Site-specific TEM Sample Preparation using Focused Ion Beam Methods

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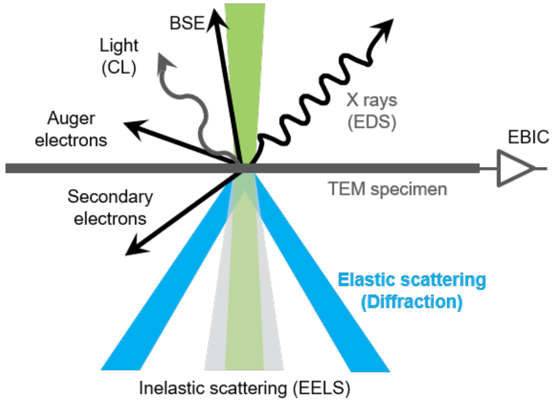
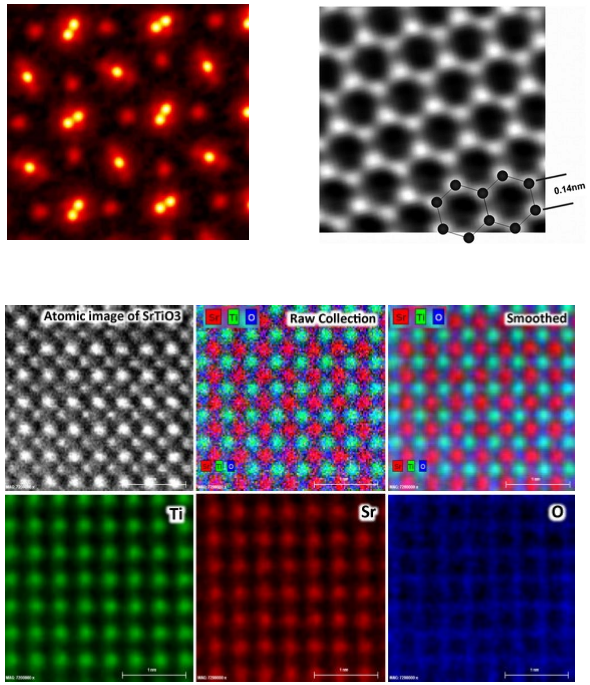
Sample Preparation is Key for Perfect (S)TEM Data

Sample IN | **(S)TEM** | **Data OUT**





What makes a good High-Quality TEM Sample?

- ✓ Area ≤ 3mm to fit into holders
- ✓ Very thin (≤100 nm)
- ✓ Large flat electron transparent region
- ✓ No additional/foreign artifacts
- ✓ No contamination

Chen, Z. *et al.*, *Science* **372** 826 (2021)
Dato A. *et al.*, *Commun.* **40**, 6095 (2009)

Sample Preparation is Key for Perfect (S)TEM Data

Garbage

~~Sample~~ IN

(S)TEM

Garbage

~~Data~~ OUT

TEM Sample Preparation & Classification

Everything in a solid state can be prepared as a TEM sample

Bulk Sample

1) Shape sample to 3 mm disc & polish/dimple/ion mill until electron transparent (typically < 100 nm)

2) Crush into powder, disperse into solvent (typically alcohol) and transfer to TEM grid

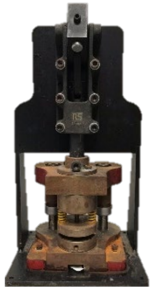
3) Electrochemically polish

4) Perform FIB-SEM liftout

cut

grind/polish

punch disk



dimple

ion milling

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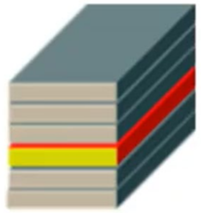
Thin Film/Region of Interest

Identify desired orientation, typically cross-section of plan-view

1) Prepare as bulk sample (mechanical thinning) ensuring correct orientation of imaging plane

2) Perform FIB-SEM liftout



Cross-section

TEM Sample Preparation & Classification

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Thin Film/Region of Interest

Identify desired orientation, typically cross-section of plan-view

1) Prepare as bulk sample (mechanical thinning) ensuring correct orientation of imaging plane
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Powders, NPs & Fibers

Either:

1) Disperse onto a supporting film or grid
2) Compress/embed in epoxy (bulk sample workflow), or microtome
3) In certain cases: Perform FIB-SEM liftout

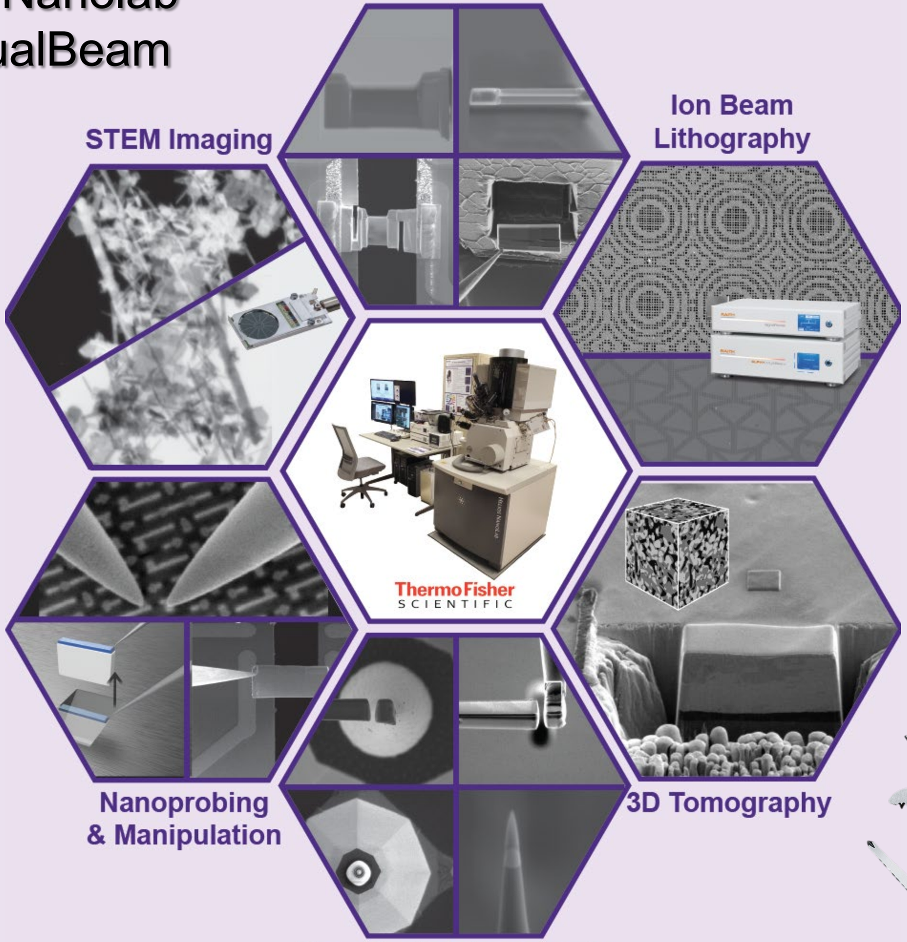
FIB-SEM DualBeam Capabilities @ NUANCE

Helios Nanolab 600 DualBeam

TEM Sample Preparation

STEM Imaging

Ion Beam Lithography



Nanoprobing & Manipulation

3D Tomography

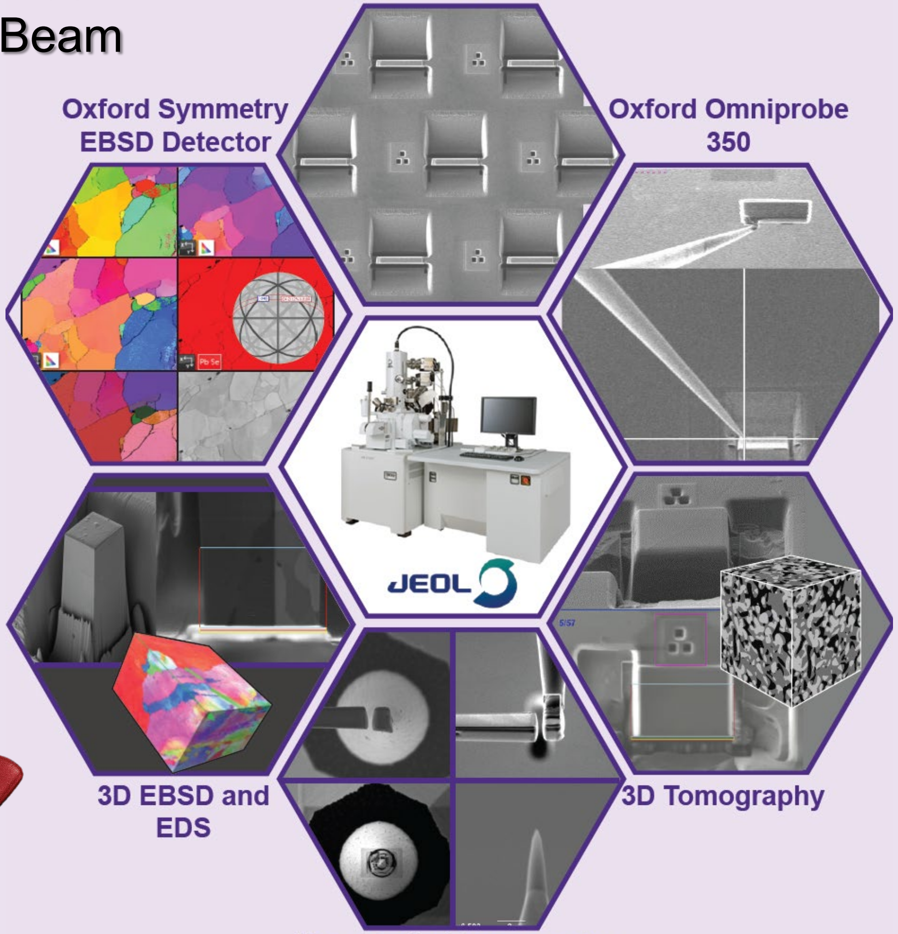
Atom Probe Tomography Preparation

JIB-4700F MultiBeam

Automated TEM Sample Preparation

Oxford Symmetry EBSD Detector

Oxford Omniprobe 350



3D EBSD and EDS

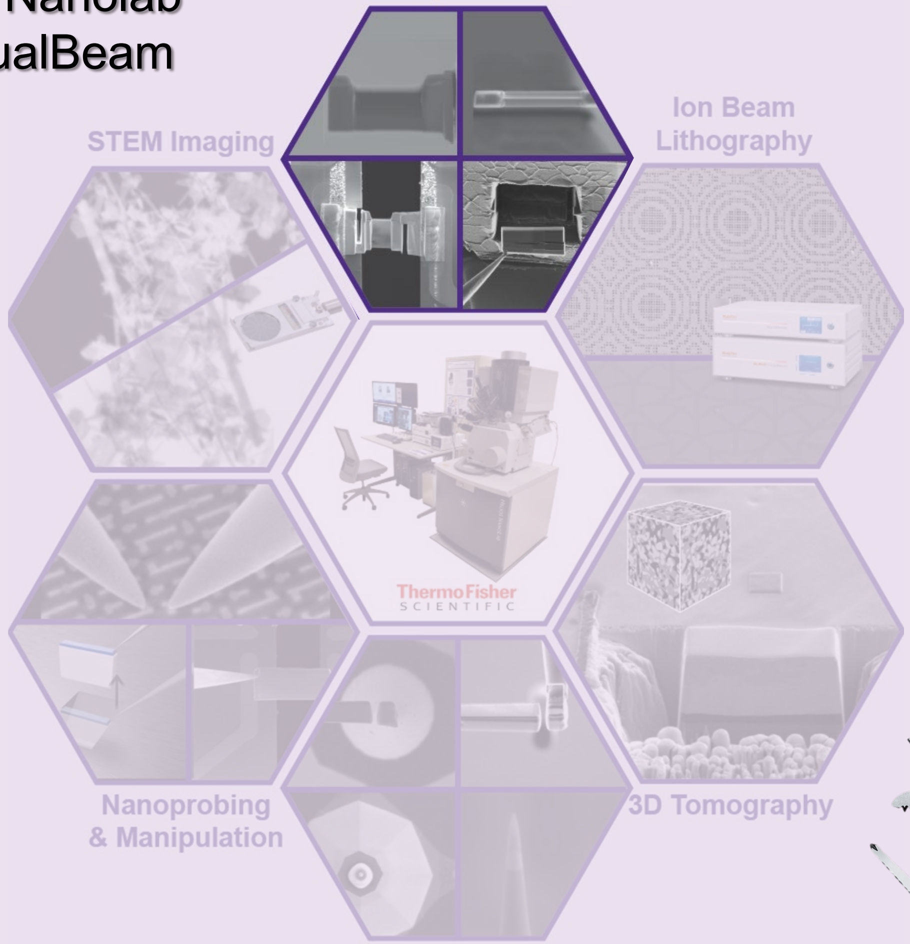
3D Tomography

Atom Probe Tomography Preparation

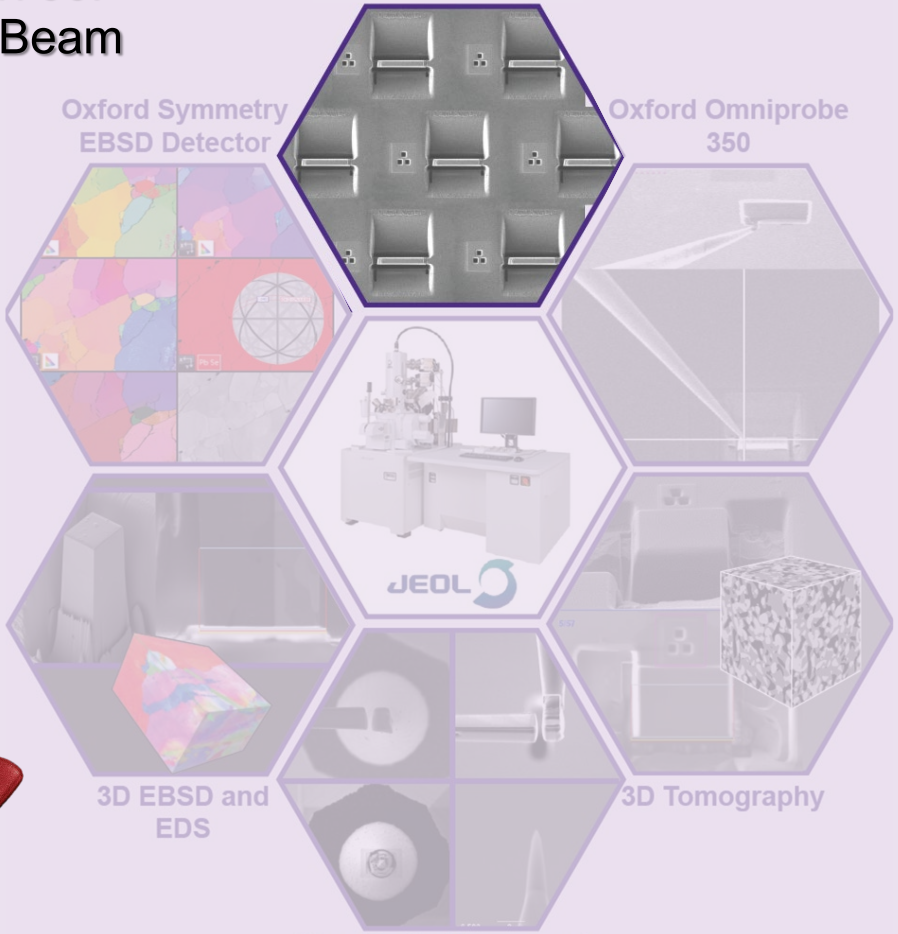
“Swiss Army Knife”

FIB-SEM DualBeam Capabilities @ NUANCE

Helios Nanolab
600 DualBeam



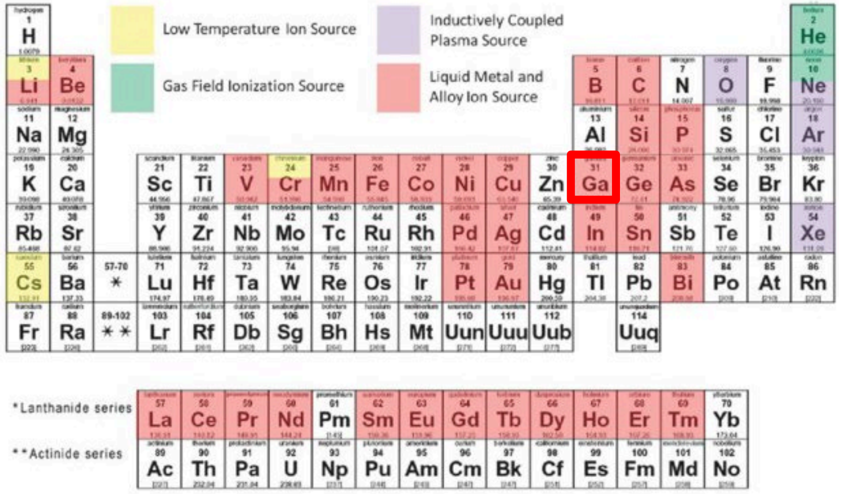
JIB-4700F
MultiBeam



"Swiss Army Knife"

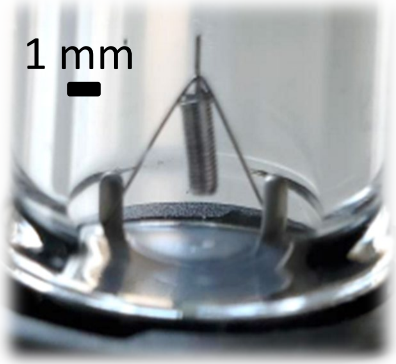
FIB-SEM DualBeam Fundamentals

Formation of a Focused Ion Beam – Ga^+ LMIS

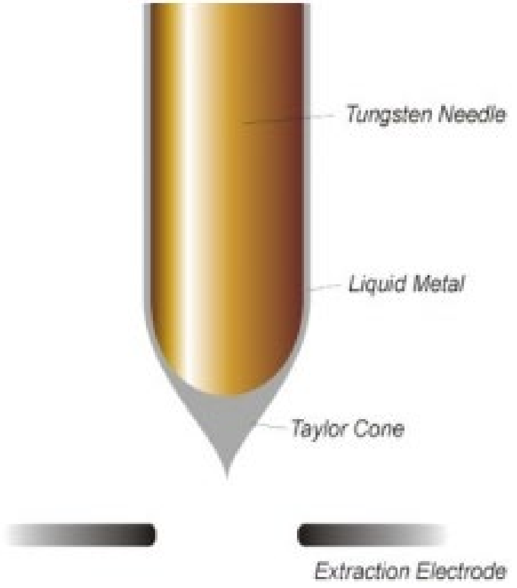


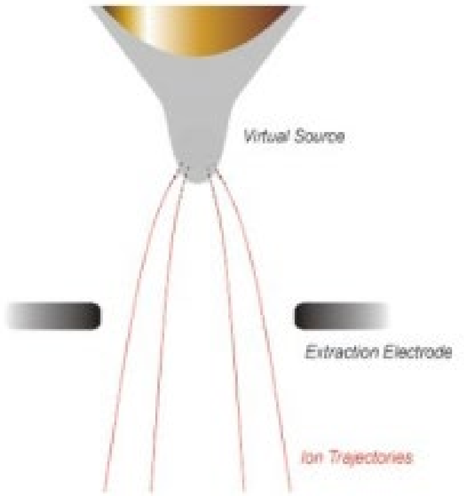
Liquid **M**etal **I**on **S**ource – Why Ga^+?

- Low melting point/vapor pressure → vacuum compatible
- Low volatility
- Good emission characteristics (low energy spread & high brightness)
- Single ion species Ga^+
- No diffusion/reaction with W tip

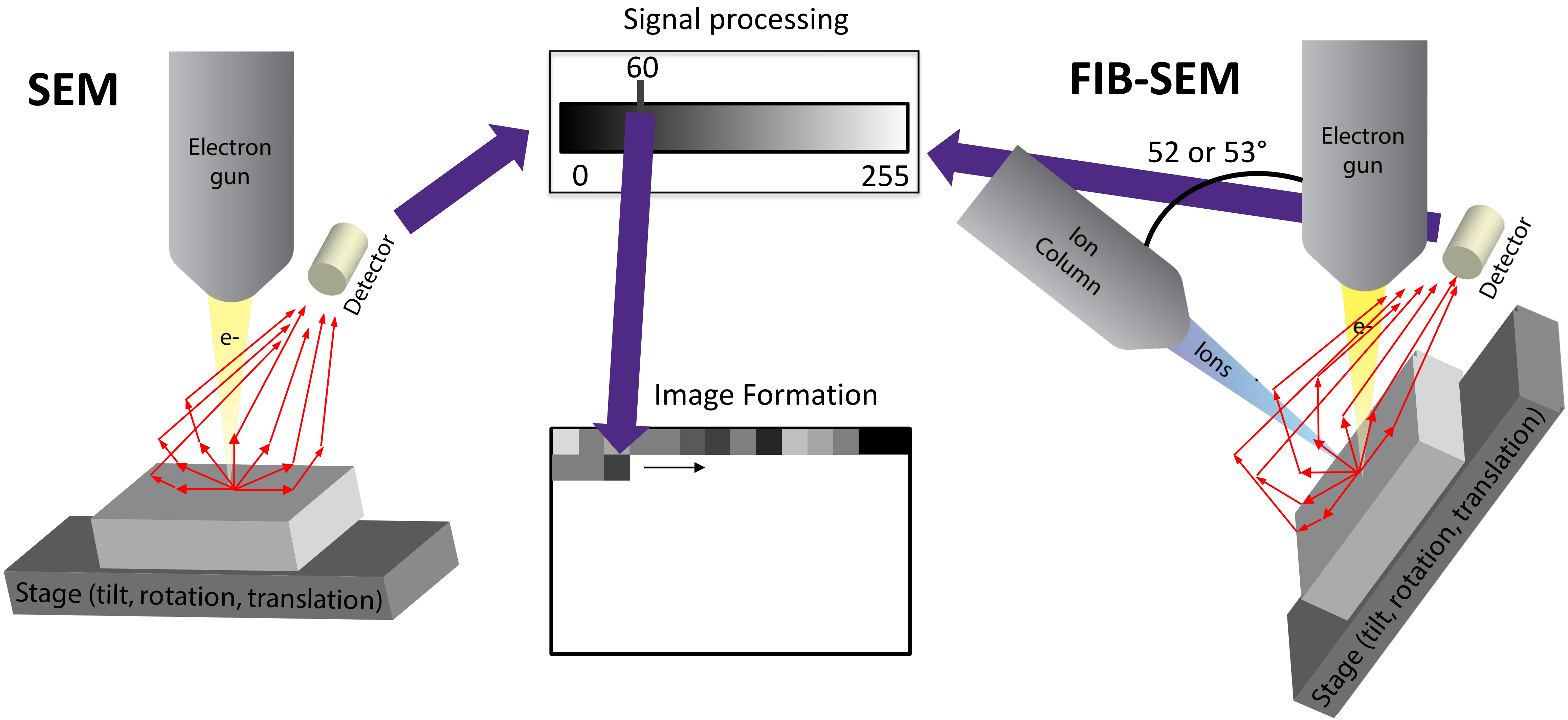


- Pure Ga metal is heated to liquid at >30°C and wets tip of W needle
- Electric field pulls liquid Ga into sharp cone
- Cone tip field emits Ga^+ ions
- Accelerated 1keV – 30keV typically, focused by electrostatic lenses



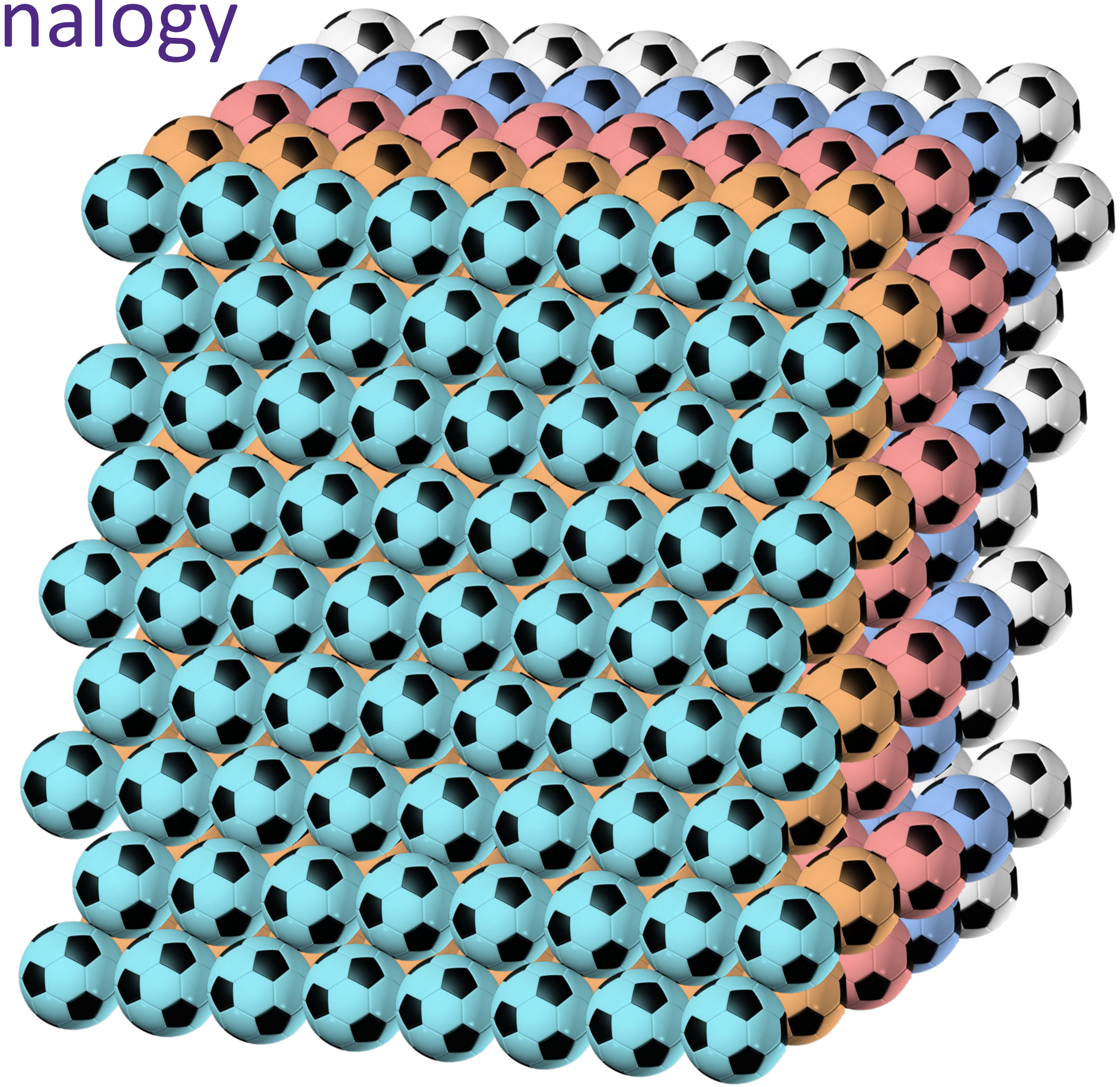


SEM and FIB Image Formation



Ion Bombardement – A Soccer Analogy

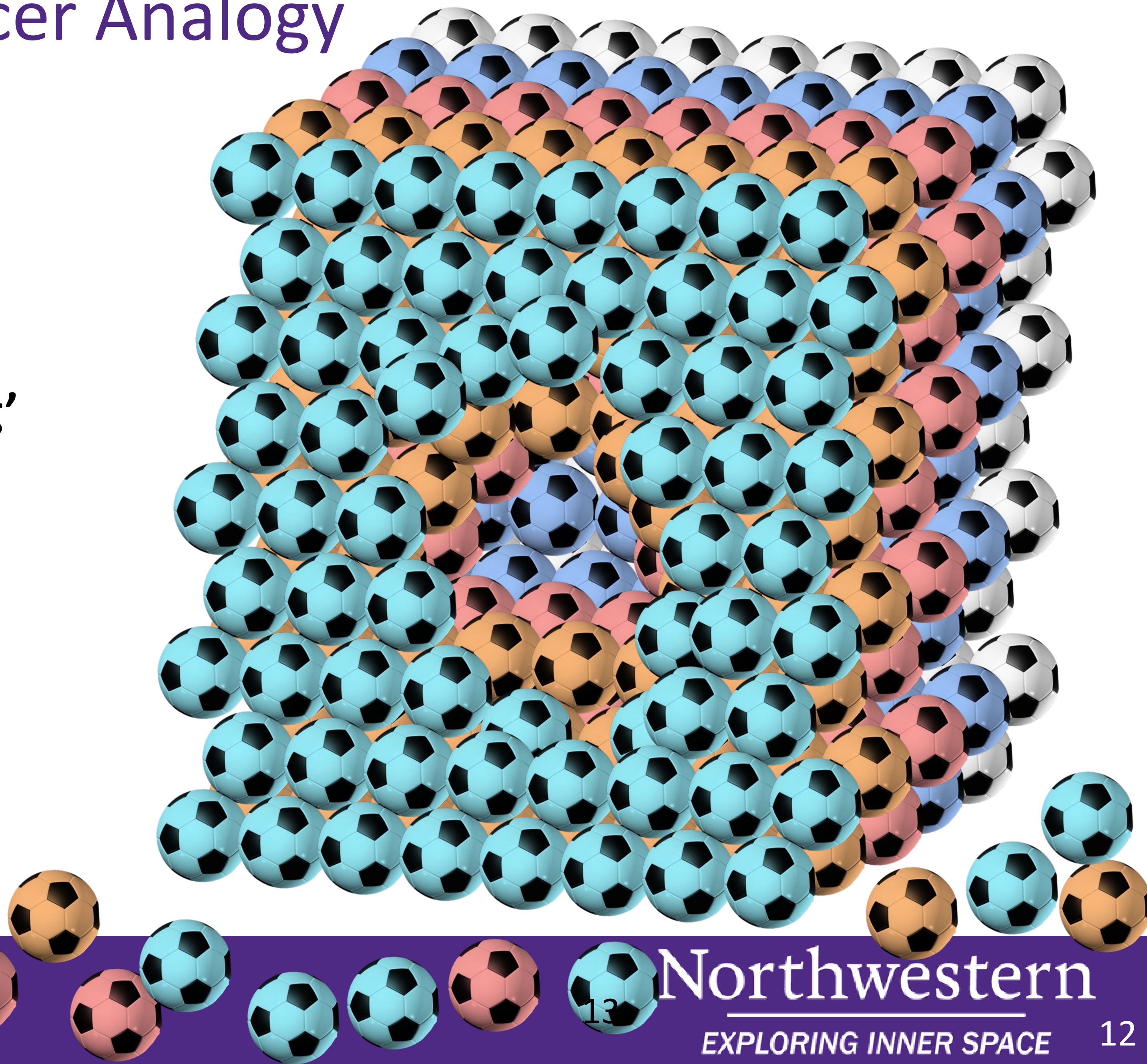
Me



Ion Bombardement – A Soccer Analogy

Me

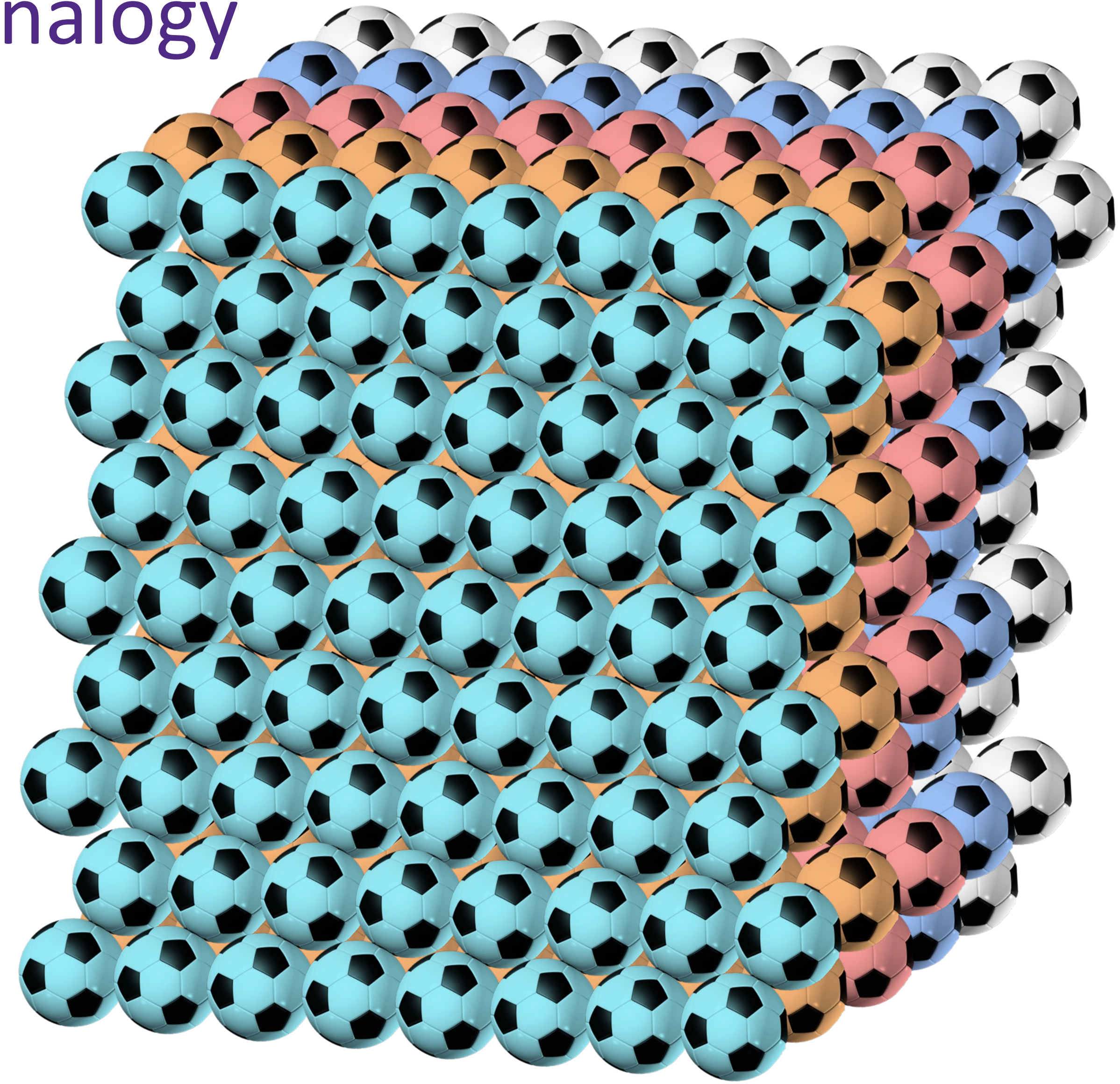
‘sputtering’
‘milling’



Ion Bombardement – A Soccer Analogy

“High Voltage”

Professional soccer player



Ion Bombardement – A Soccer Analogy

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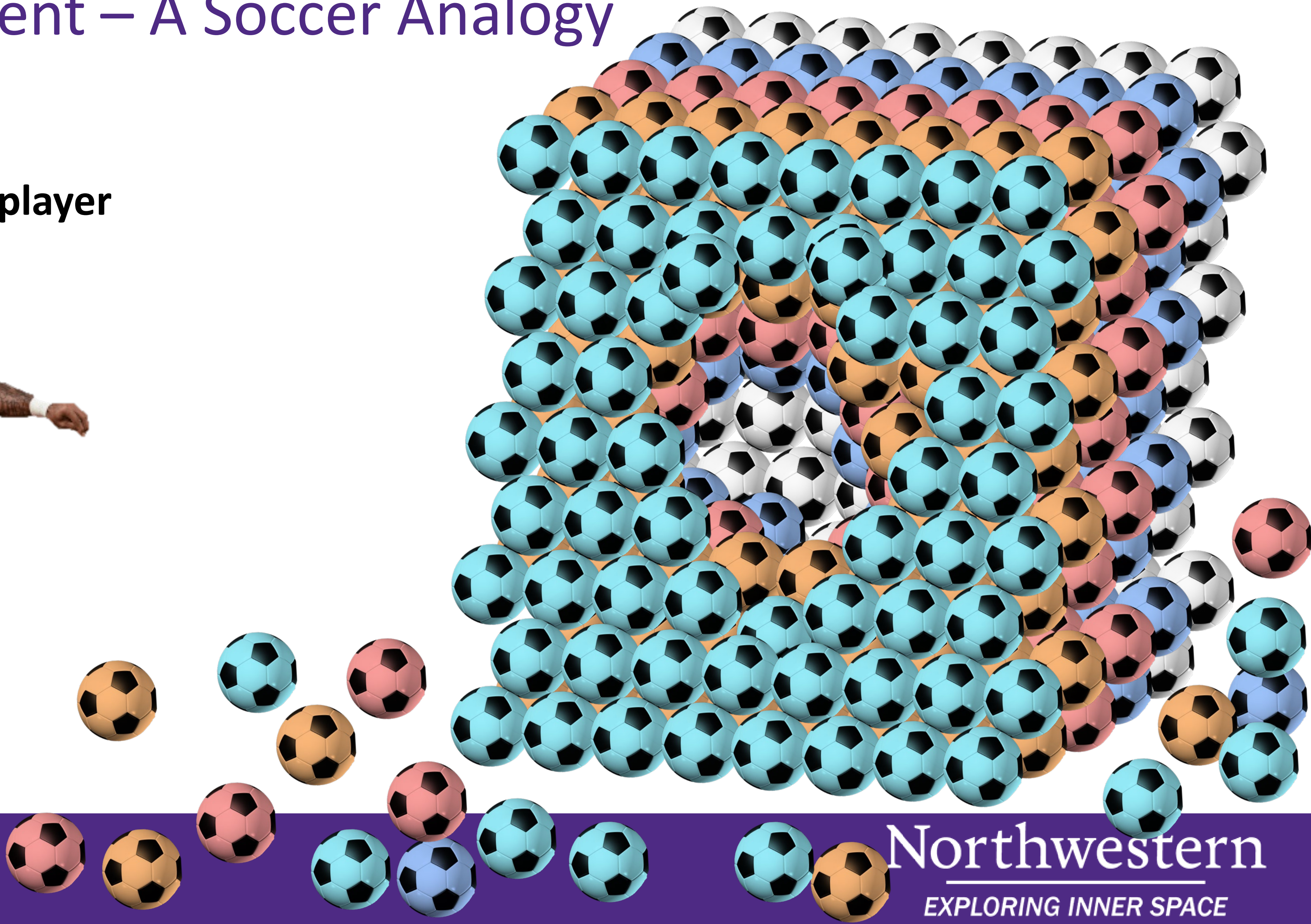
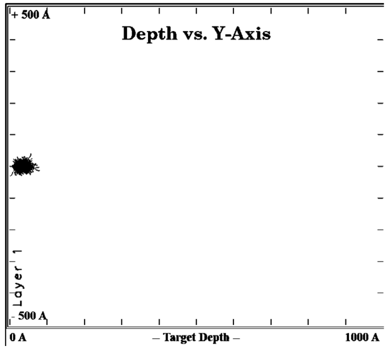


FIB-Sample Interaction – Effect of keV and Current

- Influence of altering Accelerating Voltage

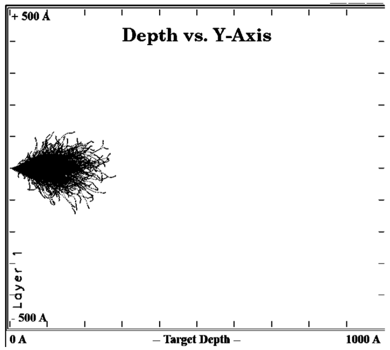
Increase keV

1 keV Gallium



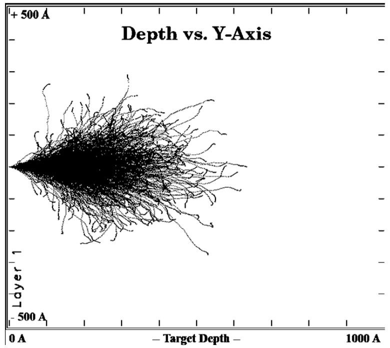
10 nm

10 keV Gallium



30 nm

30 keV Gallium

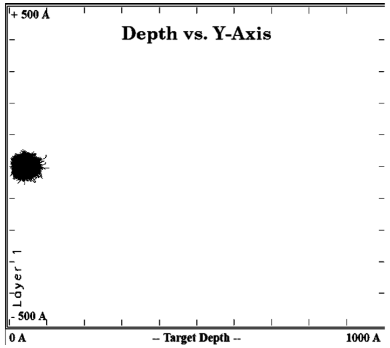


60 nm

- Influence of altering Current

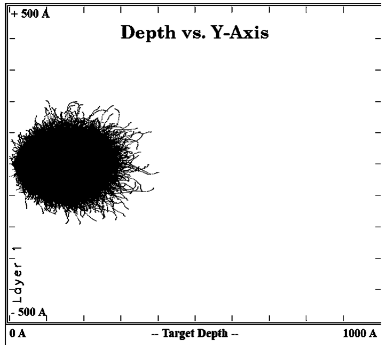
Increase current

1 keV Gallium



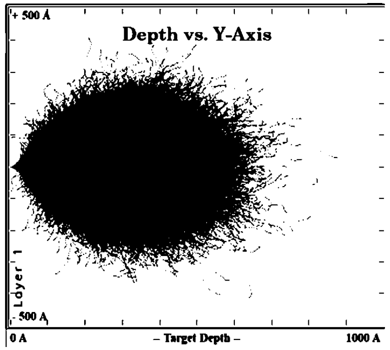
10 nm

10 keV Gallium



30 nm

30 keV Gallium

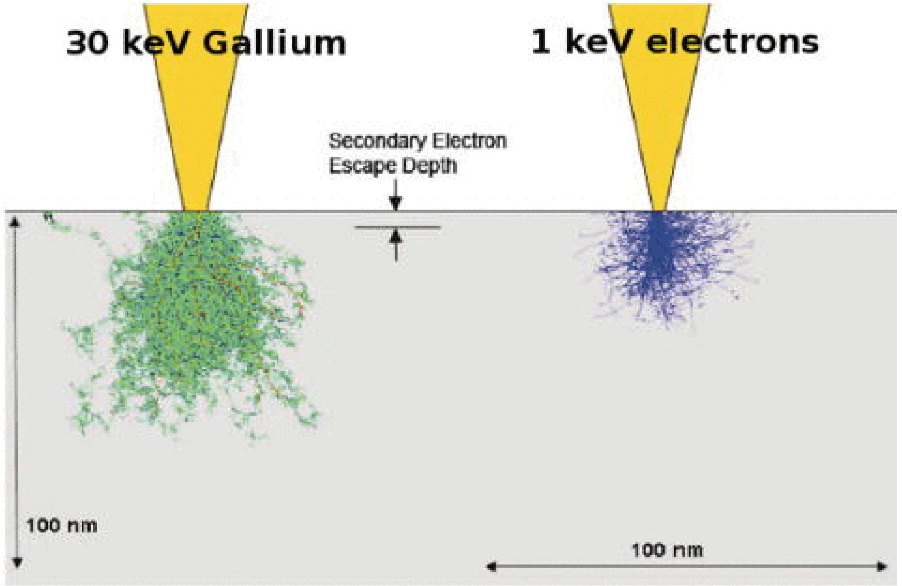


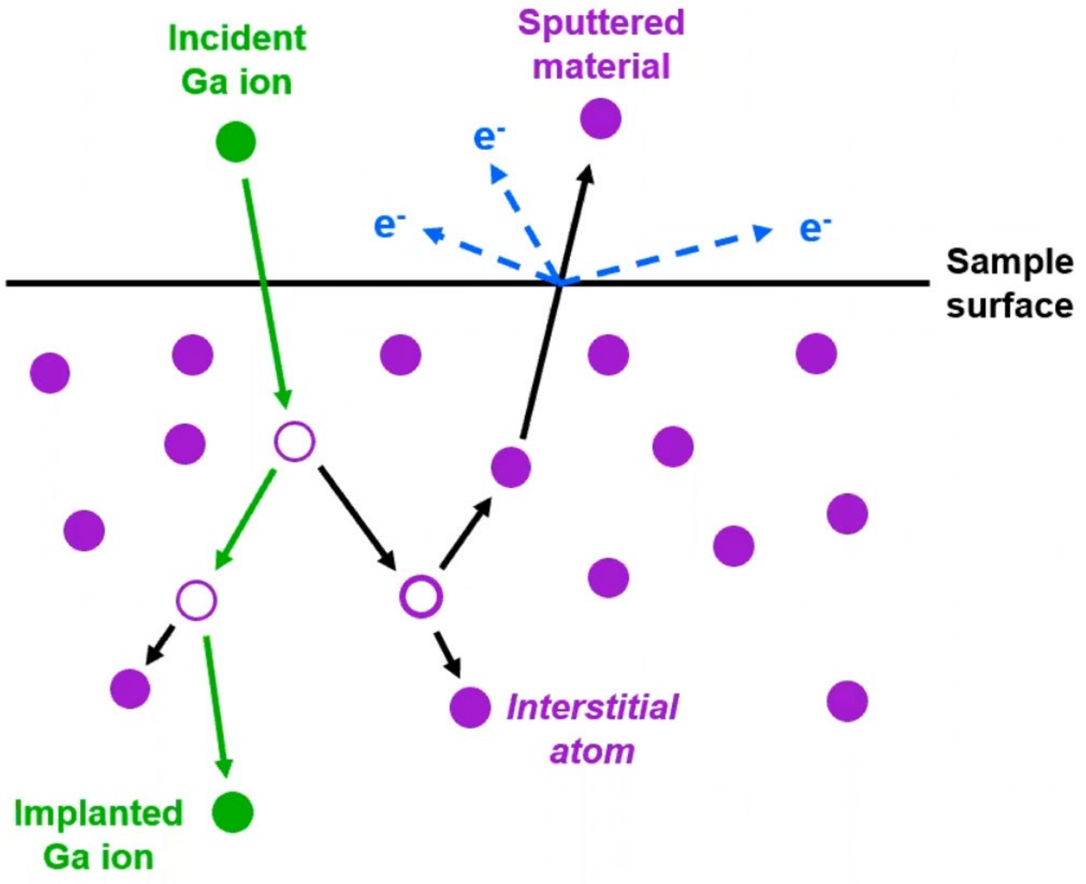
70 nm

TRIM simultations - **S**topping and **R**ange of **I**ons in **M**atter (SRIM) 2013

Why can't Electrons Sputter Atoms?

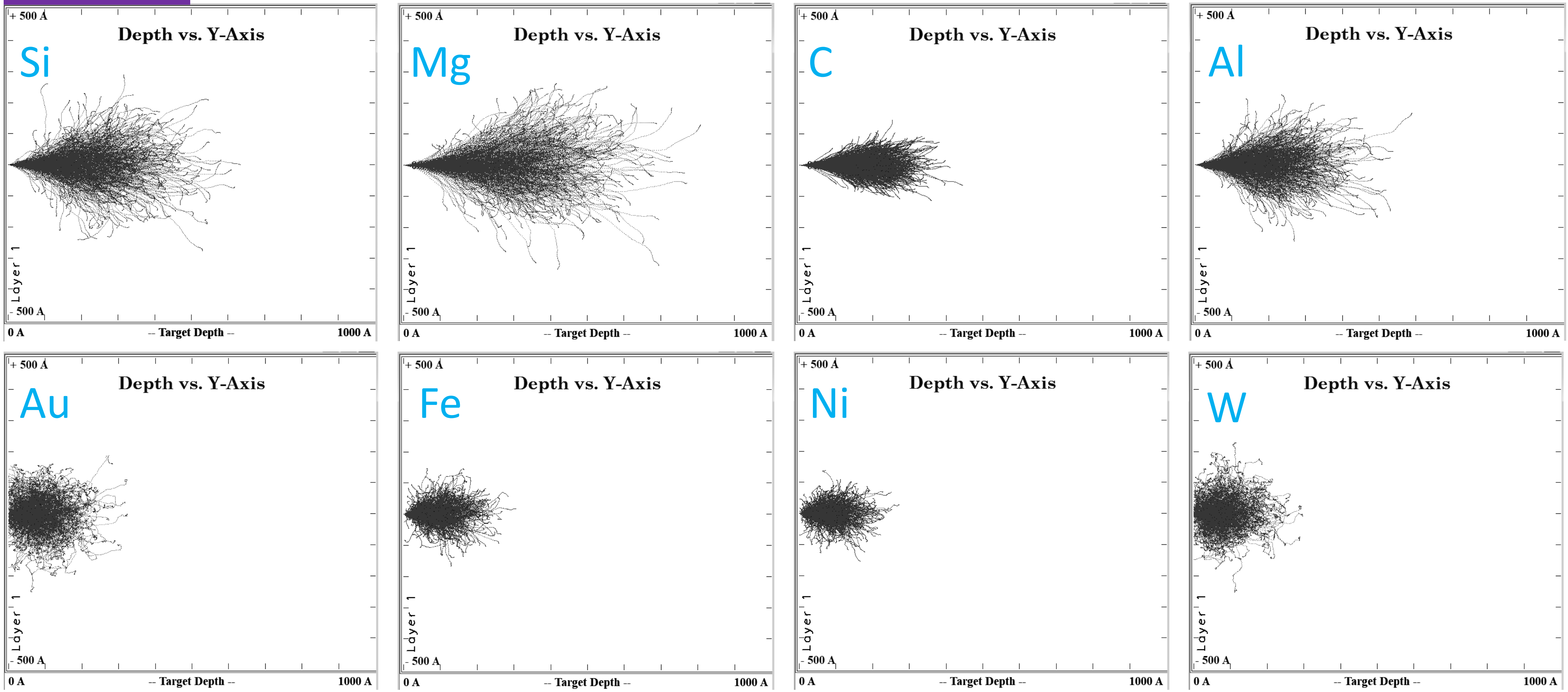
- Ga is much heavier (m_{Ga}/m_e = 1.27 E5) → much more momentum
- Ga is much, much larger than an electron





FIB-Sample Interaction – Material Dependent Penetration Depth

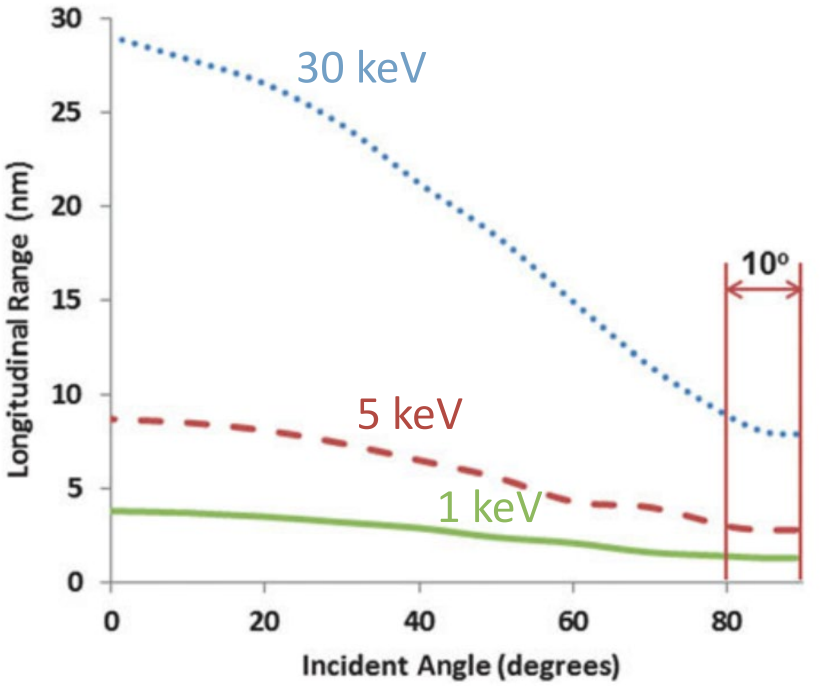
30 keV Ga$^+$



Stopping power is <u>highly</u> correlated with density and materials properties such as melting T; not directly with atomic number

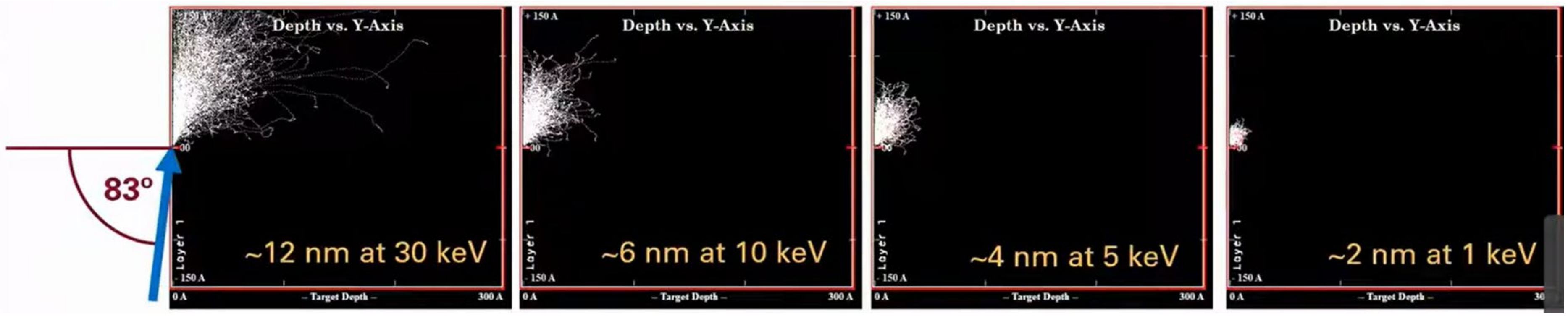
TRIM simultations - **S**topping and **R**ange of **I**ons in **M**atter (SRIM) 2013

FIB-Sample Interaction Depth – Control with Voltage & Angle



- Combination of low voltage and high angle minimizes ion penetration depth
- ~80-90 is optimal and nearly equivalent
- Especially important for final thinning of TEM Sample

x keV Ga^{+} in Ni

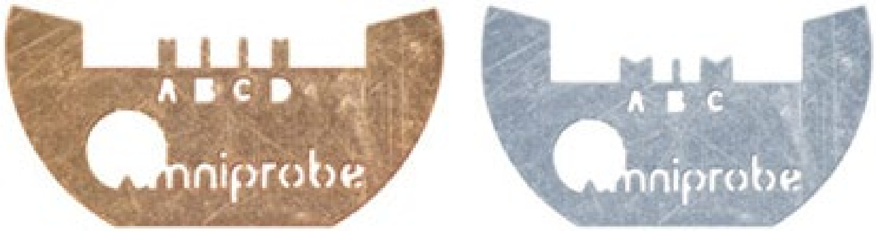


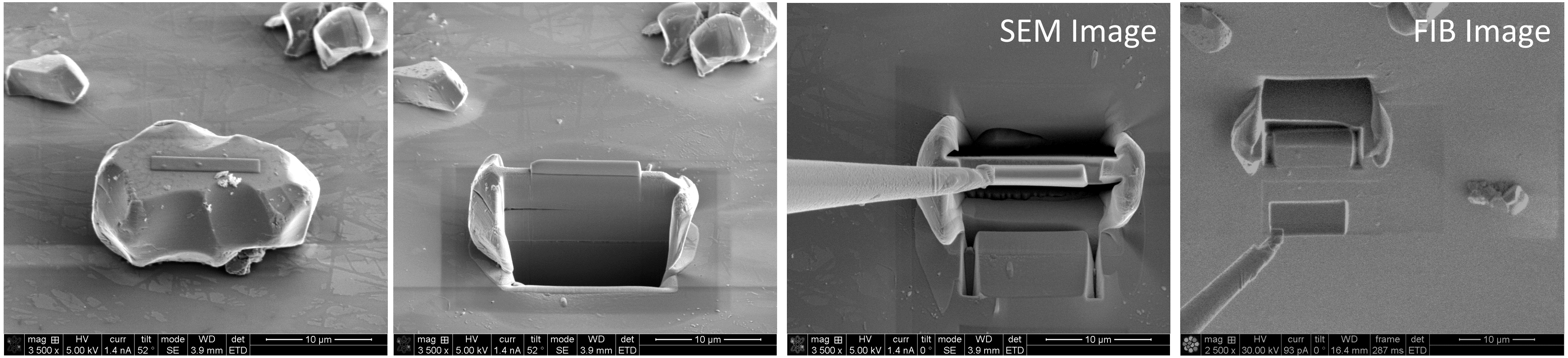
Bassim, N., Scott, K., Gianuzzi, L.A., *MRS Bulletin*, **39** (2014)

TEM Sample Preparation using FIB-SEM

Processes used in Site-Specific TEM Sample Prep

- SEM and FIB **Imaging**
- **Deposition**: Solid protection layer of ROI using a <u>G</u>as <u>I</u>njection <u>S</u>ystem (GIS)
- **Ion Milling**: Bulk material removal & sample thinning
- ***In Situ* Liftout** of TEM Lamella using W micromanipulator
- Attachment of lamella to a **TEM (half)grid**
  - Grids can be made from various materials: **Cu, Mo,** Au, Be, Si, Ni…





FIB-Sample Interaction – Control Processes with Current

- Ga FIB has Gaussian shape
  - Lower current narrower beam distribution (higher spatial res)
  - Higher current broader beam distribution (lower spatial res)

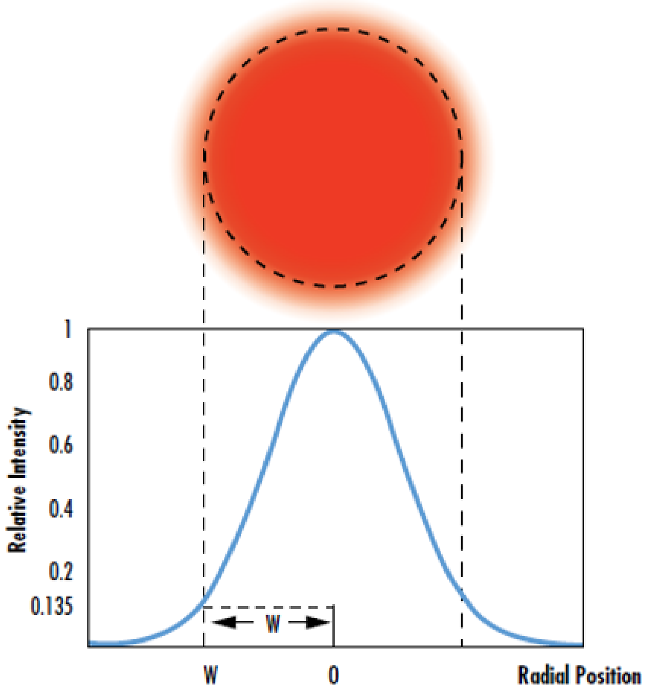
- Lower current for precise milling; higher current for faster milling

- Control of currents through aperture selection

(rough) milling: beams 01–08

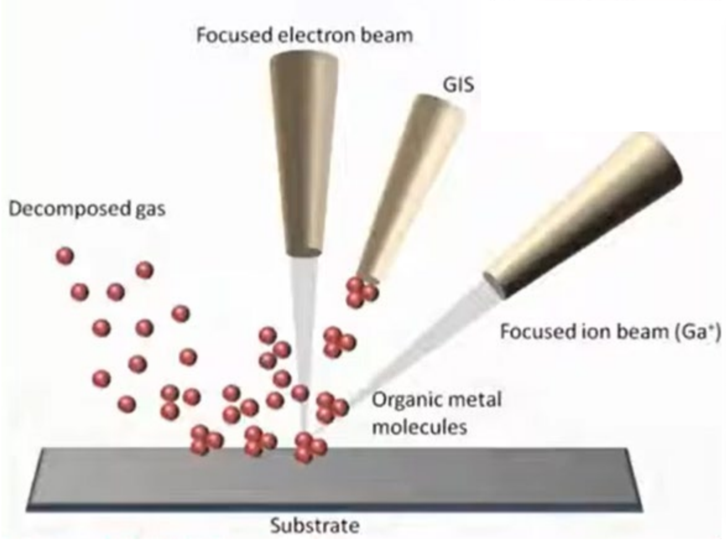
imaging & deposition / thinning / liftout: beams 09–13

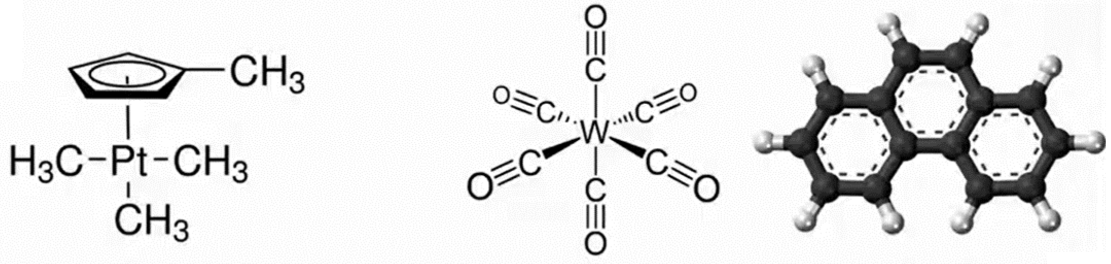
Beam number	01	02	03	04	05	06	07	08	09	10	11	12	13
Ip (pA)	90.000	50.000	30.000	10.000	3.000	1.000	500	300	100	30	10	3	≤ 1



Deposition using GIS

- Solid material deposition at site-specific location through precursor gas injection
- Through **e-beam** or **i-beam** interaction with the surface, adsorbed molecules decompose into volatile fragments carried away by vacuum system, and a metallic deposit remains
- Gas source needs to adsorb readily on surfaces and decompose faster than it sputters away
- Usually **Pt**, **W** or **C**

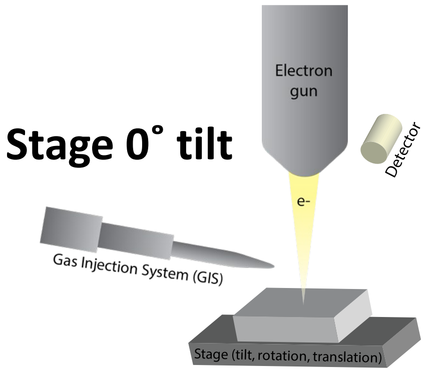




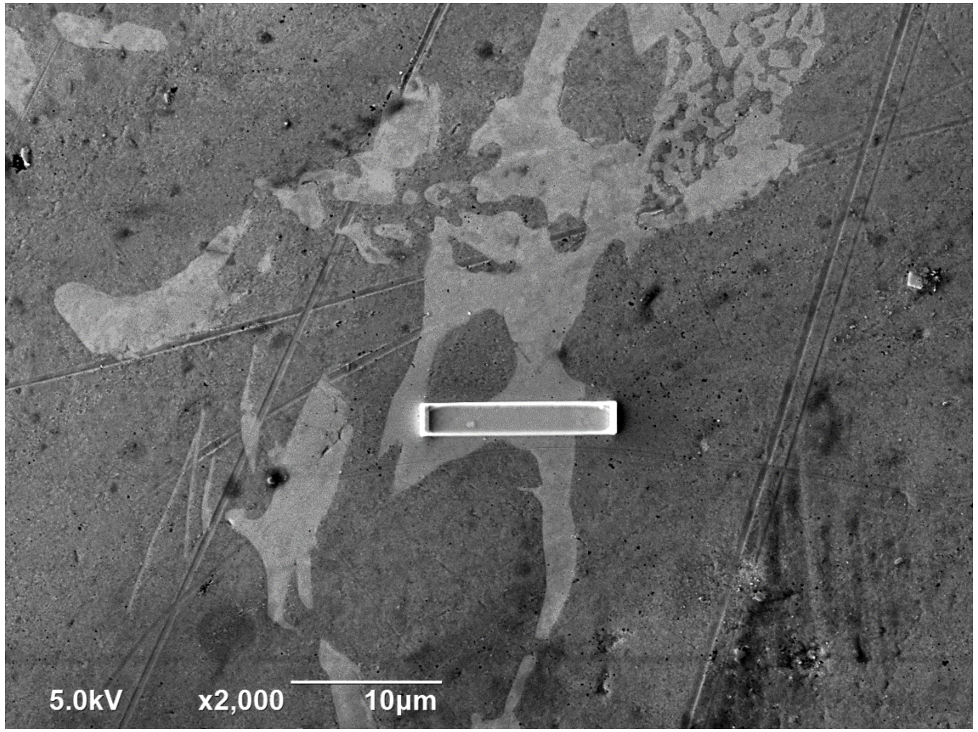
Platinum:
Trimethyl(methylcyclopentadienyl) platinum(IV)
$C_5H_4CH_3Pt(CH_3)_3$

Tungsten:
Tungsten Hexacarbonyl
$W(CO)_6$

Carbon:
Phenanthrene
$C_{14}H_{10}$

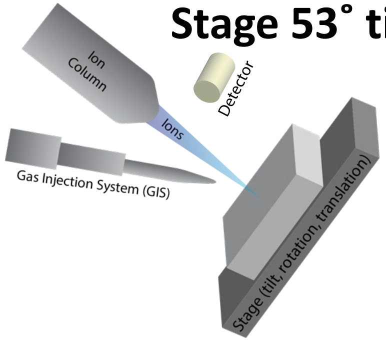


TEM Sample Prep: Conventional TEM Lamella



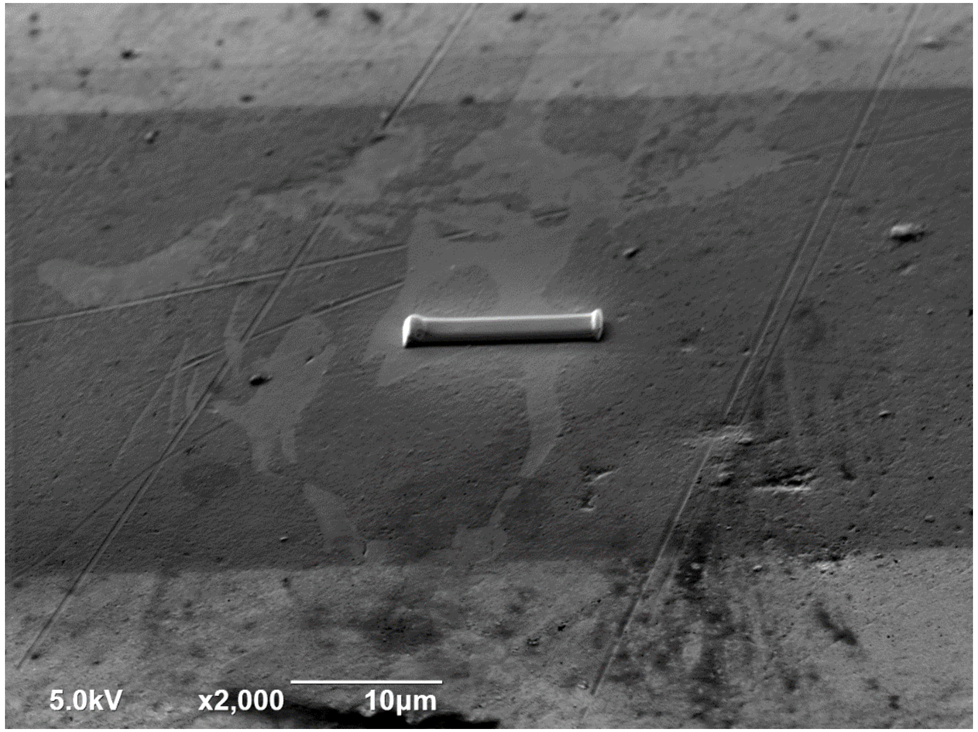
i) Electron beam-assisted Pt deposition (Pt edep)

5 kV, 10 pA → **~300 nm Pt layer**

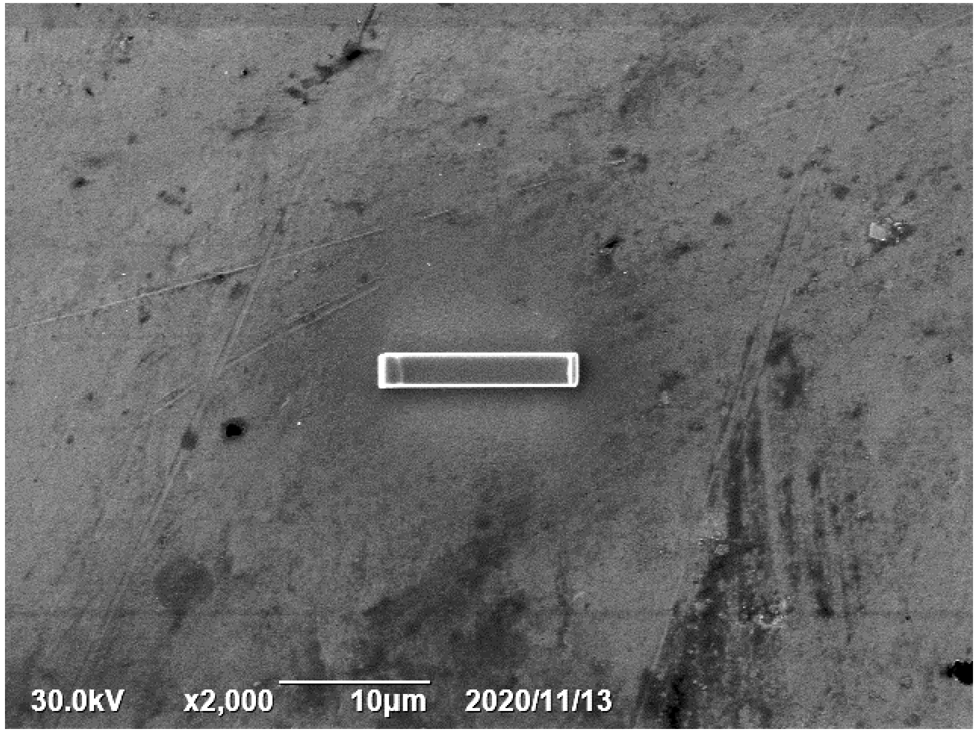


TEM Sample Prep: Conventional TEM Lamella

SEM image



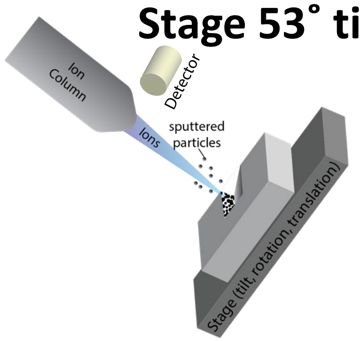
FIB image



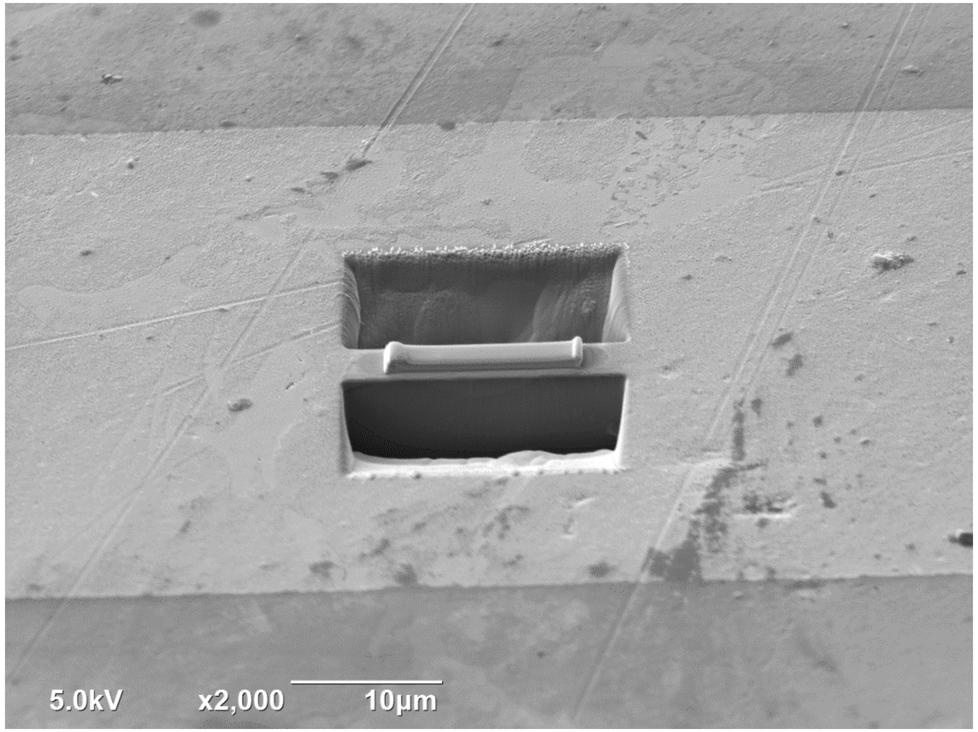
ii) Ion beam-assisted Pt deposition (Pt dep)

*30 kV, 30 pA → **~1 µm Pt layer**

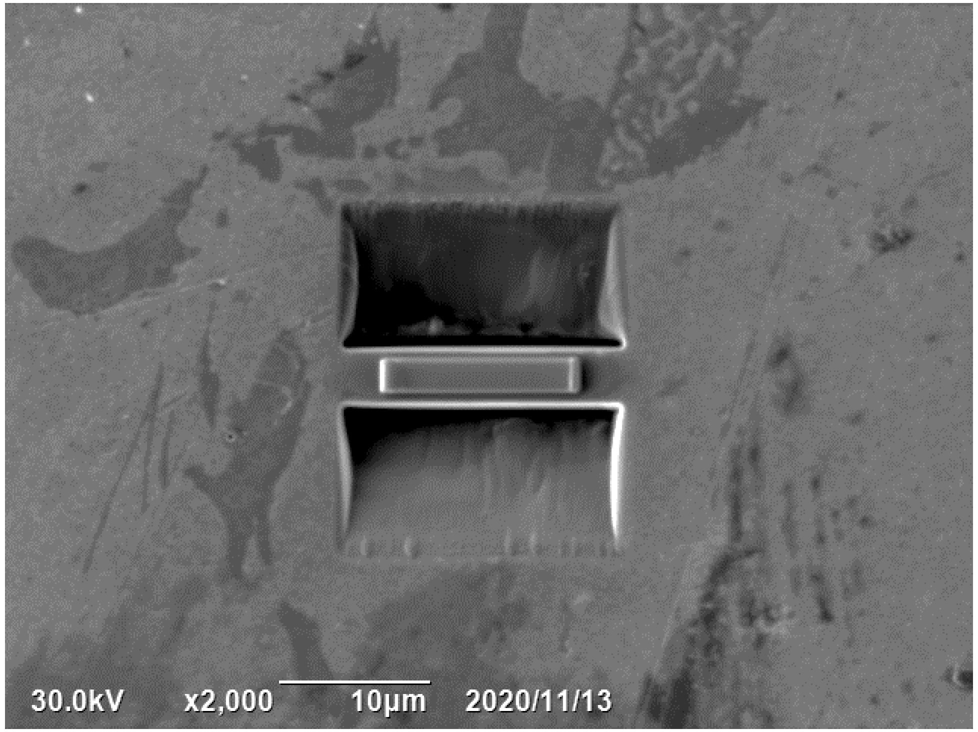
TEM Sample Prep: Conventional TEM Lamella



SEM image



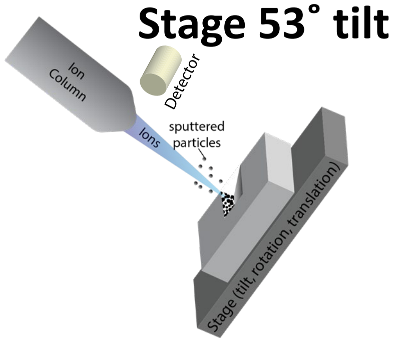
FIB image



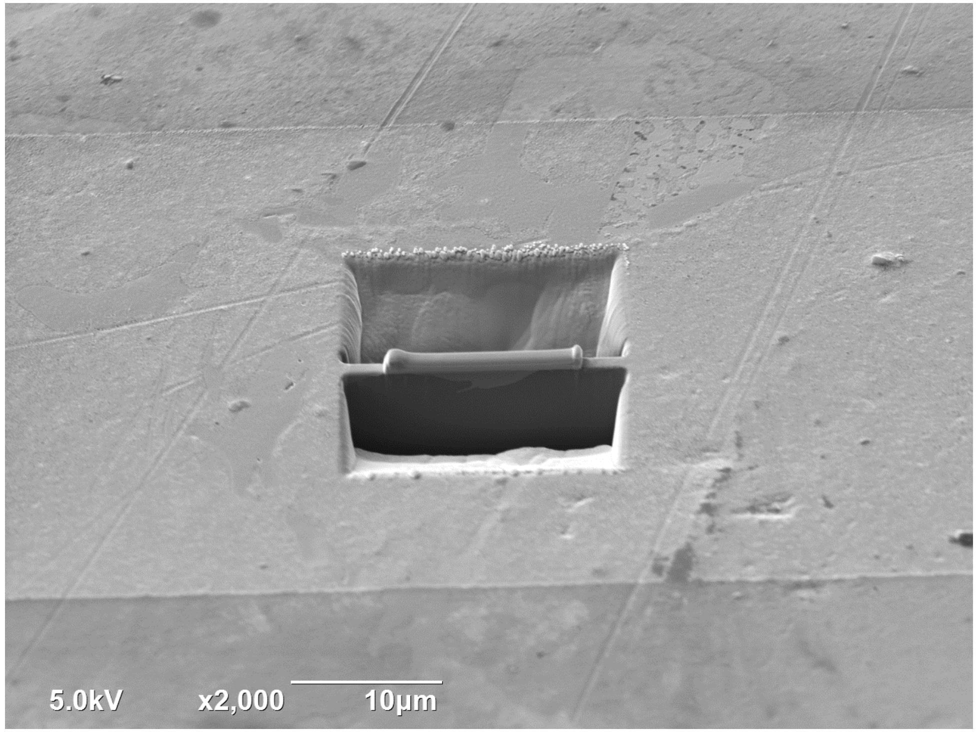
iii) Bulk out (×2)

*30 kV, 6.5 nA

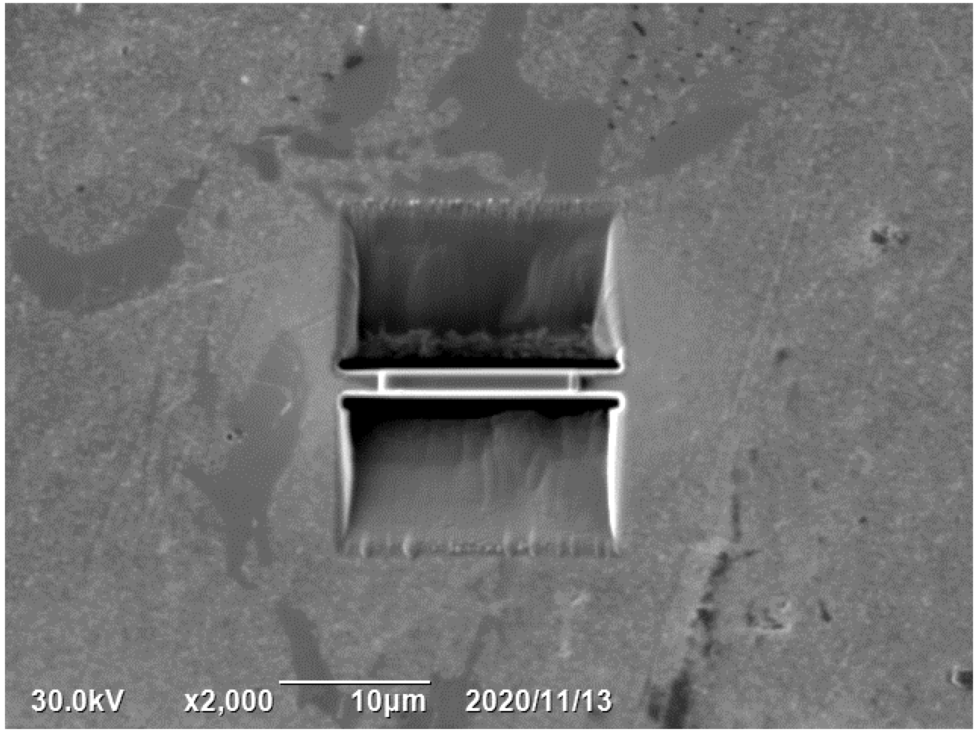
TEM Sample Prep: Conventional TEM Lamella



SEM image

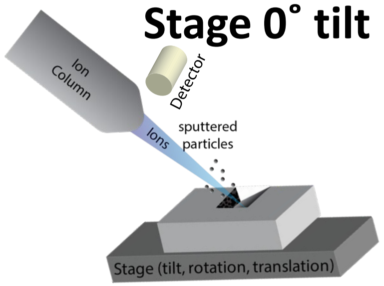


FIB image



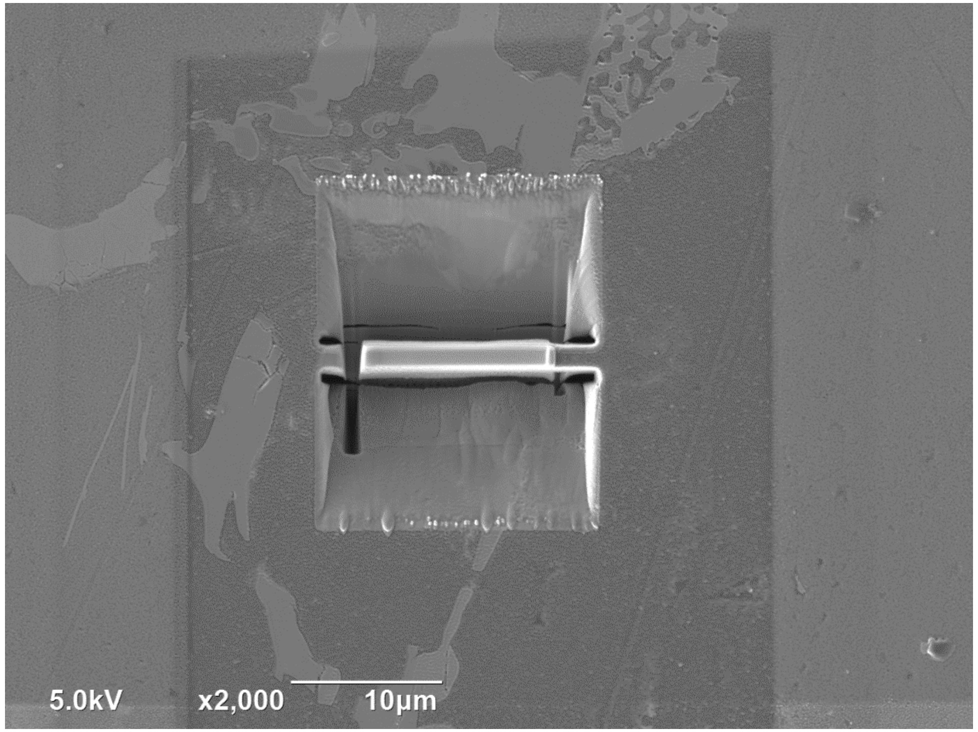
iv) Clean-up edges (×2)

*30 kV, 3 nA→ ~**1.5 µm thick** lamella

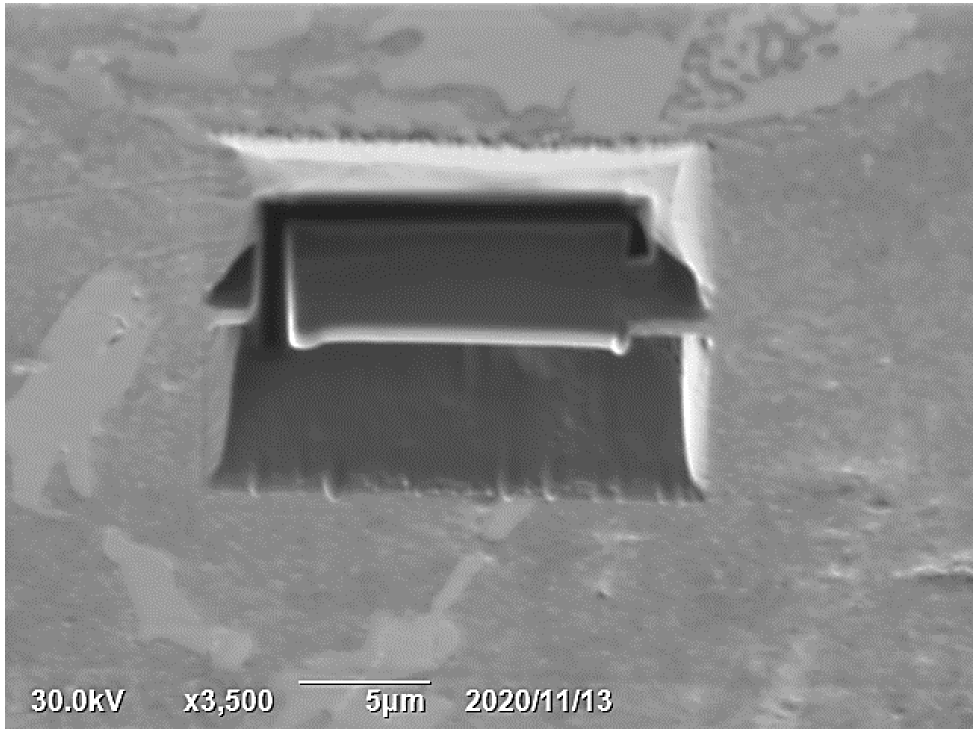


TEM Sample Prep: Conventional TEM Lamella

SEM image



FIB image

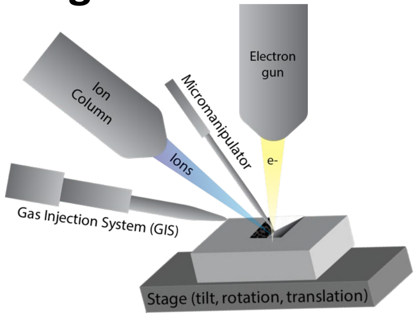


v) J-cut

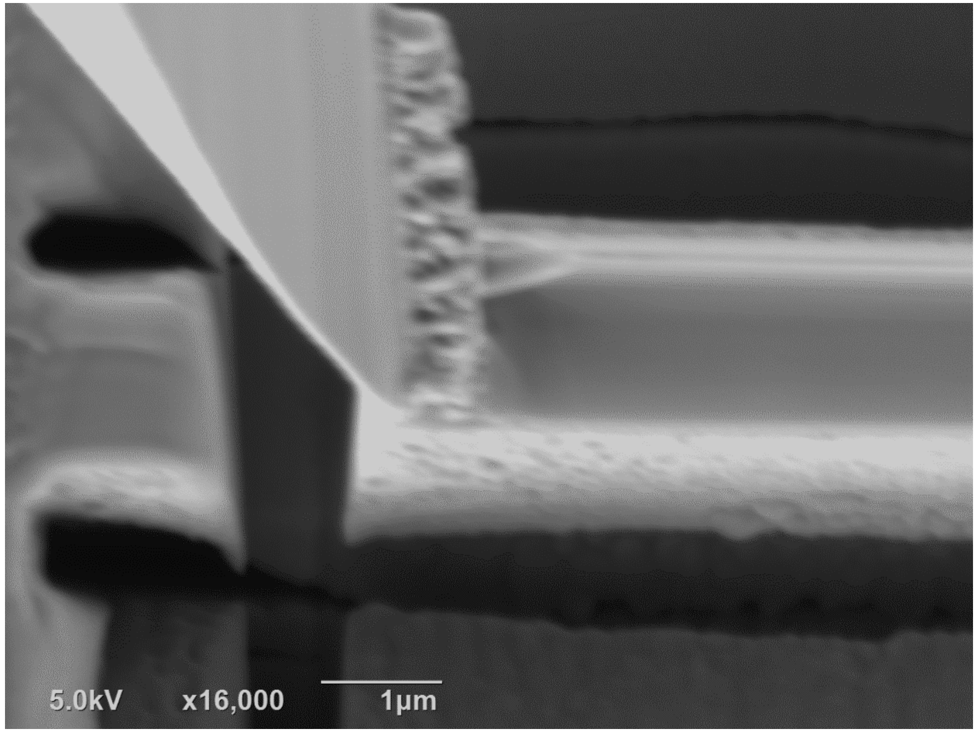
*30 kV, 3 nA

TEM Sample Prep: Conventional TEM Lamella

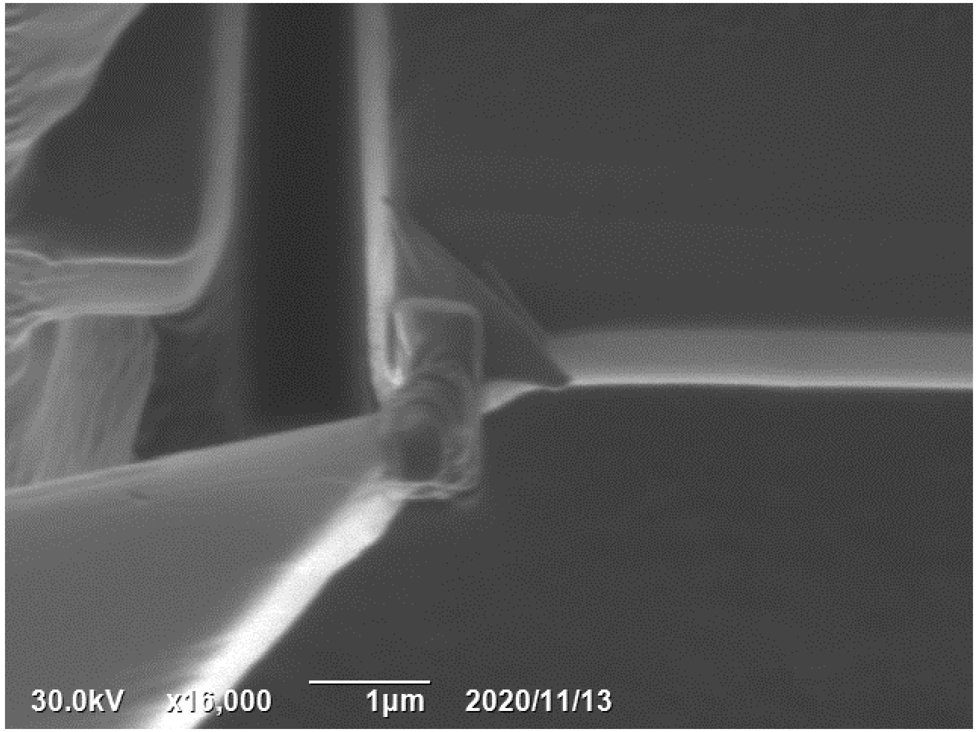
Stage 0˚ tilt



SEM image

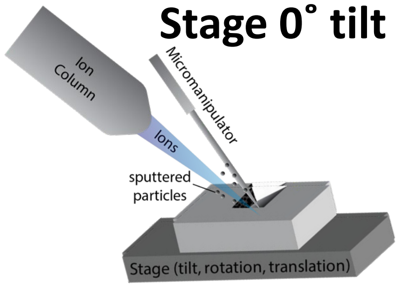


FIB image



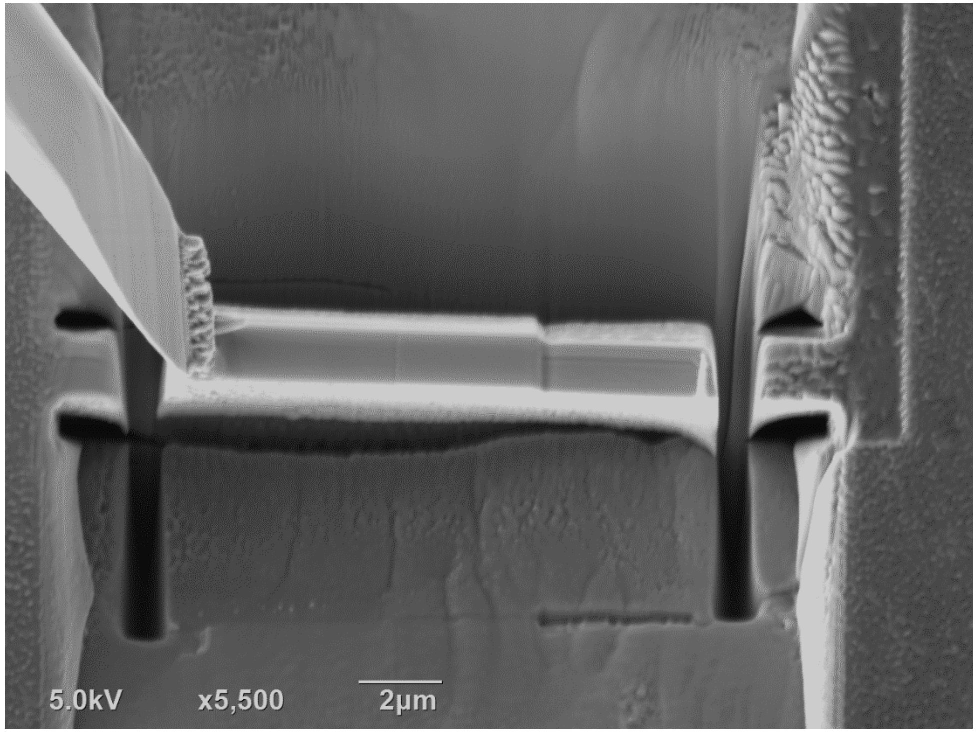
vi) Weld Omniprobe to sample

*30 kV, 30 pA → **~0.2 µm Pt layer**

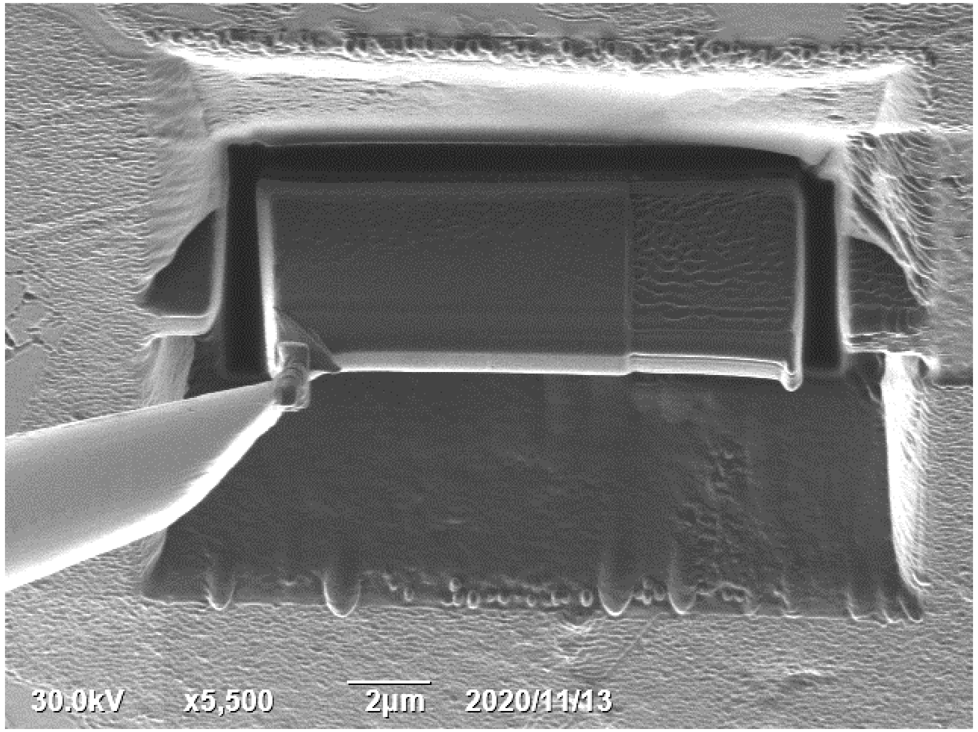


TEM Sample Prep: Conventional TEM Lamella

SEM image



FIB image



vii) Mill dangling side

*30 kV, 3 nA

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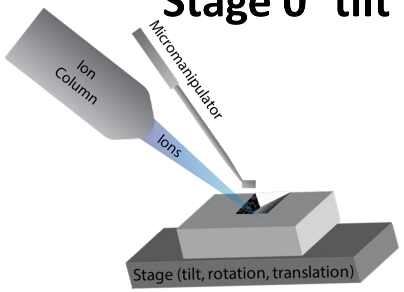
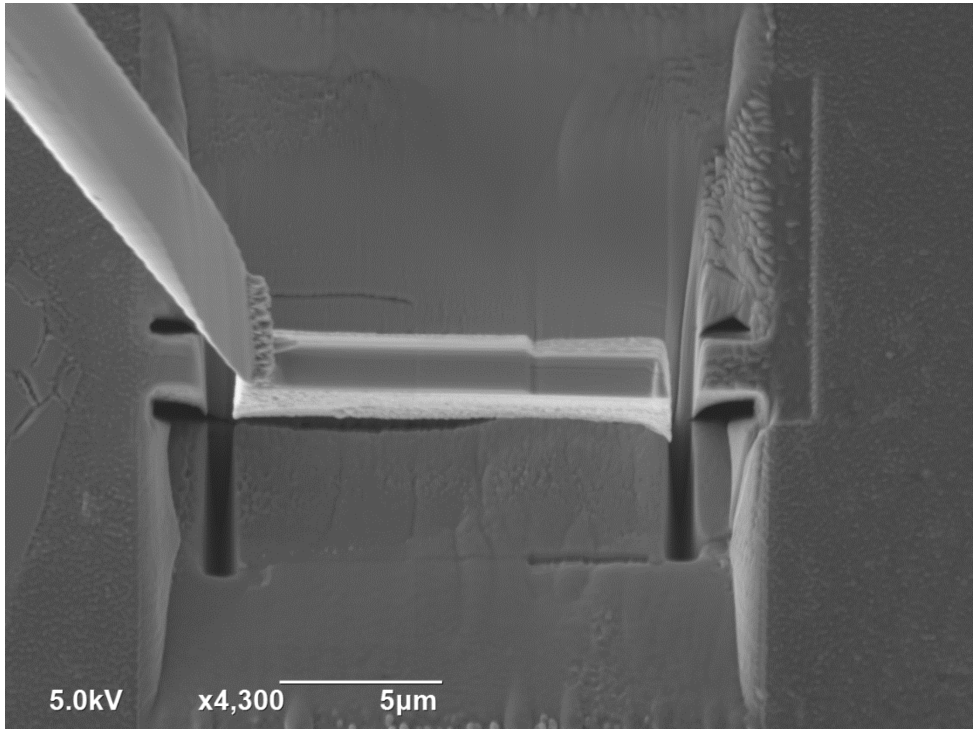
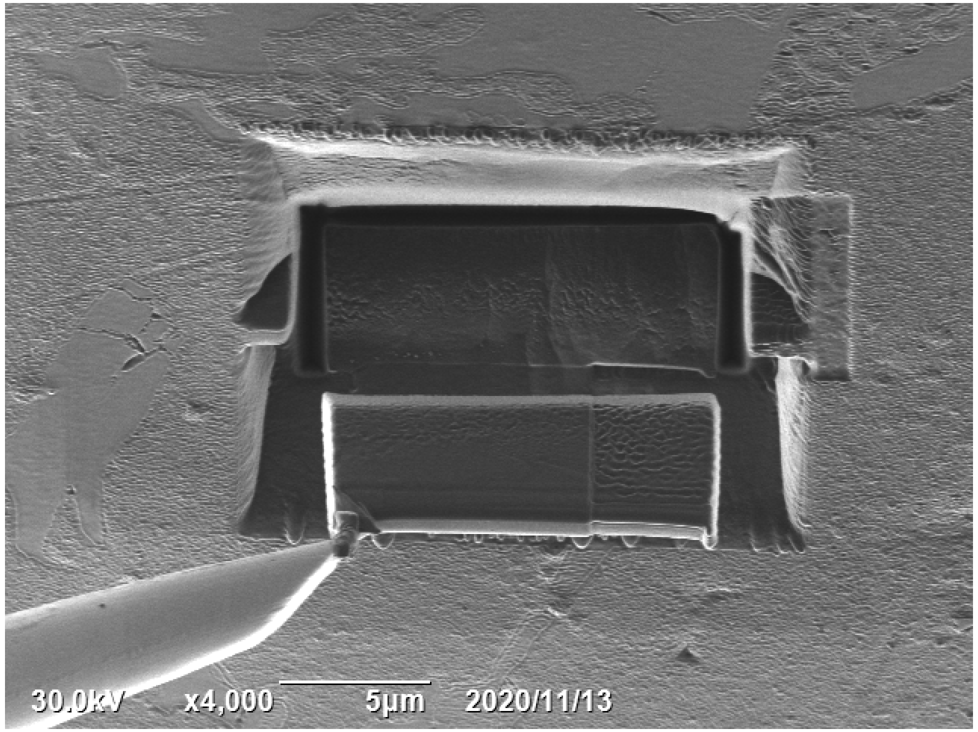


SEM image



FIB image

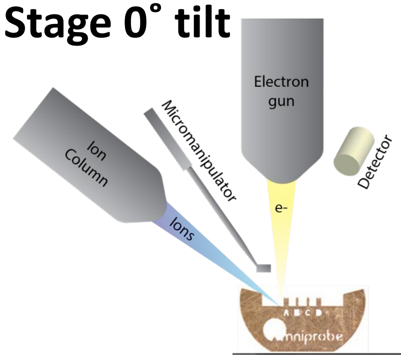


viii) Liftout using Omniprobe

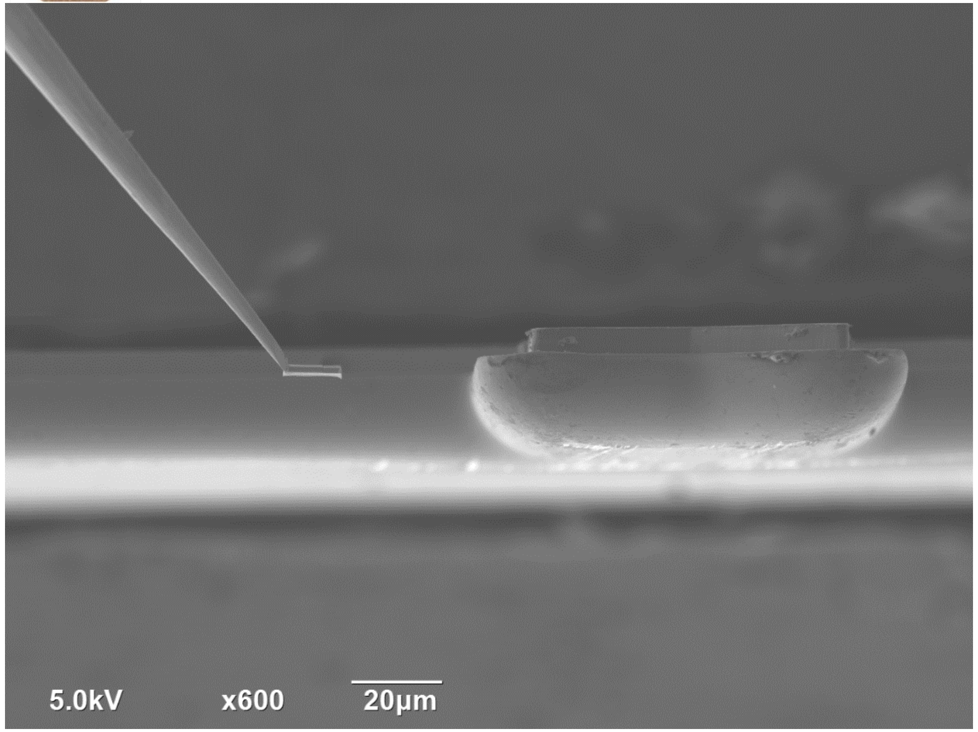
*30 kV, 30 pA

TEM Sample Prep: Conventional TEM Lamella

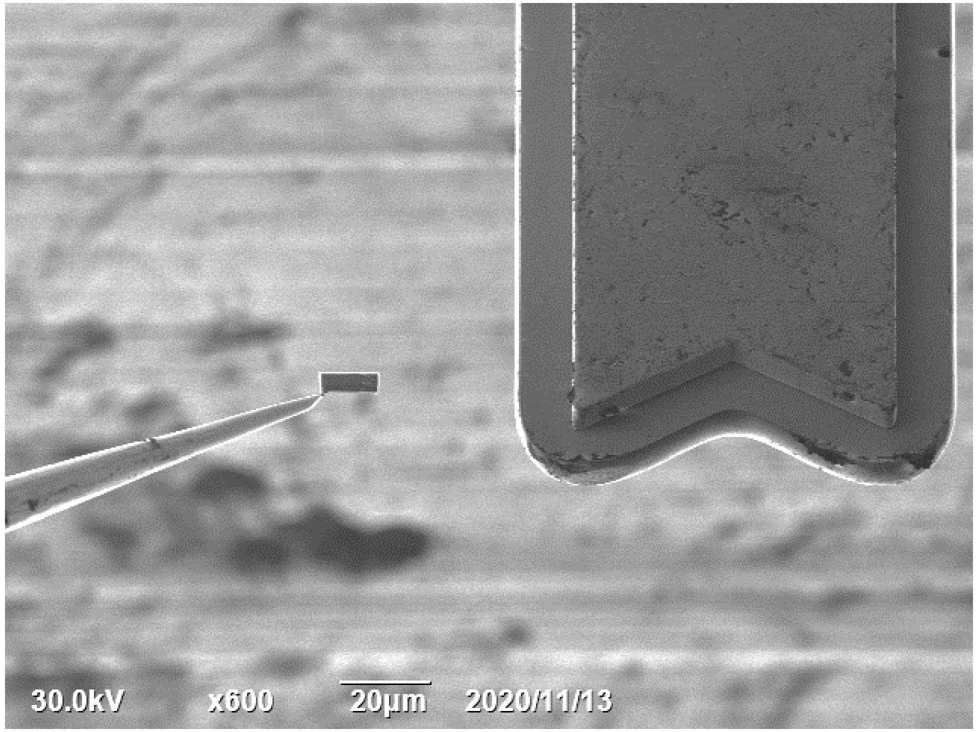
Stage 0˚ tilt



SEM image



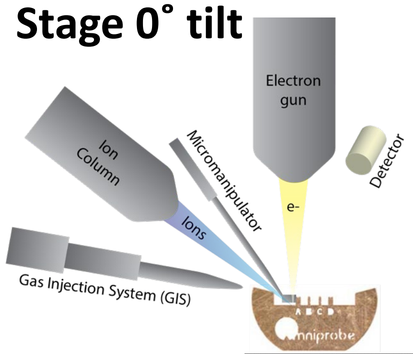
FIB image



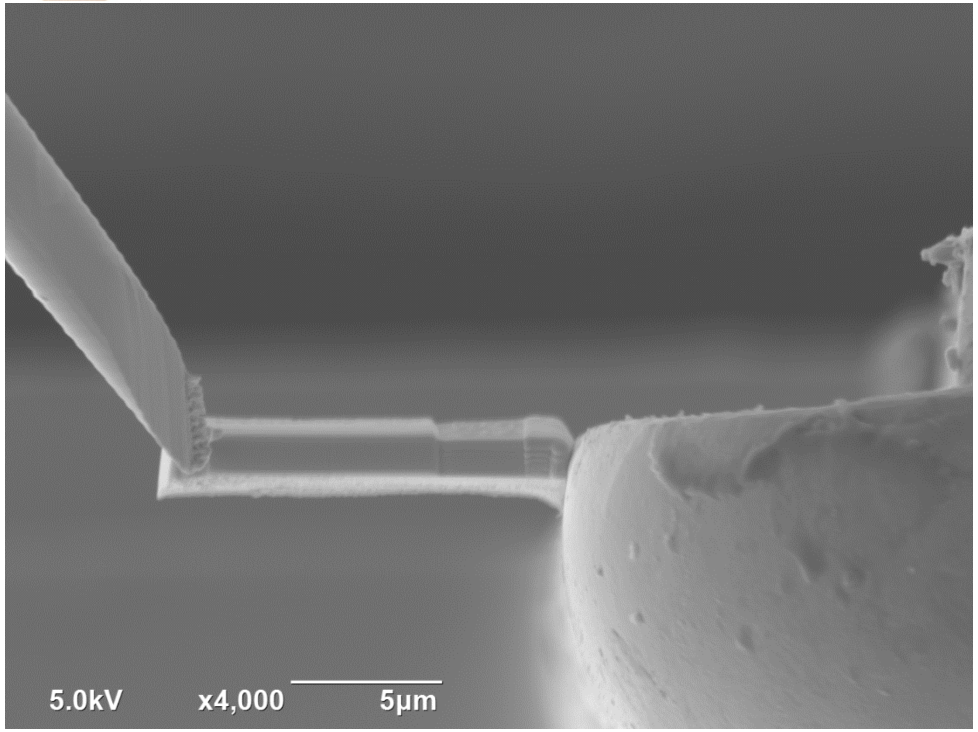
ix) Omniprobe approach to TEM half-grid (Cu)

*30 kV, 30 pA

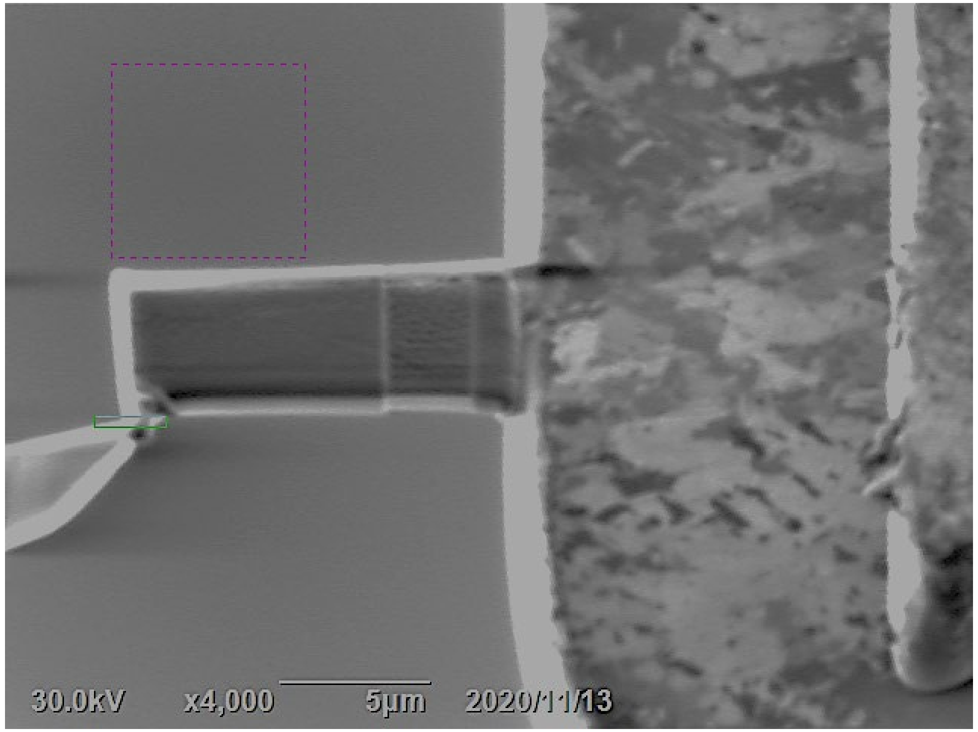
TEM Sample Prep: Conventional TEM Lamella



SEM image



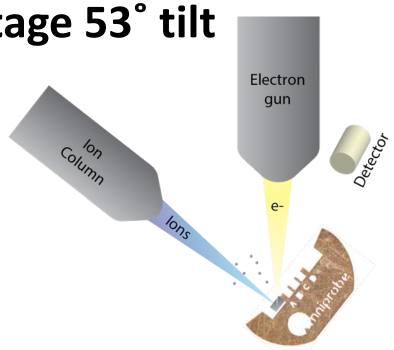
FIB image



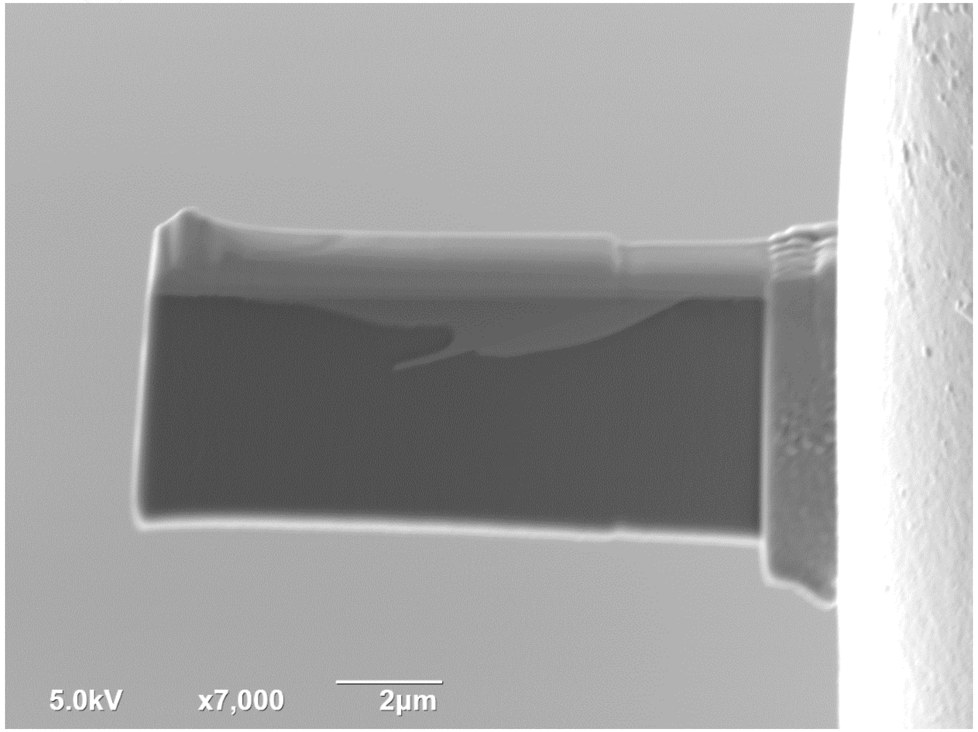
ix) TEM Lamella attachment to TEM half-grid (Cu)

TEM Sample Prep: Conventional TEM Lamella

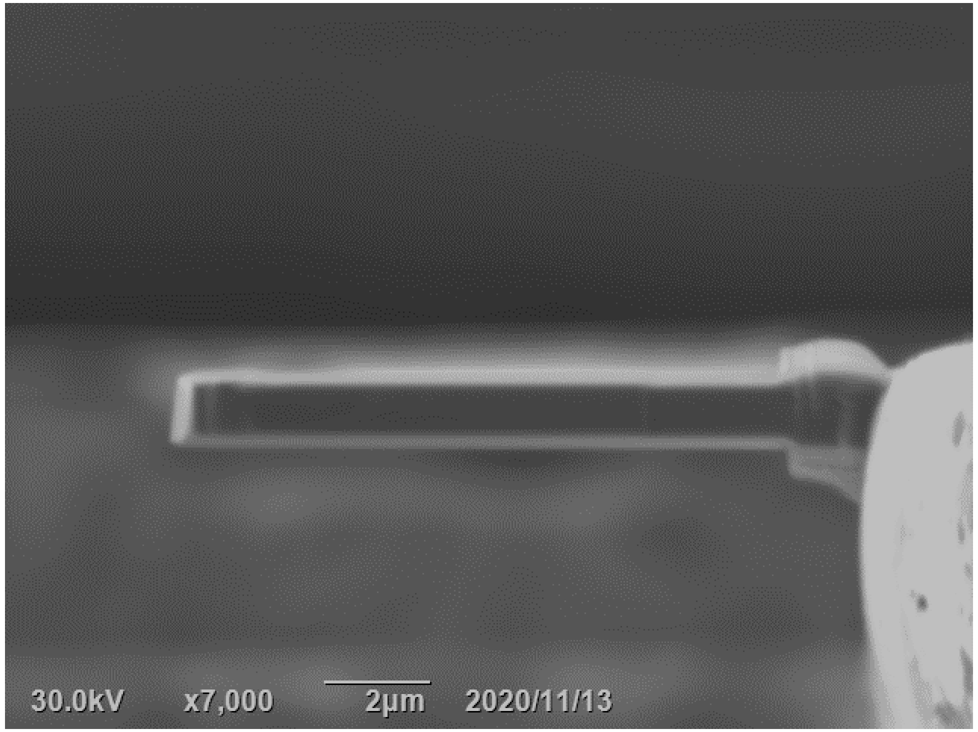
Stage 53° tilt



SEM image



FIB image

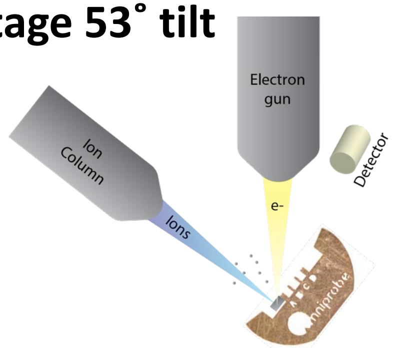


x) Trim lamella (×2)

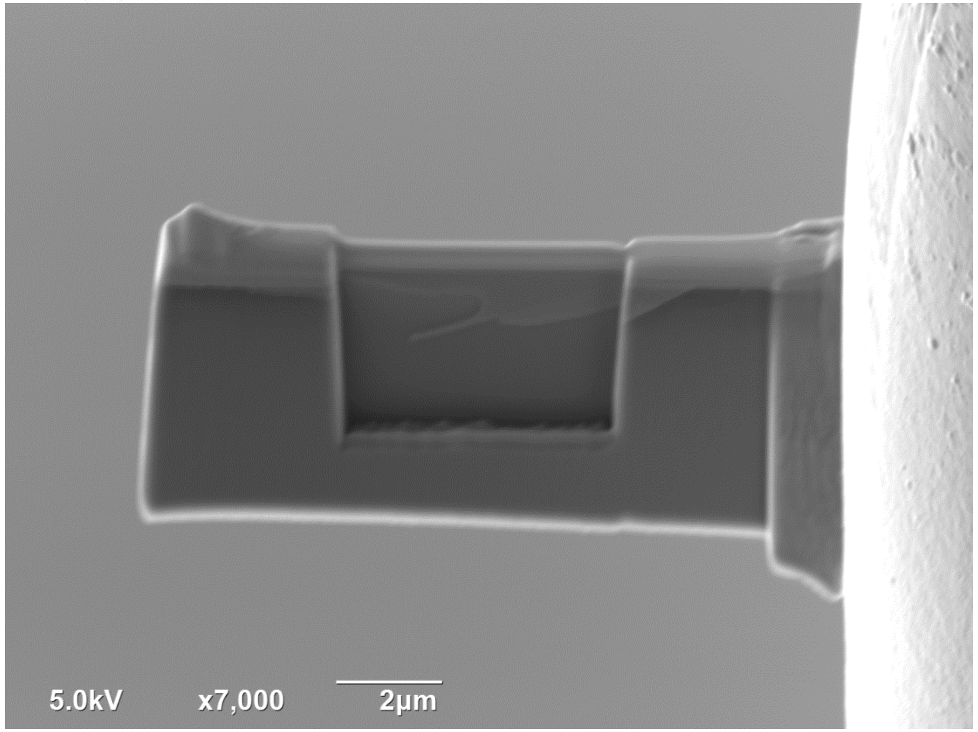
*30 kV, 500 pA

TEM Sample Prep: Conventional TEM Lamella

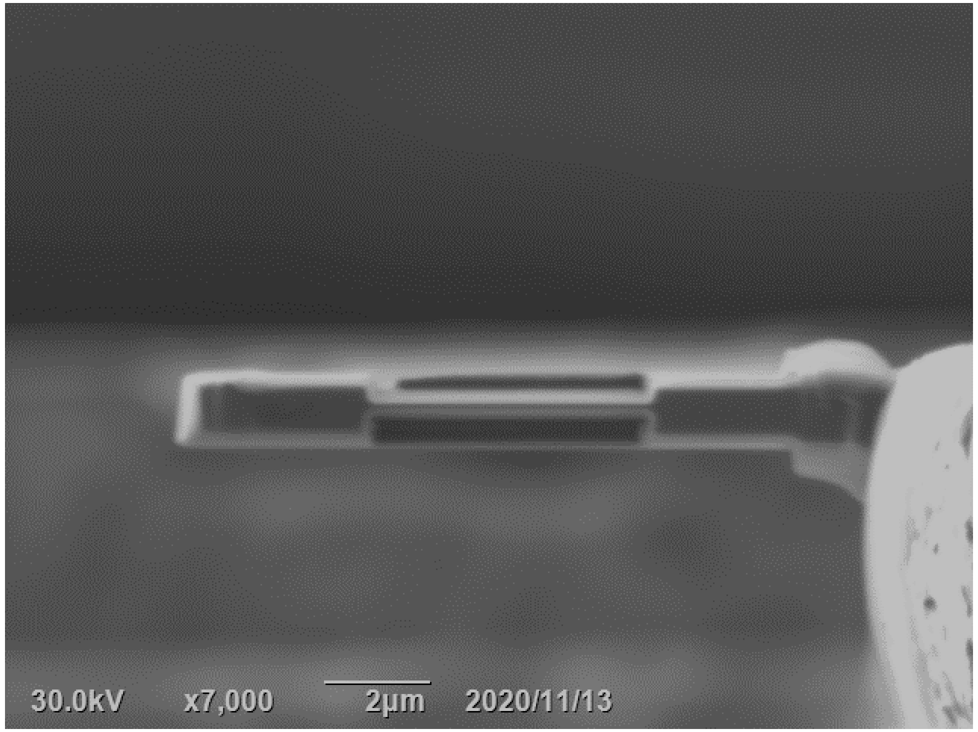
Stage 53° tilt



SEM image



FIB image

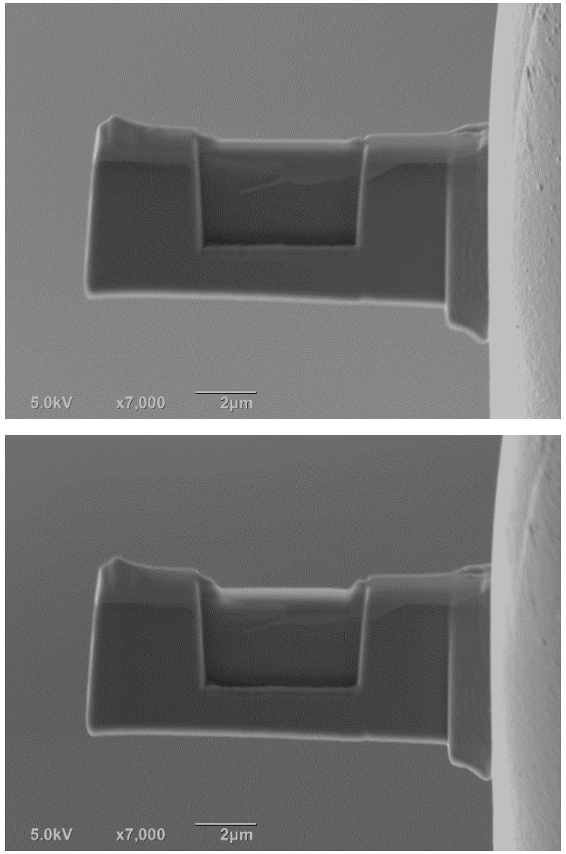


xi) **Mill window (×2)**

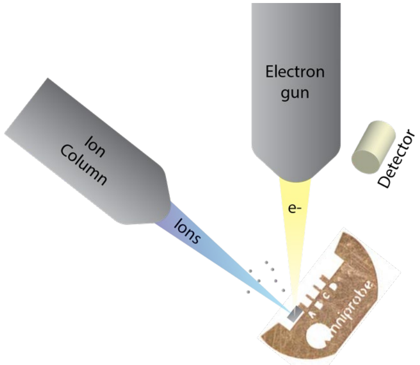
*30 kV, 500 pA

TEM Sample Prep: Conventional TEM Lamella

SEM image

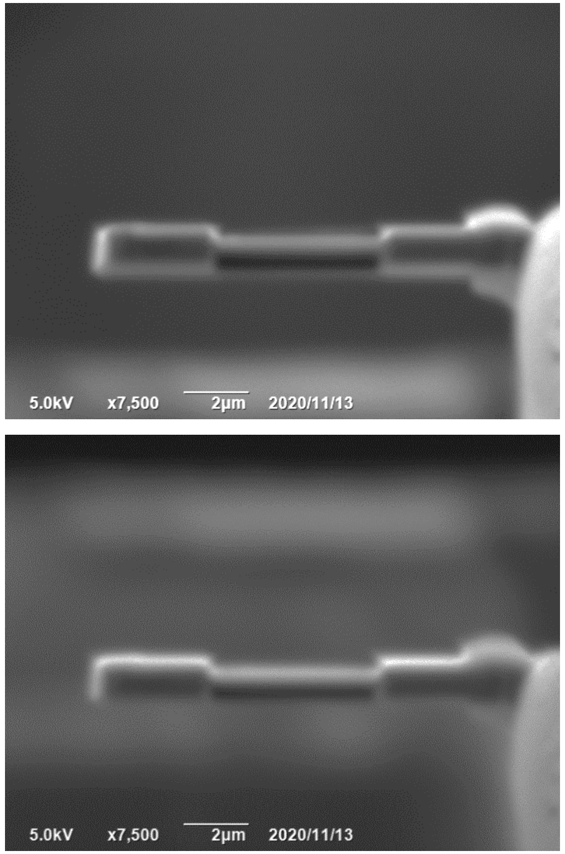


53˚ + 1.5˚ tilt



53˚ - 1.5˚ tilt

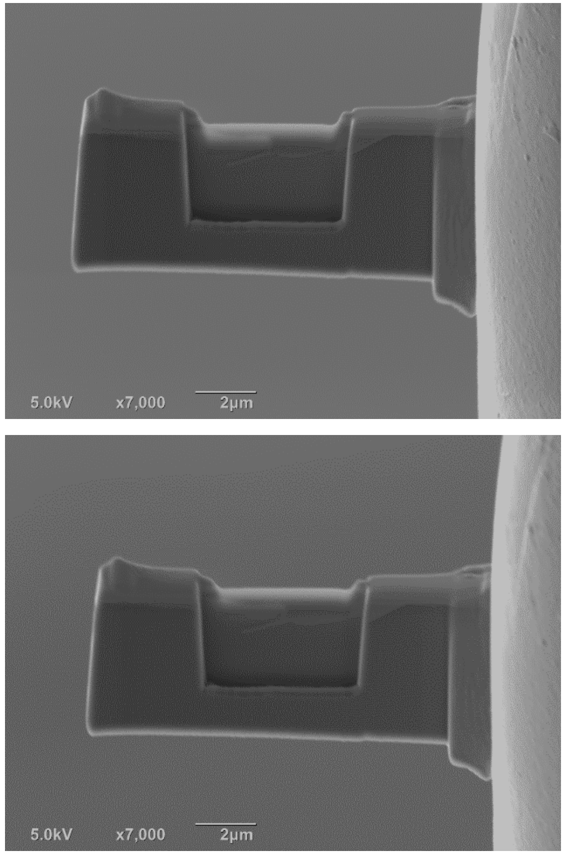
FIB image



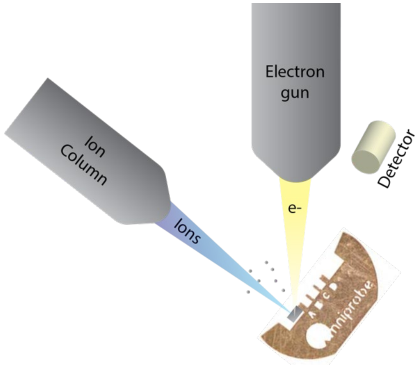
xii) Low energy window milling (×2)

TEM Sample Prep: Conventional TEM Lamella

SEM image

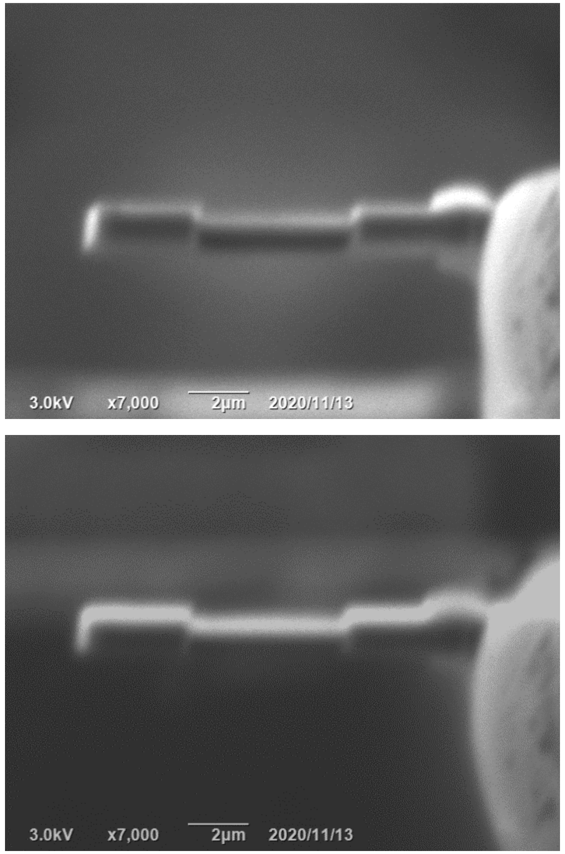


53˚ + 2.5˚ tilt



53˚ - 2.5˚ tilt

FIB image



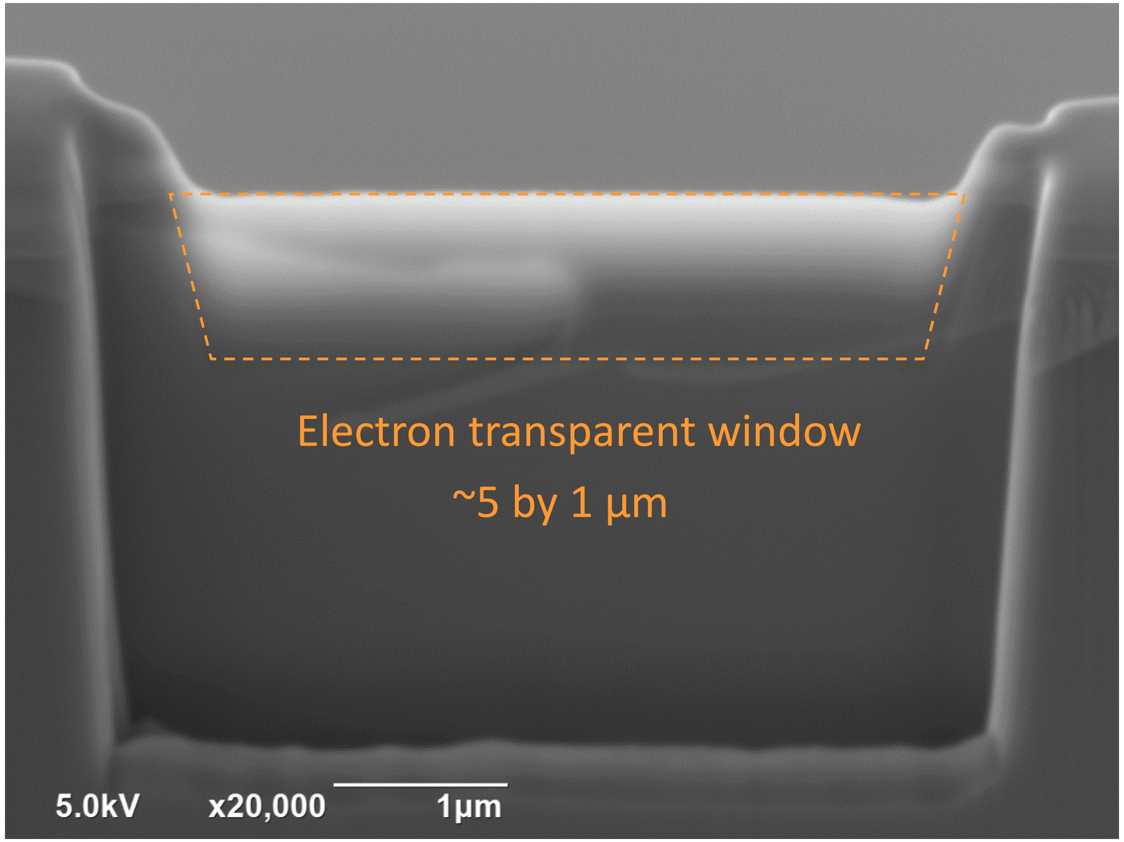
xiii) Low energy window cleaning (×2)

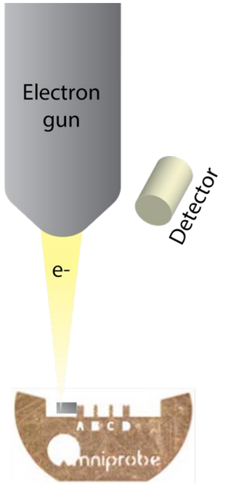
*3 kV, 30 pA

TEM Sample Prep: Conventional TEM Lamella

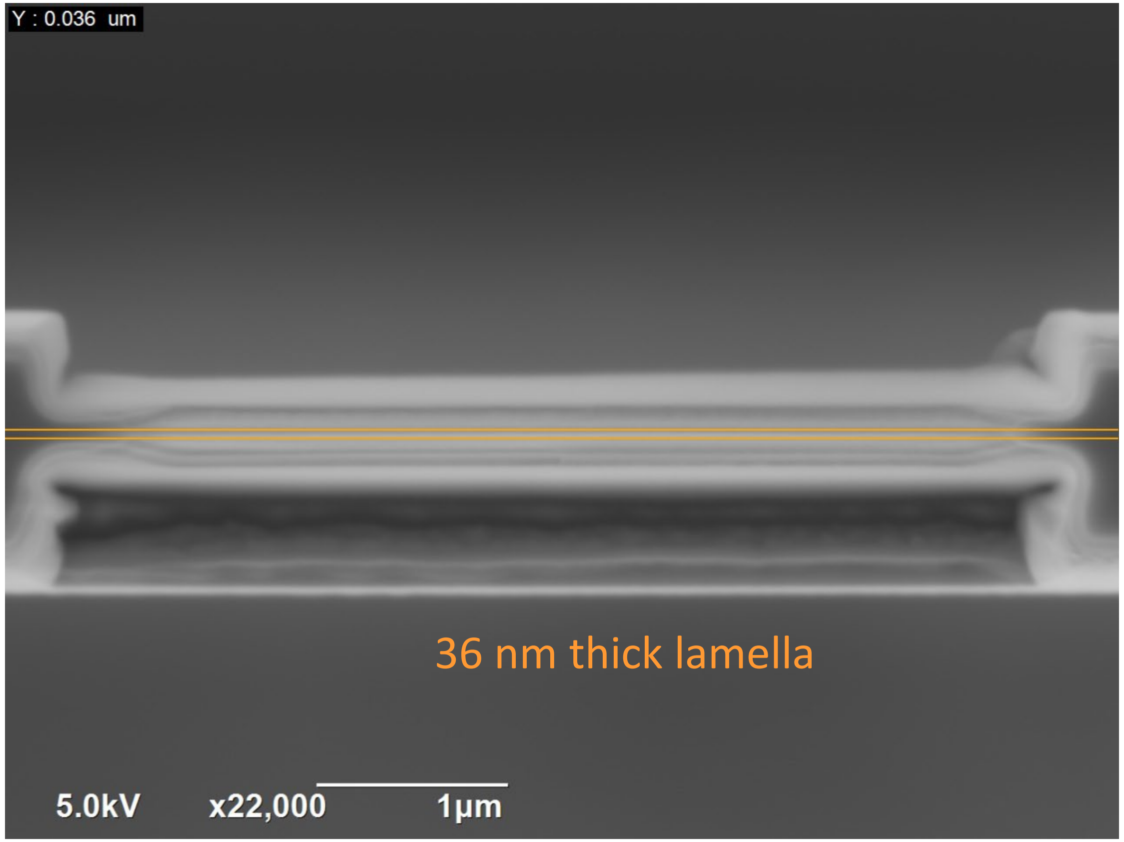
SEM image

53˚ tilt





0˚ tilt

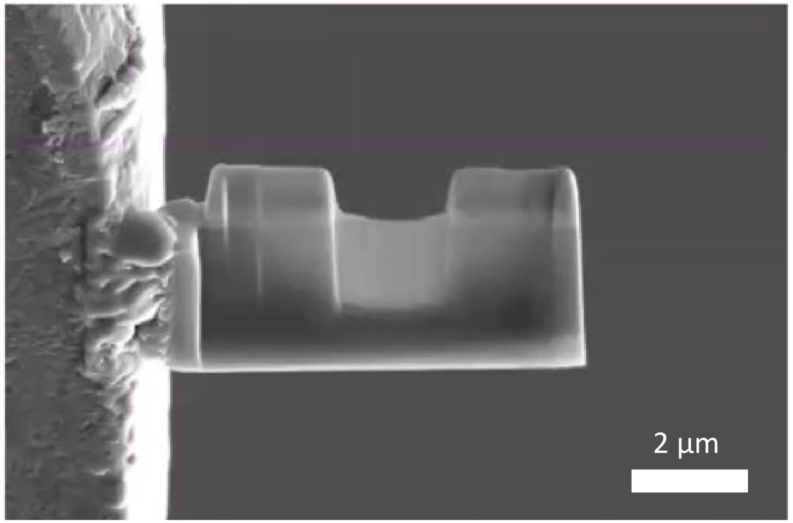
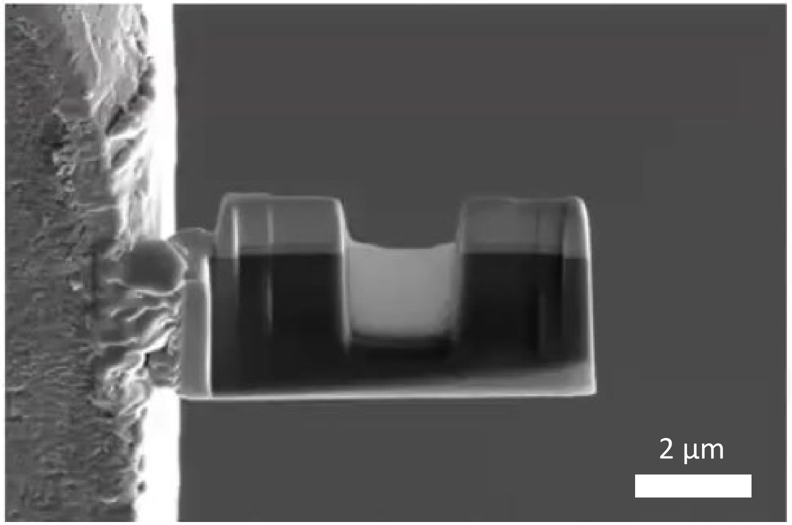
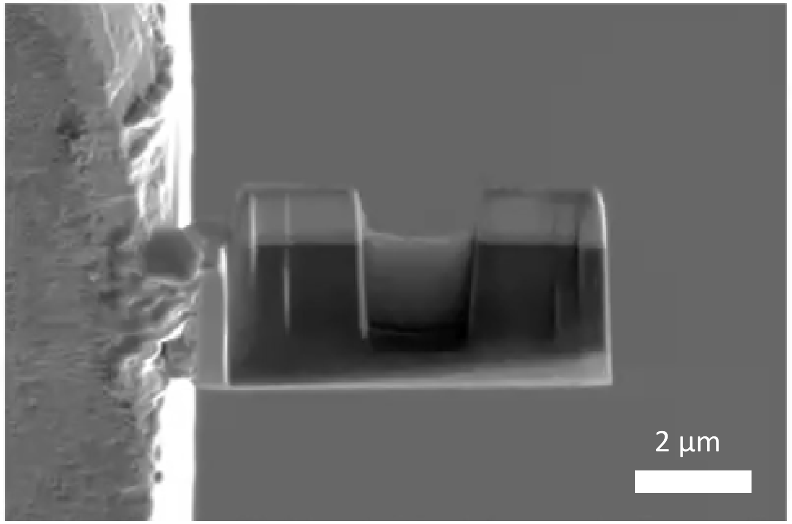


xiv) Final lamella imaging

*5 kV, 20 pA

TEM Lamella – Electron Transparency and Thickness

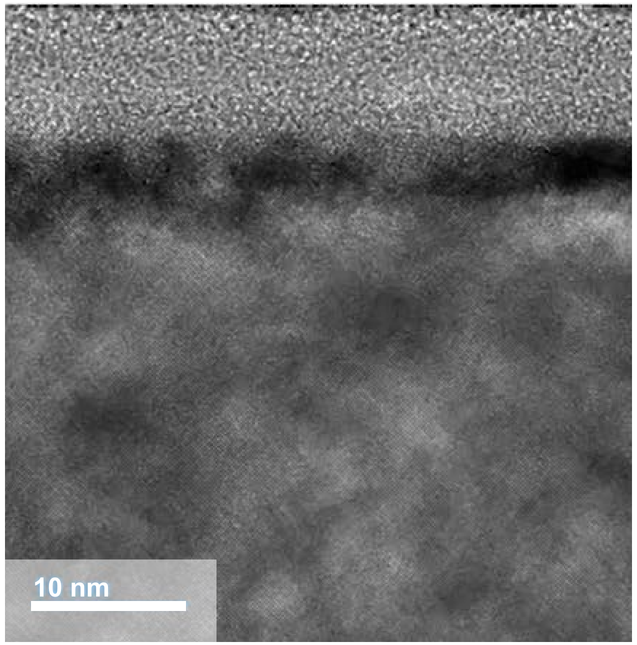
- **Si**

10 keV	**5 keV**	**2 keV**
		
~350 nm	**~80 nm**	**~30 nm**

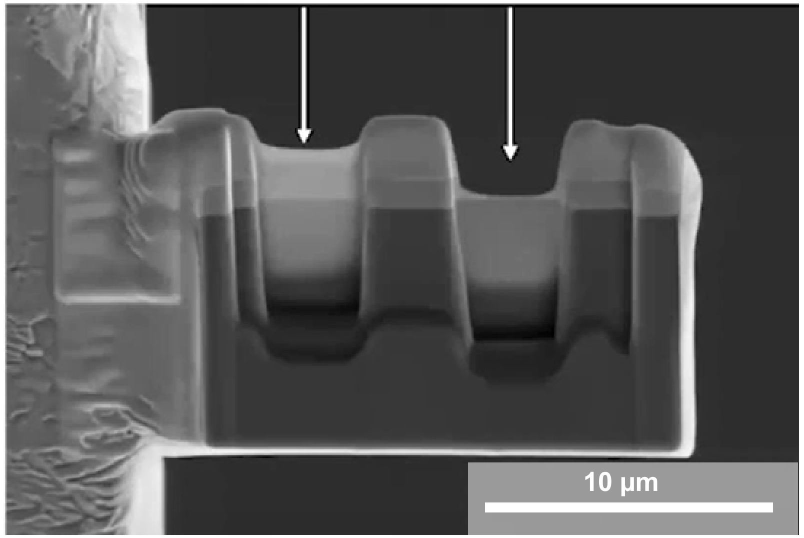
- Heavier elements become electron transparent at smaller thickness

100nm vs. 40nm Thick Lamella

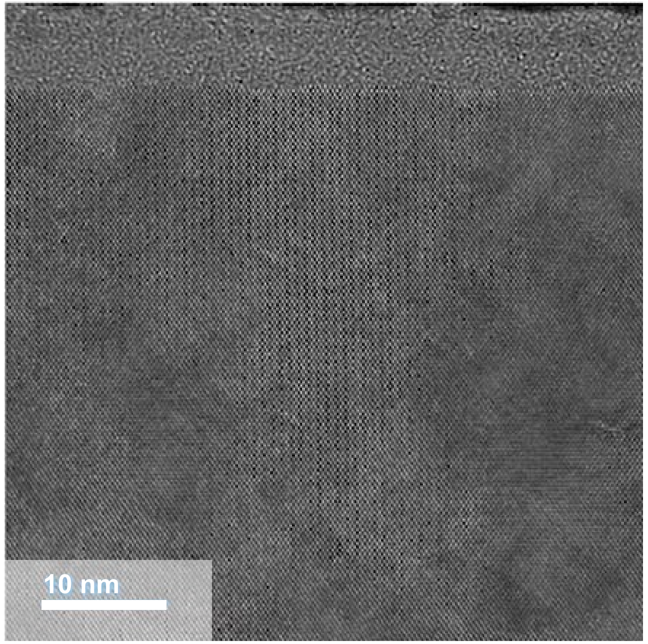
100 nm



100 nm 40 nm

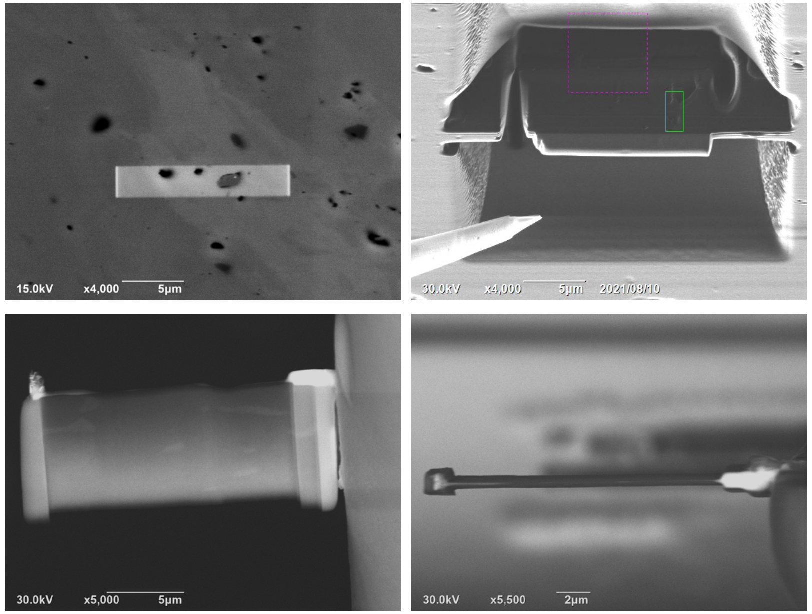


40 nm



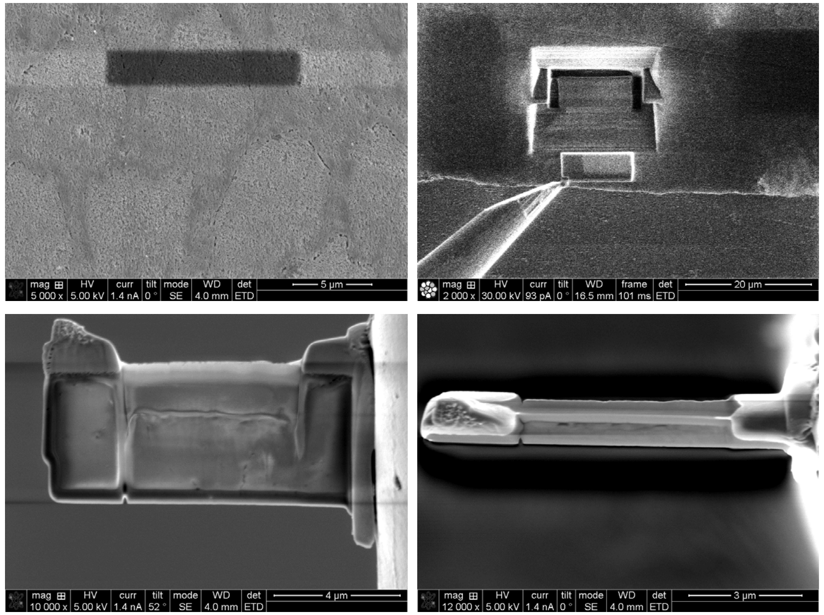
TEM Sample Prep – From Hard to Soft Materials

meteorite



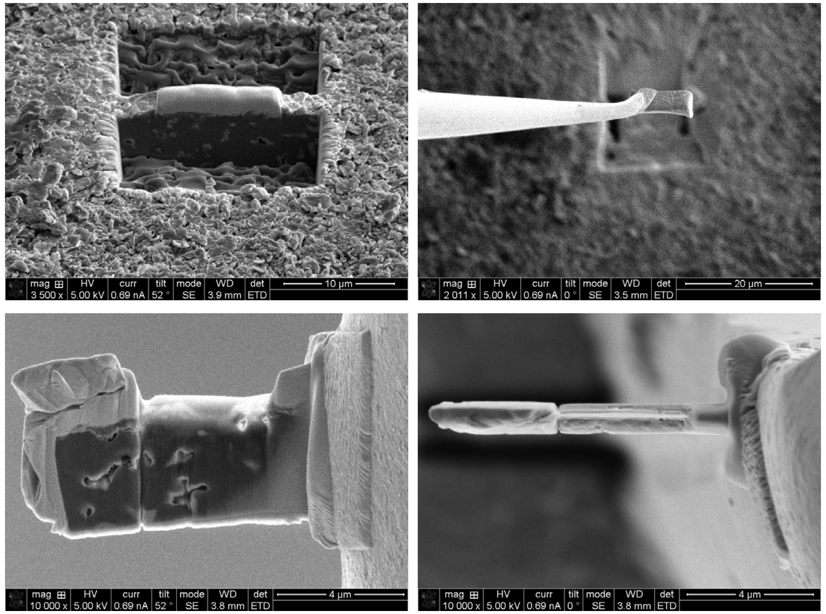
HARD

enamel



HYBRID

graphite

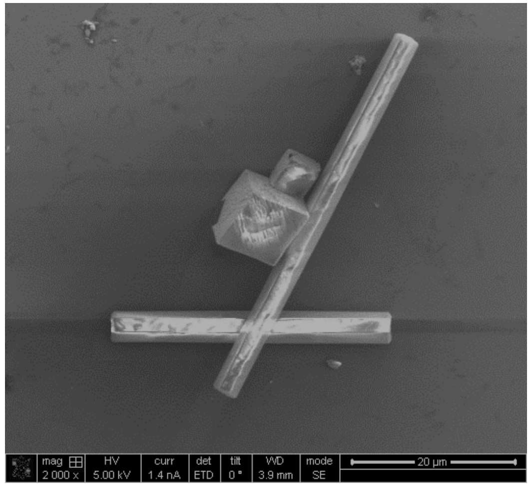


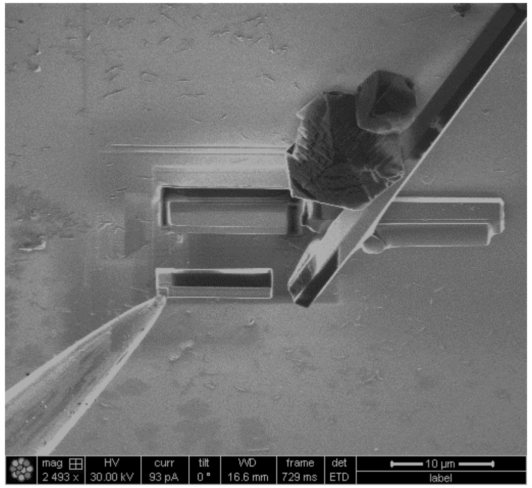
SOFT

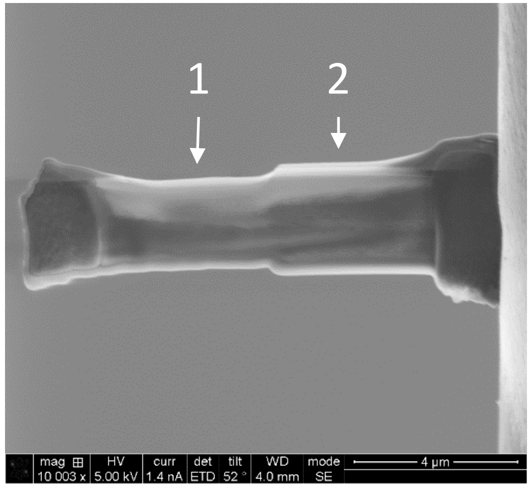
Mg^{2+}

$Ca_{10}(PO_4)_6(OH)_2$

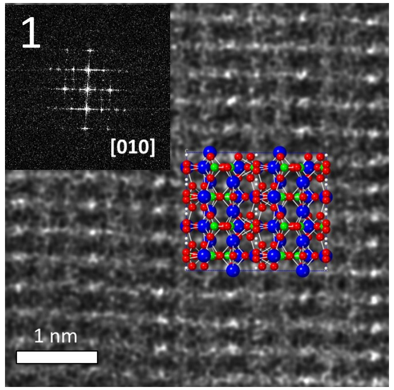
Exotic shape TEM sample prep





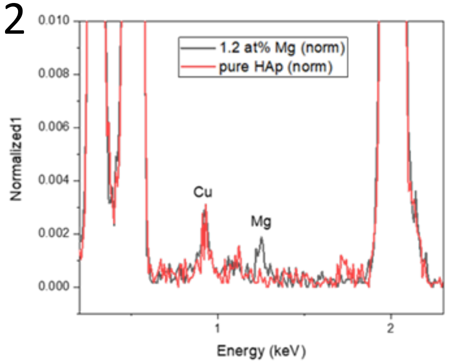


STEM imaging

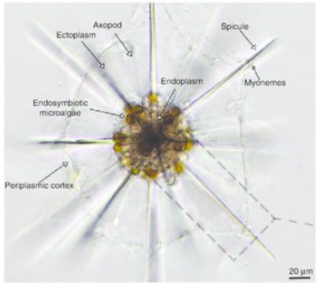


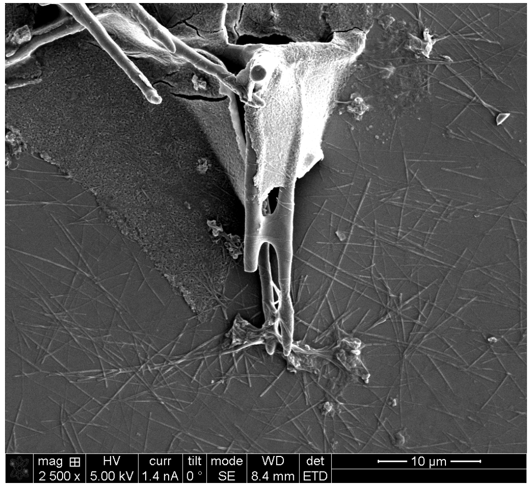
EDS acquisition

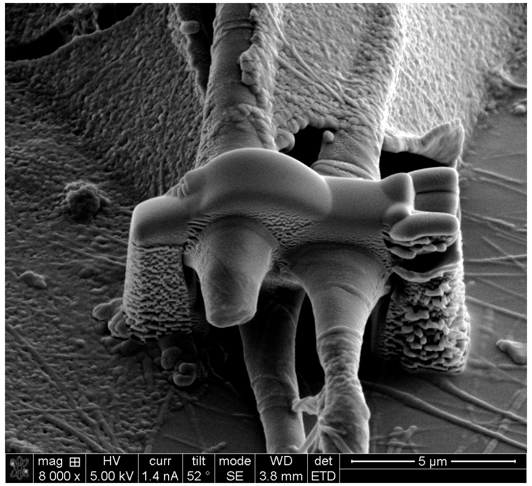
2

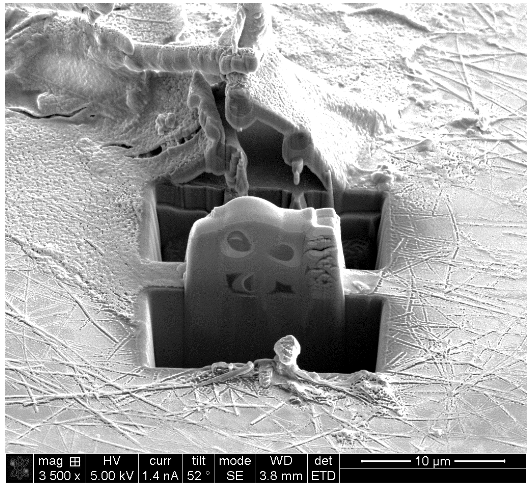


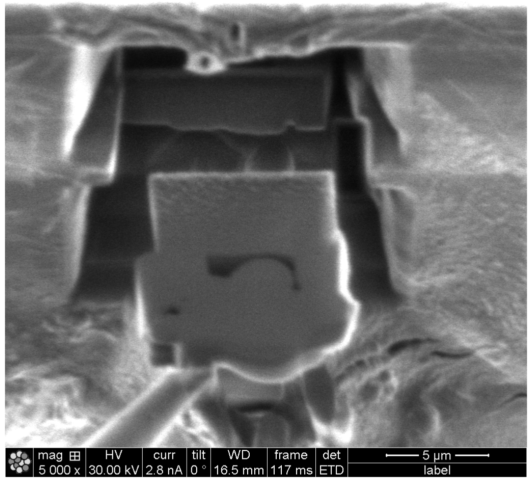
~1.2 at% Mg



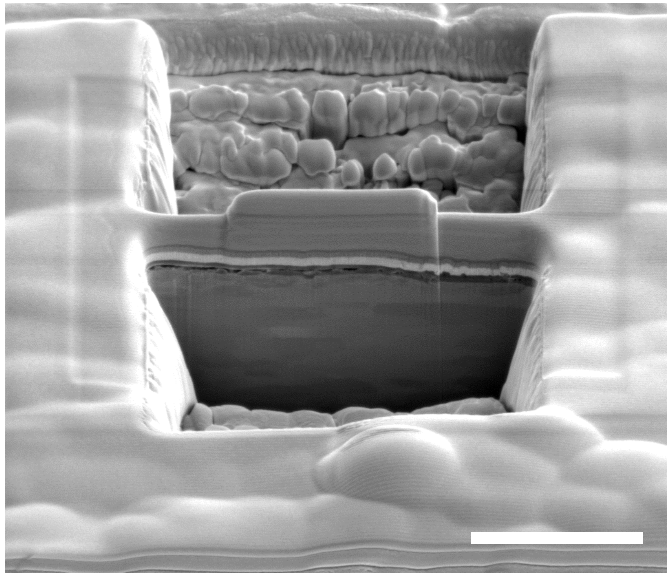


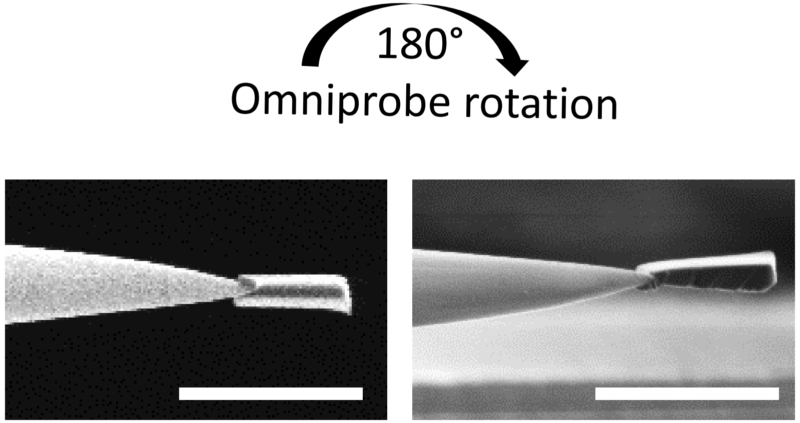


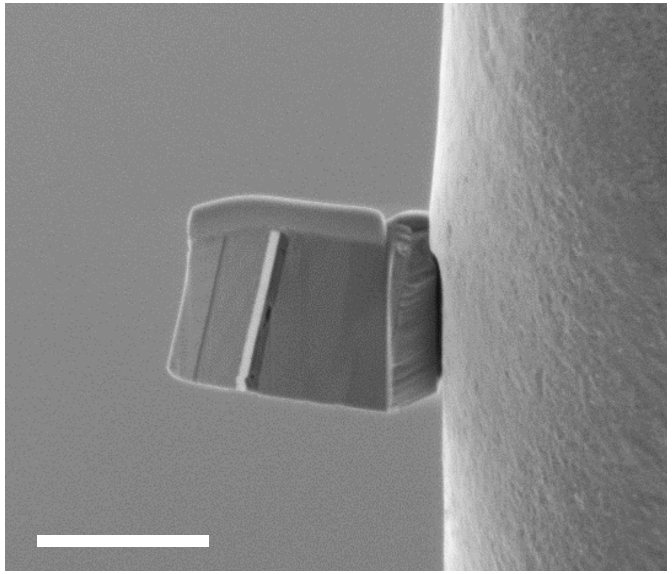




Lamella Orientation through Omniprobe Rotation



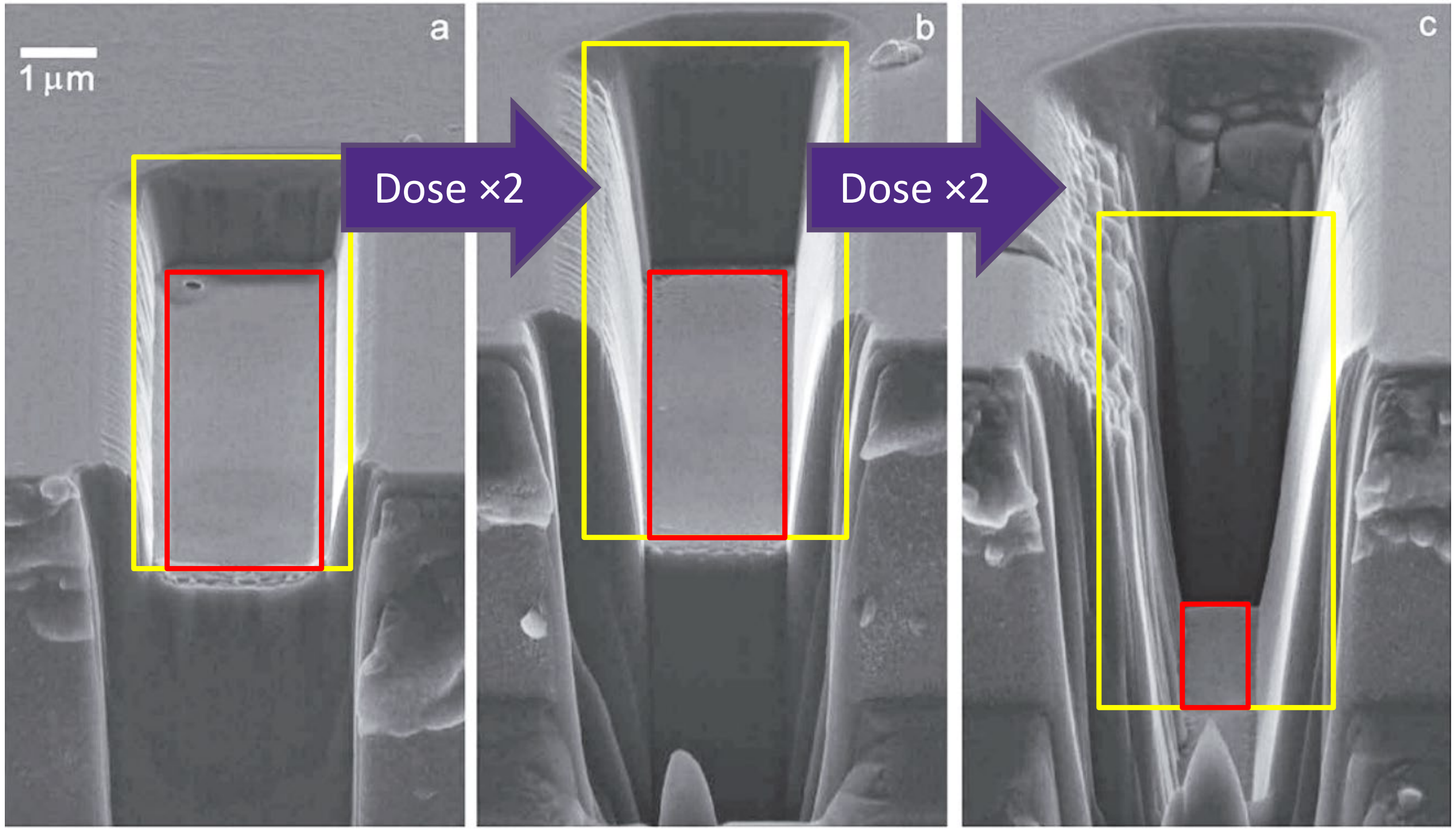




Common Problems & Artifacts

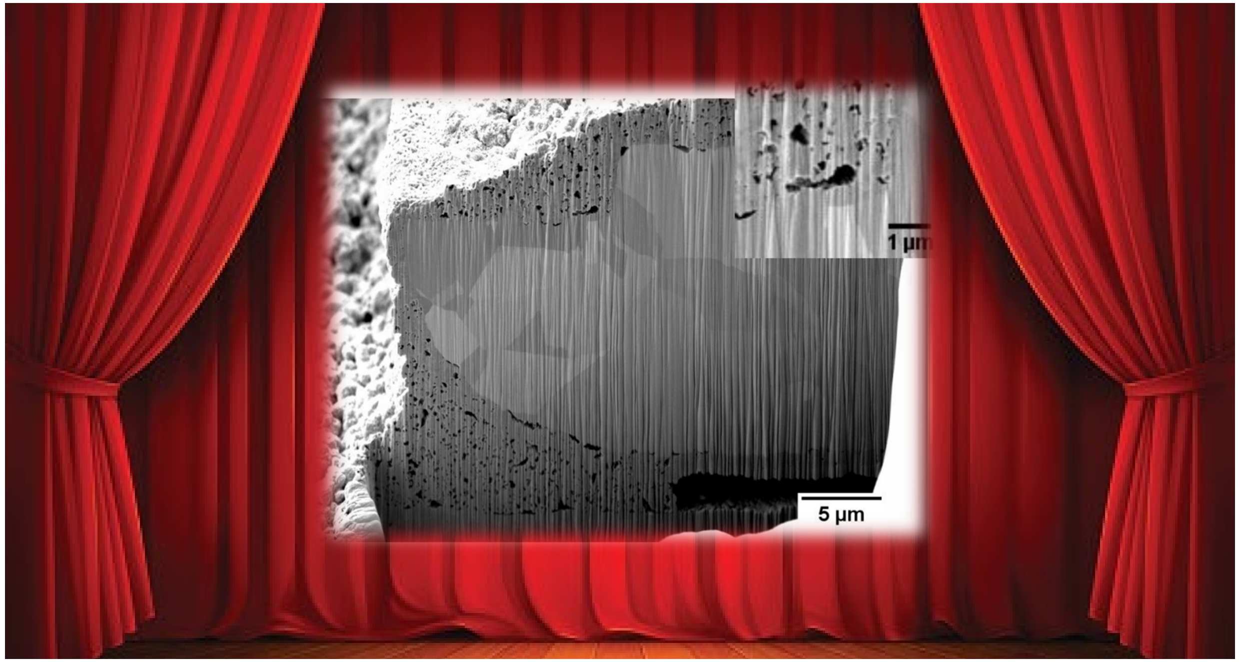
- General FIB-SEM artifacts in TEM sample prep
  - Ion Implantation
  - Redeposition
  - Curtaining
  - Amorphization
- Artifacts specific to TEM sample thinning
  - Lamella bending
  - Hole formation in lamella

General FIB-SEM Artifacts - Redeposition

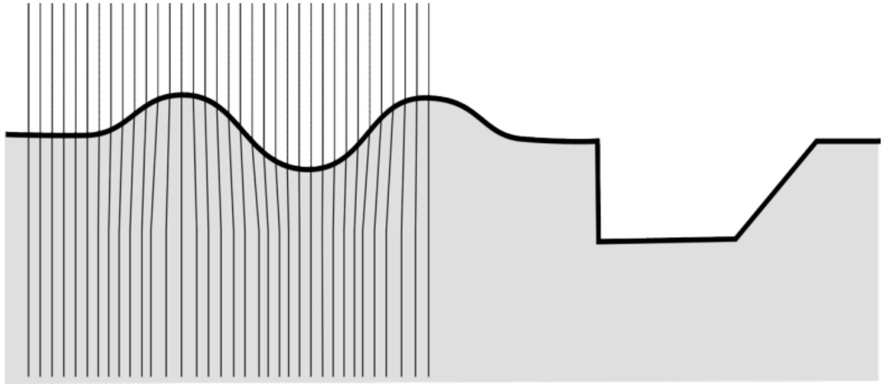


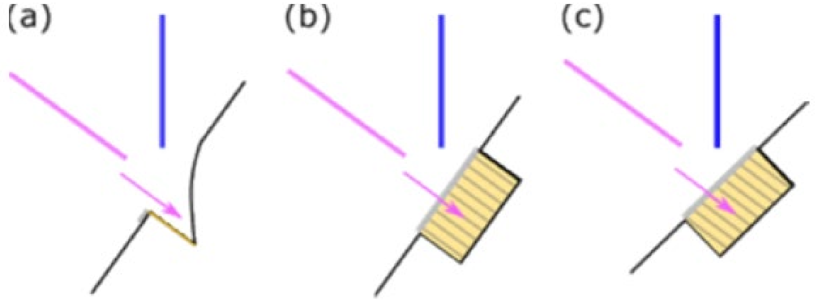
- Sputtered material can deposit nearby
- Dependent on energy of sputtered particles and their sticking coefficient
- Limits milling of deep and narrow features (milling rate approaches redeposition rate)
- Mitigate through 1) lowering ion current, 2) changing milling geometry, 3) angle

General FIB-SEM Artifacts - Curtaining



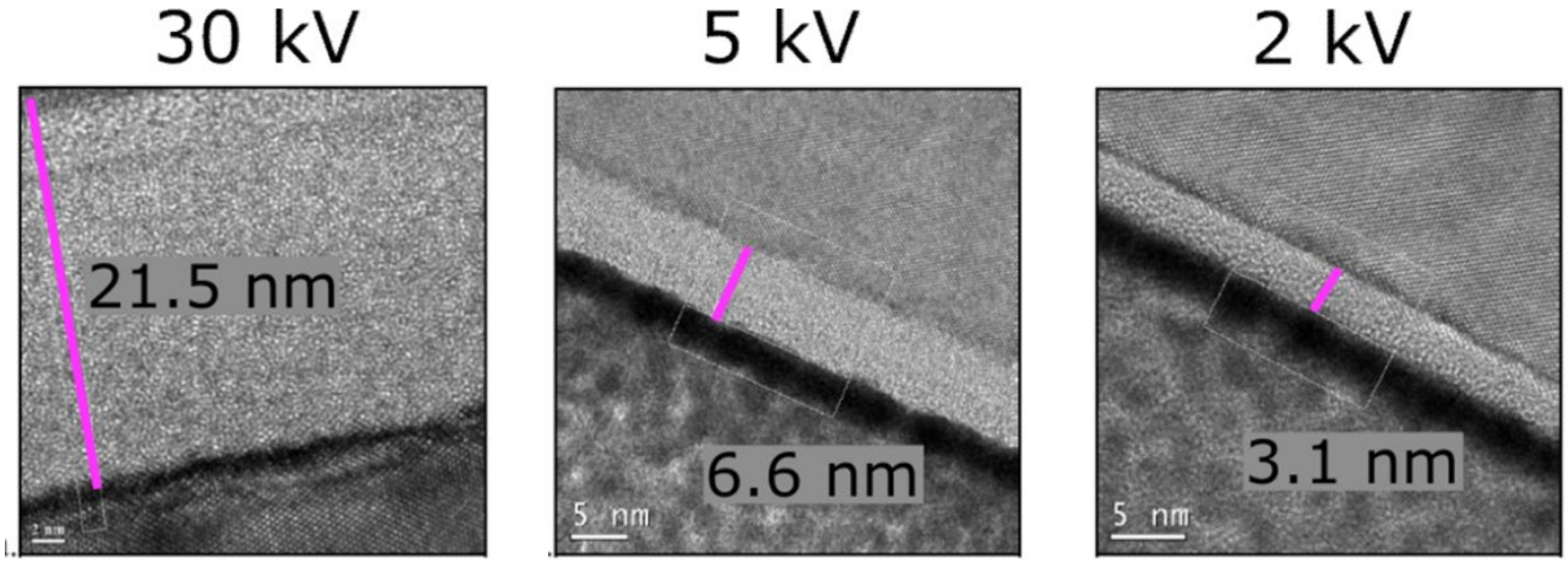
- Appears as vertical streaks in image (classic theatre curtain)
- **Mechanism**: Created by spatial variation of sputter rate (ion beam gets deflected by tilted faces modulating local dose)
- Porous materials, rough surfaces, composites of hard/soft materials
- Mitigate through 1) hiding it (BSE or postprocessing) 2) thicker deposition layer 3) lowering current 4) backside TEM sample milling 5) stage rocking



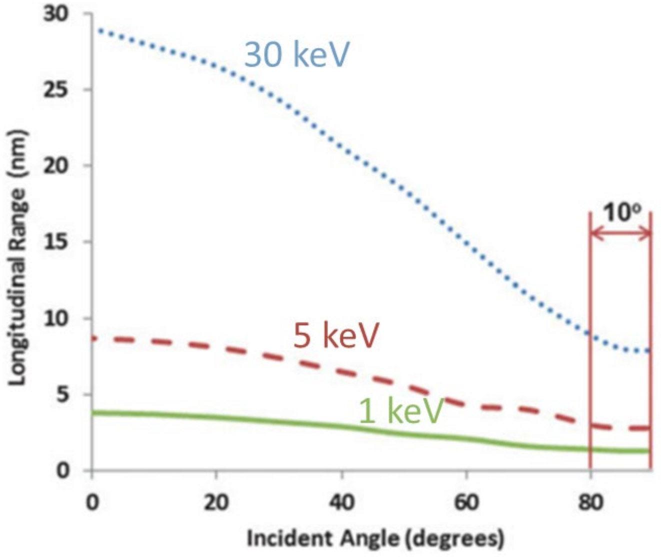


Reuteler, J. *FIB artifacts and how to overcome them*, ETH Zurich (2016)

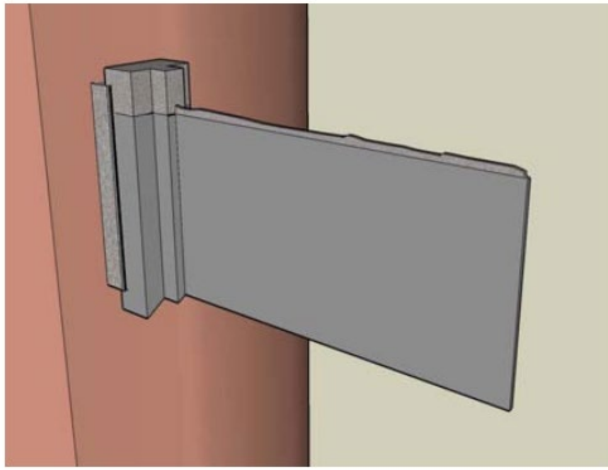
General FIB-SEM Artifacts - Amorphization

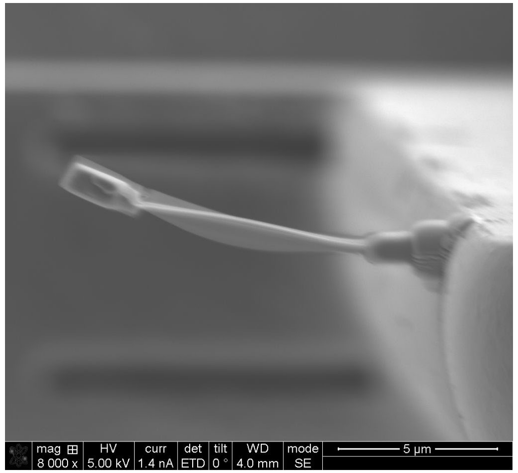


- Damage in Si
- Reduce thickness of amorphous layer:
  - Low kV
  - Lower angle

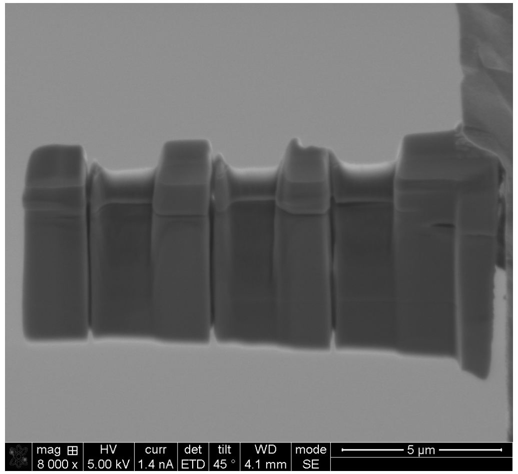


TEM Prep Specific Artifacts – Lamella Bending

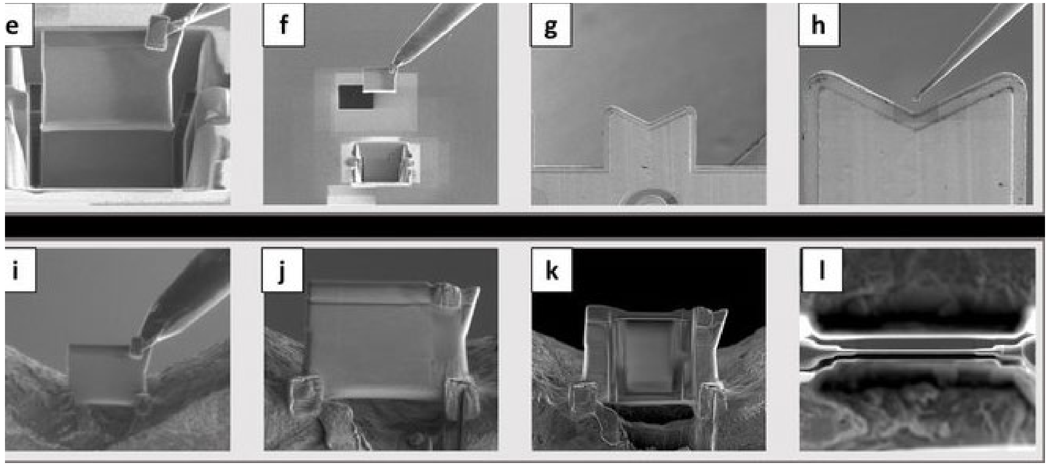




‘flag’ with multiple windows



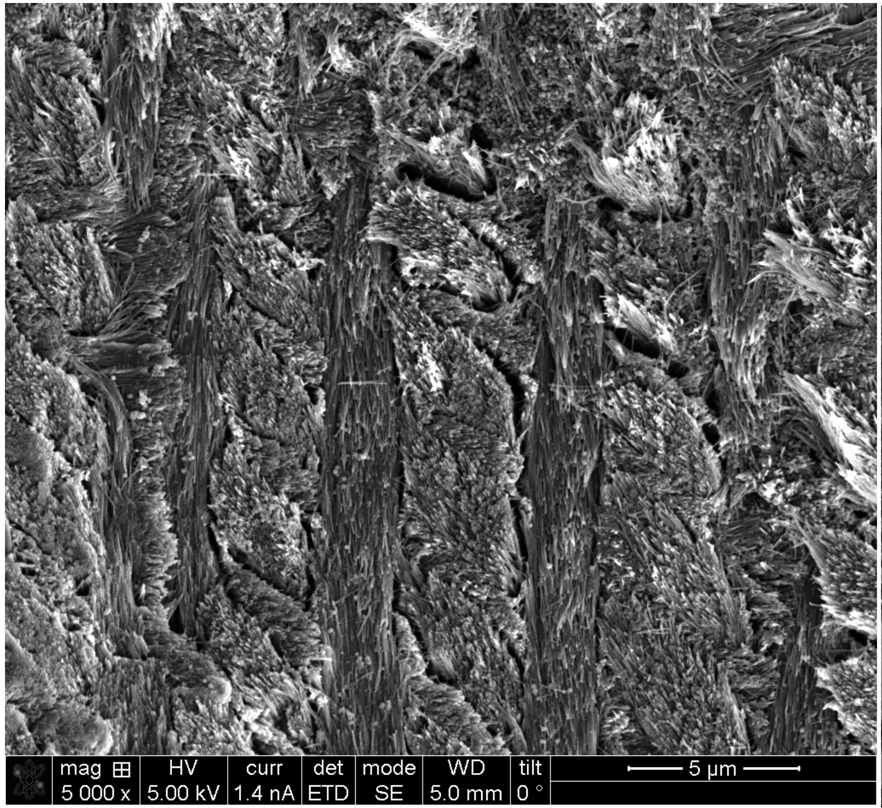
Top mount

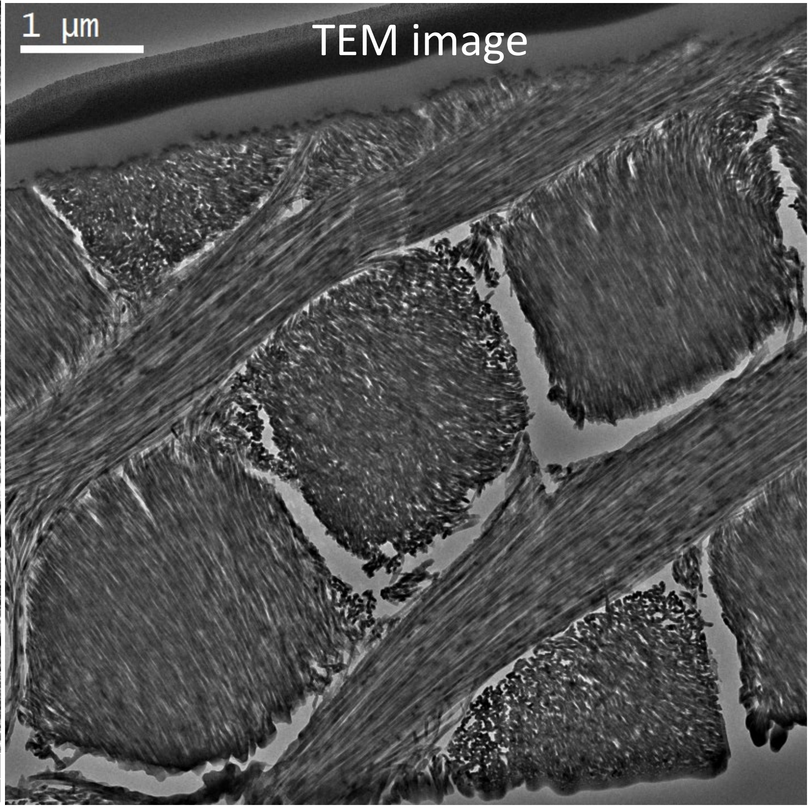


- Lamella may bend at various thicknesses dependent on sample material
- Mitigate through 1) lower FIB current/voltage milling 2) minimize beam dwell time 3) use a different lamella geometry and/or attach lamella differently to the TEM grid

TEM Prep Specific Artifacts – Holes

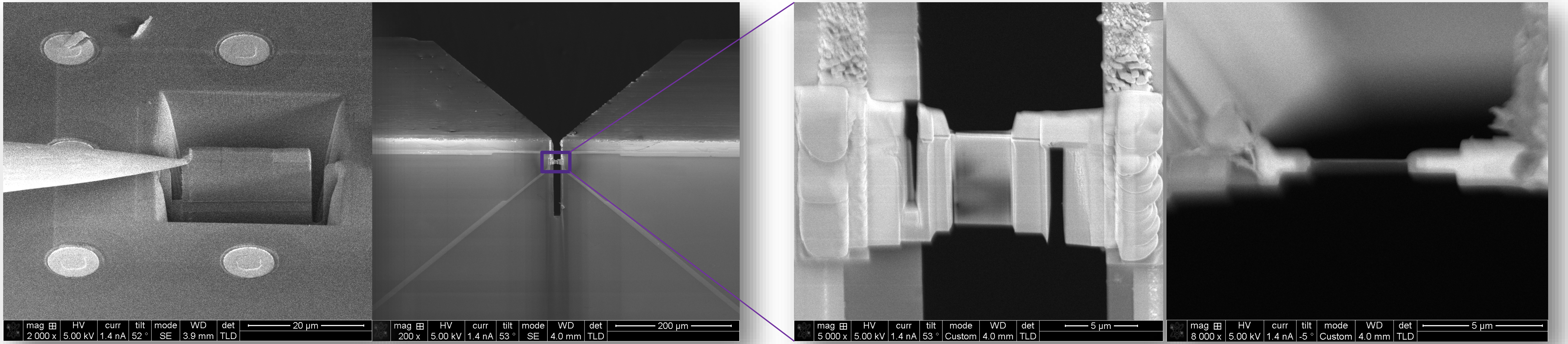
- Porous samples may be problematic for liftout – holes may widen
- Holes may contribute to other lemalla defects, such as bending/curling, or even destroying the ROI
- Mitigate through 1) lower FIB current/voltage milling 2) minimize beam dwell time 3) use a different lamella geometry 4) leaving final lamella a bit thicker

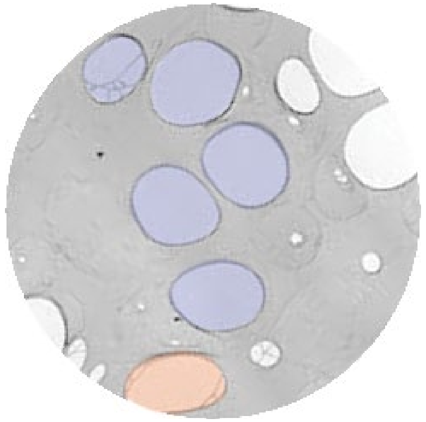




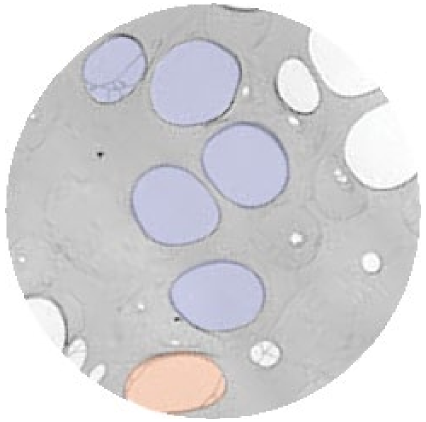
Take-away Messages

- Sample preparation is crucial to (S)TEM characterization
- FIB-SEM is a great tool in the (S)TEM sample prep arsenal to site-specifically prepare samples at various orientations for a wide range of materials
- TEM lamella preparation is not only science, but a form of art





Questions?



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