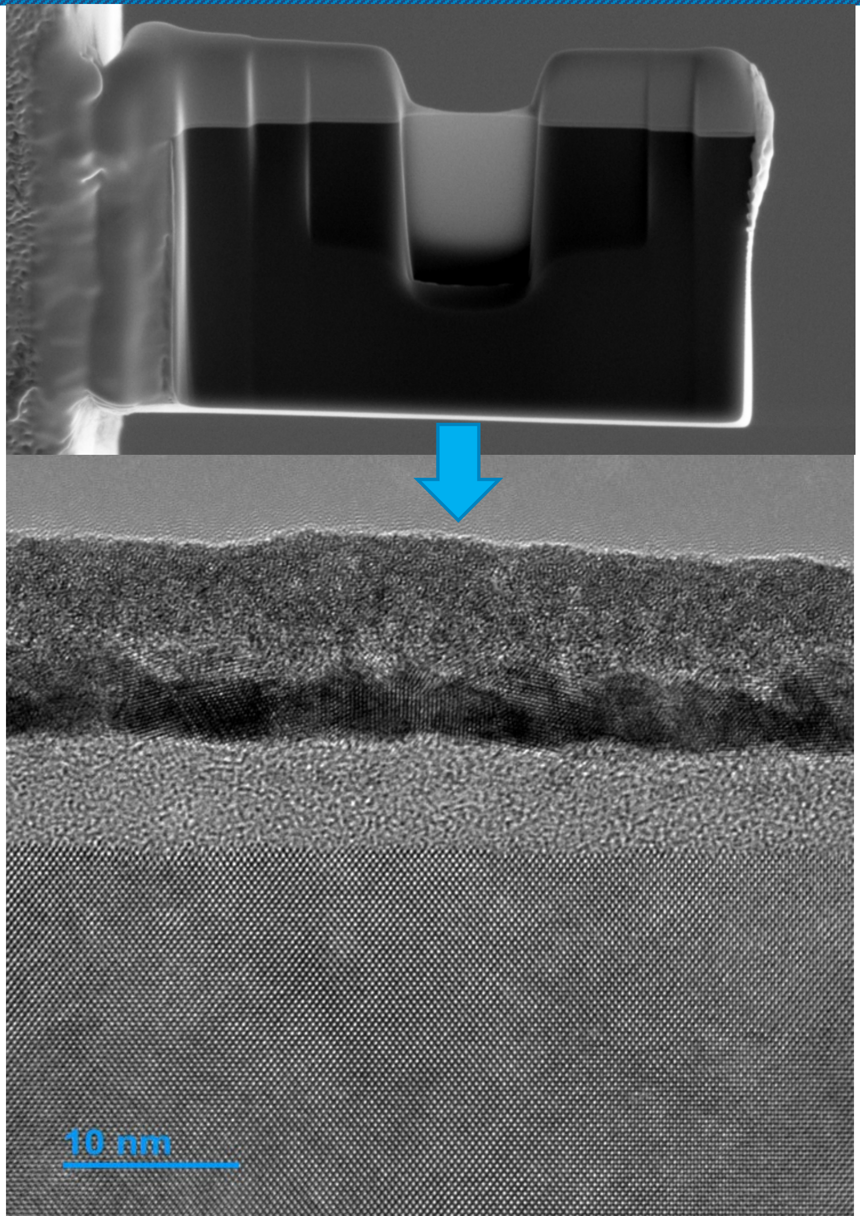
Lamella Defects – TEM Images

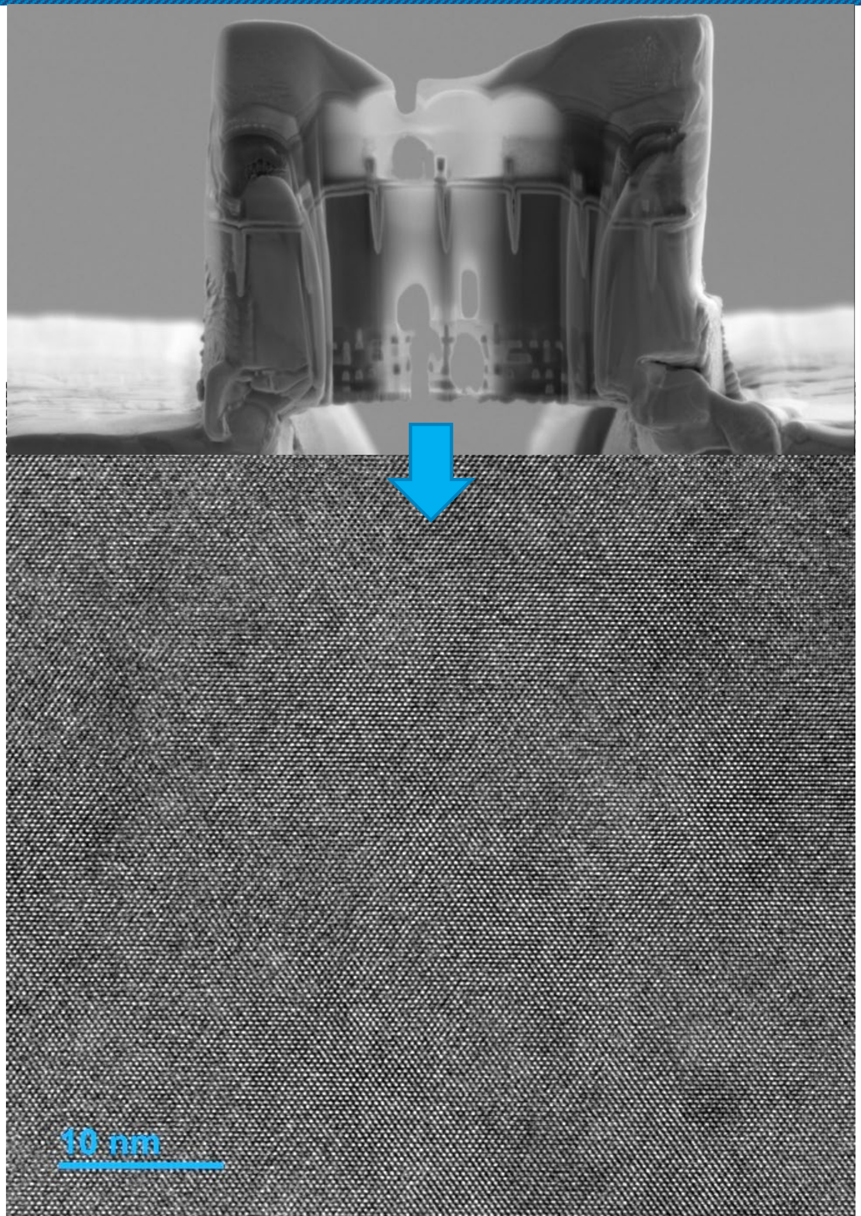






Defects Don't Have to Stop a Good TEM Image





Conclusion

- Lamella preparation is not only science, but a form of art
- Sample preparation is crucial to TEM imaging
- Understand the material being processed
- Select grids that do not contain materials of the specimen
- Sample orientation
- Several different way to thin a lamella
- Electron transparency differs amongst lining energy and material type
- Lamella defects have numerous root causes negatively impacting TEM image quality

Future webinars about TEM sample preparation will focus on advanced lamella thinning techniques such as cross-section lamella for TEM imaging, rocking mill, targeted samples, etc.

References

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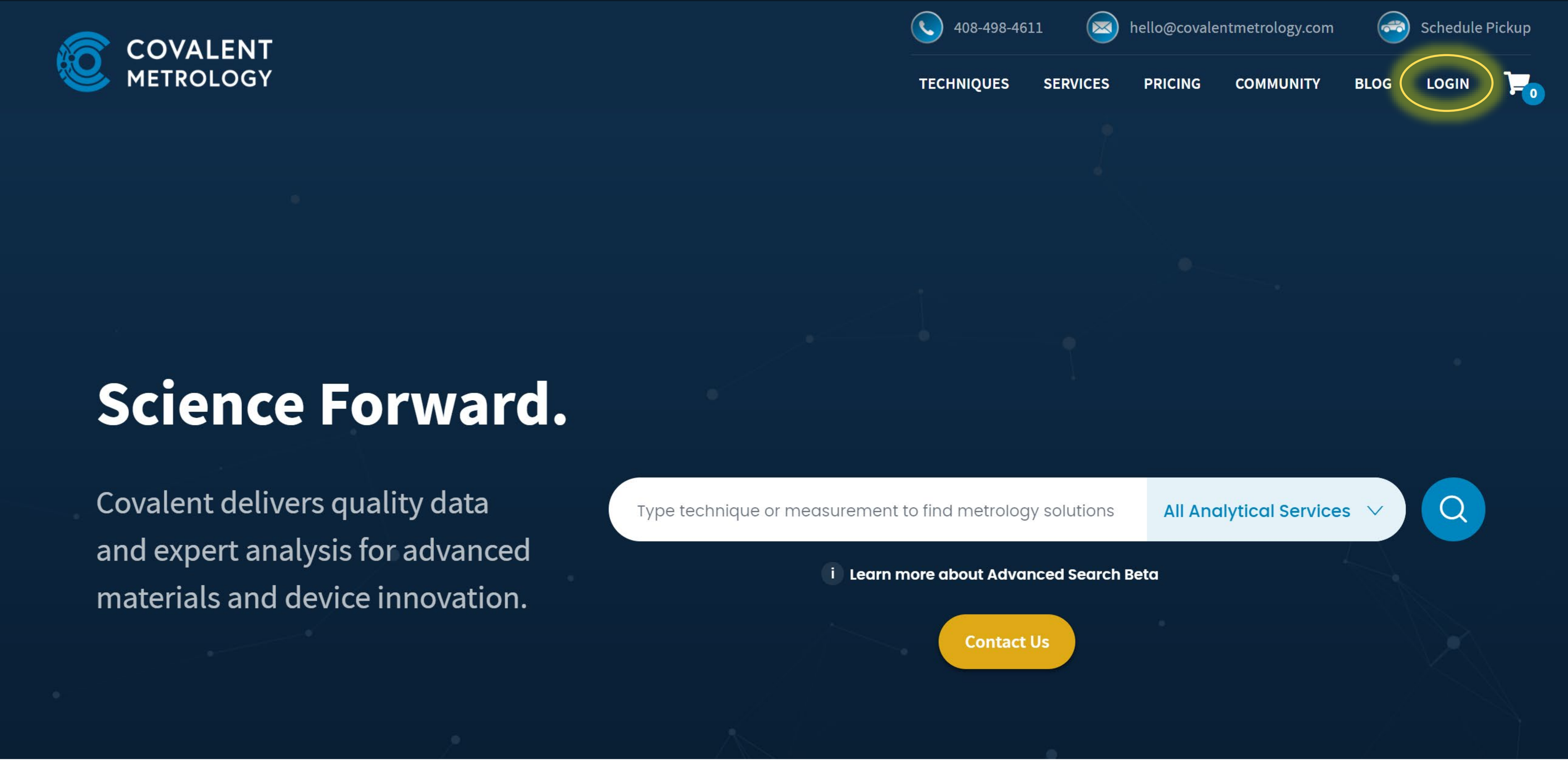

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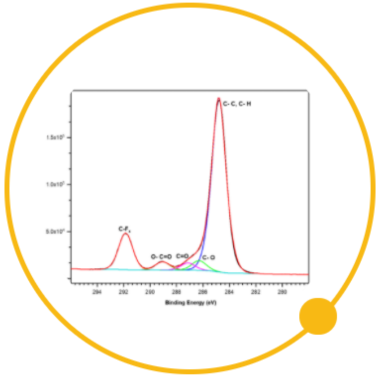

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