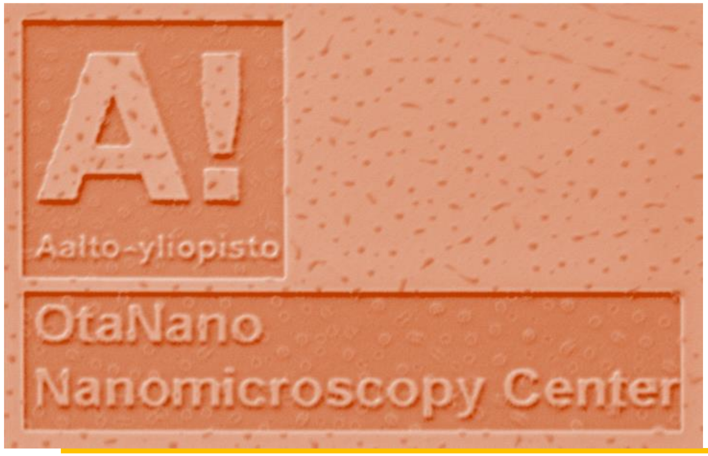
Lecture: PHYS-E0525

Microscopy of Nanomaterials

Focused ion beam (FIB) microscopy and applications



Lide Yao
lide.yao@aalto.fi

Learning goals

❑ What is FIB?

Ion source, ion-solid interaction,
basic working principal……

❑ Why use FIB?

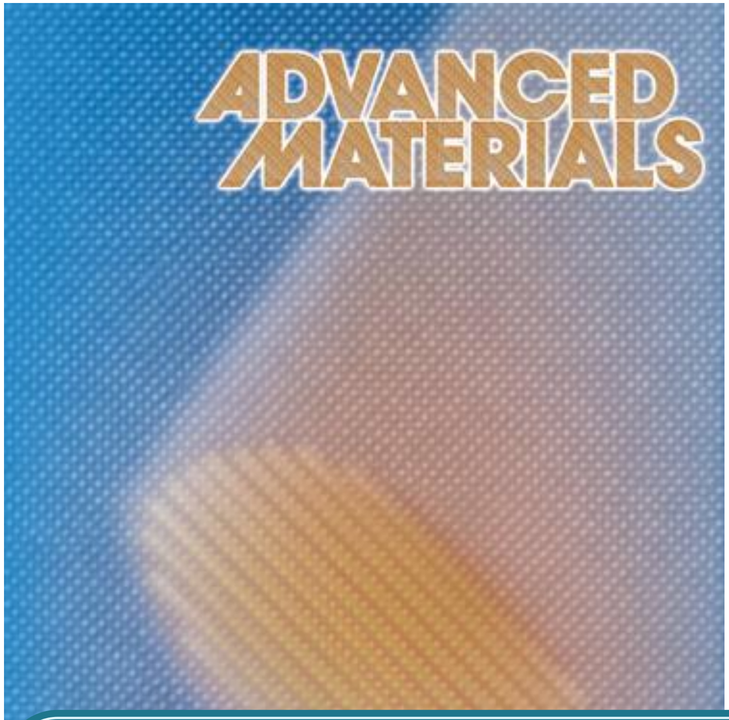
Basic functions with a FIB
Imaging, milling, deposition and implantation

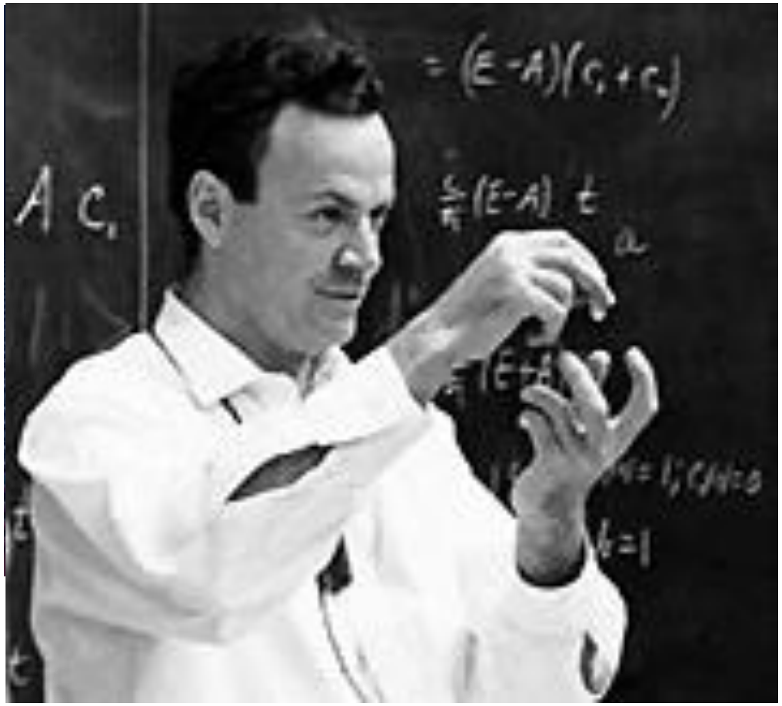
❑ For what by a FIB?

Main applications by a FIB

Patterning/etching, cross-sectional SEM, circuit modification,
SEM/TEM sample preparation, EBSD, and 3D slicing/EDX mapping

From electron beam to ion beam





Focused ion beam (FIB)

- ✓ Using highly focused ion beams such as Ga^+ beam to scan and cut a solid material inside a vacuum chamber.
- ✓ Imaging and micro/nano fabricating technique.

Nanoscience to nanotechnology

Development of FIB microscope

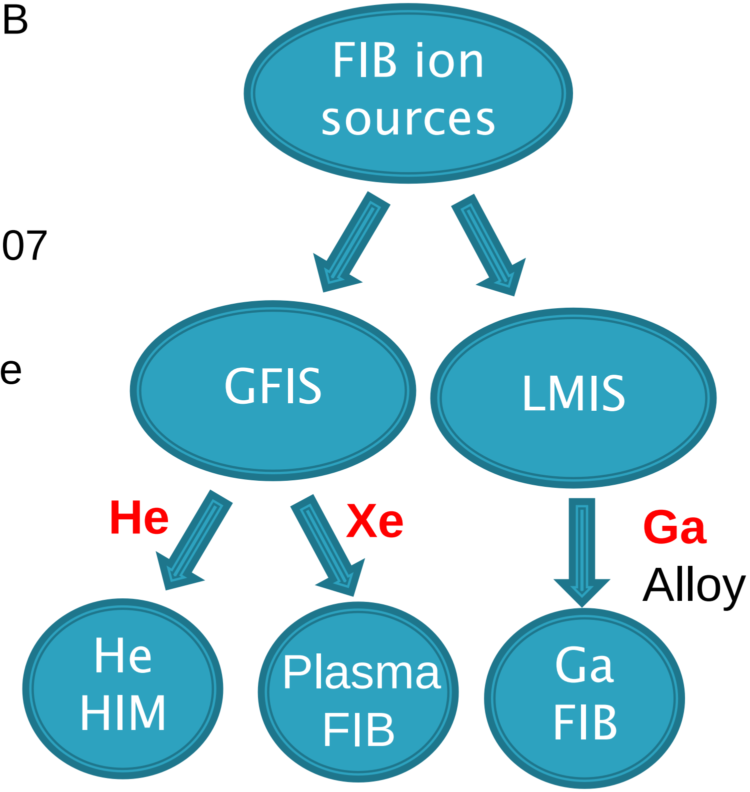
- 1975: The first gas field ionization sources (**GFIS**)-FIB systems based on field emission technology were developed [1,2].

  Gas ion sources: **He**, Ne, Ar, N,……

  Helium ion microscope (HIM) on the market since 2007

  Plasma (**Xe**, Ar, O, N) FIB
- 1978: The first FIB based on a liquid metal ion source (**LMIS**) was built by Seliger et al. [3]

  Metal ion sources: **Ga**, Alloy
- 1988: First dual beam FIB/SEM appears [4]
- In the early 1990s: Dual beam commercial systems were available on the market.



FIB ion sources

GFIS

LMIS

He

Xe

Ga

Alloy

He HIM

Plasma FIB

Ga FIB

[1] Orloff, J. et.al, J. Vac. Sci. Tech. 12 (6), 1209, (1975).
[2] Levi-Setti, R. Scanning Electron Microscopy: 125 (1974).
[3] Seliger, R. et.al, Appl. Phys. Lett. 34, 310 (1979)
[4] Sudraud P, et.al, J. Vac. Sci. Technol. B6, 234 (1988)

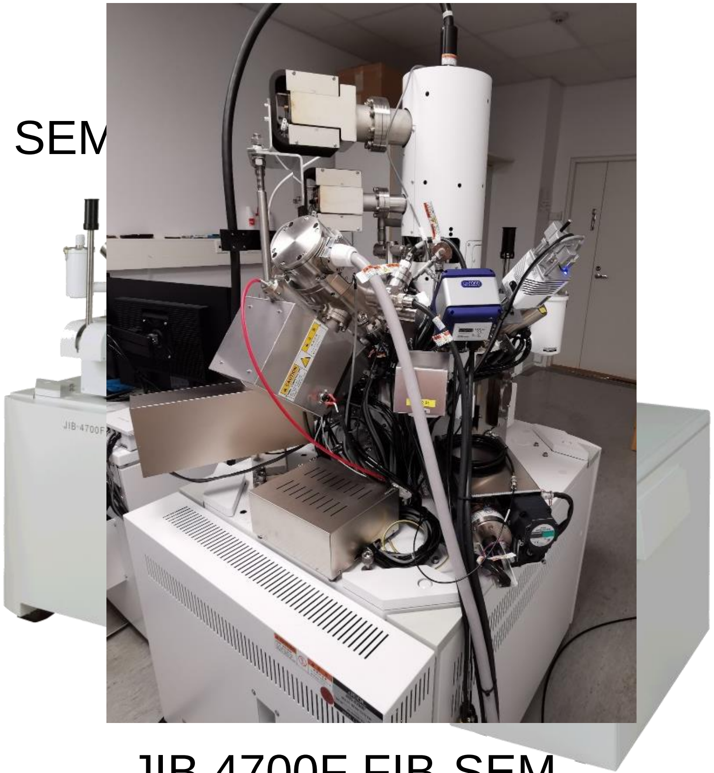
SEM vs FIB

Single electron beam system

JSM 7500F SEM

Dual beam system

SEM

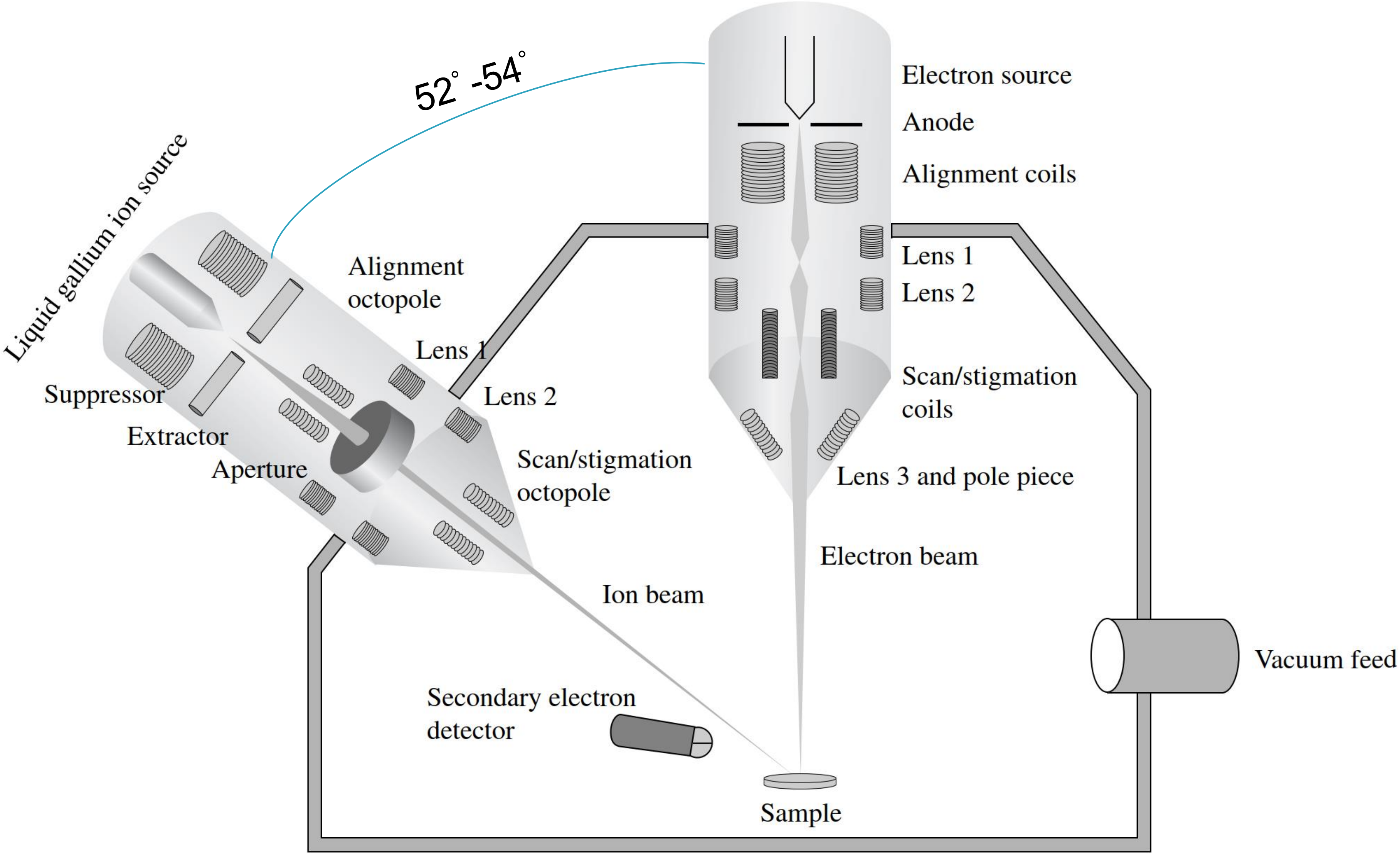


JIB 4700F FIB-SEM

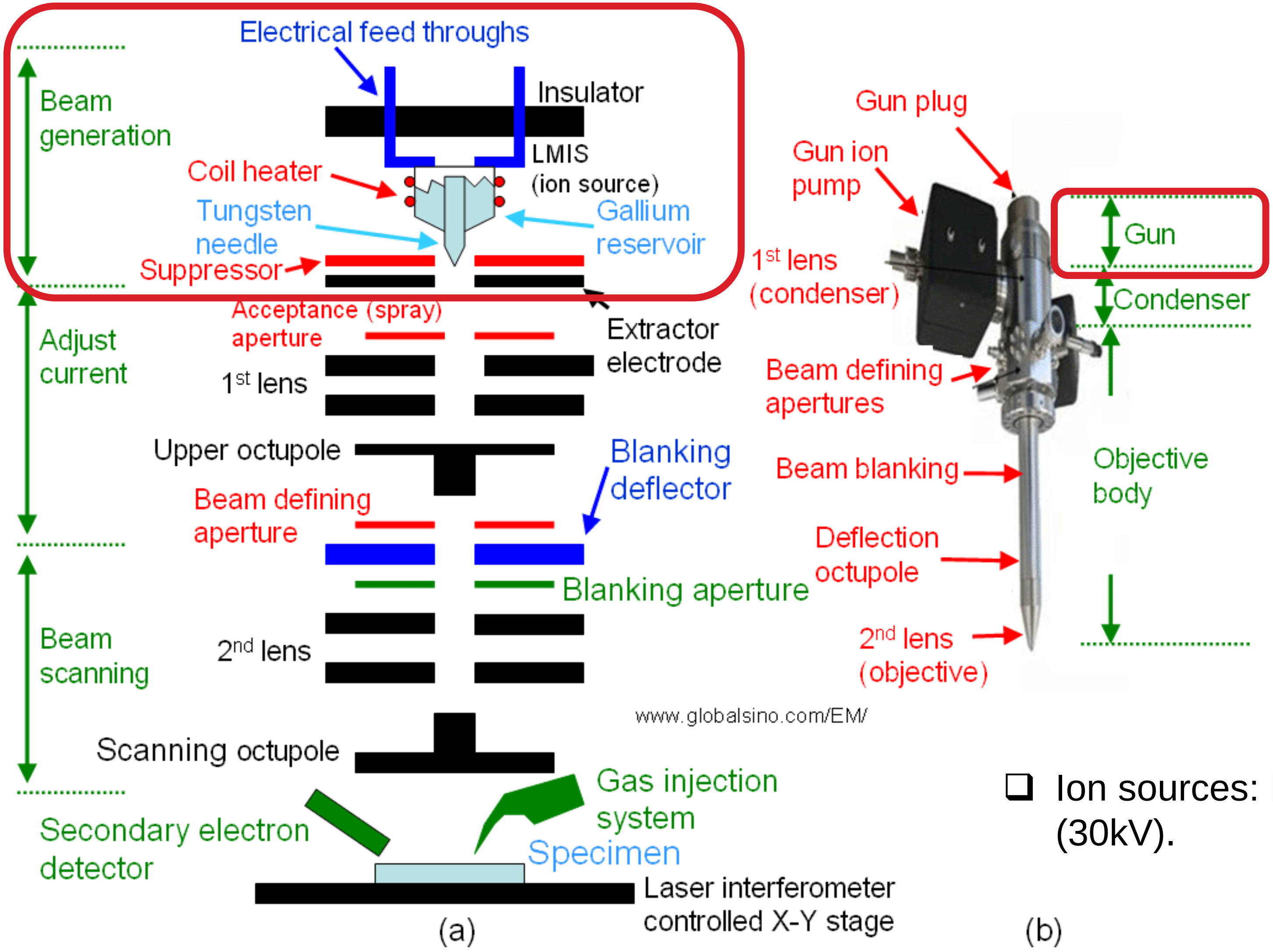
@ OtaNano-Nanomicrosopcy Center (NMC), Aalto University

Main difference: additional ion beam column

Dual-beam columns



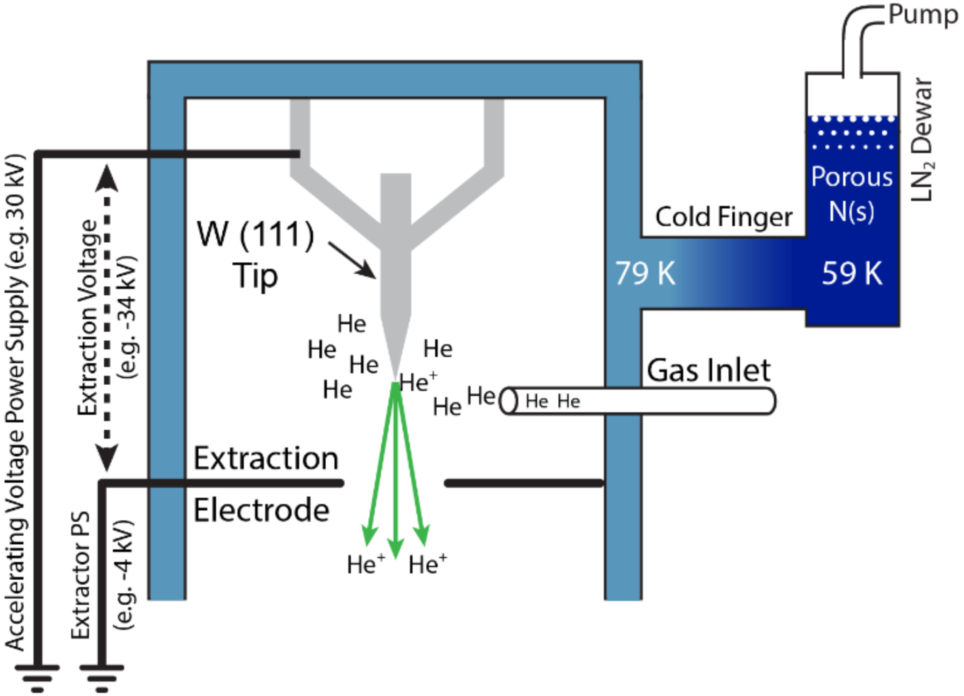
Ion beam column



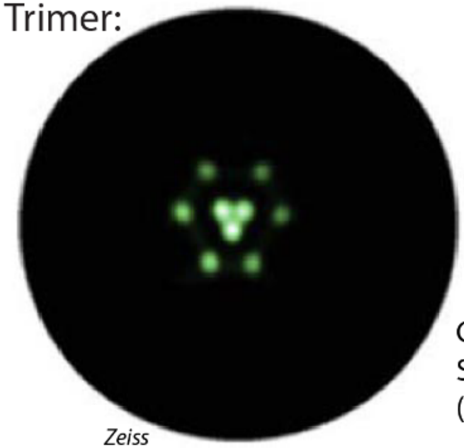
www.globalsino.com/EM/

- ☐ Ion sources: LMIS, GFIS (30kV).

Gas field ion source (GFIS)



Trimer:

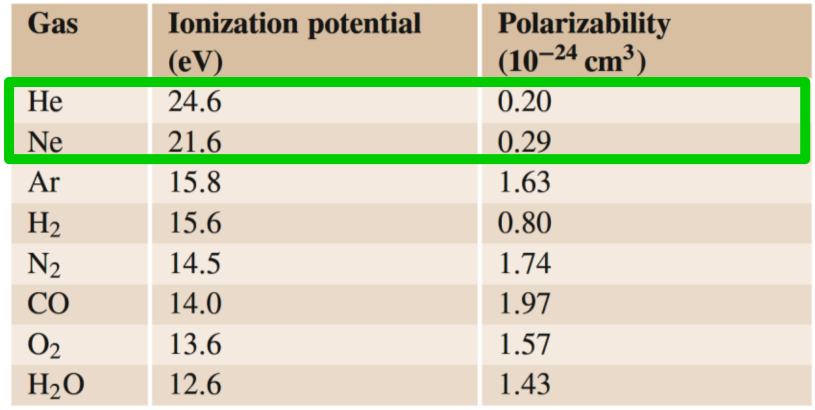


Zeiss

Choose one for
Single Atom Source
(<1 nm)

http://www.orsayphysics.com/what-is-fib

Ionization potential and polarizability of common gases in GFIS vacuum

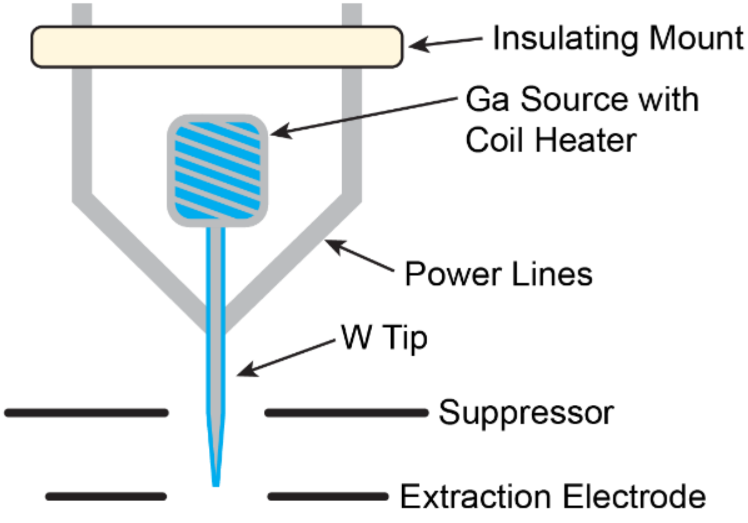


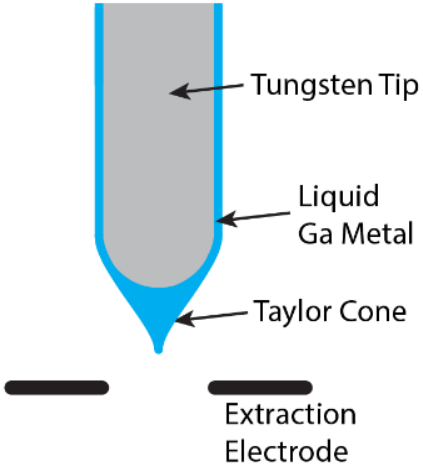
Gas	Ionization potential (eV)	Polarizability (10^{-24} cm^3)
He	24.6	0.20
Ne	21.6	0.29
Ar	15.8	1.63
H_2	15.6	0.80
N_2	14.5	1.74
CO	14.0	1.97
O_2	13.6	1.57
H_2O	12.6	1.43

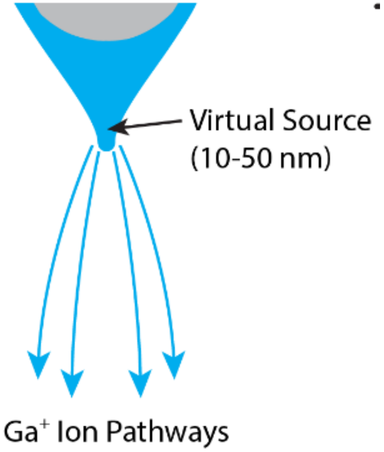
For He ion GFIS:

- ✓ Spot size: 0.35 nm for He
- ✓ Brightness: 5.0E9 A/cm^2·sr

Liquid metal ion system(LMIS)







http://www.orsayphysics.com/what-is-fib

- By applying an electric potential (such as 30kV) between the needle and a downstream metallic extractor.
- A structure known as Taylor cone is formed at the tip of needle.
- Once exceeding a threshold voltage, ion and droplets are extracted from the cone (E> 1 x 10^8 V/cm)
- The extracted ions pass through the hold of extractor.

With Ga ion LMIS (JIB 4700 @Otanano-NMC):

☐ Spot size: 3-5 nm (imaging resolution: 5 nm)
☐ Brightness: ~3.0×10^6 A/cm^2·sr
☐ Probe current 1pA-90nA

Other alloy ion sources?

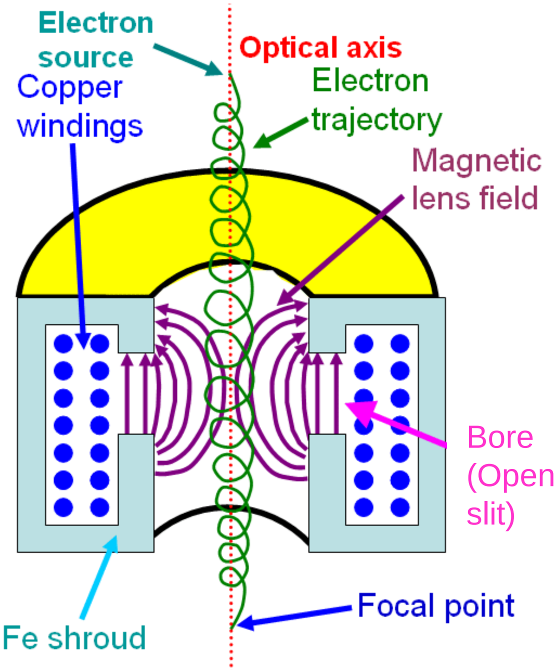
Au-Si, Au-Ge and Au-Si-Ge

Why Ga ion in LMIS?

- **Low melting** point (29.8°C)
- **Heavy enough** for milling the heavier elements
- **Low volatility** at the melting point (a long source life of about 400 mA-hours/mg)
- **Low vapor pressure**

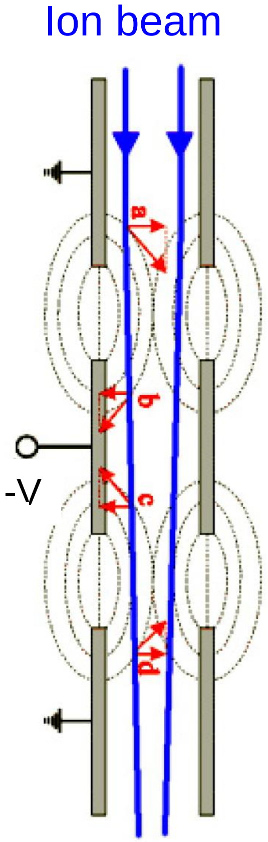
--allowing Ga to be used in its pure form instead of in the form of an alloy source.

Electrostatic lenses in FIB column



www.globalsino.com/EM/

Magnetic lens



Electrostatic lens

Table 1.1 *Quantitative comparison of FIB ions and SEM electrons*

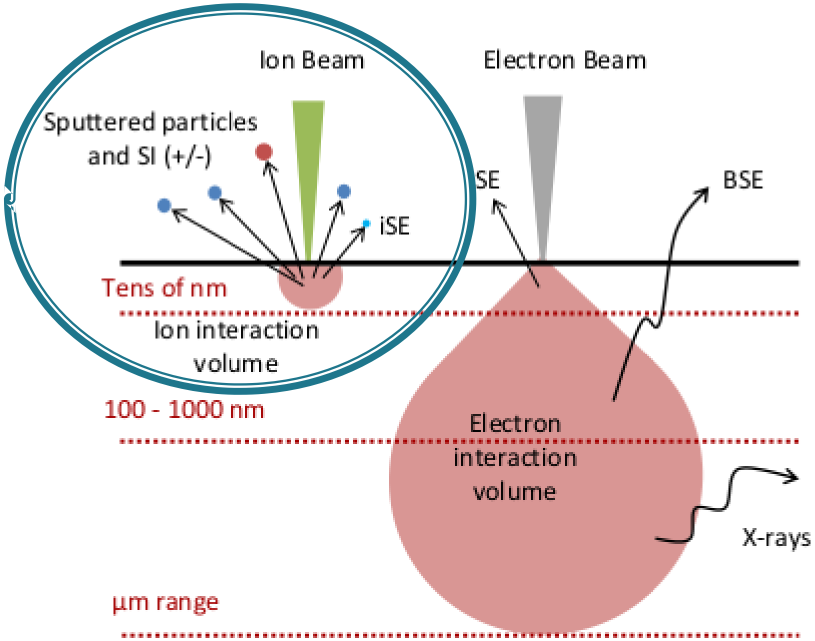
Particle	FIB	SEM	Ratio
Type	Ga^+ ion	Electron	
Elementary charge	+ 1	− 1	
Particle size	0.2 nm	0.00001 nm	20 000
Mass	1.2×10^{-25} kg	9.1×10^{-31} kg	130 000
Velocity at 30 kV	2.8×10^5 m/s	1.0×10^8 m/s	0.0028
Velocity at 2 kV	7.3×10^4 m/s	2.6×10^7 m/s	0.0028
Velocity at 1 kV	5.2×10^4 m/s	1.8×10^7 m/s	0.0028

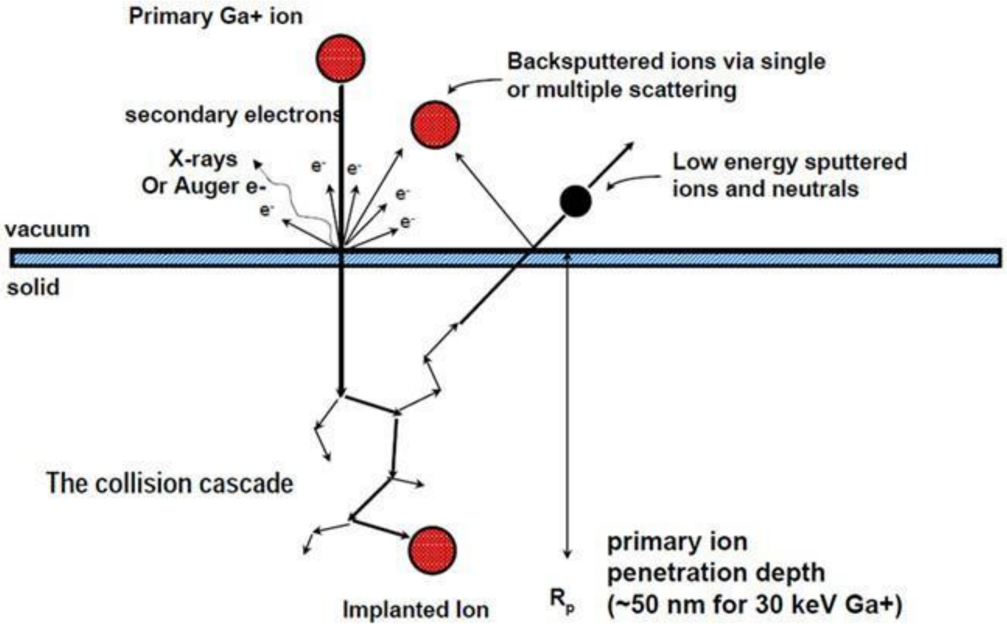
Why electrostatic lens?

- ✓ The ion (positively charged) is much larger and more massive than the electron. m_I ~$\mathbf{10^5}$ m_e
- ✓ Ions travel more slowly
- ✓ Larger fields to focus and control ions than electrons:
  Lorentz force: $$\vec{F} = \underbrace{q\vec{E}}_{\text{Electric force}} + \underbrace{q\vec{v}\ x\ \vec{B}}_{\text{Magnetic force}}$$
- ✓ If magnetic lens, Lorenz force is weaker, so a few **km** coils will be needed.

Aberrations in ion and electron optics depend on the same factors.

Ion–solid interactions in FIB–SEM





Bernd Schmidt, Klaus Wetzig. Ion Beams in Materials Processing and Analysis. Springer, 2013.

Electron-solid interaction

- ✓ Secondary electrons (SE),
- ✓ Backscattered electrons (BSE)
- ✓ Cathodolumenecence
- ✓ Auger electrons
- ✓ Characteristic x-ray
- ✓ Interaction volume: **µm**

Ion-solid interaction

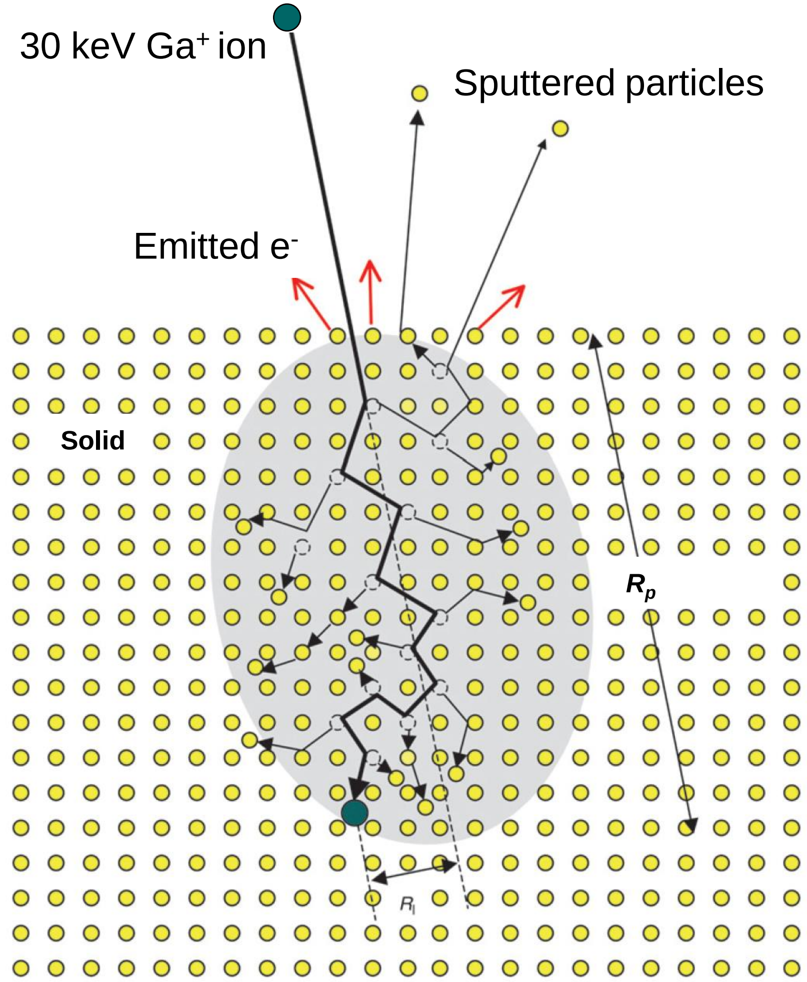
Nuclear processes

- ✓ Sputtered particles (ions or neutrals)
- ✓ Backscattered ions

Electronic processes:

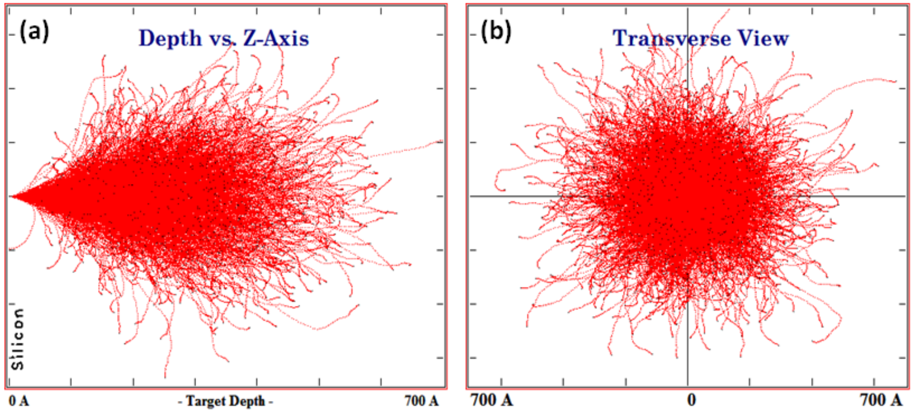
- ✓ Ion induced secondary electron (iSE)
- ✓ X-rays or Auger electrons (low yield)
- ✓ Interaction volume: **tens of nm**

Collision cascade (Ga ions)



Collision cascade model

TRIM or SRIM (transport, or stopping range ions in matter)—Monte Carlo simulation



- ✓ Projected range R_p :10-100 nm
- ✓ Lateral range R_l : 5-50 nm

C. A. Volkert et al., MRS bulletin 32, 389, 2007

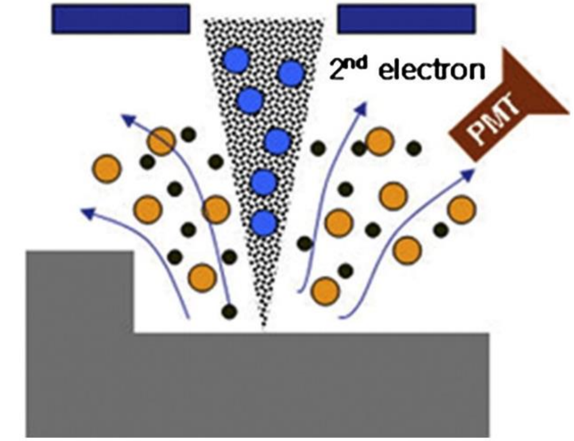
Basic functions in a FIB

✓ **FIB imaging**
iSEs

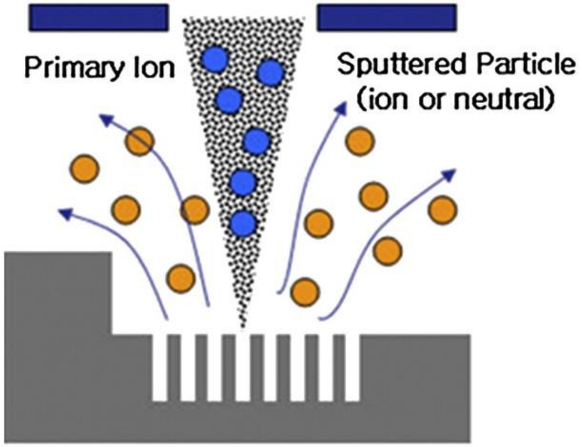
✓ **FIB sputtering (milling)**
Primary ion

✓ **FIB assisted deposition**
Primary ion

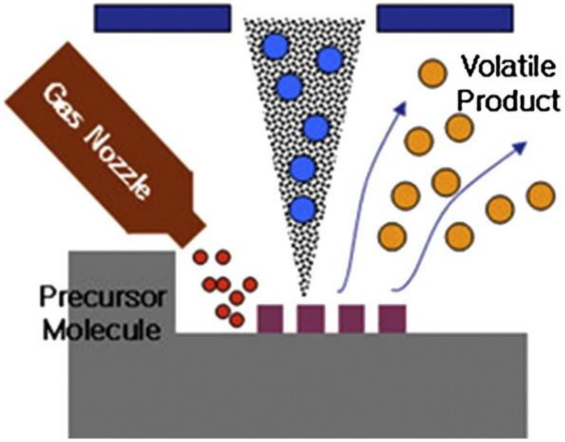
✓ **Ion implantation**
Primary ion



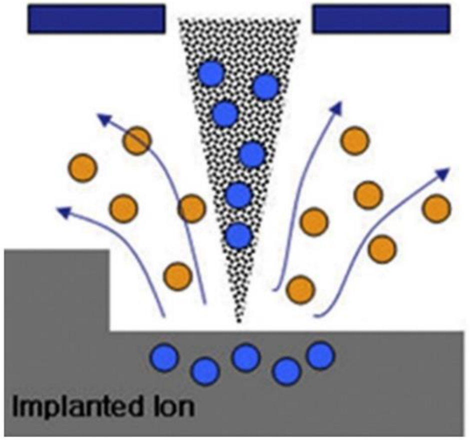
a imaging



b milling

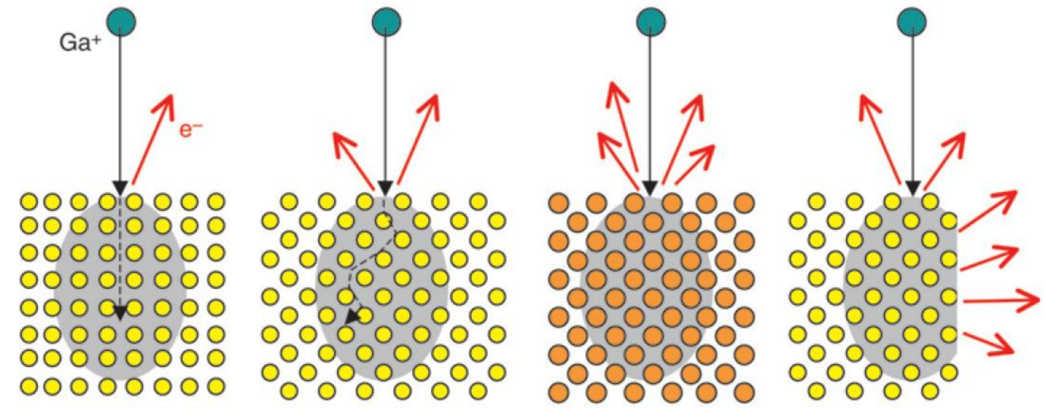


c deposition



d implantation

ISE imaging in a FIB–SEM

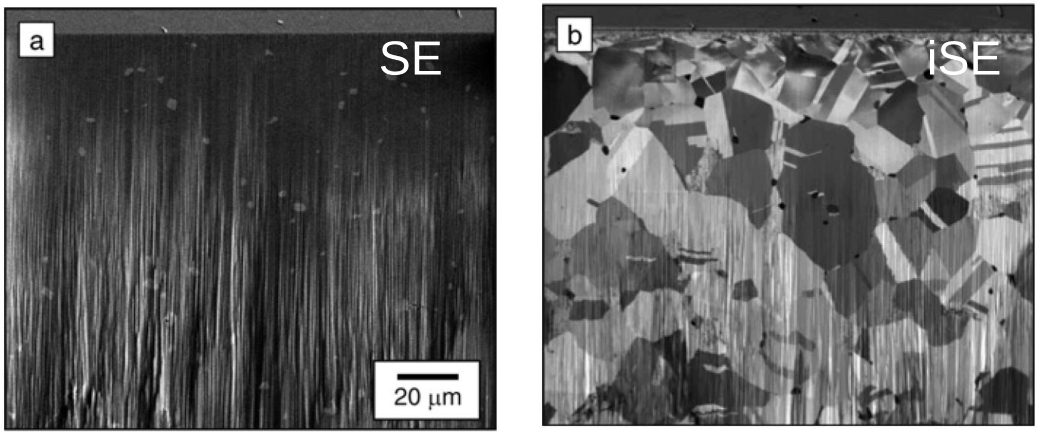


Crystal orientation (100)

Crystal orientation (110)

Heavier atoms (110)

Surface geometry (110)



SE and iSE images from a FIB-cut brass

C.A.Volkert et al., MRS bulletin 32, 389, 2007

- ➢ Mainly detecting iSEs for imaging in a FIB
- ➢ A few 1-10 iSEs (10eV) / Ga ion (5-30kV)

Contrast mechanism of iSE imaging

- ✓ Ion “channels” parallel to crystal planes, fewer electrons are emitted.
- ✓ Heavier samples typically result in more ISEs (and SEs).
- ✓ Surface topography can lead to increases in the number of ISEs (and SEs).
- ✓ Offering complementary information about a sample surface.

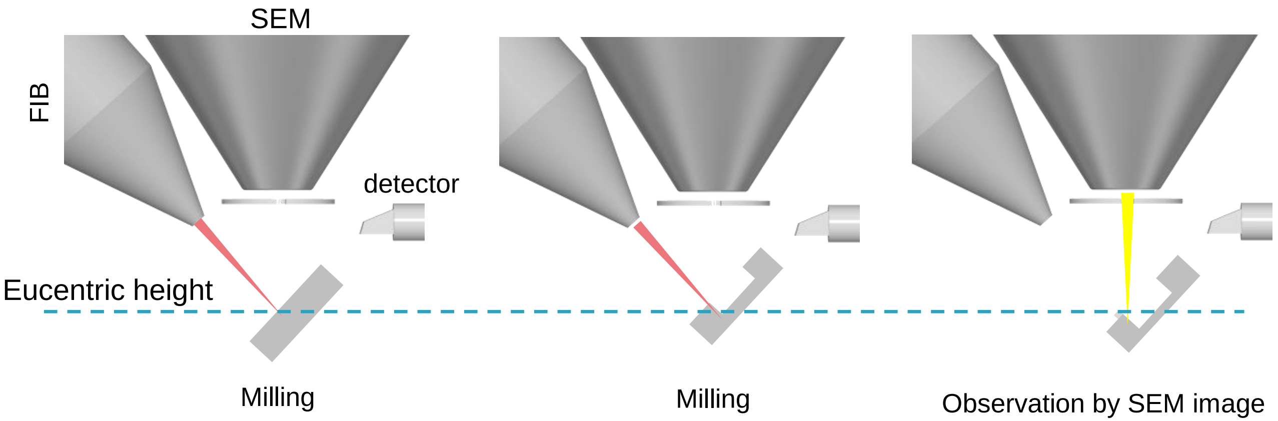
Drawbacks by iSE imaging

Surface damage and ion implantation

- ❖ **Channeling effect** in iSE imaging is obvious**!**
- ❖ **Imaging resolution:** ~5 nm

FIB milling

JIB-4700F



FIB-SEM(Dual-FIB)

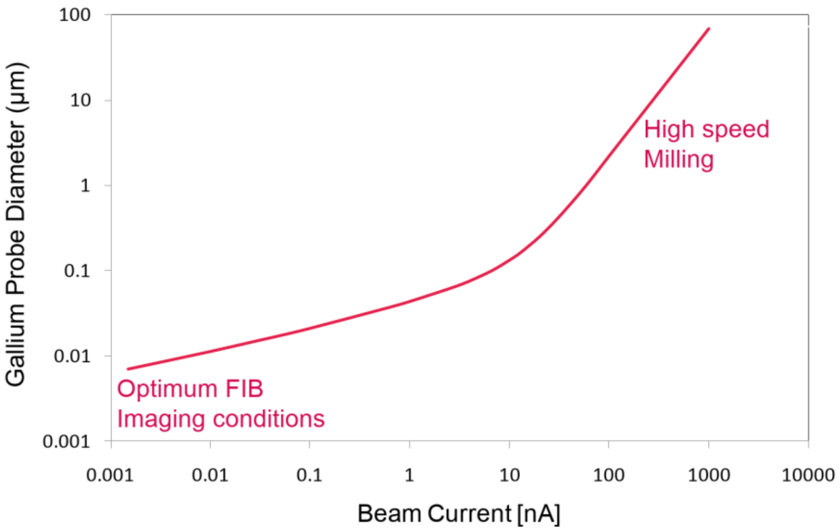
Ion Milling ⇒ Observation by SEM image

To optimize the following parameters for efficient milling:

(1) Ion beam parameters (ion energy, probe current, and beam diameter),

(2) Processing parameters (dwell time, beam overlap, ion dose, scanning mode)

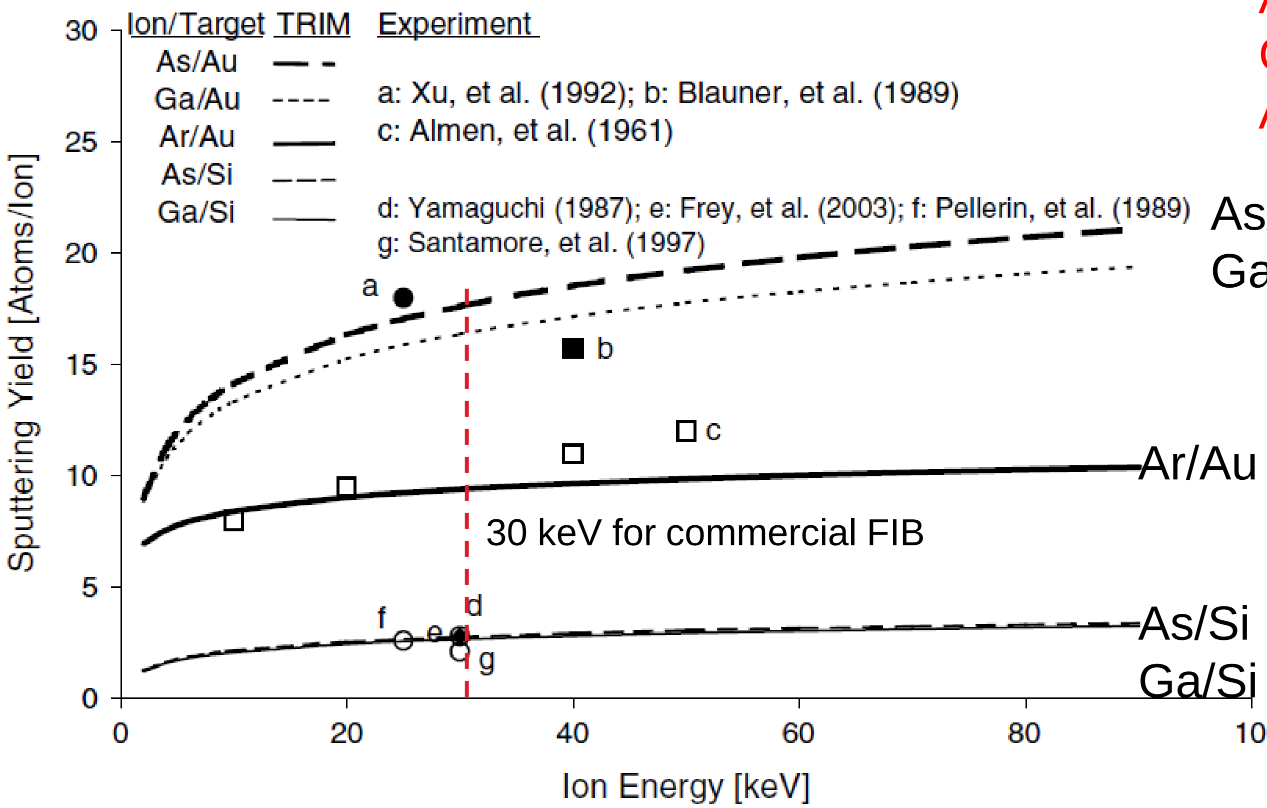
(3) Target materials (mass, density, and crystallographic orientation)



FIB milling

Energy dependence of sputtering yield

Ar: Z=18
Ga: Z=31
As: Z=33



As/Au
Ga/Au

Ar/Au

As/Si
Ga/Si

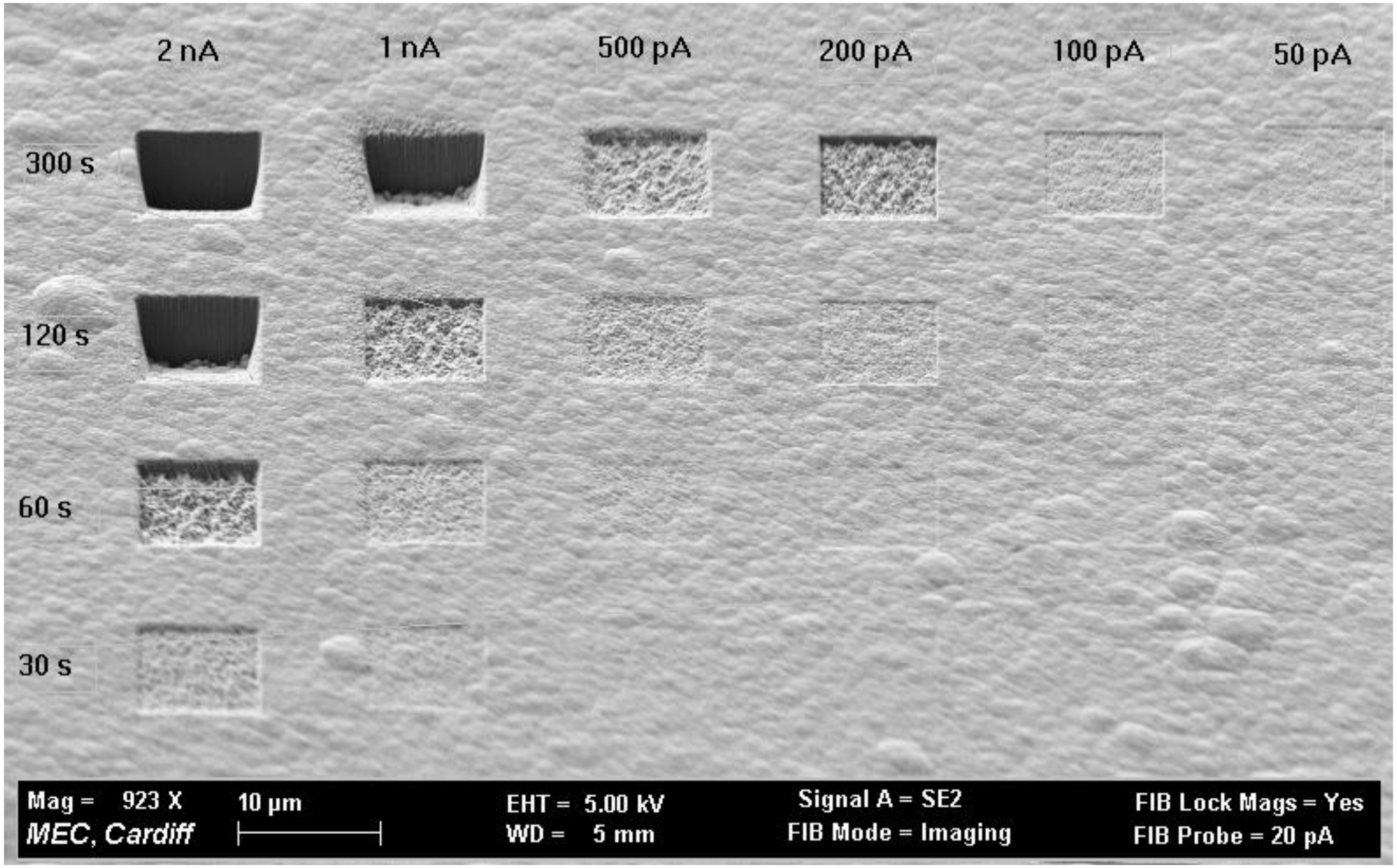
Energy dependence of sputtering yield of Au and Si target substrates by three types of ions at normal incidence.

- ✓ Sputtering yield “saturates” at ~100keV.
- ✓ Higher energy leads to significant implantation

“Recent developments in micromilling using focused ion beam technology”, Tseng, 2004

FIB milling

Probe current, beam diameter and milling time

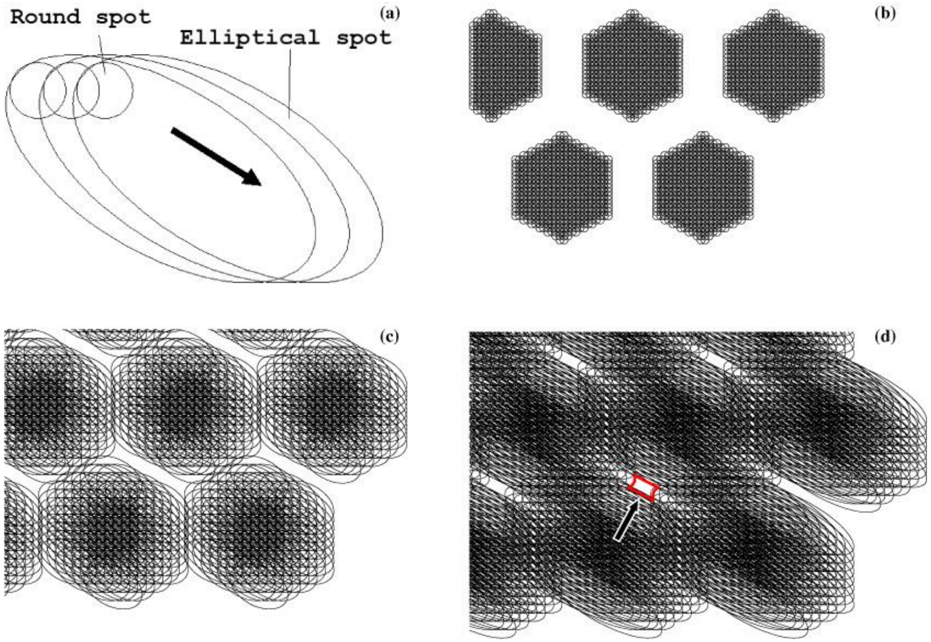


- ✓ At higher probe current (i.e. larger spot size), higher sputtering yield but lower resolution, vice versa.

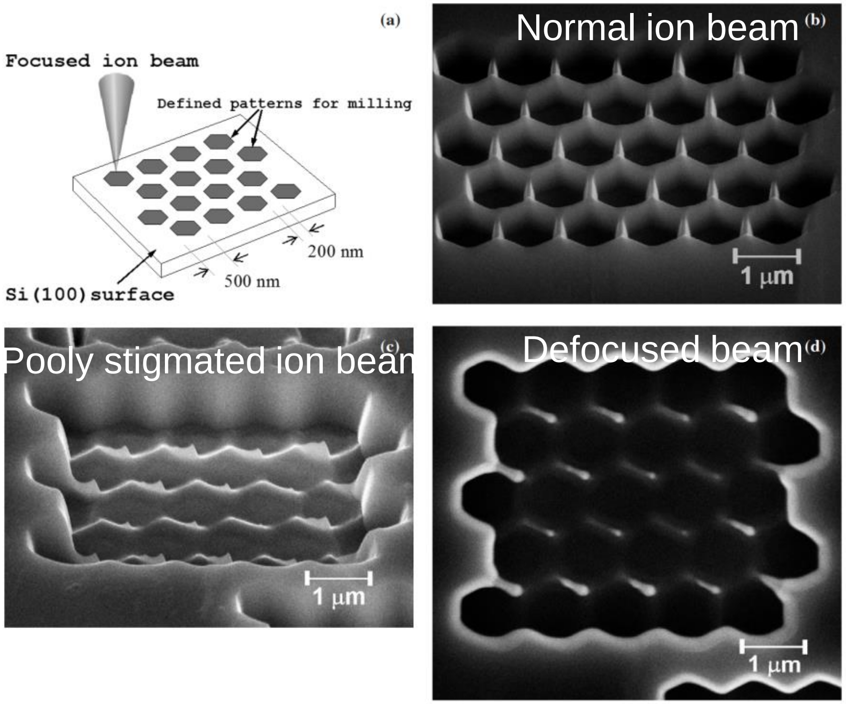
FIB milling

Focus and astigmatism

Much necessary with **good focus** and **low astigmatism** before milling!



Shape of the beam spot with and without distortion

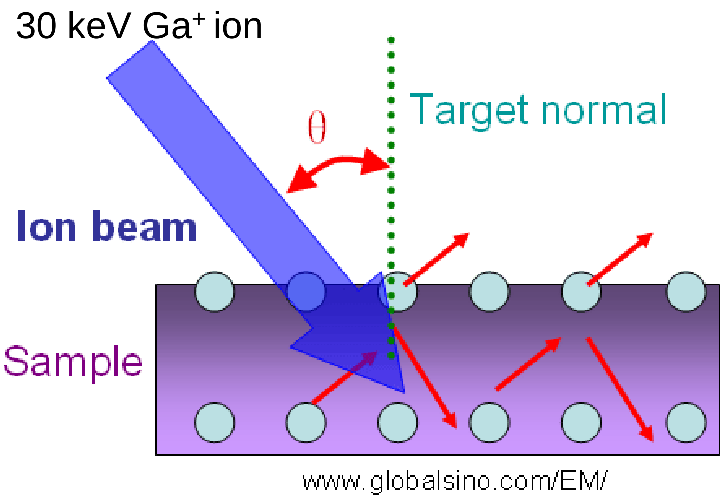


- ❖ With astigmatism and out-of-focus ion beam, each spot may become elliptical and elongated. Thus, the distorted beam finally causes the unwanted milling.

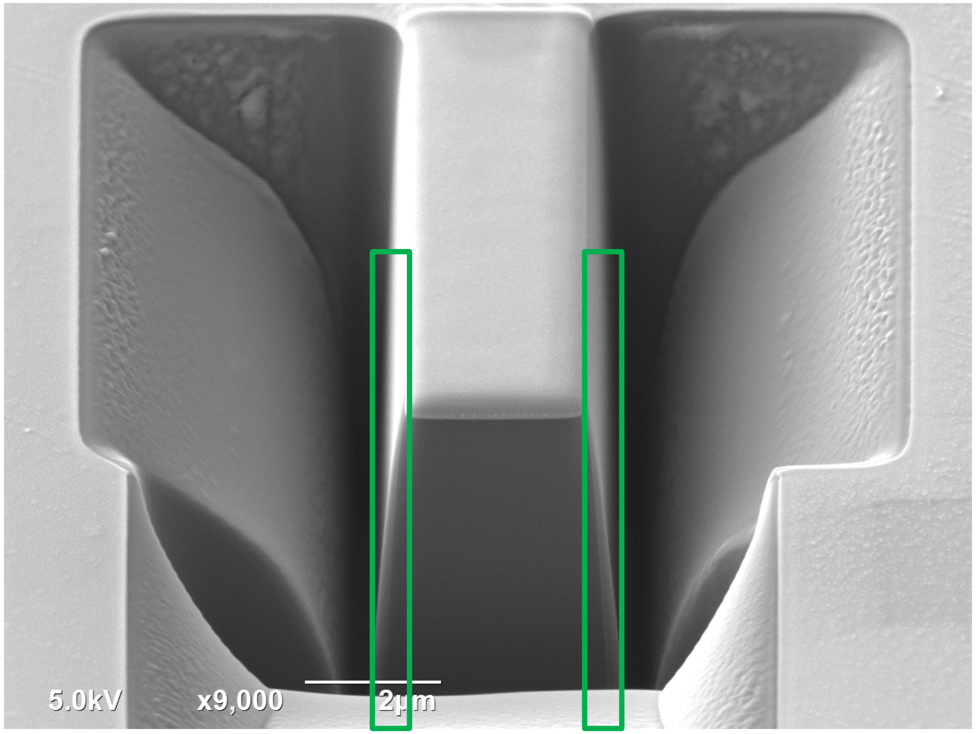
Nanotechnology 16 (2005) 2507–2511

FIB milling

Incident angle



www.globalsino.com/EM/

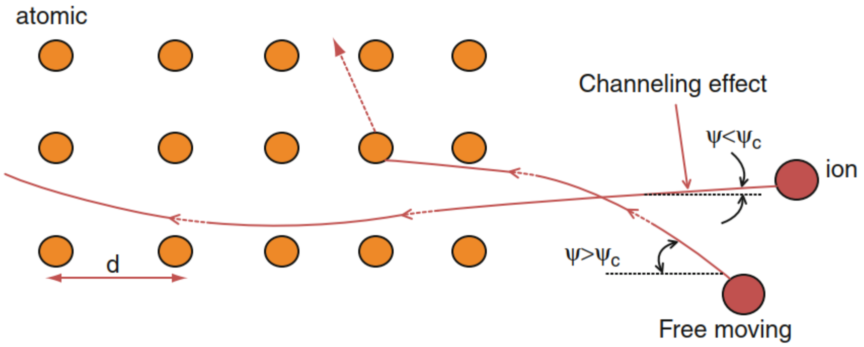


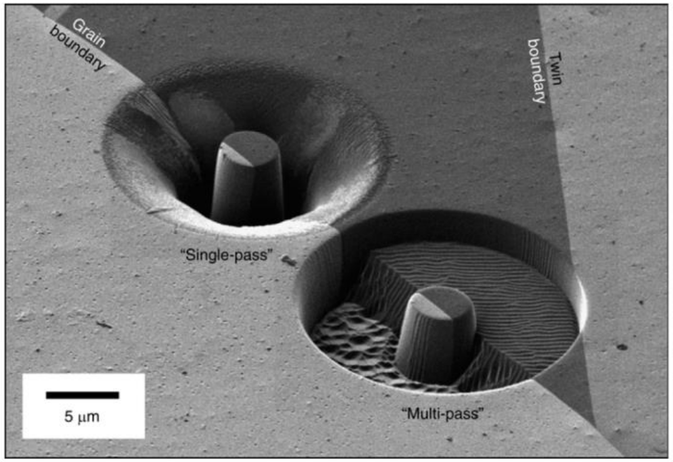
Effect of incidence angle:

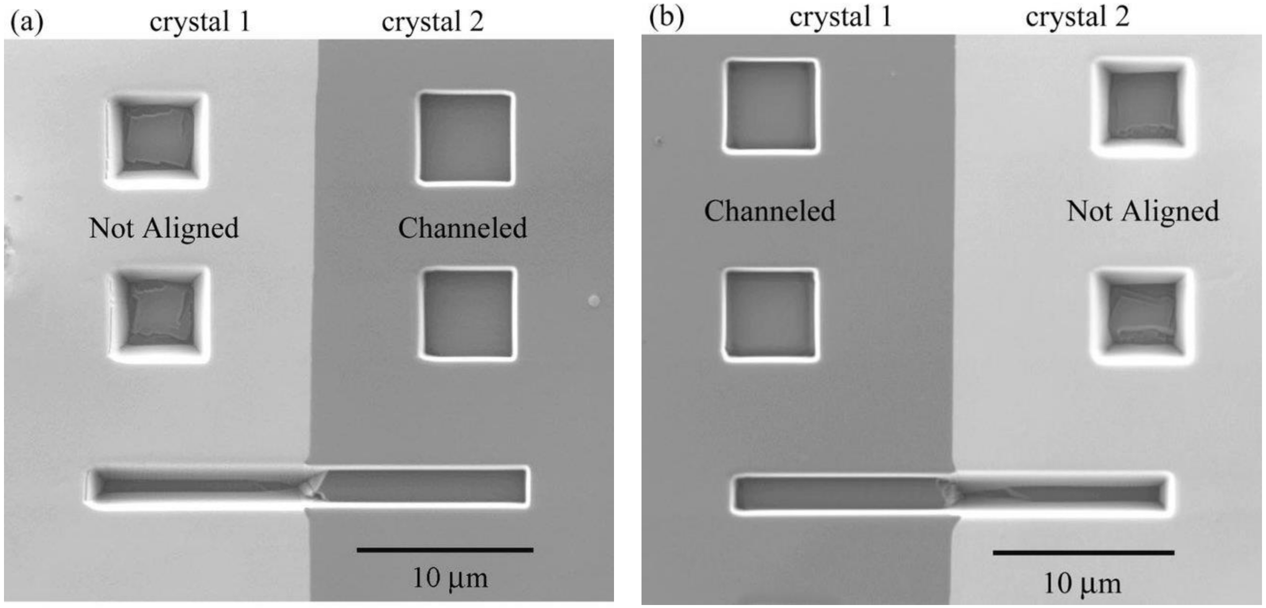
- ✓ Maximum sputtering yields (Sputtered atoms per incoming ion)at angles in the range of 75° to 80°.
- ✓ FIB milling is usually done at normal incidence for vertical trench profile.
- ✓ No longer ‘normal’ once the milling starts-inclined incidence on tampered sidewall.

FIB milling

Channeling effect







Effects of channeling on the FIB milling of a Cu 10° /100 twist bicrystal at (a) 0° tilt and (b) 10° tilt.

Ion channeling effects:

- ✓ Reducing sputtering yield,
- ✓ Low processing efficiency,
- ✓ groove-like morphology of the surface
- ✓ High surface roughness,

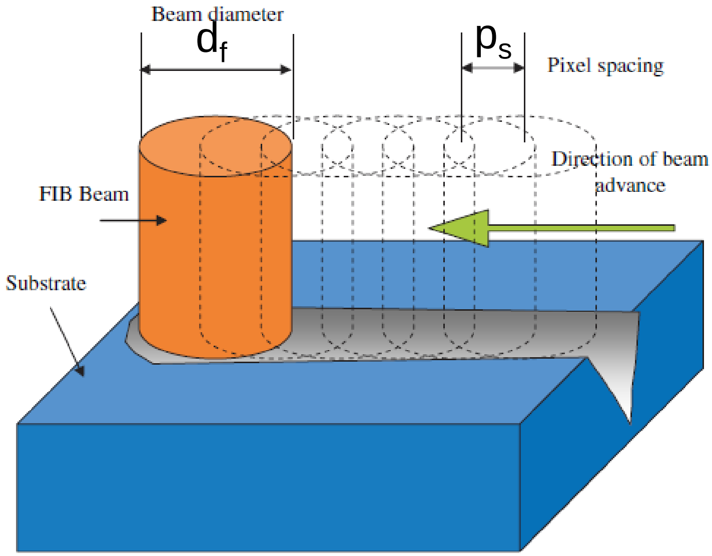
Relevant factors:

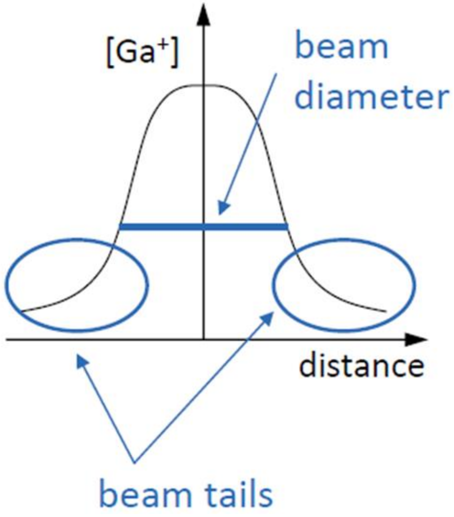
- ✓ Angle of incidence of the ion beam
- ✓ characteristics of the ion
- ✓ Orientation of the target.

C. A. Volkert et al., MRS bulletin 32, 389, 2007

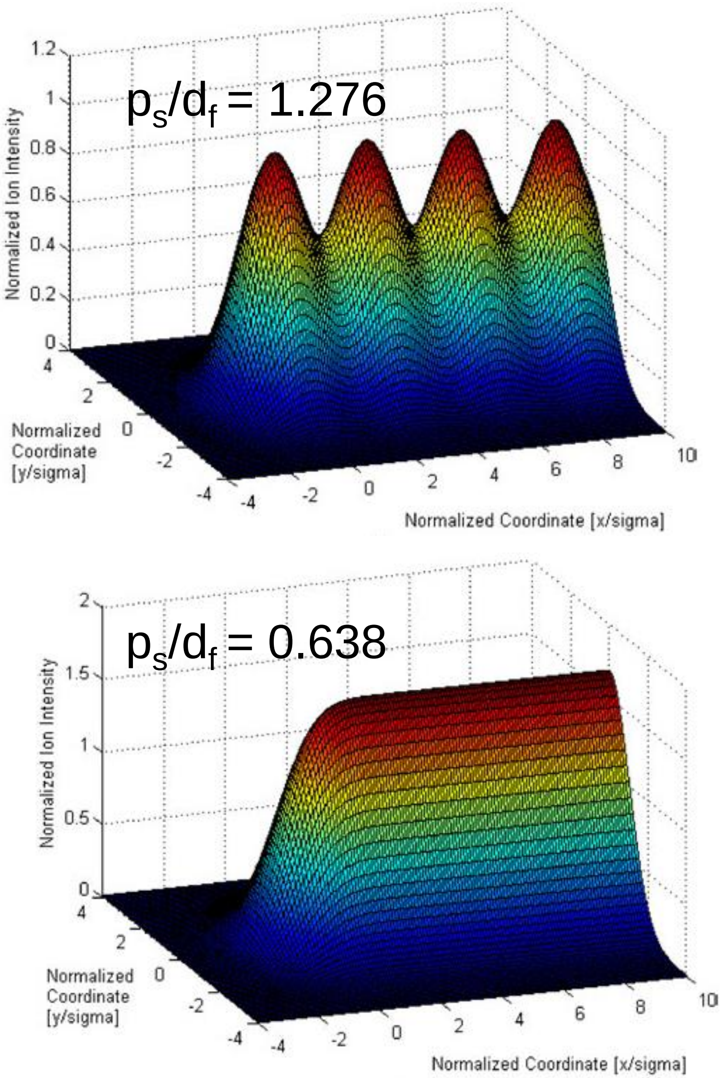
FIB milling

Pixel size vs. beam size





For continuous non-wavy milling, p_s/d_f should be less than 0.638



$p_s/d_f = 1.276$

$p_s/d_f = 0.638$

Ion flux distribution along a scan line with $p_s/\sigma = 3.0$ (top), 1.5 (bottom), $d_f = 2.35\sigma$.

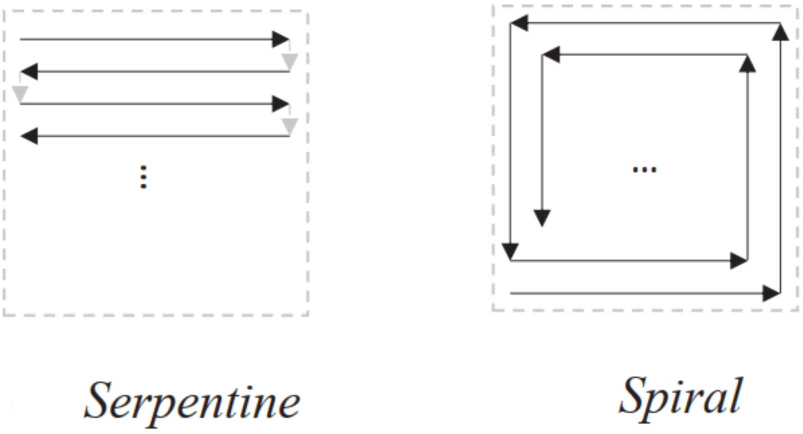
“Recent developments in micromilling using focused ion beam technology”, Tseng, 2004

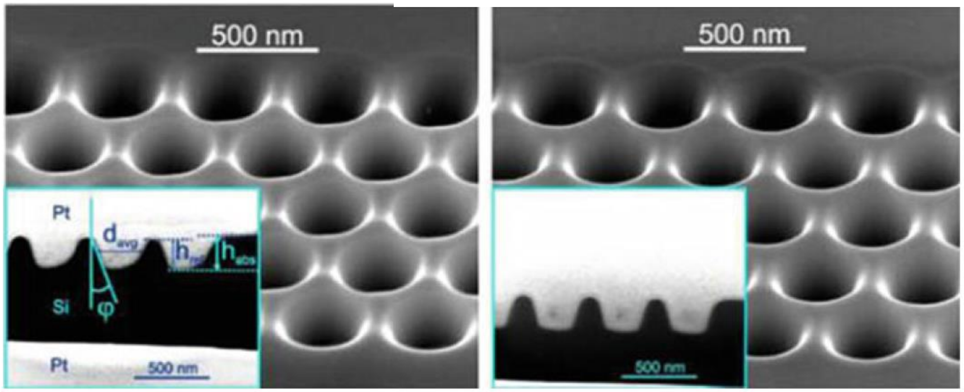
FIB milling

Scan orientation

Merits with spiral scan:

- ✓ The unwanted beam exposure or etching are much smaller in the case of the spiral scan.
- ✓ Shape produced by the spiral scan is much more symmetric.
- ✓ Redeposited material can be better removed from the sidewalls as the beam progresses from the center of a hole outward,

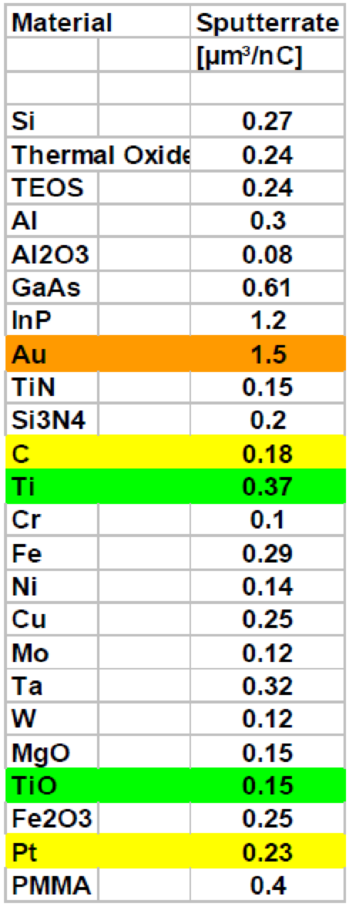




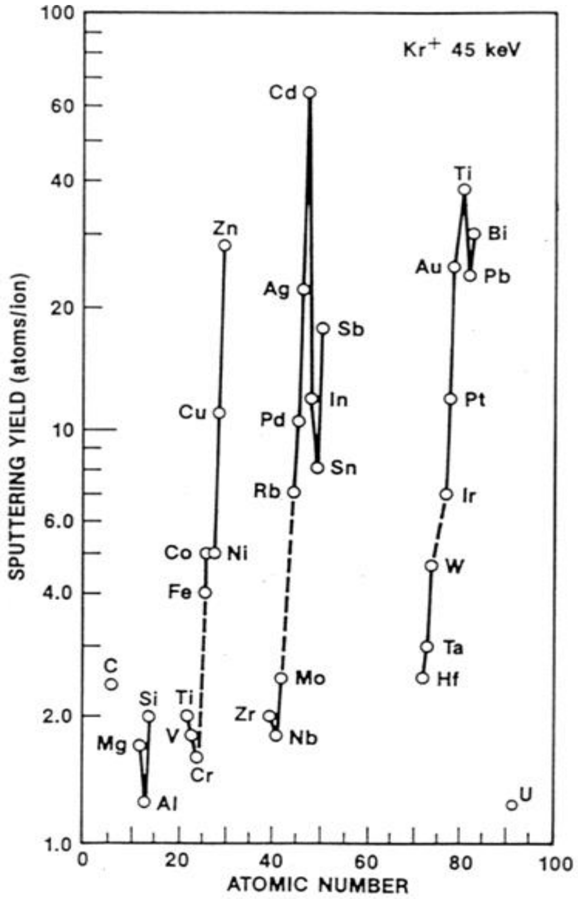
- ❖ Spiral scan -- for milling holes or complexly patterning!
- ❖ Serpentine scan-- for milling a feature with sharp angles (like square pattern).

FIB milling

Sputtering yield of different materials



Material		Sputterrate [µm³/nC]
Si		0.27
Thermal Oxide		0.24
TEOS		0.24
Al		0.3
Al2O3		0.08
GaAs		0.61
InP		1.2
Au		1.5
TiN		0.15
Si3N4		0.2
C		0.18
Ti		0.37
Cr		0.1
Fe		0.29
Ni		0.14
Cu		0.25
Mo		0.12
Ta		0.32
W		0.12
MgO		0.15
TiO		0.15
Fe2O3		0.25
Pt		0.23
PMMA		0.4



- ✓ Sputtering yield varies with material, orders of magnitude difference across periodic table.
- ✓ Actual rate much lower due to re-deposition of sputtered material.

FIB milling

Ion beam artifacts (Re-deposition)

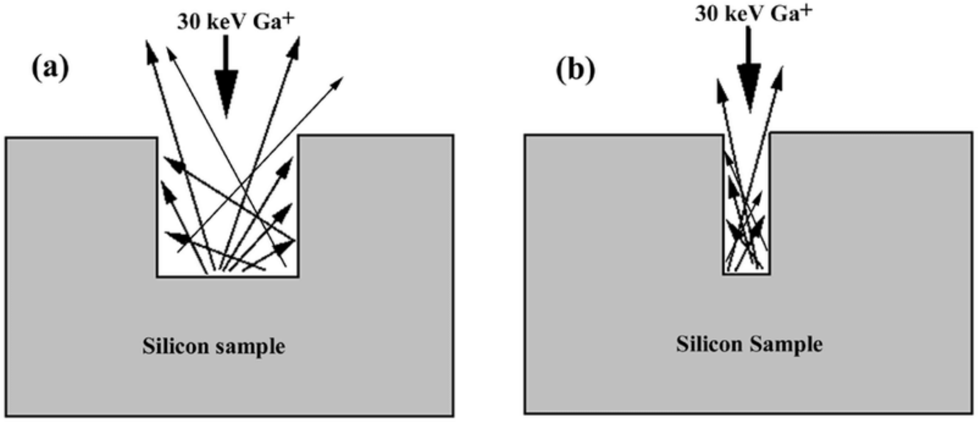
Re-deposition:

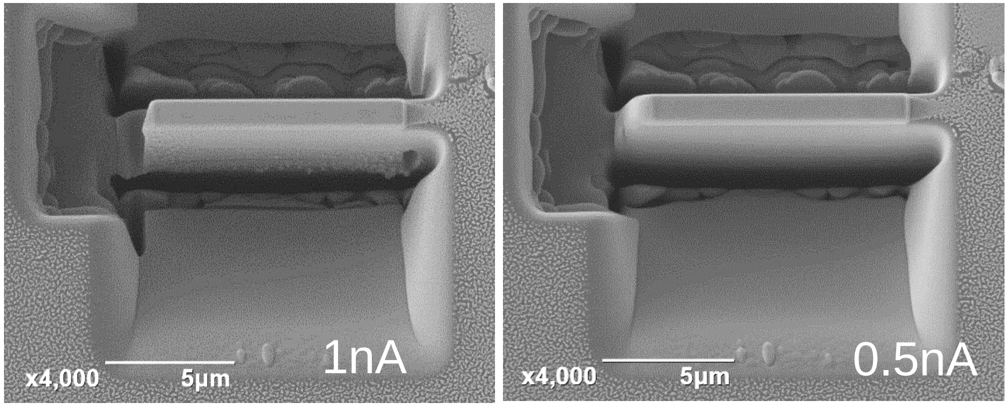
During ion milling, a portion of the ejected atoms bump back into the already sputtered surface and redeposit onto it.

Re-deposition depends on:

- Kinetic energy of atoms leaving surface
- Sticking coefficient of target
- Sputtering yield of target
- Geometry of feature being milled

Factors that increase sputtering rate tend to increase re-deposition:
i) FIB milling is performed in a confined trench.
ii) FIB milling is performed in a high-aspect-ratio trench.
iii) Higher ion beam currents are used.





Re-deposition can be greatly reduced by broadening trench width, decreasing probe current and multiple passes scanning!

FIB milling

Ion Beam artifacts–curtaining effect

Curtaining effect (by non-planar milling of the surface)

Due to competition between smoothing by surface diffusion or viscous flow and roughening because of surface curvature-dependent sputter yields.

i) Rough surface

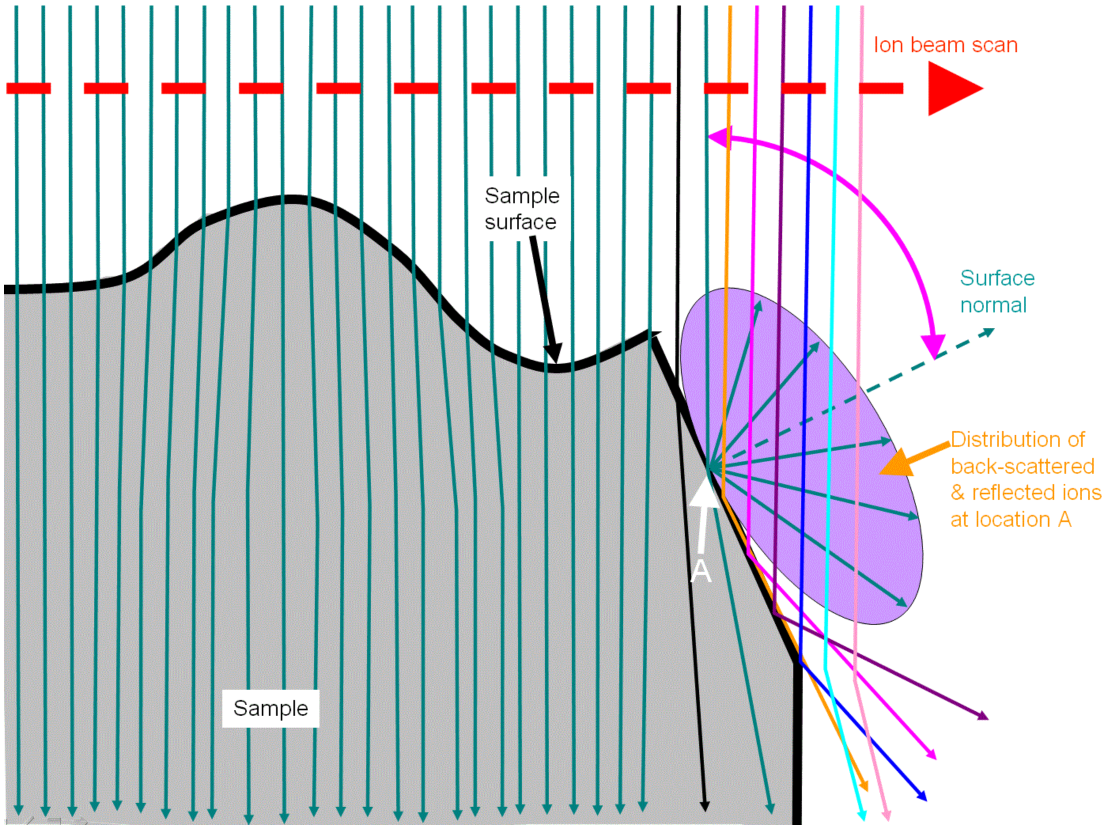
ii) A surface with uneven chemical composition.

iii) Composites of hard and soft materials.

iv) Height steps (e.g. patterned structures in semiconductors)

v) A porous structure.

vi) Curtaining effect increases with lower acceleration voltages which is used for high quality samples.



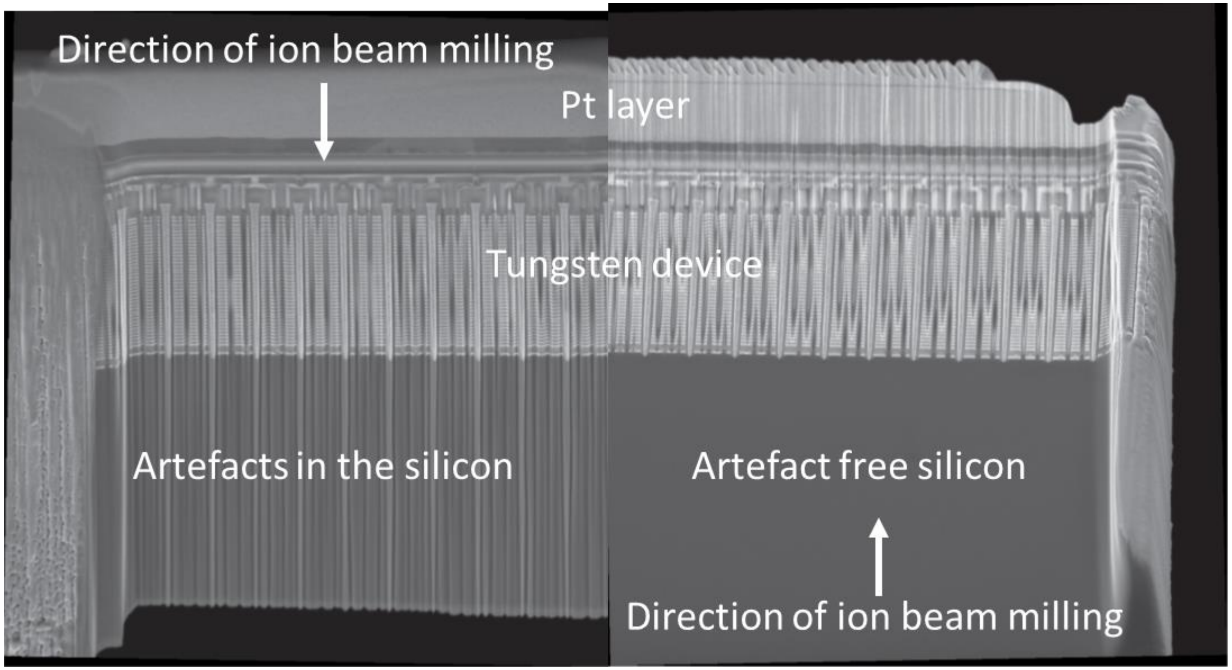
FIB milling

Ion Beam artifacts-curtaining effect

To eliminate the curtaining artifacts:

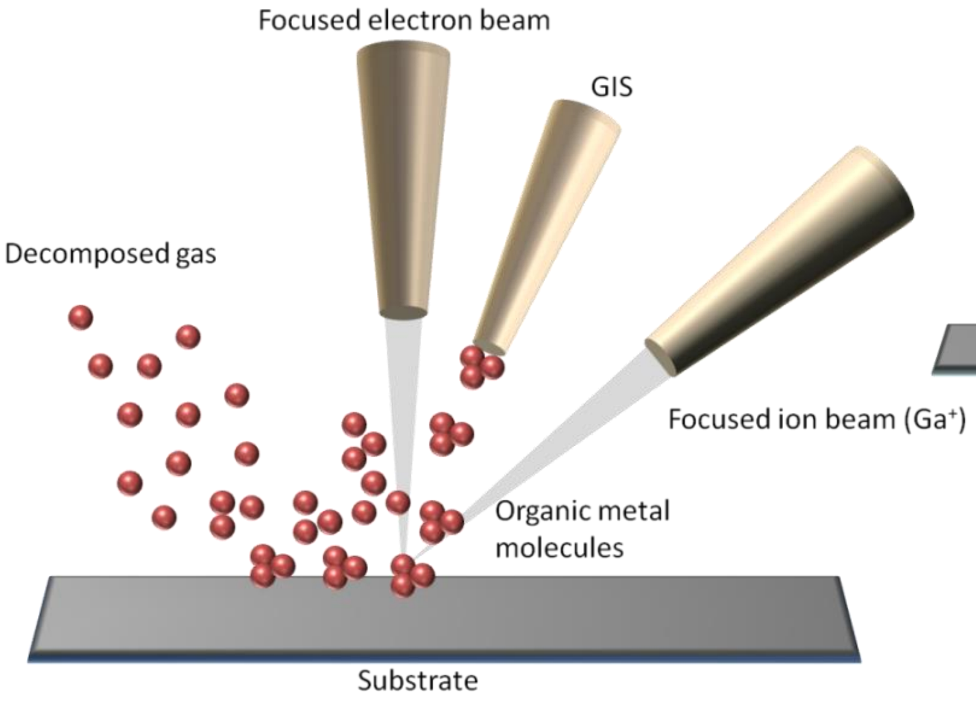
1) Ion milling direction coming from a homogeneous material.
2) Use thick, uniform and dense protection cap.
3) Rocking the sample during FIB milling process.
4) Infiltrate the samples, which have porous structures, with low viscosity resin.

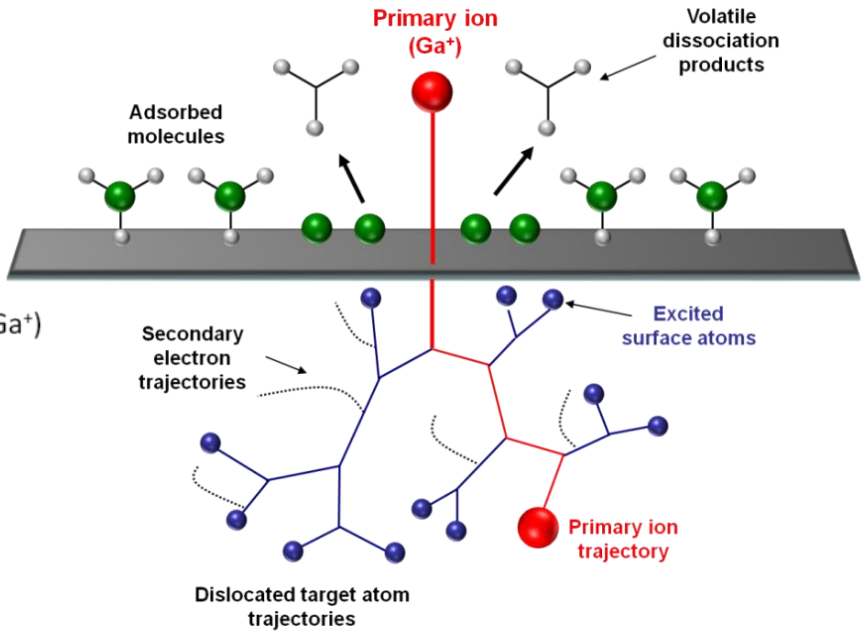
For example:



by Oxford Instruments NanoAnalysis

FIB-Deposition



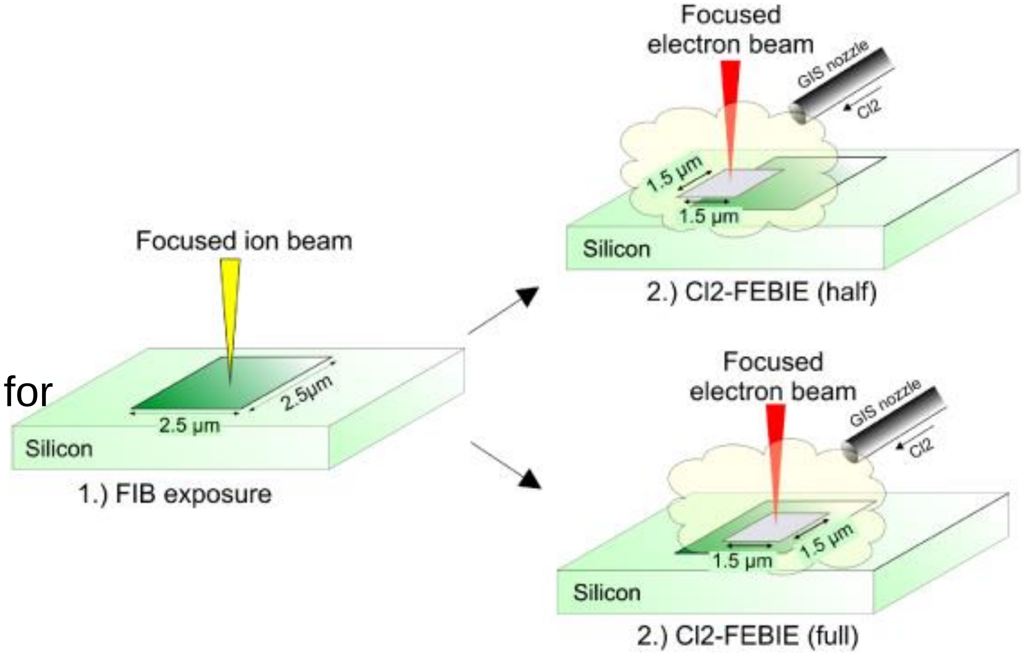


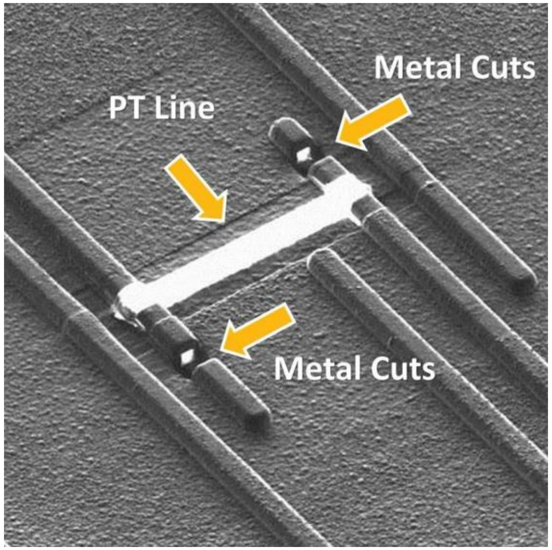
- ✓ Stream of a gaseous organometallic platinum or polymeric carbon compound
- ✓ Ga^+ beam (mild current avoiding a high rate of sputtering) causes the cleavage of the platinum or carbon from the volatile components of the precursor compounds.

FIB–Deposition

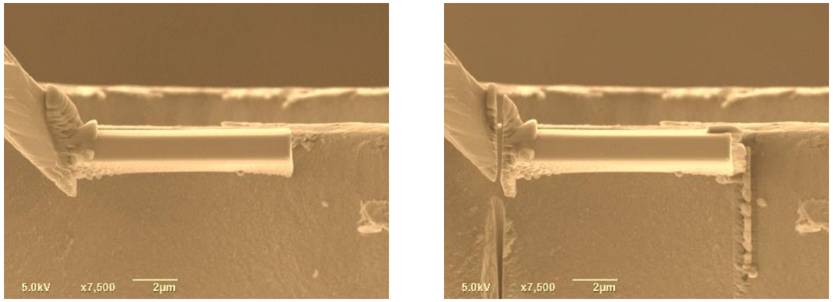
FIB deposition compared to CVD and PVD

- ✓ Locally
- ✓ Site specially
- ✓ No purity (organic residues)
- ✓ Just a few precursor gases are available for the deposition of Pt, W, SiO2, and C.





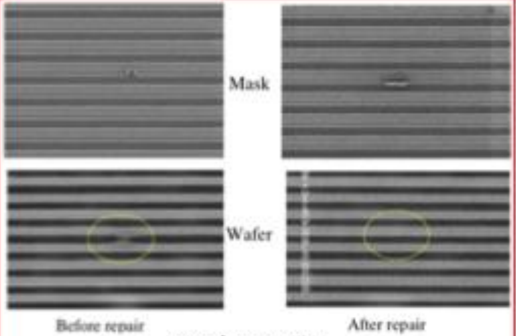
Circuit modification



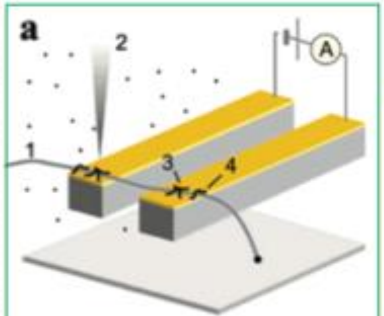
Welding for lift-out process

FIB/SEM–Deposition

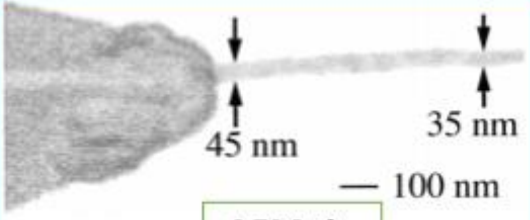
Applications of FEBID



Mask repair
Liang, T et al,. (Vol. 6283, p. 62830K). International Society for Optics and Photonics.

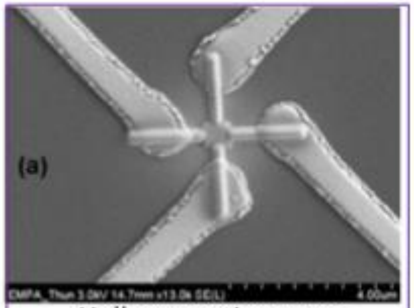


Soldering carbon nanotubes onto electrodes,
Madsen, D. N., *Nano Letters*, *3*(1), 47-49.

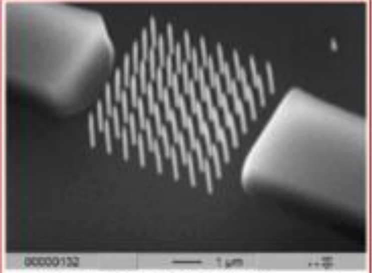


AFM tip

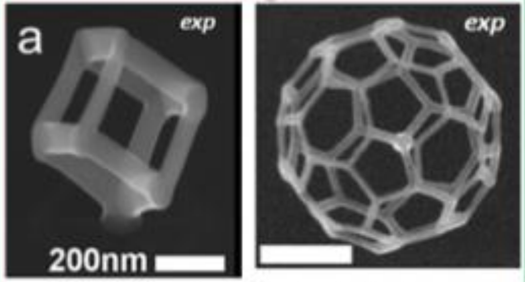
Wendel, M et al *Applied physics letters* 67.25 (1995): 3732-3734.



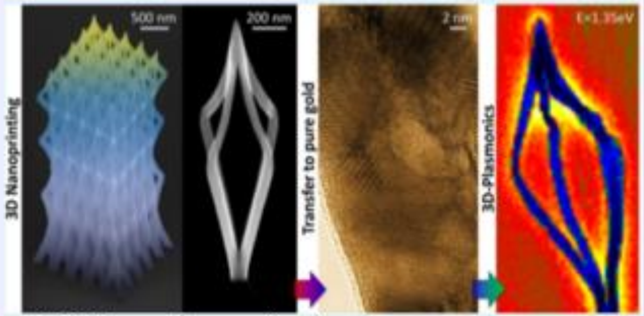
Hall magnetic sensor
Gabureac, M et al. ,
Nanotechnology 21.11 (2010): 115503.



Nano photonics, Koops, H. W. P., et al *Microelectronic engineering* 57 (2001): 995-1001.



3D Nano printing, Fowlkes, J D., et al. *ACS nano* 10.6 (2016): 6163-6172.

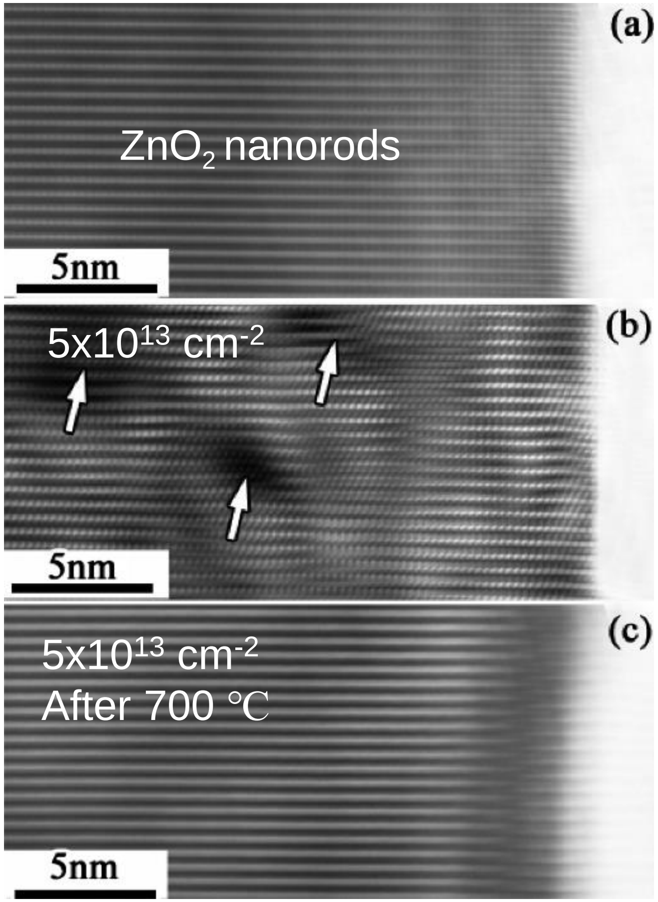


3D Nano plasmonic structures, Winkler, Robert, et al. *ACS Applied Materials & Interfaces* 9.9 (2016): 8233-8240.

FIB-implantation

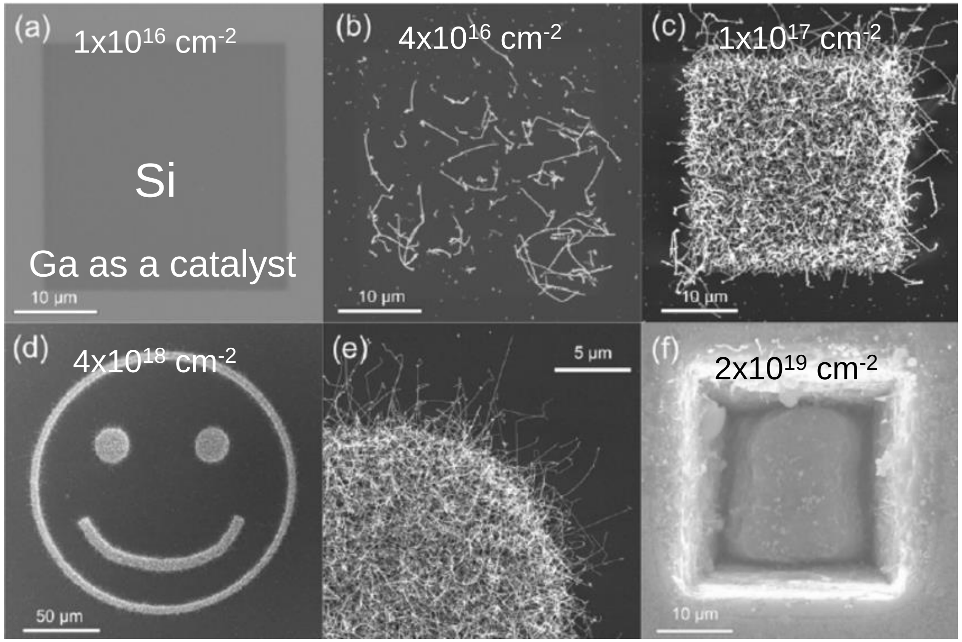
Gallium implantation

- ✓ Alternation of the specimen’s local composition within the interaction volume.
- ✓ Leading to structural changes, as well as alteration in, e.g., thermal, electrical, optical, and mechanical properties.



Annealing effect of Ga-implanted ZnO$_2$ nanorods

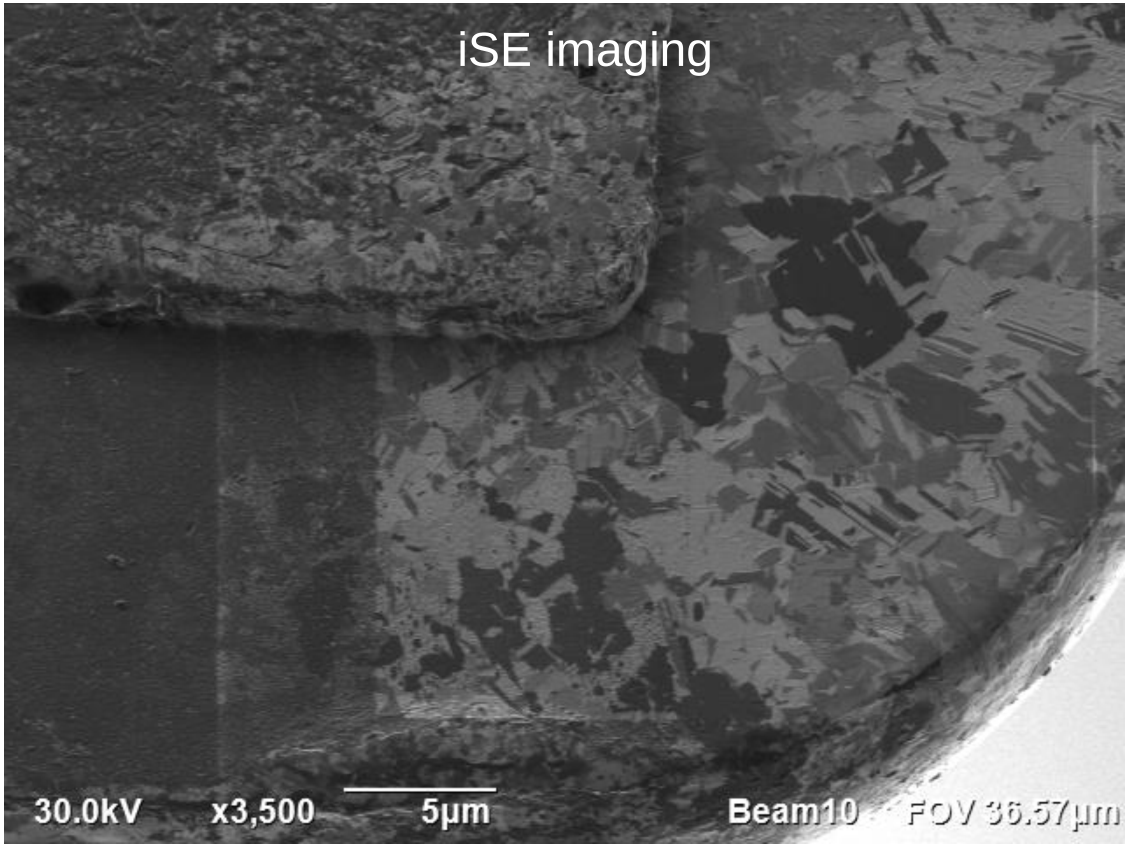
L. Yao et al., J. App. Phys,**105**, 103521 (2009),



Nanotechnology, 22(39) 395601 (2011).

Applications

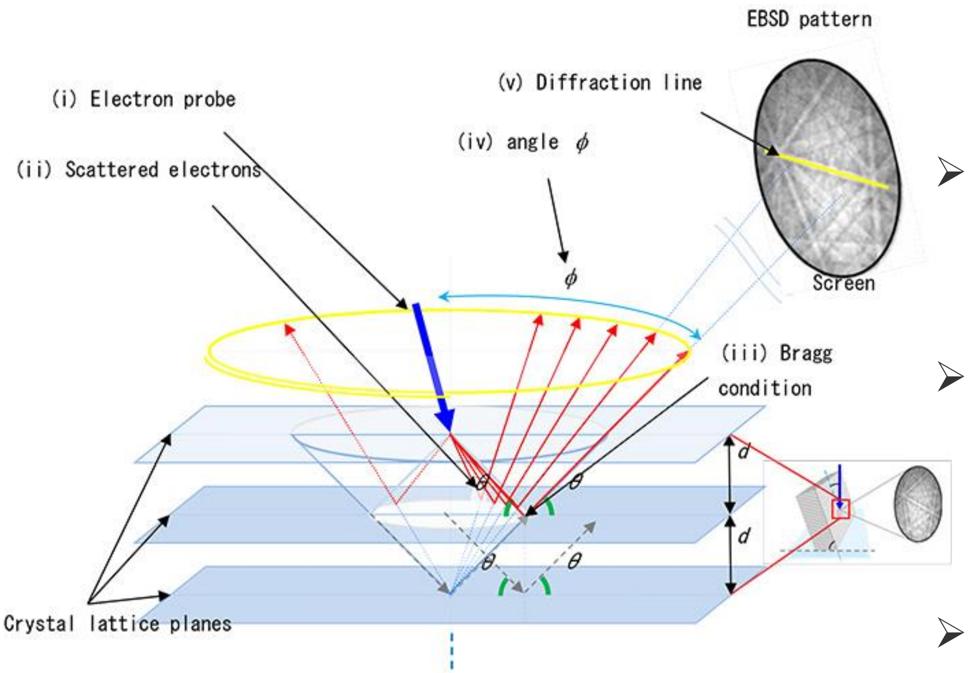
Characterization of polycrystalline micrsoctructure



Any other way to determine crystallographic information of microstructure such as crystal orientation, grain boundaries, local crystal perfection…….?
Electron backscatter diffraction (EBSD) pattern analysis

Applications

Formation of EBSD pattern

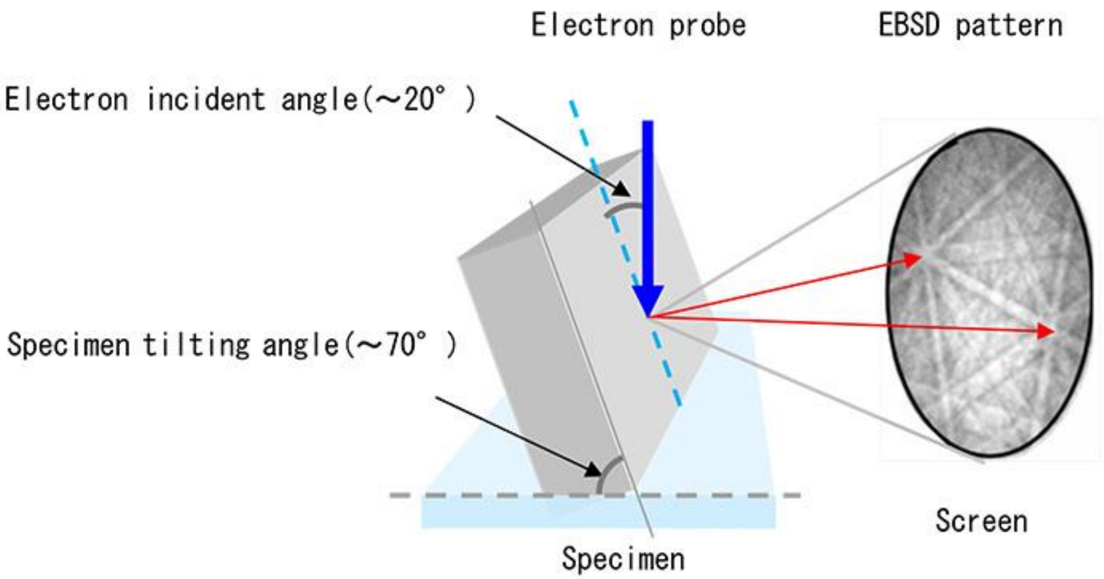


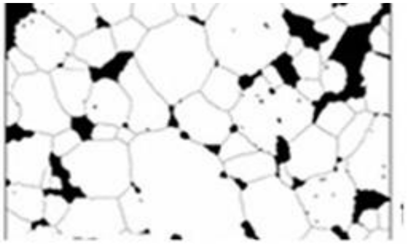
- Elastically or inelastically scattered electrons over a large angular range by sample
- The electrons satisfying the Bragg condition ($2d \cdot \sin\theta = n \cdot \lambda$) are diffracted and produce diffraction spots on the screen.
- The electrons travelling in different azimuthal direction produce successive diffracted spots to finally form a diffraction line.
- Many diffraction lines generated from the lattice planes in different orientations are superposed and thus form an EBSD pattern (well known as Kikuchi pattern).
- For acquiring high intensity of EBSD, specimen tilt angle is about 70°

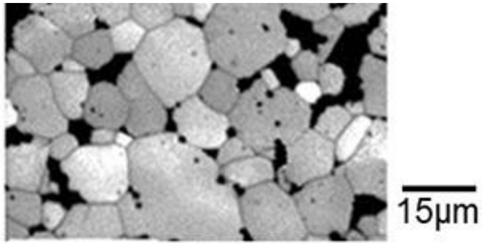
https://www.jeol.co.jp

Applications

Crystal orientation mapping




(c) Grain Boundary map



(d) Image Quality map

https://www.jeol.co.jp

- ➢ If electron probe scans an arbitrary area of the specimen, the crystal orientations of each point of the area are recognized by comparing measured and calculated EBSD patterns.

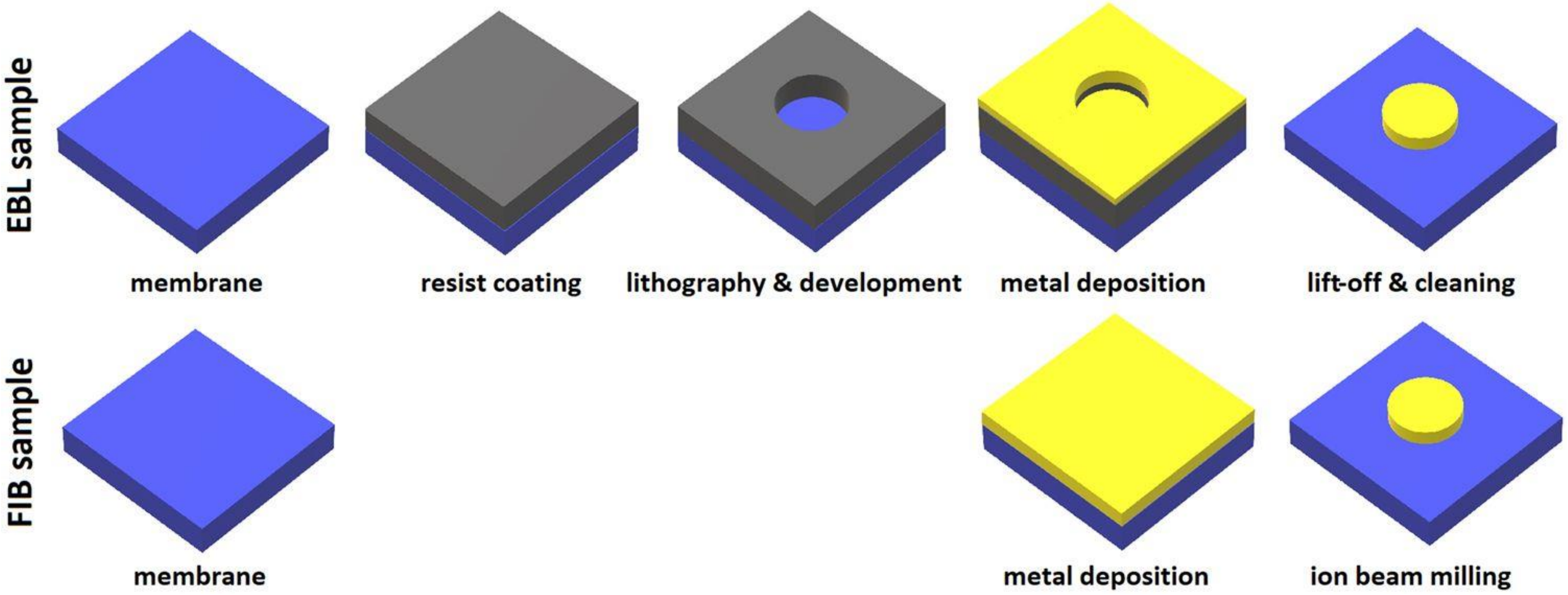
- ➢ Crystal orientation map can be obtained automatically and rapidly.
- ➢ 10-20 nm resultion in Schottky-emission electron gun SEM
  Depth: 30-50nm

If EBSD+FIB

- ✓ Microstructure orientation analysis
- ✓ No necessary with mechanical polishing
- ✓ To reveal grain orientation inside bulk by 3D EBSD

Applications

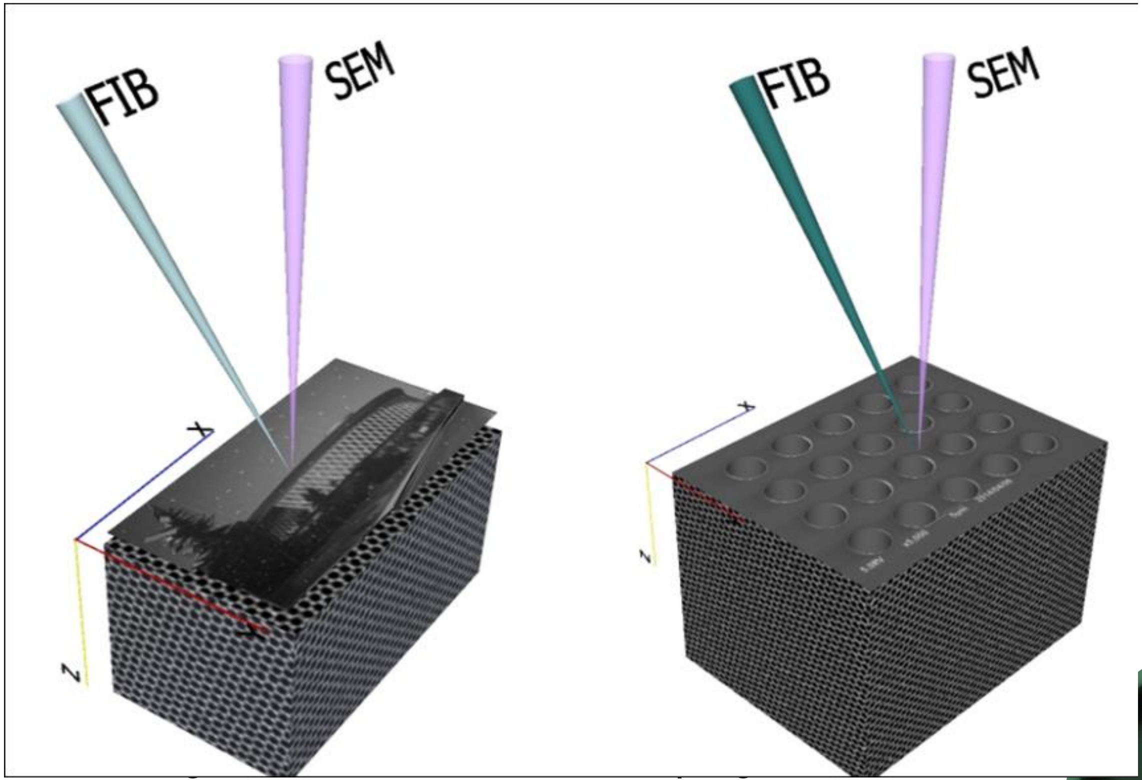
FIB patterning vs EBL patterning



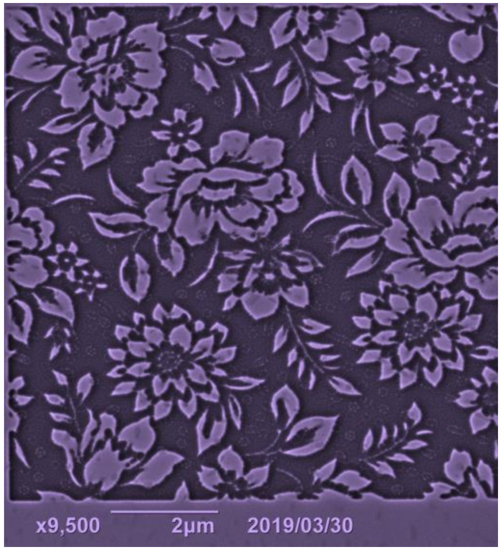
- ✓ FIB Milling allows for creating cross sections or developing structures with desired geometries to control not only the lateral position but also local depth.
- ✓ It does not require the use of masks.

Applications

Etching and patterning @OtaNano-NMC

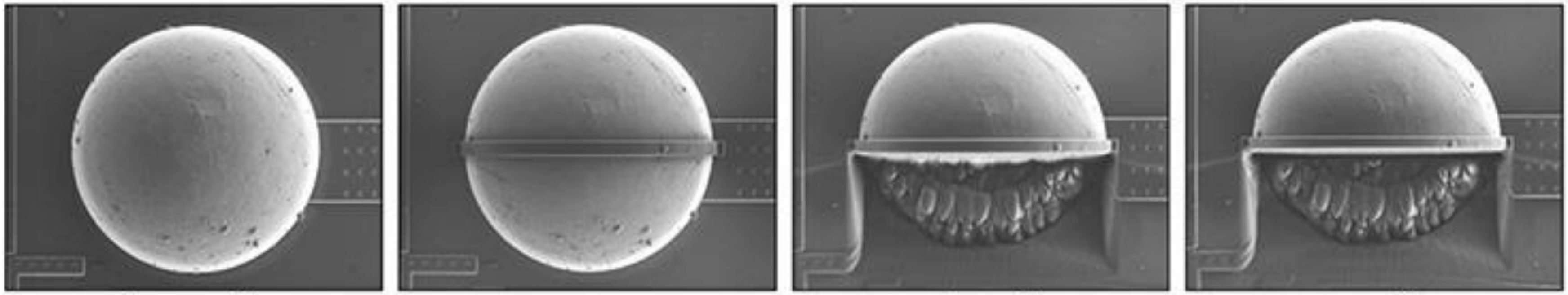


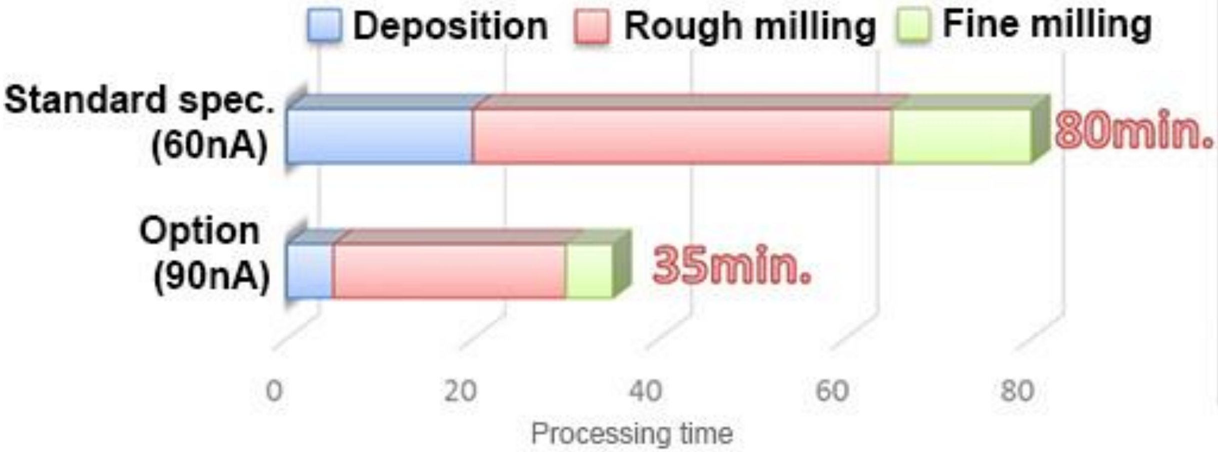
with desired geometries to control not only the lateral position but also local depth.

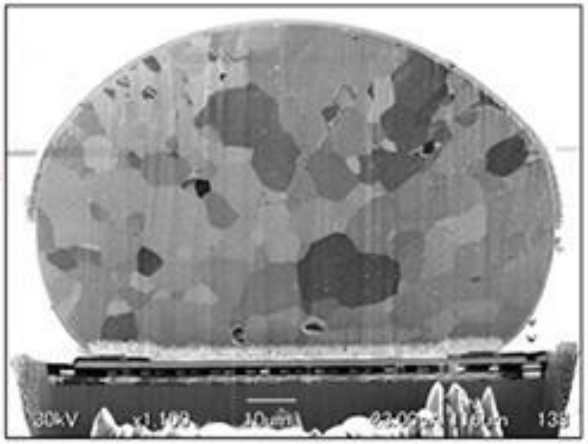


Applications

Cross Sectional SEM sample


Before milling Deposition Rough milling Fine milling

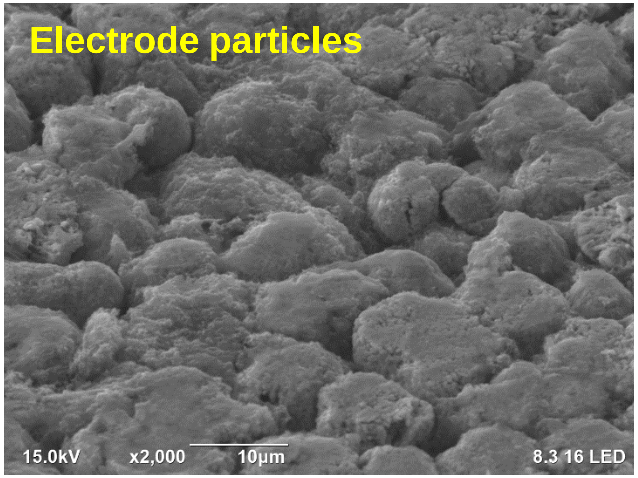


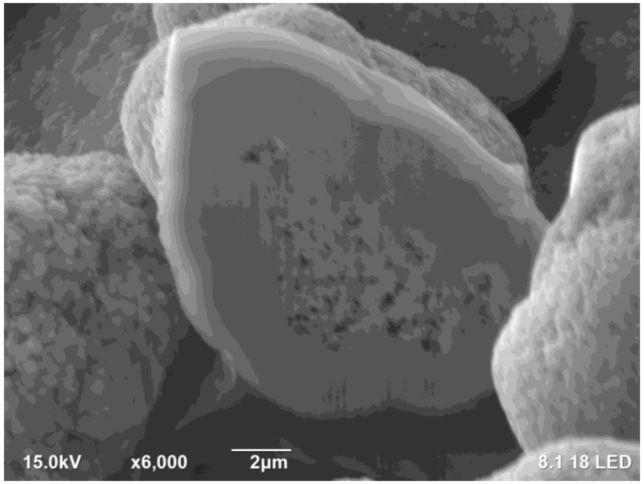

Milled cross section (SIM image)

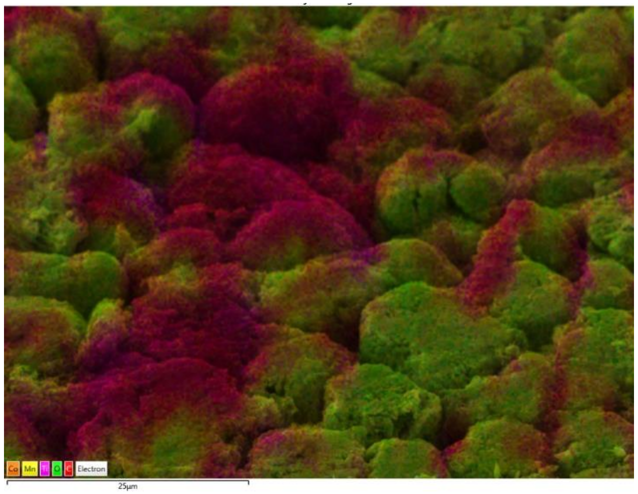
From JEOL

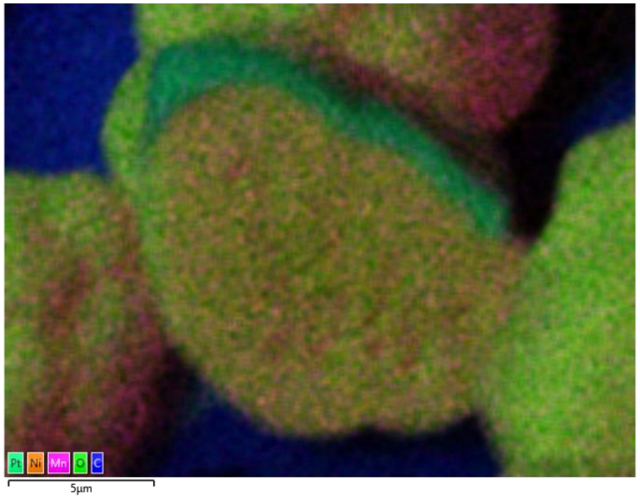
Applications

Cross Sectional SEM imaging/EDX mapping



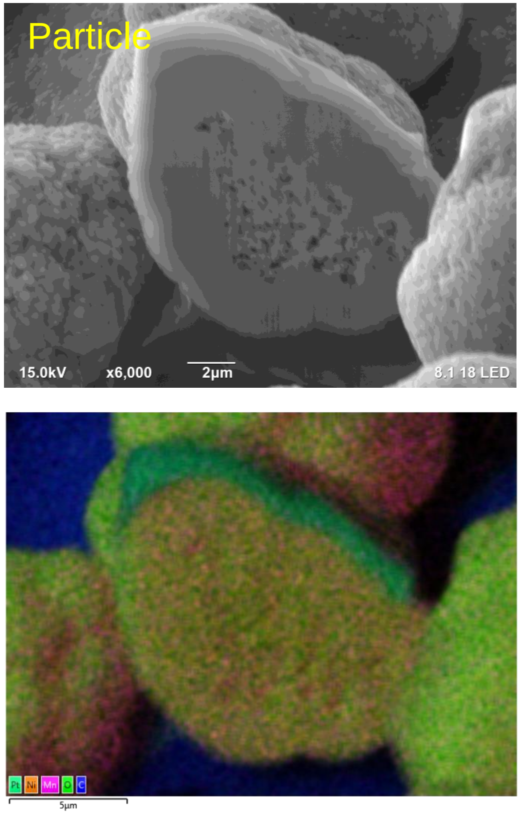


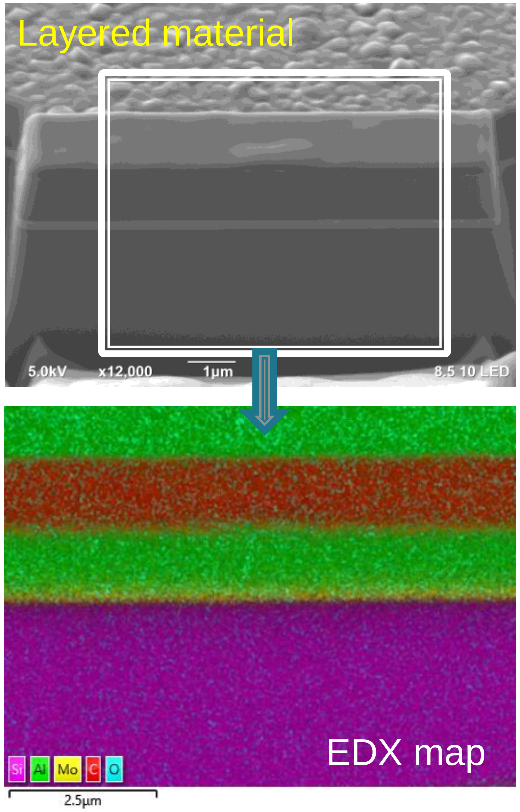


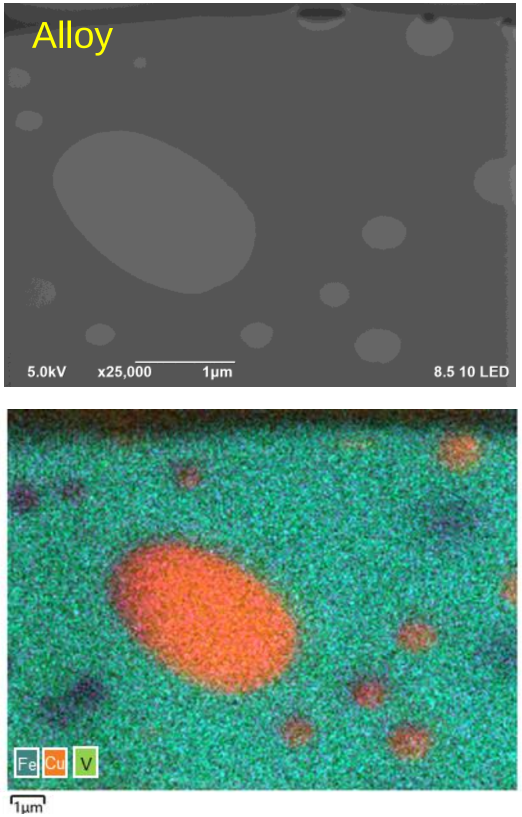


Applications

FIB-cut cross sectional SEM imaging and EDX mapping

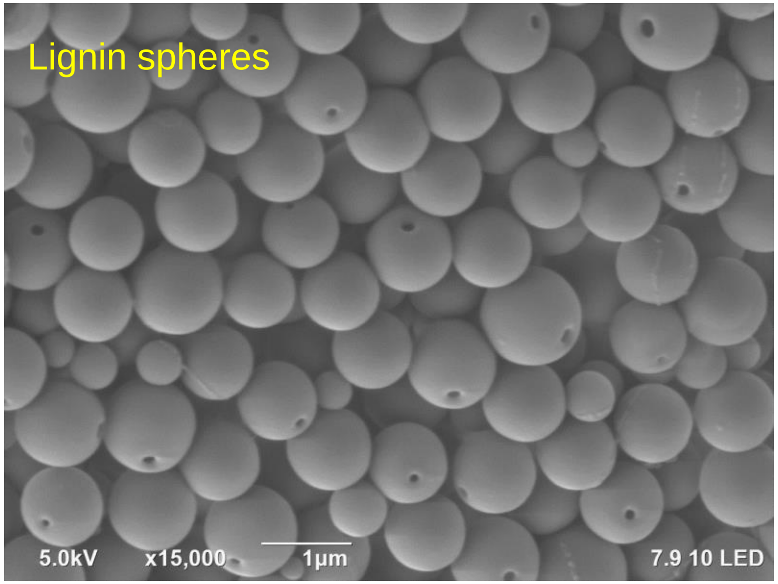




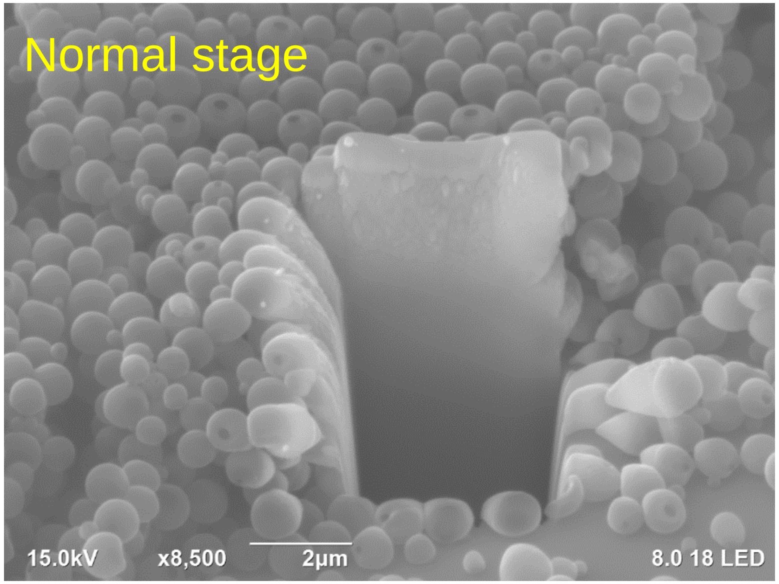


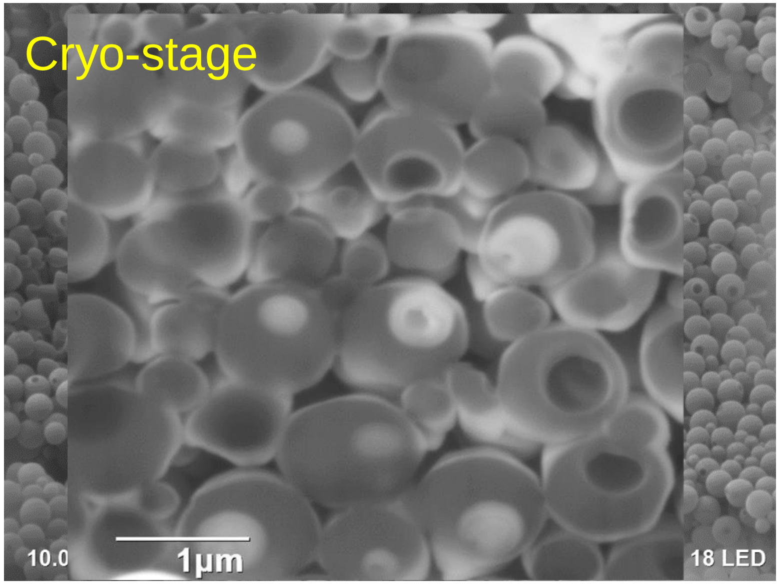
Applications

Cross Sectional SEM imaging with Cryo-stage



FIB-cut





- Cryo-SEM/FIB offers new opportunities to study soft materials, and frozen, hydrated samples from the field of life science.
- FIB-milling enables imaging additional planes by site-specific X-sectioning perpendicular to the cryo-fracture surface, thus adding a third imaging dimension to the cryo-SEM.

How to prepare a successful TEM lamella?

JEOL 2200FS TEM
with double correctors

Mechanical polishing

Ion polishing by FIB

Probe-based

HE150 electrical
probing holder
(by Nanofactory AB)

Chip-based

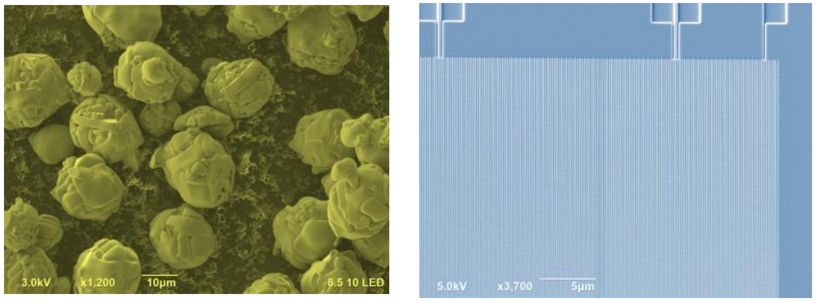
DENSsolutions

Challenging work!

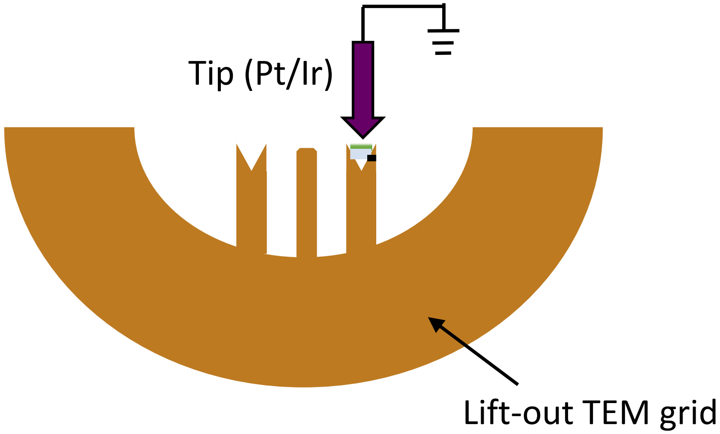
Applications

FIB-cut TEM lamella (in-situ lift-out technique)

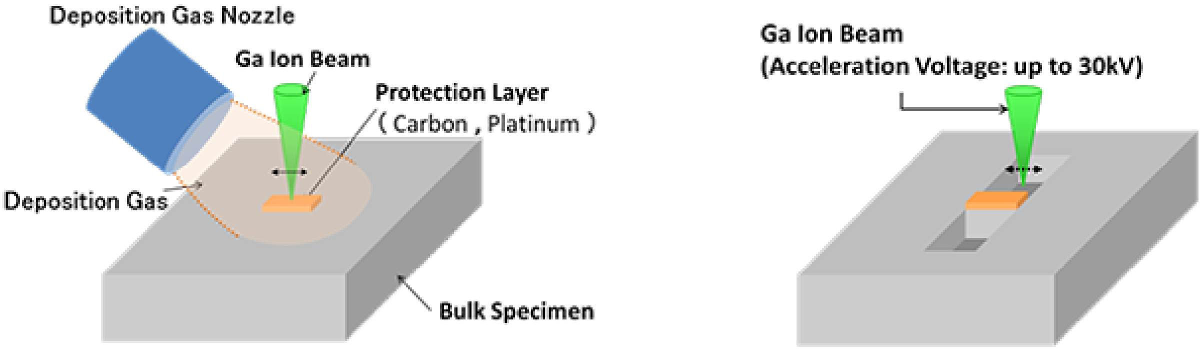
By FIB polishing



- TEM lamella can be prepared site-specifically with a spatial accuracy as fine as 30 nm.
- Compared to other techniques (microtomy, low-energy ion milling, dimpling, etc.), it costs a short time (a few hours).
- Applicable for broad material systems including hard, soft, life materials with **cryo-stage.**

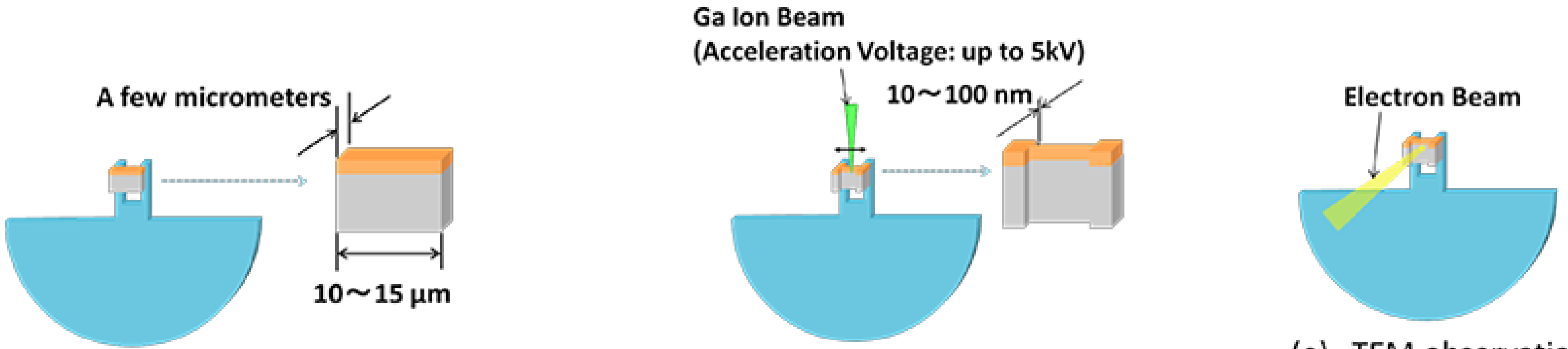


TEM specimen preparation (overview)



(a) Formation of protection layer

(b) Rough milling



(c) Fixation of thin section to TEM grid

(d) Fine milling (Thinning)

(e) TEM observation

https://www.jeol.co.jp

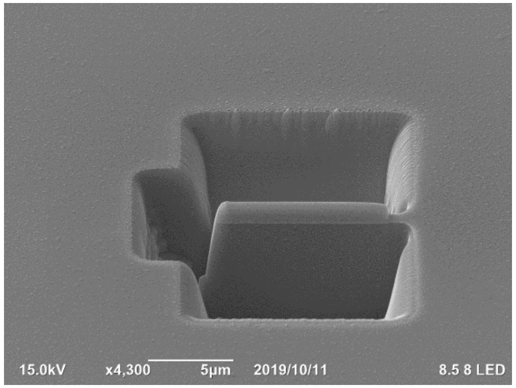
FIB-cut Steps

Prior to FIB, a ~30 nm Pt layer was pre-deposited on the top by sputtering machine

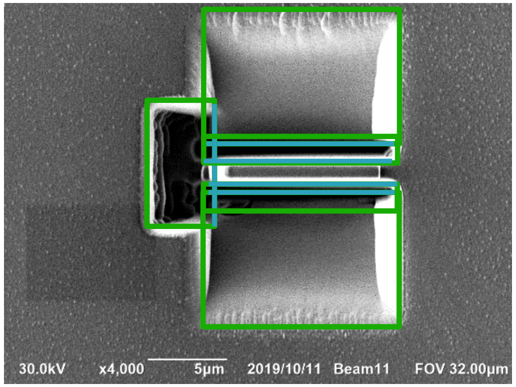
SEM

FIB

Pre-cutting



T: 53°



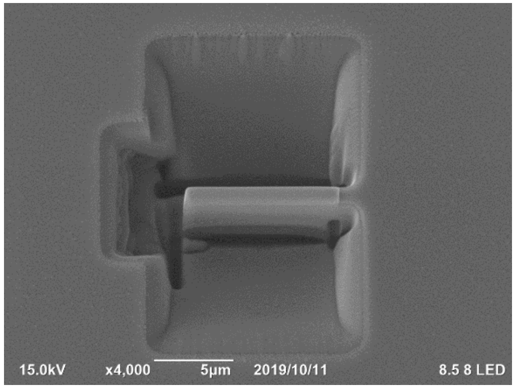
T: 53°

Pattern size:
10 μm L x 2 μm H,

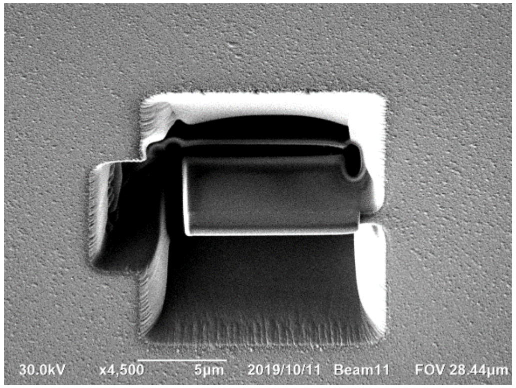
Depo (Pt):
Beam 10 (30pA)
1 μm thick

Grooving:
Beam 4 (10 nA)
Depth 6 μm

Undercutting



T: 8°



T: 8°

Side cutting
Pre-thinning
Under cutting

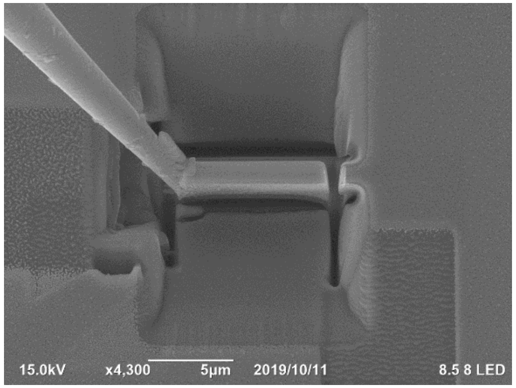
Beam 5 (3 nA)
Depth 6 μm

FIB-cut Steps

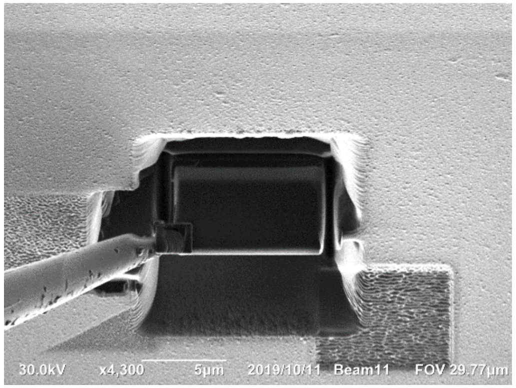
SEM

FIB

Lifting-out



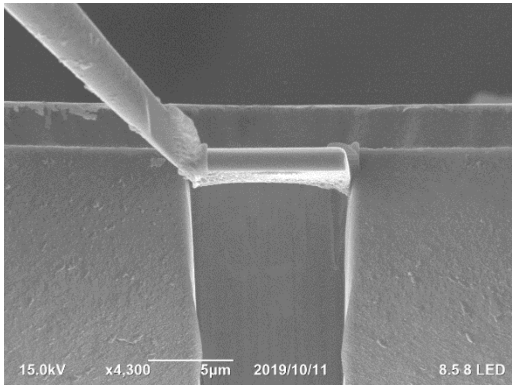
T: 0°



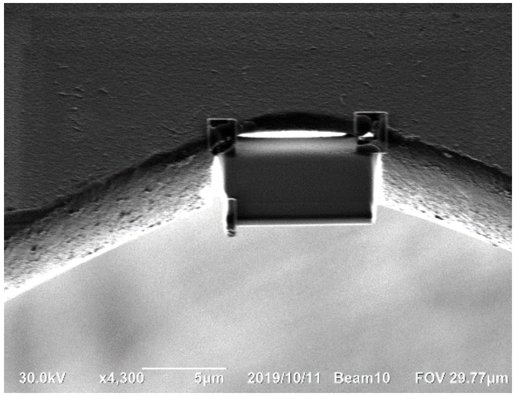
T: 0°

For welding:
Carbon deposition,
Beam 10 (30 pA),
2-3 minuntes

Deposit



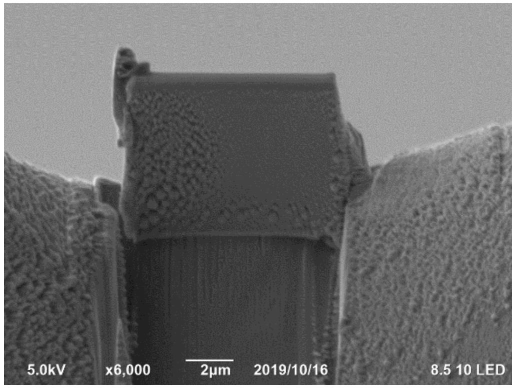
T: 0°



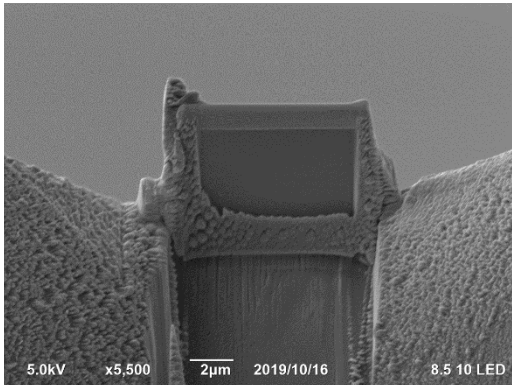
T: 0°

FIB-cut Steps

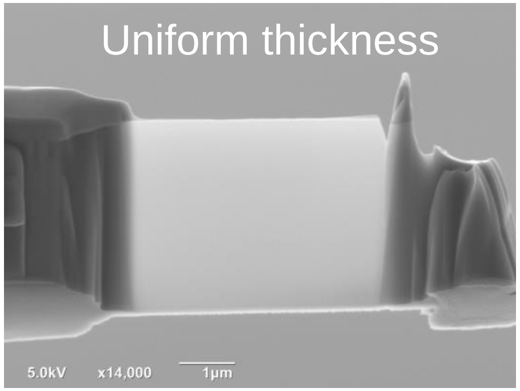
Fine milling



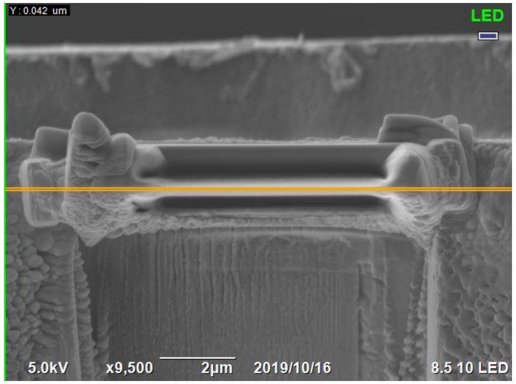
T: 53°



T: 53°


Uniform thickness

T: 53°



T: 0°

Fine milling:

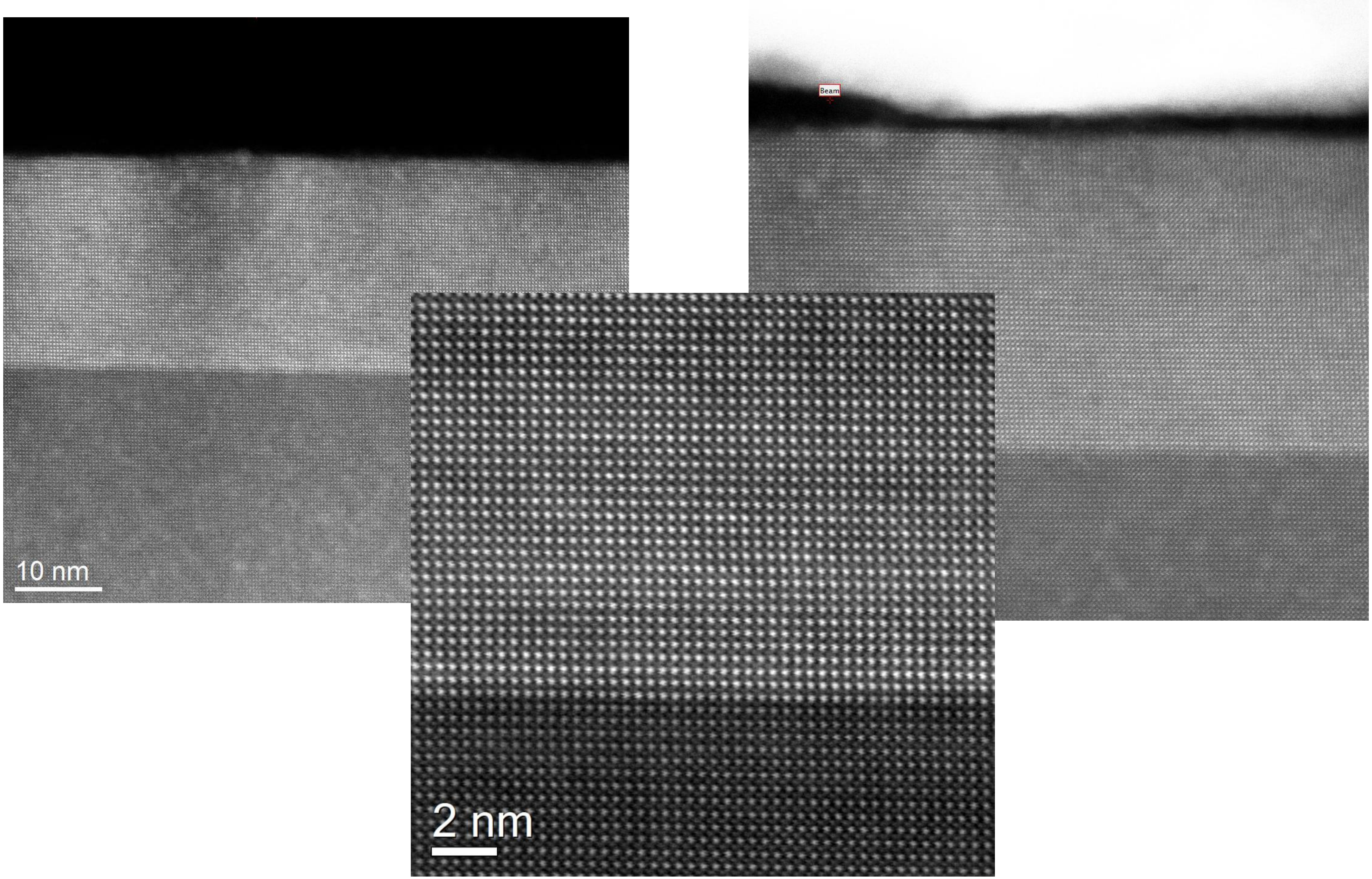
30 kV, Beam 7 (500 pA), until 750 nm
30 kV, Beam 8 (300 pA), until 300 nm
+/- 2°
Fine polishing:
5 kV, Beam 7 (50 pA) or Beam 8 (30 pA)
< 100 nm
+/- 1.5°

Cleaning:

3kV, Beam 7, < 2 mins
+/- 5°

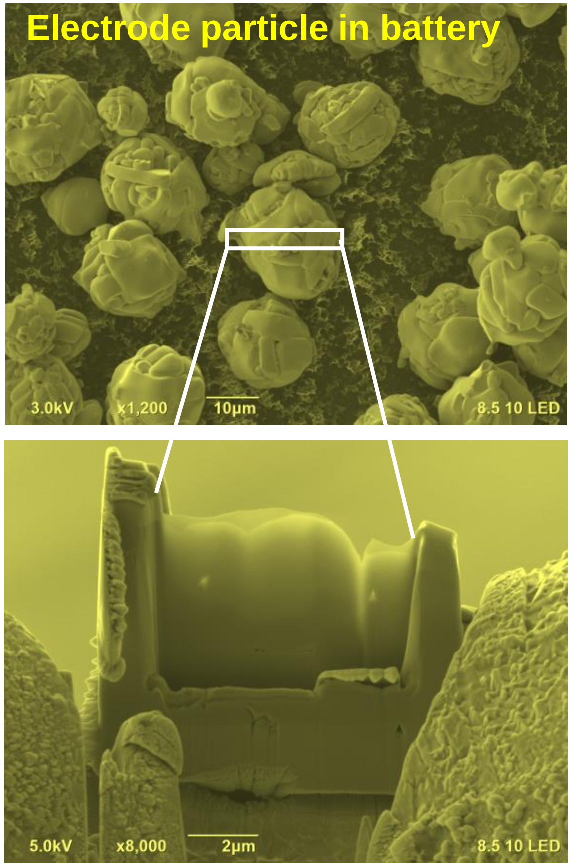
Note: in order to have a robust in-situ TEM sample, it would be better to prepare a wedging shape lamella.

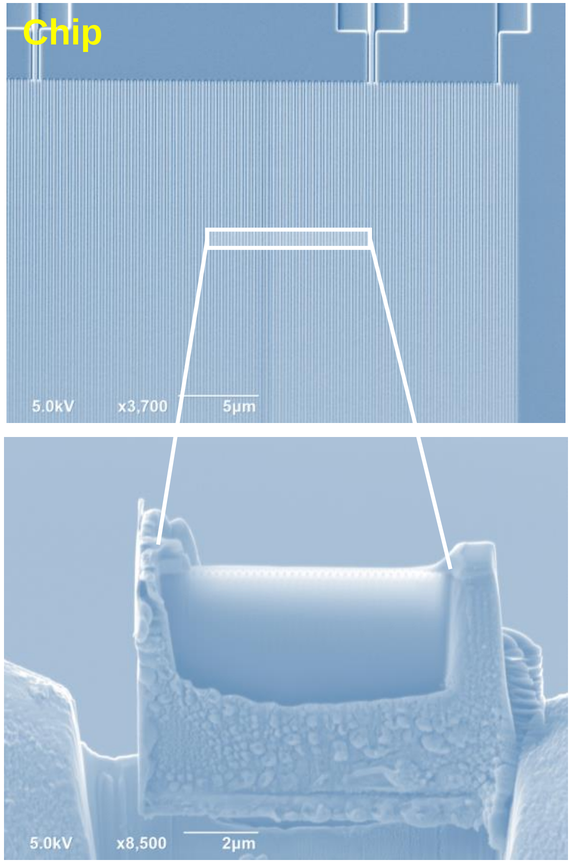
STEM imaging after cleaning

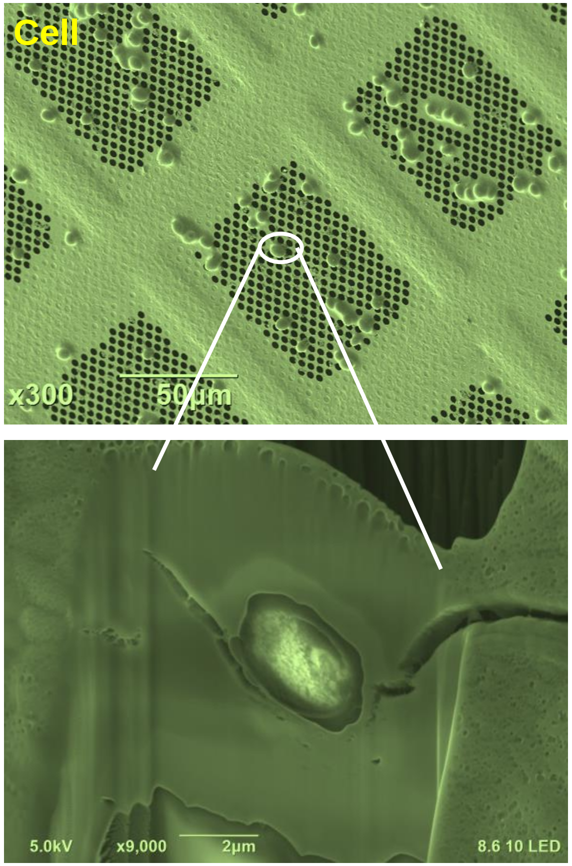


Applications

FIB-cut TEM lamellas for various materials

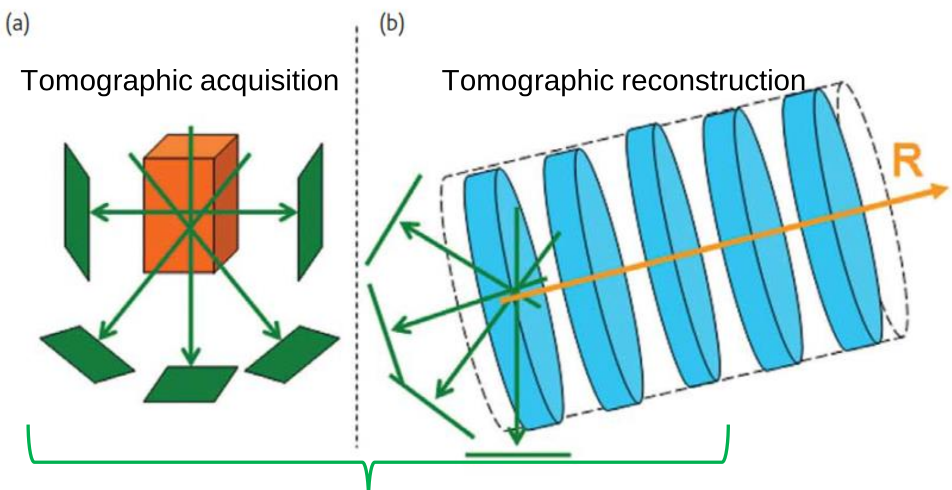




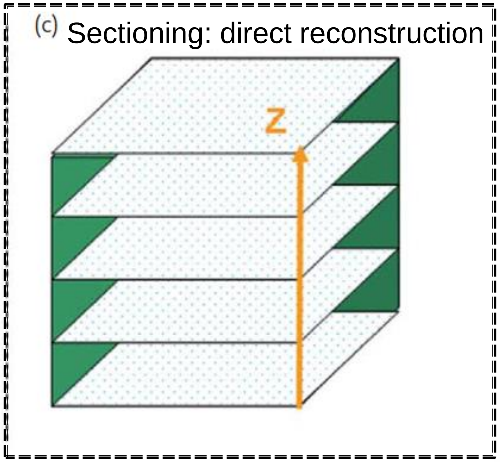


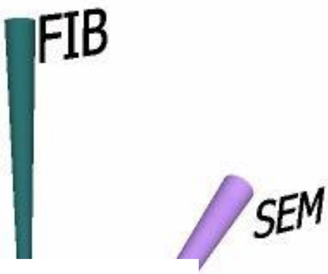
Applications

3D observation and analysis –FIB tomography



TEM tomography

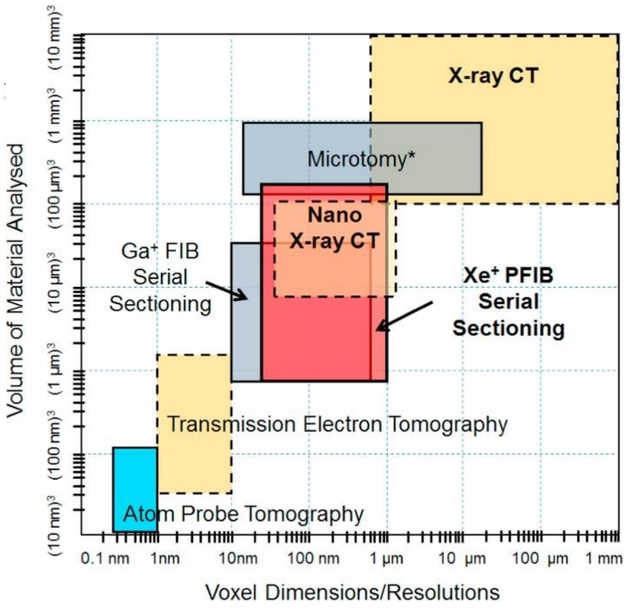




FIB tomography

Technique features:

- ✓ Simultaneously cross-sectioning (by ion beam) and monitoring /or SE or BSE imaging (by electron beam)
- ✓ Two modes
  - i) Dynamic mode: SEM imaging in real time during milling process.
  - ii) Static mode: SEM imaging, EDX mapping/FIB imaging after each milling. High resolution imaging.
- ✓ Resolution: lateral ~1nm, z- resolution 10-100nm
- ✓ FIB tomography fills in the gap between TEM tomography and X-ray tomography.



Materialstoday, 10, 20, 2007

Applications

Procedure of FIB-Tomography (video)

Narayan, K., Subramaniam, S. Focused ion beams in biology. *Nat Methods* **12,** 1021–1031 (2015)

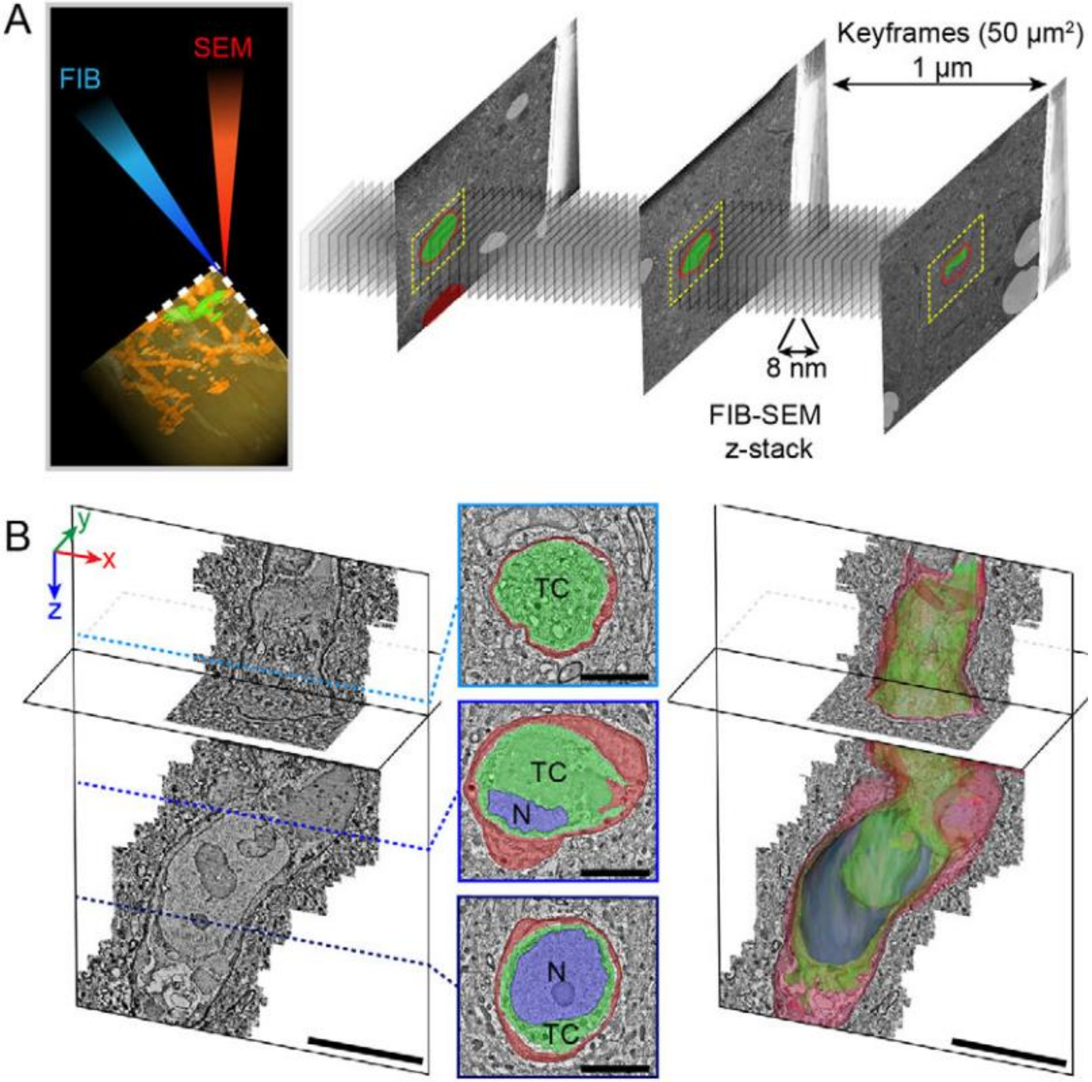
Applications

FIB tomography

Advantage:

- Compared to serial TEM or serial tomography, the main advantage is the size of the volume that can be acquired
- With a close to one thousand-fold increase in favor to the FIB-SEM.

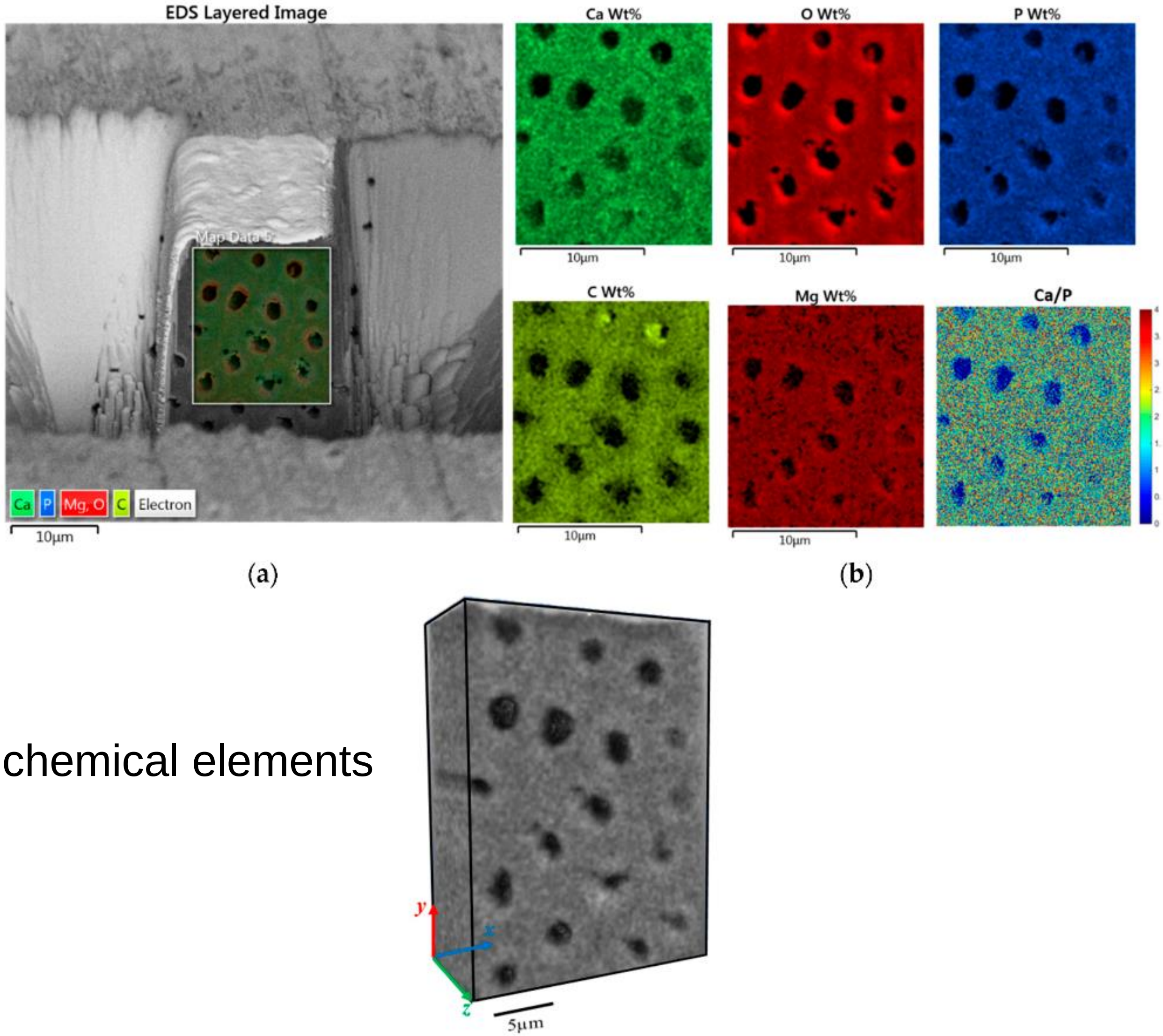
It is applicable at NMC with a Cryo-stage!



Automated 3DEM. Scale bars: 10µm. (From Karreman et al. *Journal of Cell Science* 2016)

Applications

3D EDX mappings



(a)

(b)

(c)

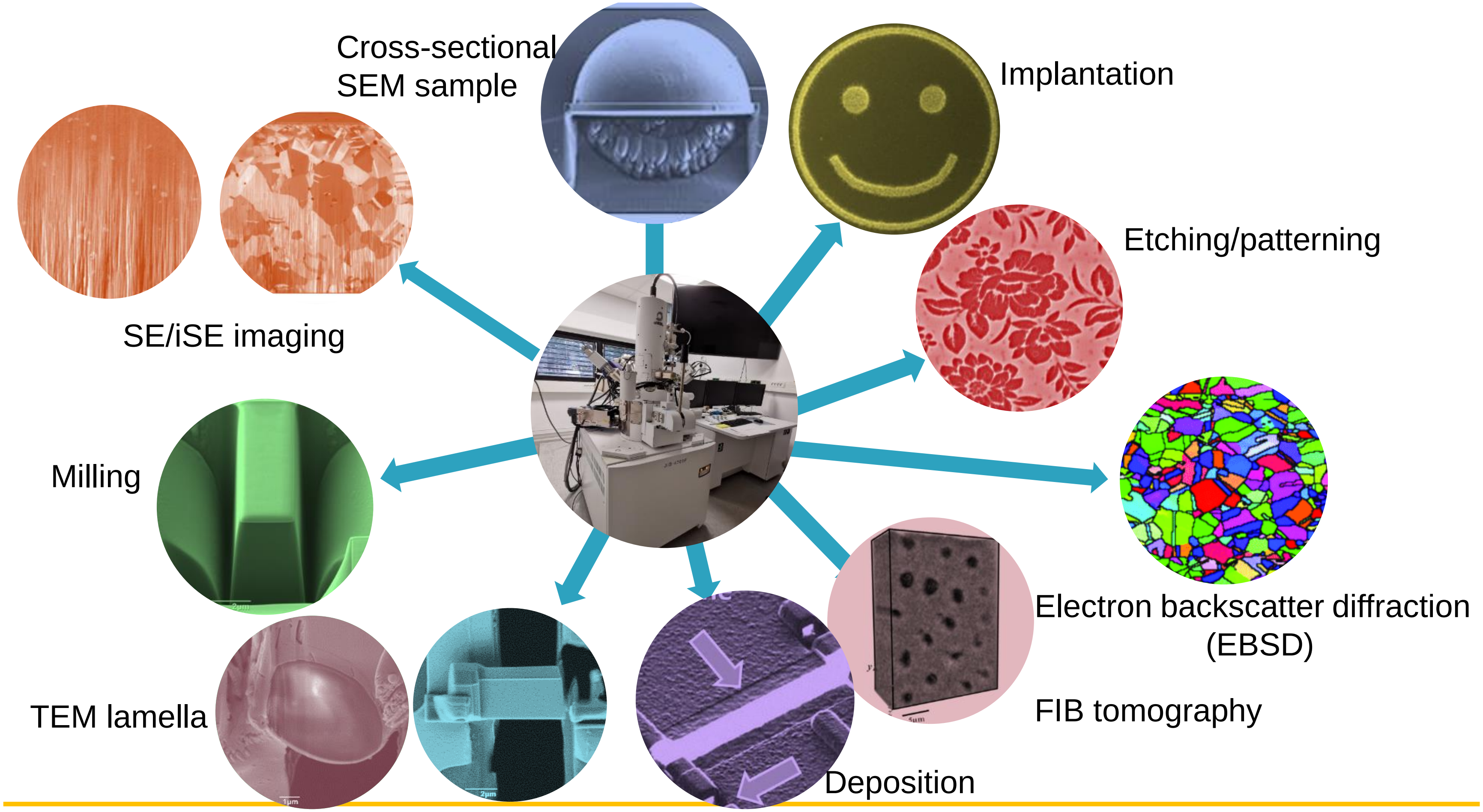
Spatial distribution of chemical elements in dentine

Materials **2018**, *11*(9), 1493

Key points to remember with FIB

- ✓ Micro/nano manufacturing technique with unique advantages of high fabrication resolution, high flexibility, maskless processing, and rapid prototyping.

- ✓ Wide applications in various areas, such as semiconductor industry, micro-/nano-optics, surface engineering, biotechnology, and nanotechnology.



Cross-sectional SEM sample

Implantation

Etching/patterning

SE/iSE imaging

Milling

Electron backscatter diffraction (EBSD)

FIB tomography

TEM lamella

Deposition

Key references

- *Focused ion beam systems–basics and applications*, edited by Nan Yao, Cambridge University, New Jersey, online 2010.
- R.J. Young, M.V. Moore, Dual-Beam (FIB-SEM) Systems. In: Giannuzzi L.A., Stevie F.A. (eds) *Introduction to Focused Ion Beams* (2005), Springer
- *Introduction to Focused Ion Beam Nanometrology*, Edited by David C. Cox, Morgan & Claypool Publishers, 2015.

  (available in Aalto library)