Optical, Electron, and Scanning Probe Microscopy
Online Workshop November 6th, 2024

THE BASIS OF FOCUSED ION BEAM

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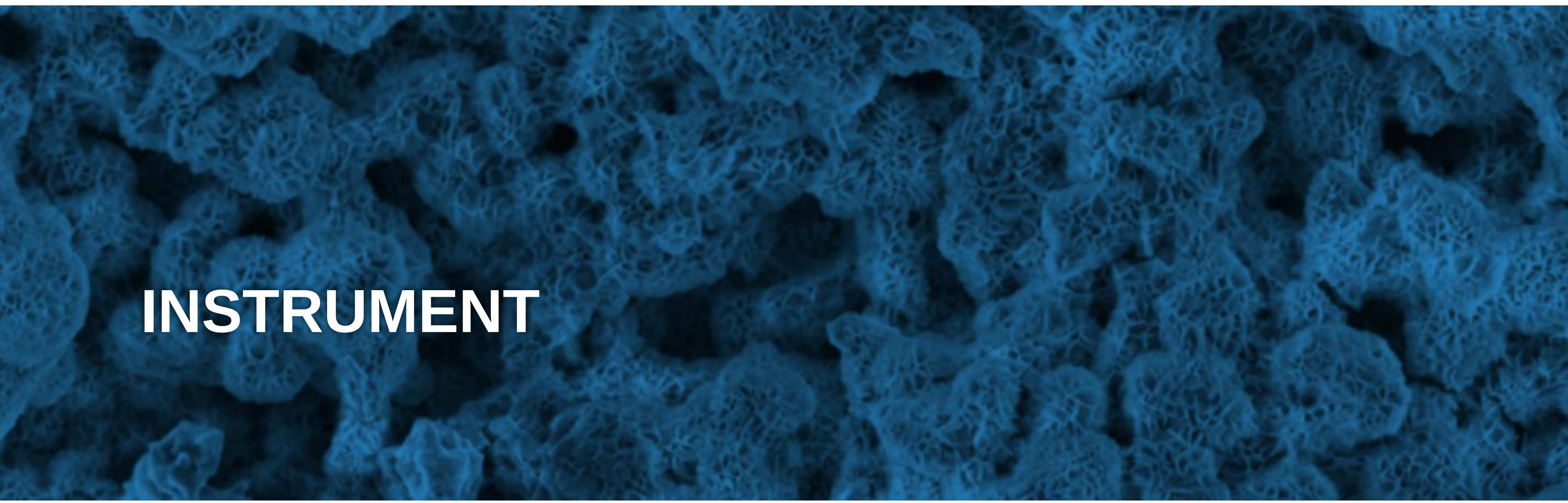
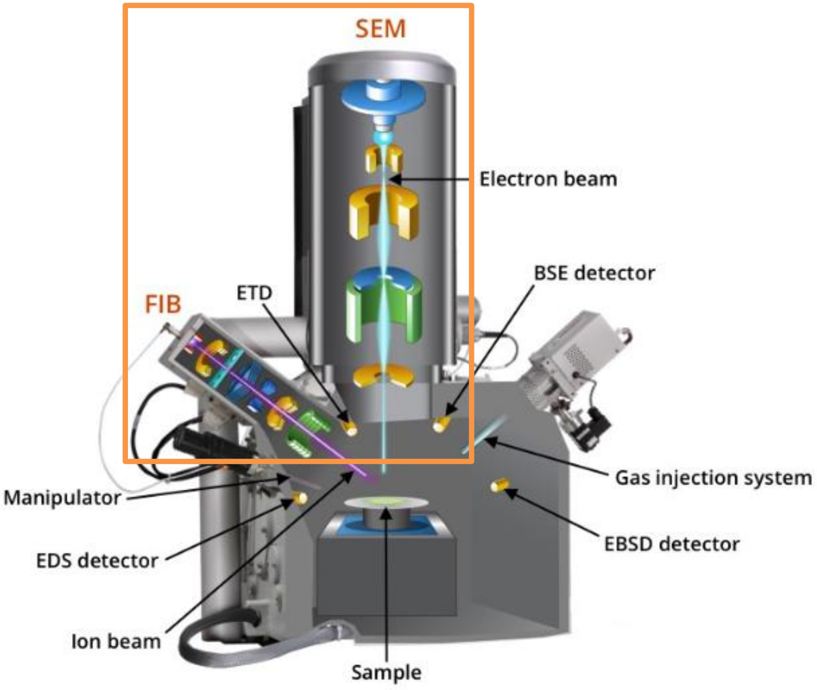
- **FIB** instrument and **Dual Beam (FIB-SEM)** setup
  - Source
  - Column
  - Chamber
- **Ion-solid interaction**
  - Dose, Signals,
  - Sputtering
- **Gas chemistry**
  - GIS, Beam Induced Deposition,
  - Gas Assisted Sputtering
- **FIB Imaging and Artefacts**
  - Charging
  - Channeling contrast
- **Applications**



INSTRUMENT

- Focused Ion Beam (FIB) is a stream of energetic ions focused into a fine beam
- The beam is scanned onto a target material
- Ions interacts with the specimen leading to atoms removal with nanometric precision (high depth and spacial accuracy)
- In addition to the ion beam, most FIB instruments have an electron beam (FIB-SEM).

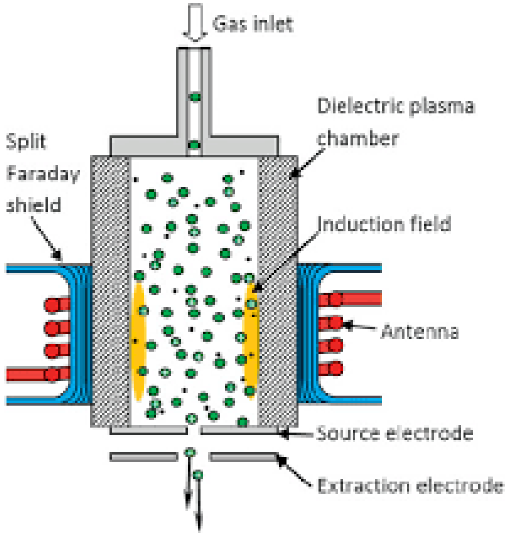


FIB columns typically employ a Liquid Metal Ion Source (**LMIS**), with the Ga+ ion source being the most common in microscopy applications, operating from 500 eV to 30 keV and 1 pA to 65 nA beam current.

Other sources:

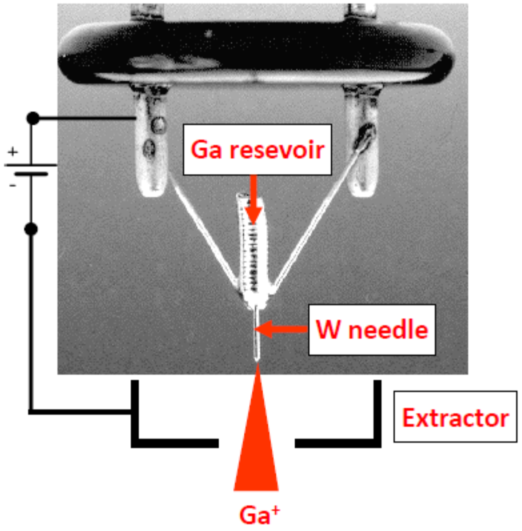
- Gas Field Ion sources (GFIS), He and Ne;
- Inductively coupled plasma ion source using noble gases, such as Xe;
- Low Temperature Ion Source (LoTIS);
- magneto-optical trap ion source (MoTIs)
- Liquid Metal Alloy Ion Sources (LMAIS).

Xe **Plasma FIB** are becoming increasingly common for large volume milling.

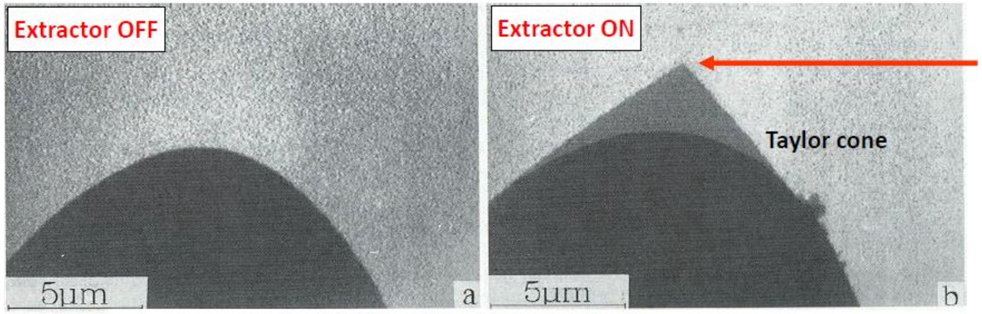


For high imaging resolution and reduced specimen damage and advanced nano-fabrication, multi-beam FIB systems are now used.

Liquid metal Ion source (LMIS)



- It’s a **Cold Field-Emission** source.
- Liquid Ga wets a W **needle** by capillary flow. The **extractor** electrode in front of the needle is biased at ~ – 10 KV.
- The extractor pulls the gallium into a small droplet called a “Taylor cone” which has a radius of few nm.
- At the cone tip (highest field strength), ions are extracted by tunneling.

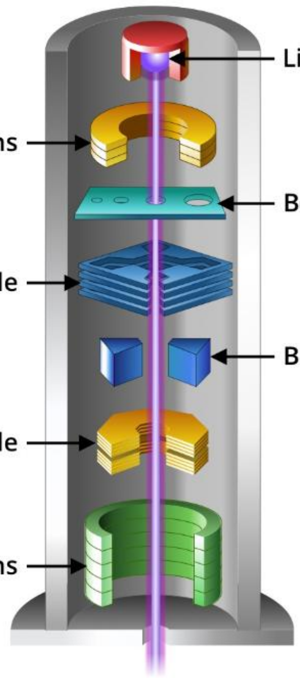


TEM image of W needle tip

Why Gallium?

Liquid at 30° C (no interdiffusion)
Low vapour pressure (long life)
Efficient sputtering (heavy)

Ga^+ ions extracted from the LMIS are accelerated through a potential and travels through lenes and aperture placed along the column.



Condenser lens: it forms the beam.

Beam Selecting Apertures: allow to control the current. The larger the aperture, the larger the ion beam current.

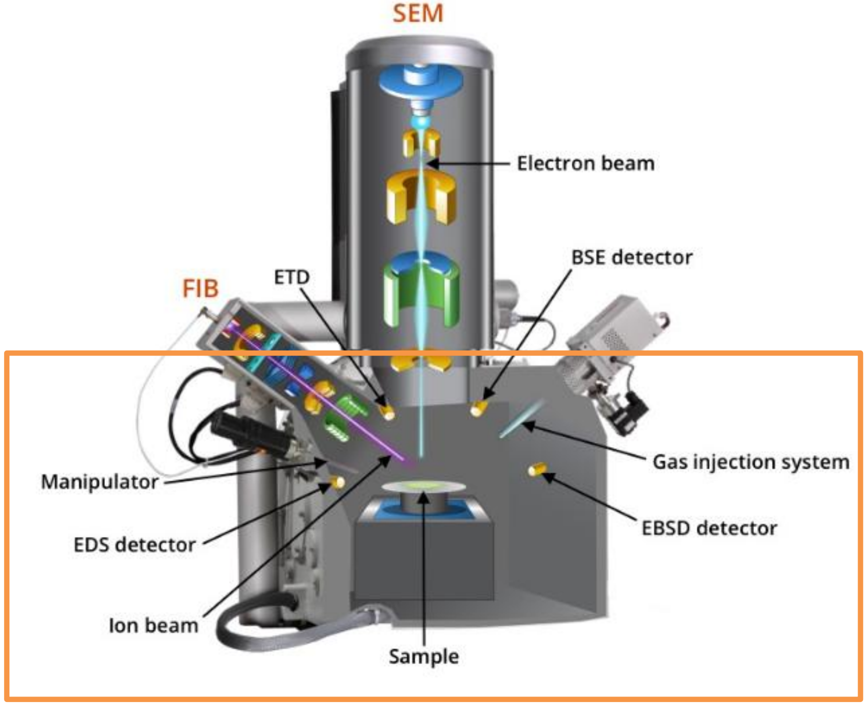
Quadrupole: controls the mid column steering of the beam.

Blanking plate: deflects the beam to avoid unnecessary exposure of the sample.

Octupole: scans the beam across the sample surface (scan coils) and corrects for astigmatism.

Objective Lens: focuses the ion beam onto the sample.

Electrostatic lenses are used (independent from particle velocity) since ions travels slower than electrons.



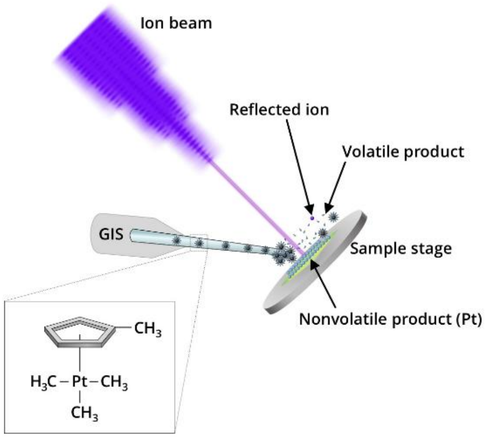
Why do we need high vacuum?

Minimize collisions of the charged particles with air molecules.

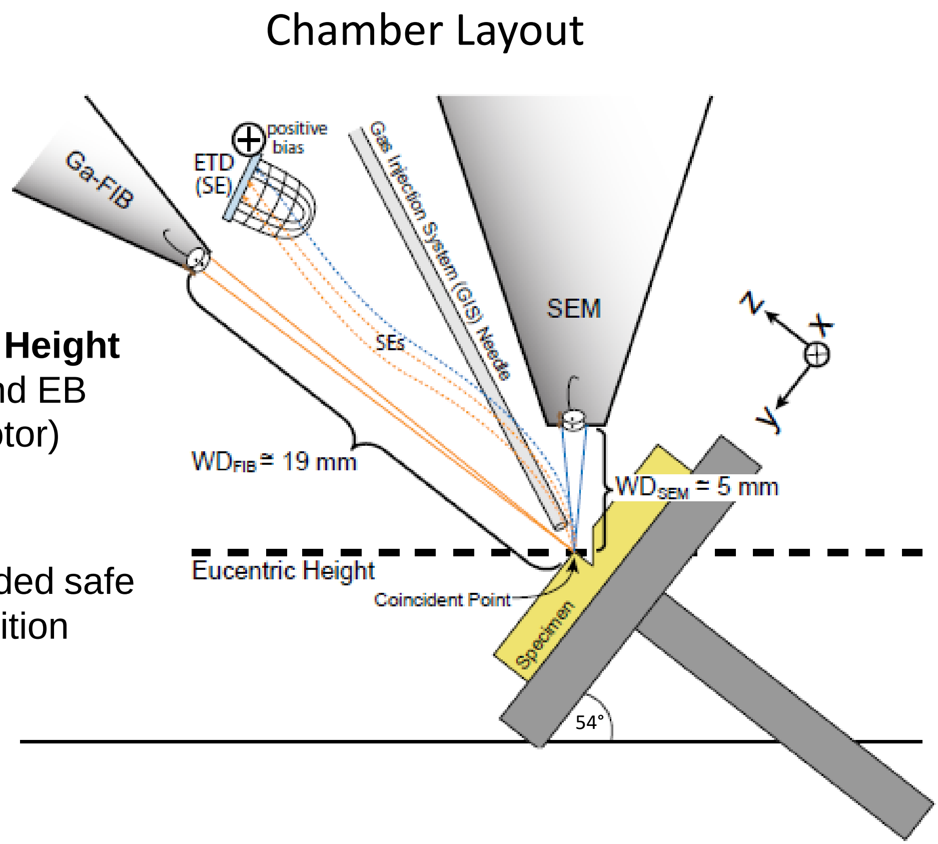
Preventing contamination of the source.

Ion pumps are normally used for the columns and a turbomolecular pump in conjunction with a dry pre-vacuum pump is commonly used for the specimen chamber.

In the chamber are placed: **Detectors** (imaging), the Gas Injection system (**GIS**), the **manipulators** and a five-axis **sample stage**



Different FIB-SEM systems have different geometries

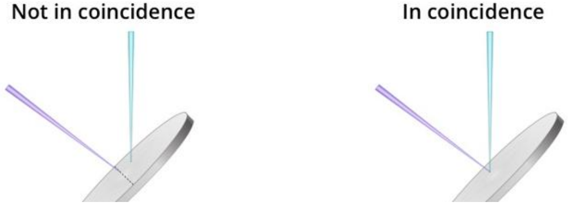


The Eucentric Height is where FIB and EB intersect (M motor)

Recommended safe working position

The Coincidence Point when FIB and EB point at the same place (Z motor)

Correct M value for eucentricity



ION SOLID INTERACTION

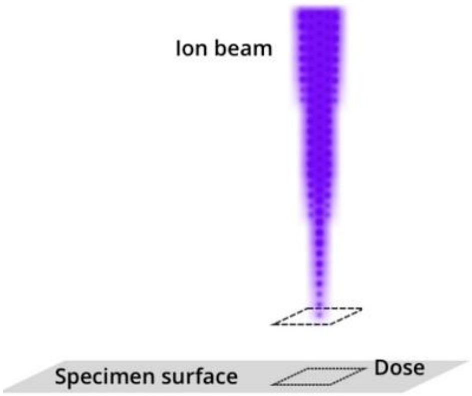
The main interaction process of **FIB ions** (medium-high Z: Ga, Xe; E < 100 keV,) are the elastic collisions with atoms (elastic scattering with screened atomic nucleus), Nuclear E-Loss.

How many ions?

$$N = \frac{I*t}{C}$$

Where N = number of ions
I = current in amperes
t = time in seconds
C = charge

The number of ions that have travelled into the sample surface is called **Dose,** while **Dose Rate** describes the number of ions that are going through a specific area into the sample per unit of time.



$$Dose = \frac{N}{A}$$

Where N = number of ions
A = area in square centimeters

$$Dose\ rate = \frac{N}{A*t}$$

Where N = number of ions
A = area in square centimeters
t = time in seconds

The ion-solid interaction leads to:

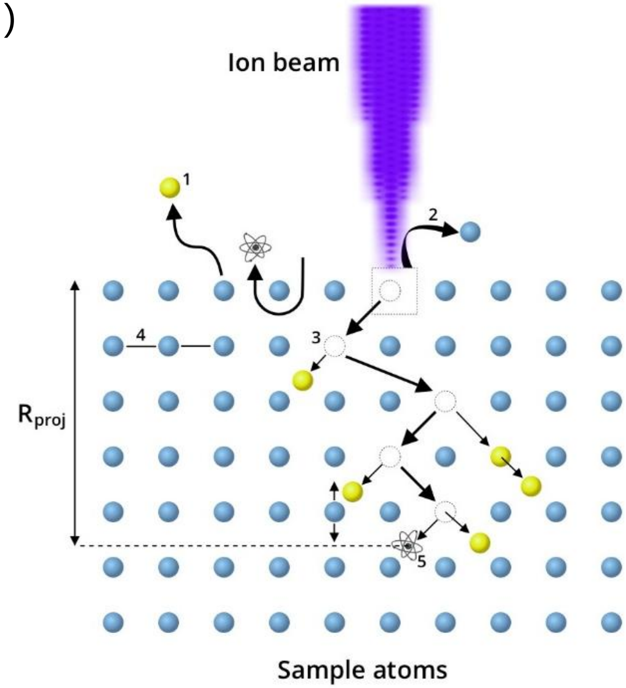
- emission of **secondary electrons** (SE) and **secondary Ions** (SI)

  Imaging

- Removal of surface atoms (**Sputtering).**

- Creation of vacancies, Disolocation and Interstitials

- Polymerisaztion

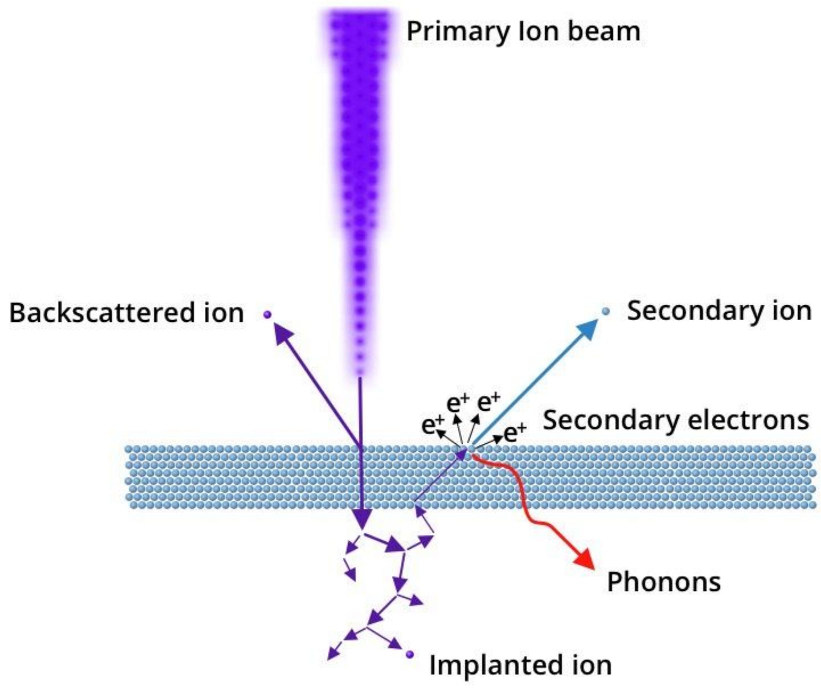


The interactions take place until the ion has lost all its energy and is **implanted** in the sample at a specific depth, often referred to as the projected range.

Gallium or Xenon implantation could potentially modify the crystal structure, grain boundary chemistry or some other characteristics of the specimen!

- An electron from the sample is ejected during the ion-beam sample interaction and detected by a standard **secondary electron (SE) detector**.

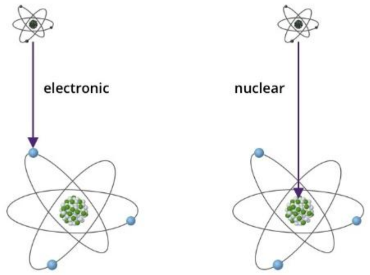
The **SE yield** for ion beams is around 10 times higher in comparison to electron beams!



- **Secondary ions** are sample atoms which are ionized after the interaction with the primary beam and emitted from the surface.

- **Backscattered Ions (BSI)** are ions from the primary beam that “bounce” back from the sample surface, but their yield is low (0.1-10 %).

- **Phonons** are atomic vibrations or waves within the sample crystal structure → **heat**



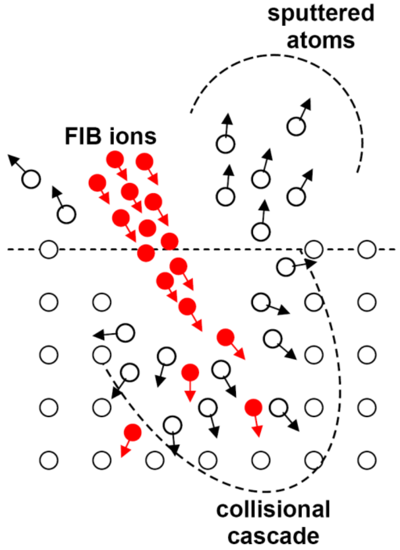
Sputtering occurs when a sample atom is **ejected** from the sample **surface**; more collisions means more chance of sputtering.

Atoms that are located **deeper** within the sample are less likely to be sputtered.

Sputtering requires:

transferred energy > binding energy

transferred momentum with outward direction



Affected by

- Incident angle, trajectory of the collision cascade
- Initial ion energy (voltage)

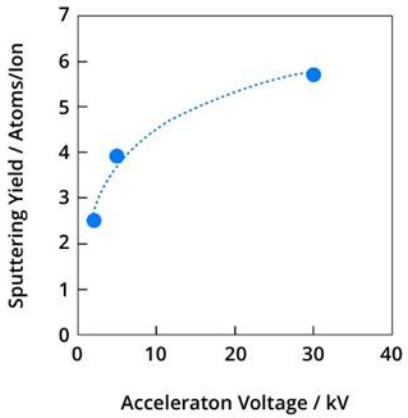
Sputtering yield (Y_S)

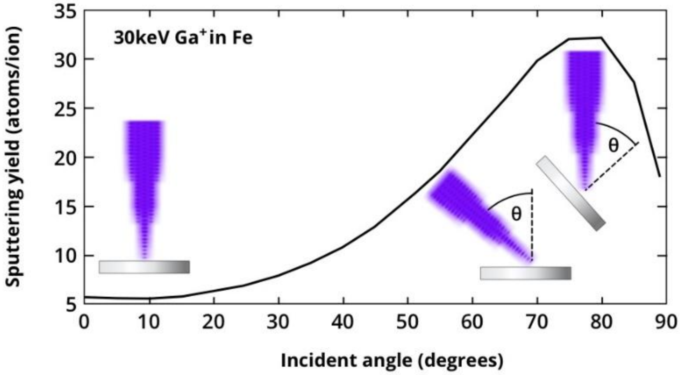
$$\textit{Sputtering yield} = \frac{\textit{number of sputtered atoms}}{\textit{number of incident ions}}$$

for Ga-FIBs is between 1 and 10

The **Sputtering Yield** increases with:

- Acceleration voltage
- Weak bonds in the sample
- Light target elements
- High incident angle





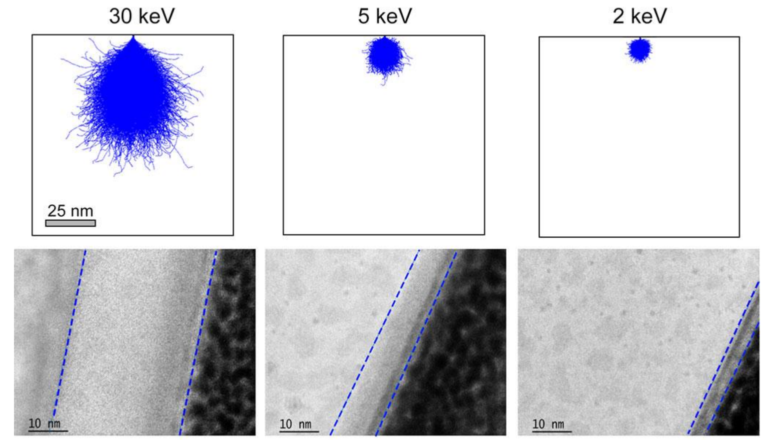
What matters most is the sputtering rate, the volume removed per unit time or charge, taking into account the target density, N.

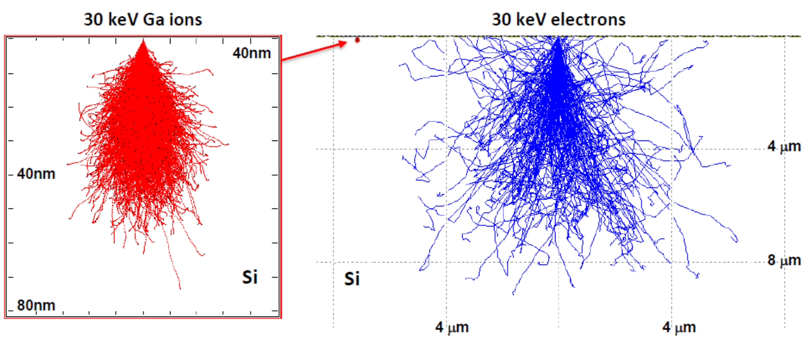
$$S_R\left[\frac{\text{Volume}}{\text{Time}}\right] = \frac{I}{e}\frac{Y_S}{N} \qquad S_R\left[\frac{\text{Volume}}{\text{Charge}}\right] = \frac{Y_S}{eN}$$

Ga FIB is suited to remove volumes within **tens of µm** in X, Y, Z.
Recent noble-gas **plasma FIB** have currents up to 2 µA, speeding up milling rates **100x**.

higher energy → larger interaction volume.
The ion–solid interactions cause amorphization.

The larger the interaction volume, the thicker the amorphous layer.



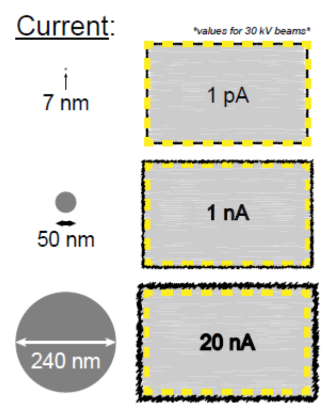


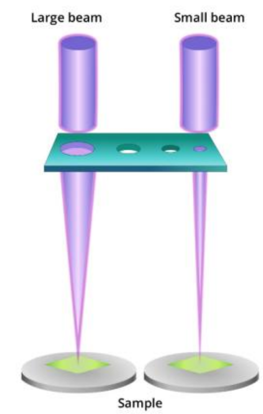
Ion Range R_I
the distance travelled into the solid until all ion energy is lost, and the ion becomes **implanted**

$$R_e/R_I \sim 100$$

More current

- Larger probe diameter
- higher material removal rate
- loss of 2D resolution



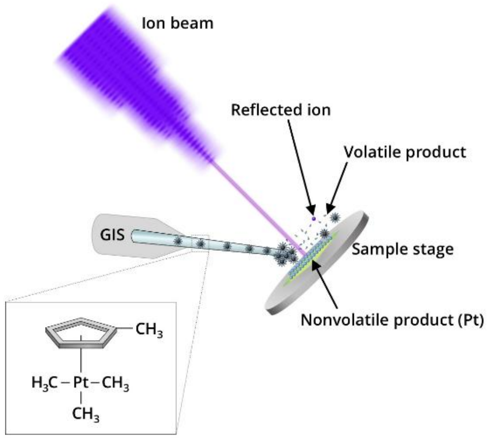


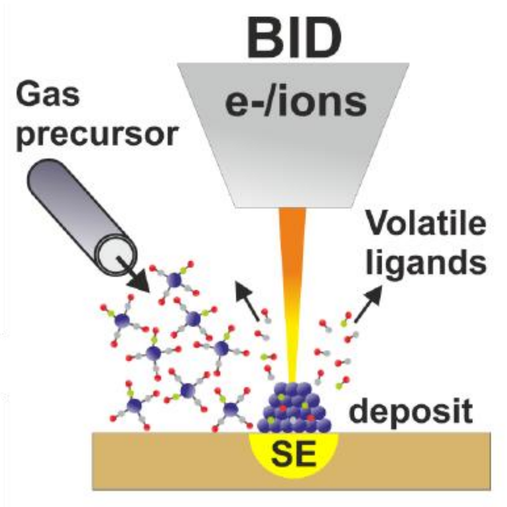
GAS SPUTTERING

Gas injection systems (GIS), introduce reactive gases to the sample surface, are used for enhanced etching, preferential etching or material deposition.

GIS are usually inserted to within ~200µm of the sample surfaceand delivers a controlled flow of gas though a nozzle.

The precursor is a solid (metallorganic) so the reservoir needs to be preheated to allow its transition to gas phase.



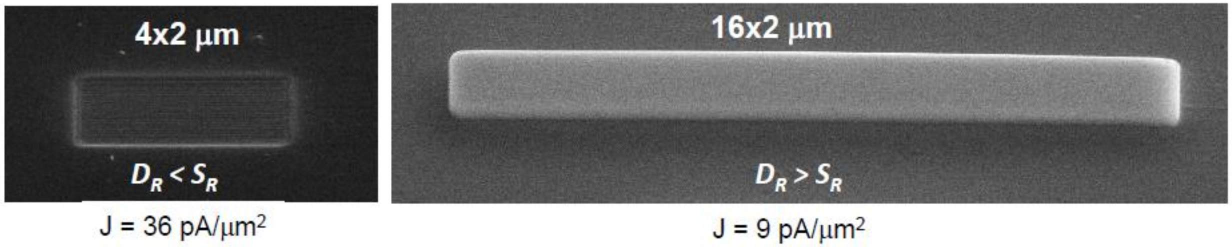


1. Precursor is injected and adsorbed on the surface.
2. Beam irradiation (e-/ions) decomposes the molecules.
3. The metal is deposited, volatile parts are pumped out.

4) Particles with energy ≈ molecular binding energy (few eV) are the most efficient bond-breakers → secondary electrons.

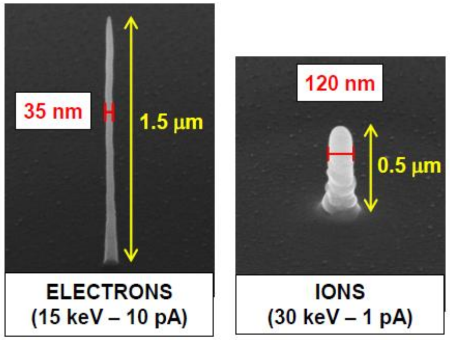
Most Common Deposited Species:
Metallic: Pt, W, Au, Co, …. Insulating: TEOS, C

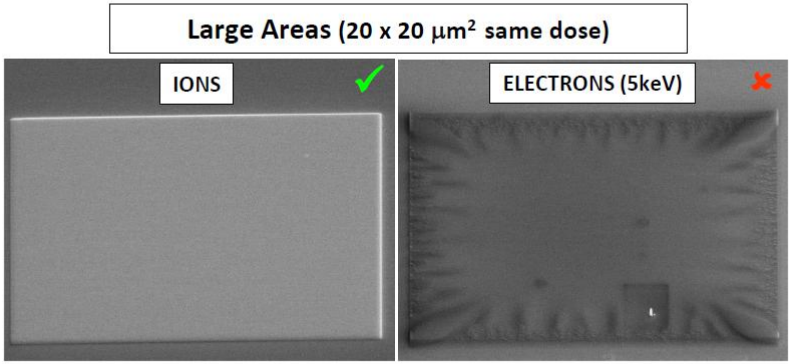
Careful! For ions, sputtering and deposition or competitive processes which depends on gas flux, current density and scanning parameters



High current density favors **Sputtering Rate (S_R)** over **Deposition Rate (D_R)**

Electrons produce **higher-resolution** deposition because there's no ion damage/intermixing → better for smaller areas or as a thin coating

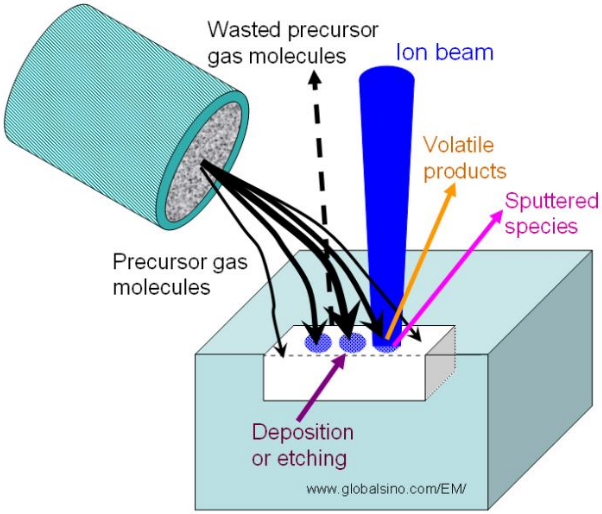


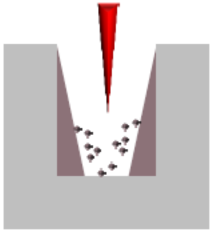


Ion **Deposition Yield** is 40 times greater since ions produce more SE

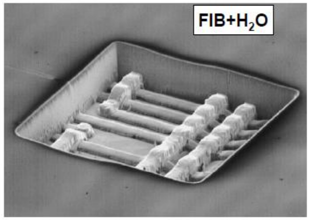
Inject a chemically reactive gas to:

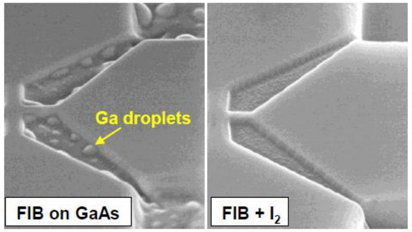
- **Enhance etch rate**.
- Reduce Ga^{+} **implantation**
- Reduce **redeposition** (cleaner structures)
- **Selective** etch





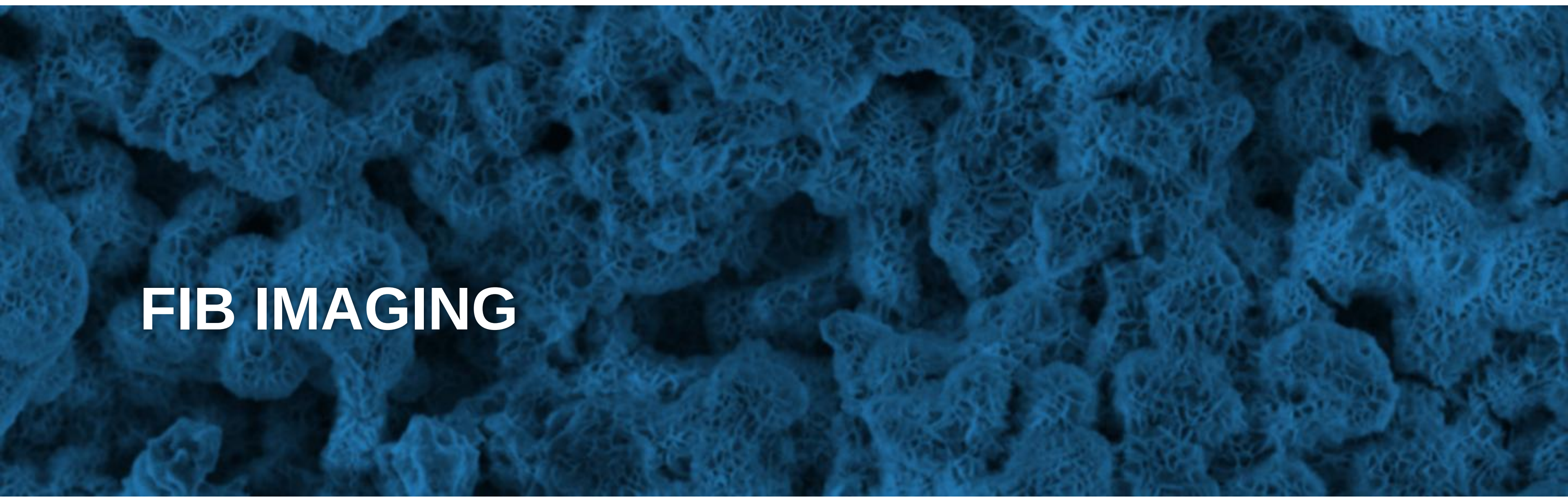
Redeposition in high aspect ratio trenches





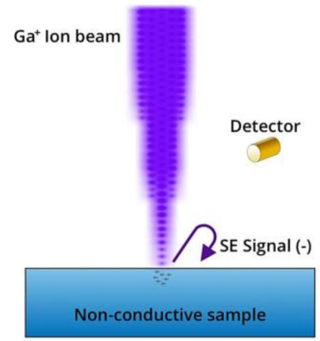
Examples :
Cl_2 for etching GaAs, Si, and InP
XeF_2 for etching SiO_2, W or diamond
H_2O for etching for carbonaceous materials

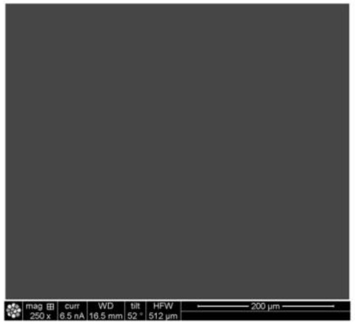
FIB IMAGING

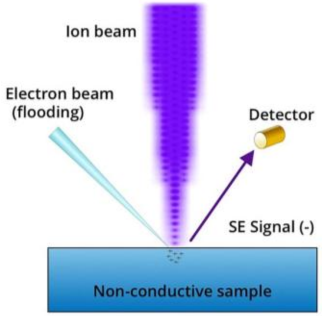


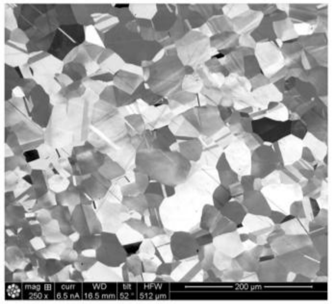
Charging in the ion beam image appears as (growing) black areas, positive charge from Ga^+.

SE electrons are back attracted to the sample and do not reach the detector.





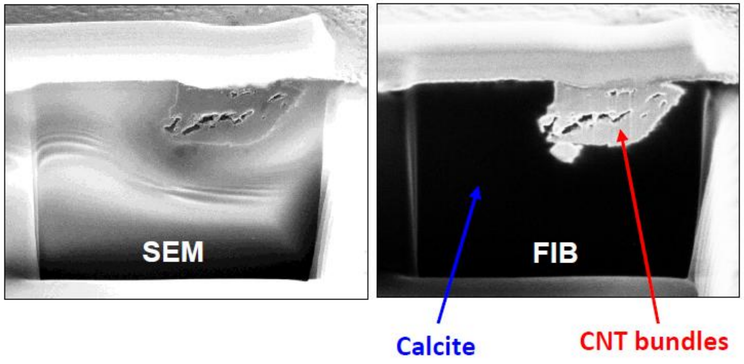




Scanning the e-beam while imaging with the FIB to neutralize the charge.

FIB **insulator contrast**:
insulators appear **totally dark**

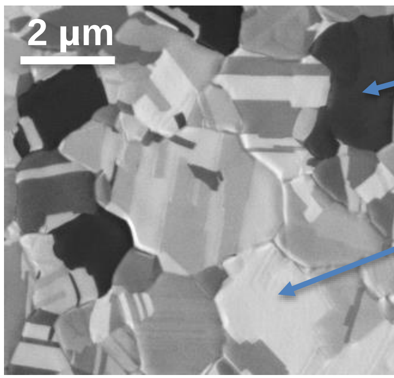
Looking for CNT inclusions (conductor) in Calcite (CaCO3, insulator)



M. Calvaresi et al., Nanoscale 5, 6944 (2013)

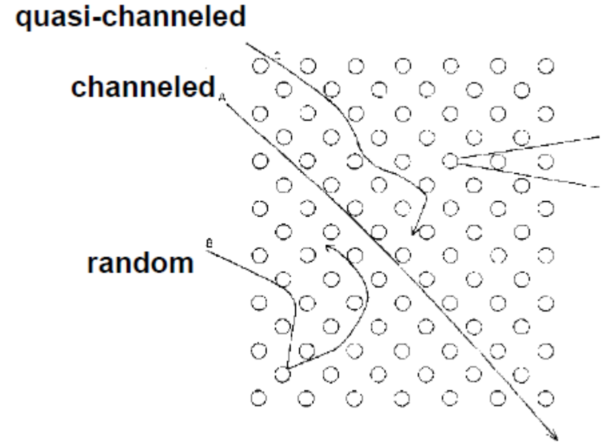
FiB Imaging: Channeling Contrast

- **Channeling contrast** is created due to different ion–sample interactions in specific crystal orientations → angle-dependent effect.
- Ions penetrate deeper in low index direction [100], [110] (wider atomic “channels”)



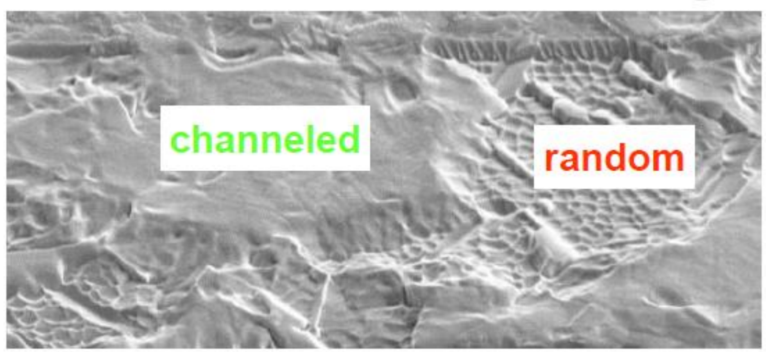
Dark “channeled”

Bright “random”

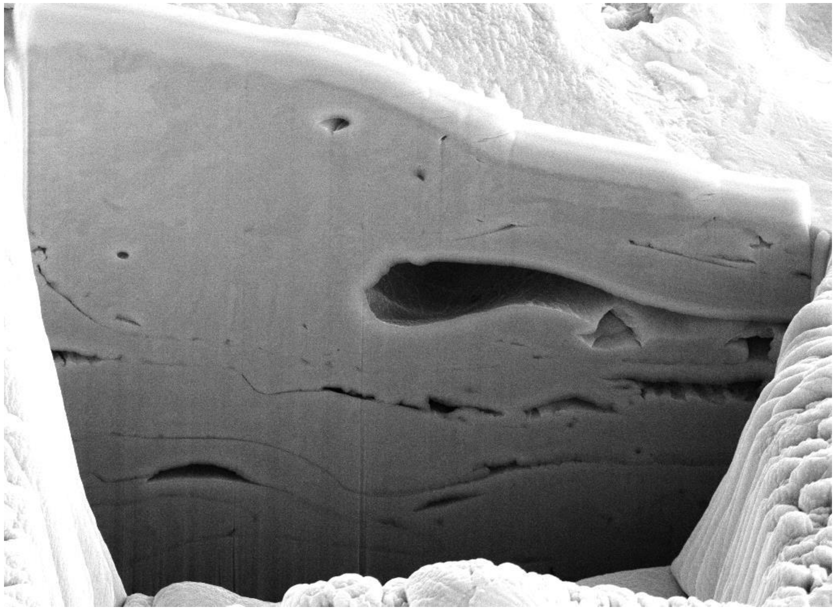


Enhanced contrast in policrystalline metals or ceramics

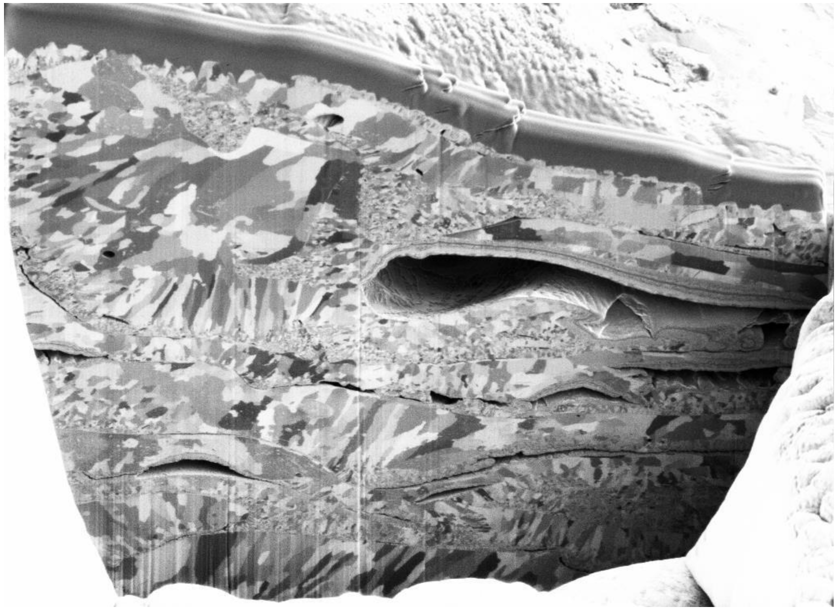
Reduced sputtering



Compared to SEM imaging, FIB imaging on polycrystalline metal (Spray coating) gives a lot more information



SEM



FIB

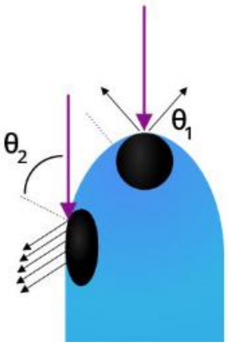
ARTEFACTS

What is an Artefact?

Effect that that is not naturally present but occurs as a result of the **preparative** or **investigative** procedure.

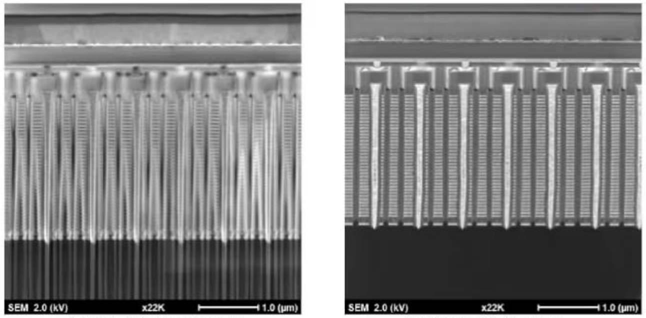
Curtaining

Topographic features and voids within the sample create different incident angles for the ion beam, and hence different sputtering yields



Possible solutions

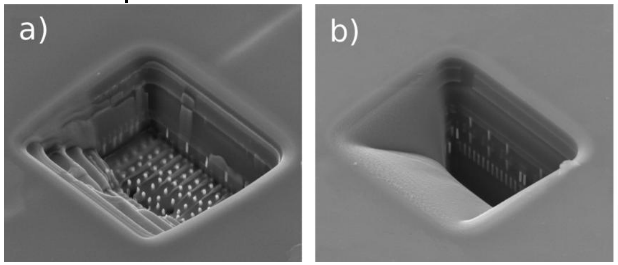
- Use homogenous protective layers
- Lower the beam current
- Rocking stage
- Embedding in resin (for porous materials)



(a) Without double-tilt system (b) With double-tilt system

Redeposition

Sputtered material can deposit back onto the sample



Possible solutions

- Reduce the milling rate
- Cut in parallel mode

Implantation

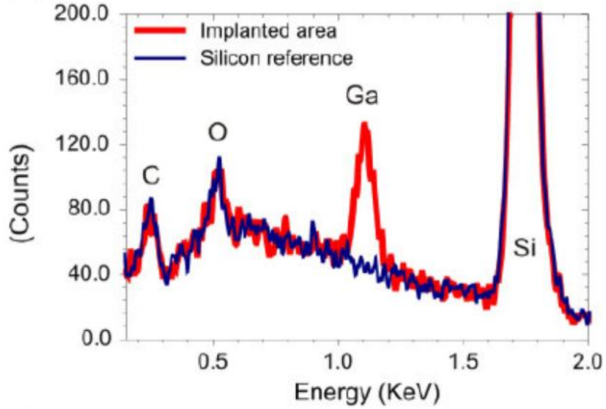
The beam ions get stuck in the sample

↓

Alters the properties in semiconductors, Ga peak in EDS

Possible solutions

- Lower Voltage
- Plasma



Amorphization

The i-beam dislodge the sample atom from their position in the crystal

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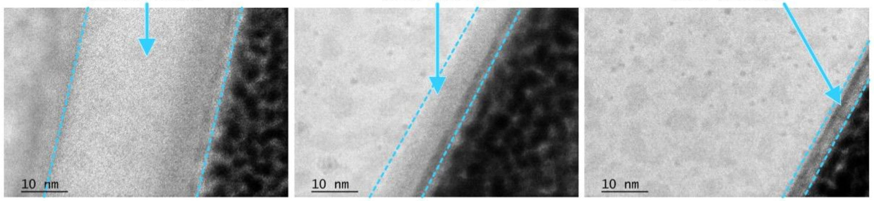
polishing at low Voltage

Amorphous layer (TEM lamella) | Amorphous layer (TEM lamella) | Amorphous layer (TEM lamella)



30kV cutting | 5kV polish | 2kV polish

Phase Transformation

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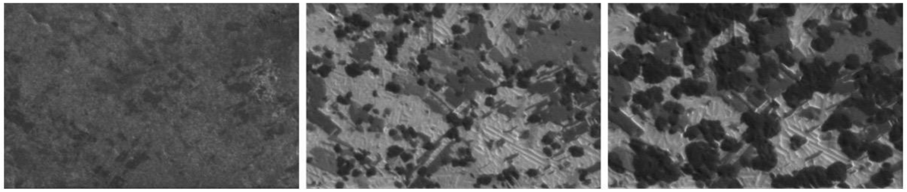
Reduce the exposure to FIB

t=0min | t~1min | t~3min

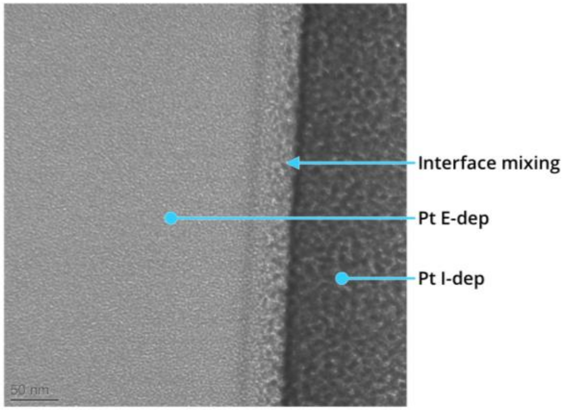


Interface Mixing

Ions from the i-beam recoil atoms deeper into the substrate or towards the surface leading to layer contamination

Possible solution

Deposit a protective layer with the e-beam



Heat Damage

Phonons produced by the i-beam and energy loss → heat
Big issue in insulator, Biological samples and polymers

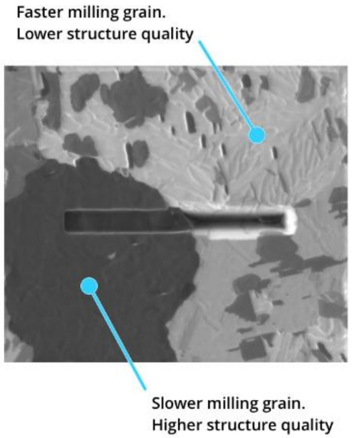
↓

Cryo-FIB

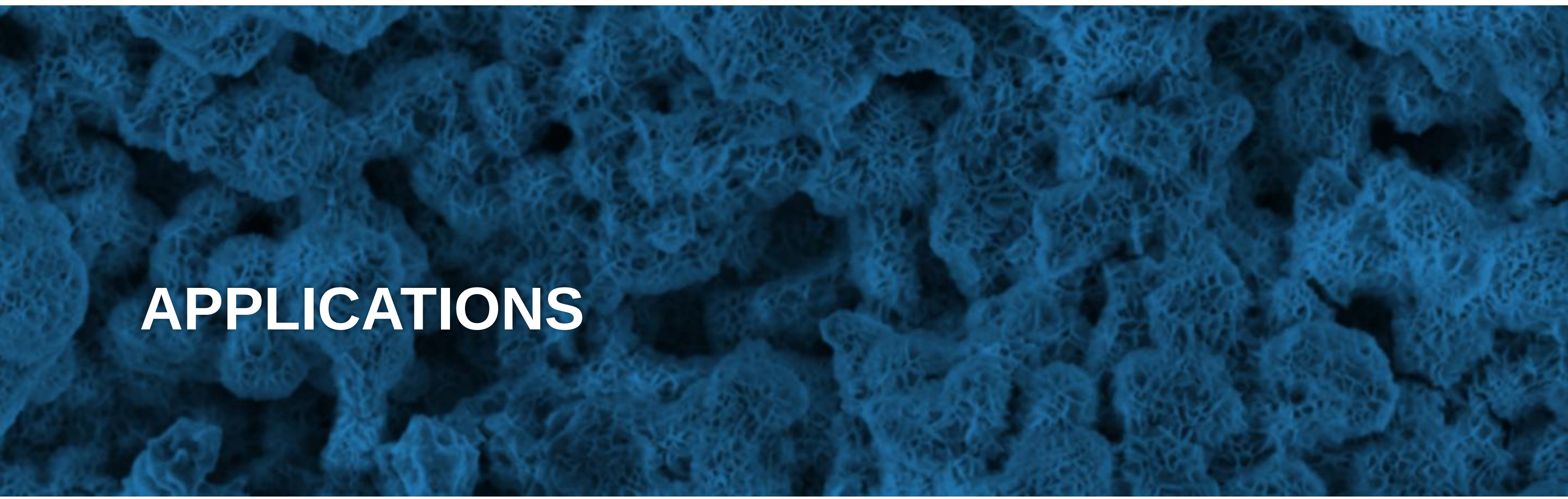
Channeling

↓

In polycristalline materials the sputtering rate depends on the orientation of the grains.

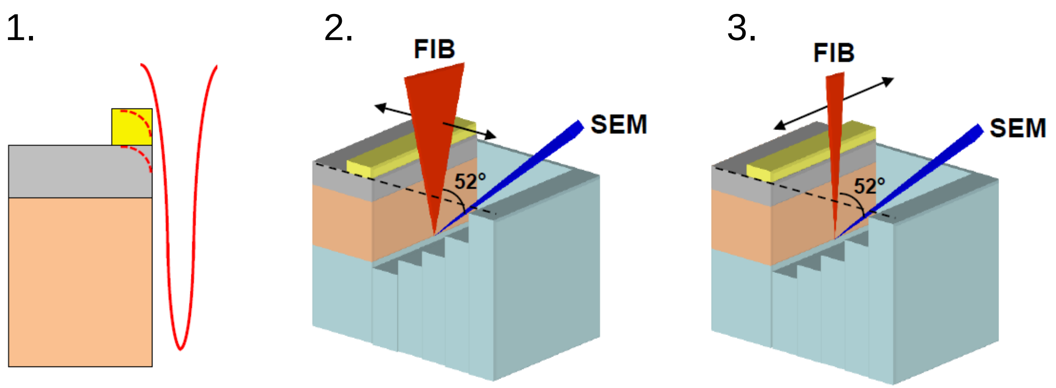


APPLICATIONS



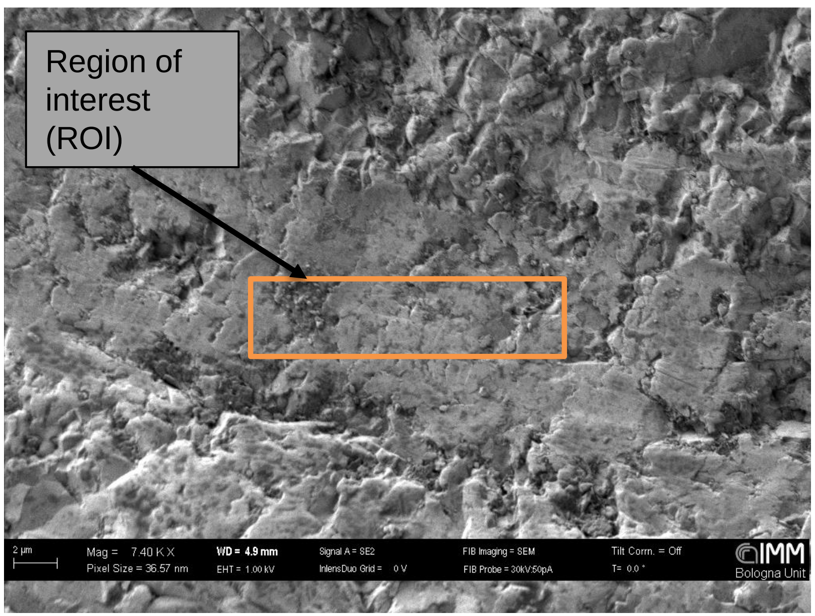
Applications: Cross-Section Analysis

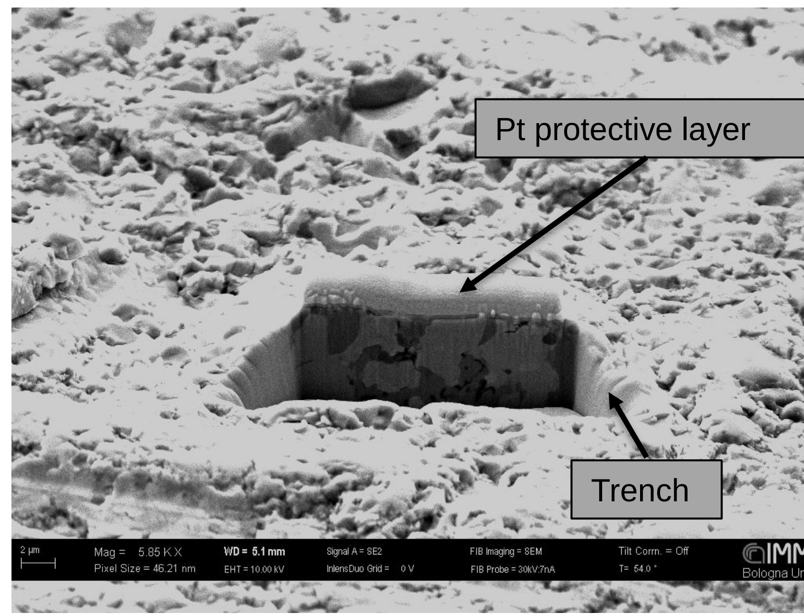
Choice of cross-section site with submicron precision



1. Deposition of a protective layer to protect the edges (Gaussian Beam)
2. Create the trench with high-current beam.
3. Polish with low current beam.

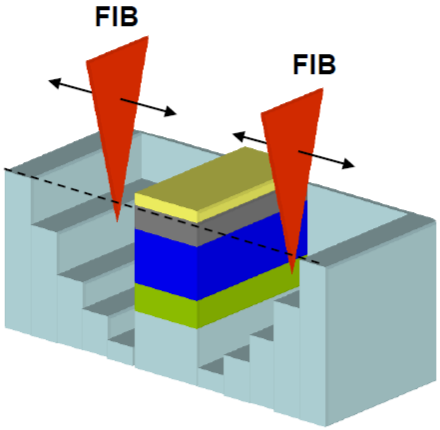
Cross section of a ceramic after oxidation test reveals the hidden microstructure

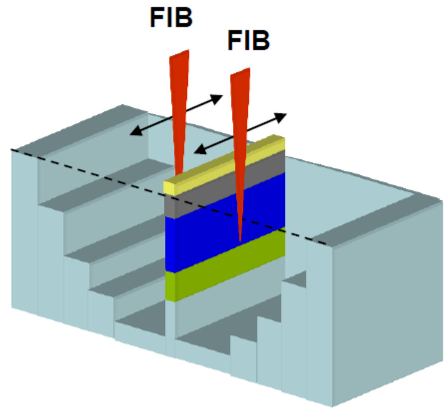




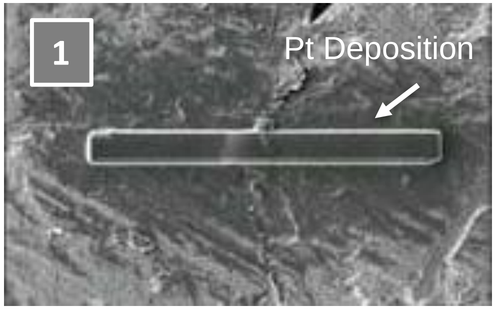
Applications: TEM Preparation

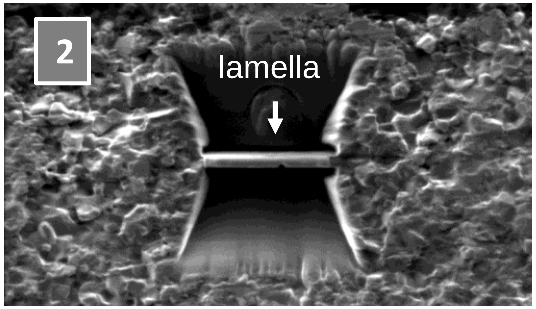
Key advantages of FIB: **Site selectivity** (Failure Analysis in semiconductor industry), **faster** and **easier**.

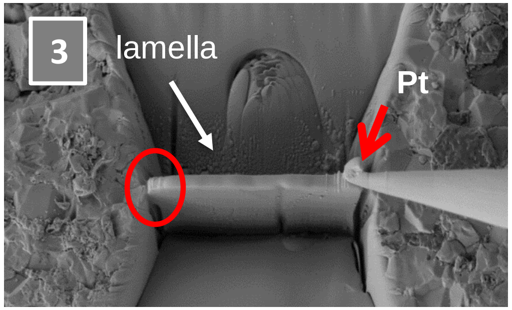


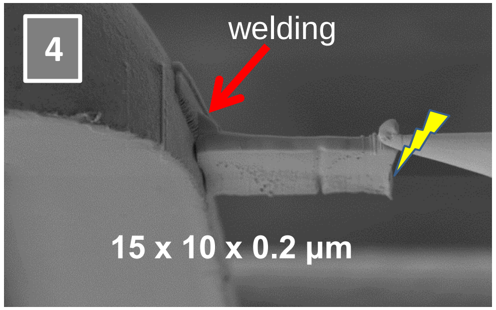


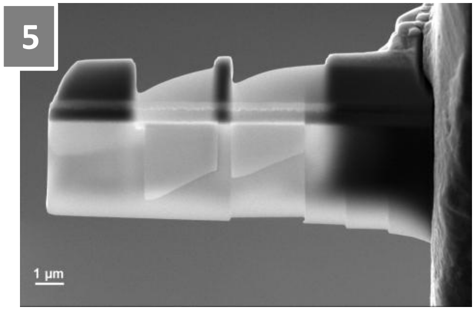
1. Pt deposition
2. Thinning & cutting
3. Lift out
4. Weld on the holder
5. Final thinning and polishing





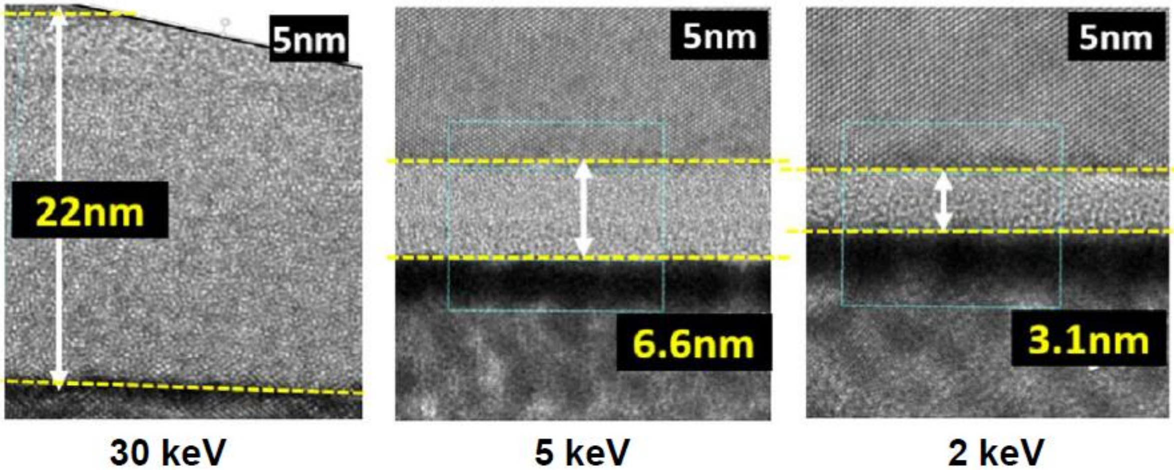




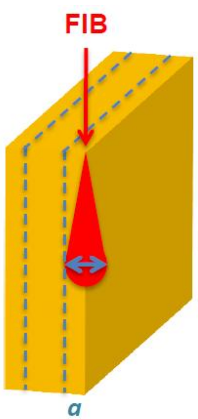


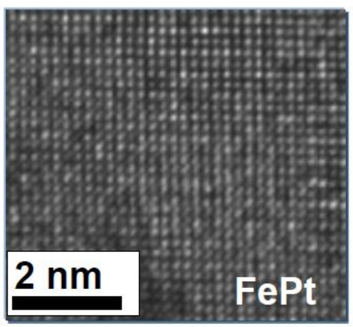
Sidewall Damage: Lateral ion straggling (∝Energy) damages lamella faces during thinning

T.L. Burnett et al., *Ultramicroscopy* 161(2016)119.



Solution: Final thinning at the lowest KeV available





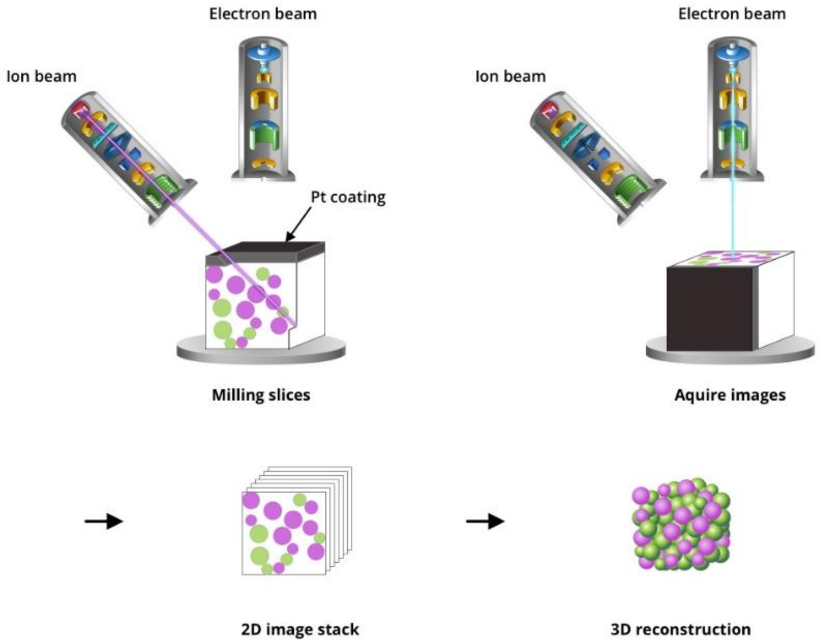
HRTEM

Atomic resolution

A. Di Bona et al.,
Acta Mater. 61 (2013) 4840

Reconstruction of a 3-dimensional volume from a series of slices → **serial sectioning**

- **Distructive Technique**
- FIB → cut, SEM → imaging (SE, EDS,EBSD)
- Pt deposition to avoid curtaining and large tranches to reduce redeposition
- Slice Thickness from 10nm to µm
- A lot of FIB-SEMs allow automatization

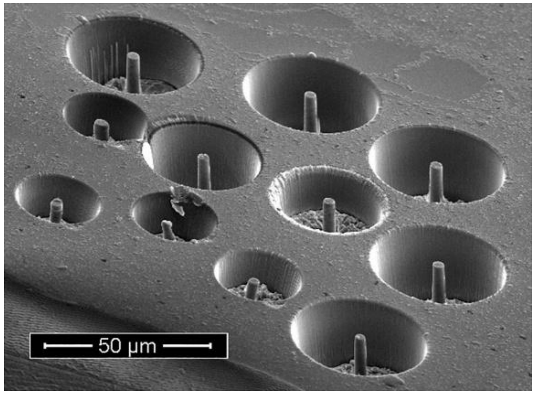


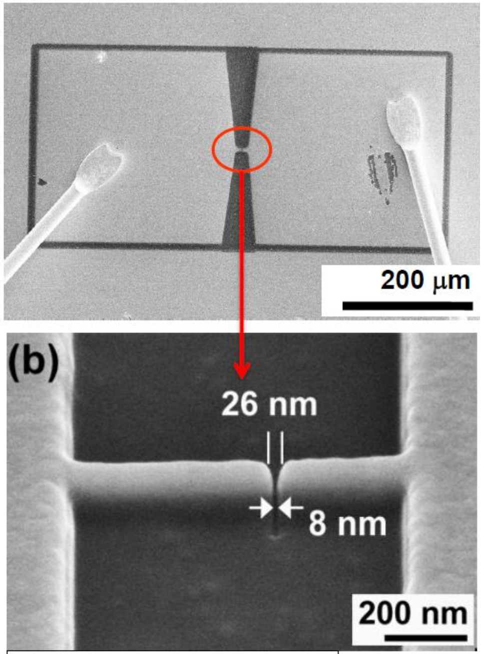
Conventional 3D stack sizes:
Ga^{+} ion < 50µm x 50µm x 50µm
Plasma <500µm x 500µm x 500µm

Applications: Micro-machining and nanopatterning

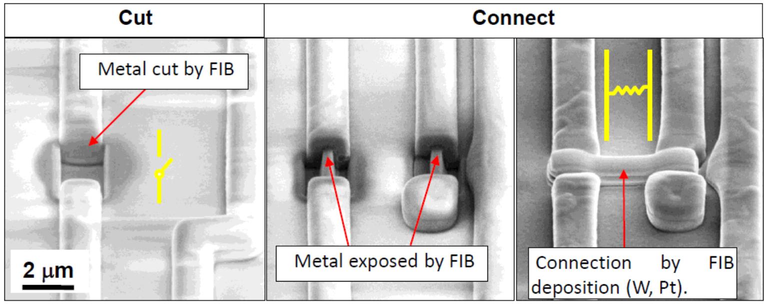
FIBs are able to structure a wide range of patterns and structure from the nanometer to micrometer scale

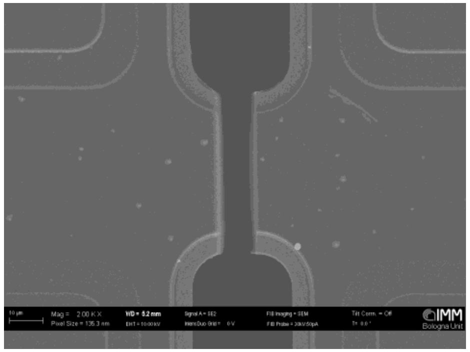
- Circuit Edit (the application at the origin of FIB development)
- arrays of nanopores, nanopillars
- plasmonic devices
- microfluidic channels
- complex 2D and 3D structures
- Nanofabrication (nanolithography, AFM tip)

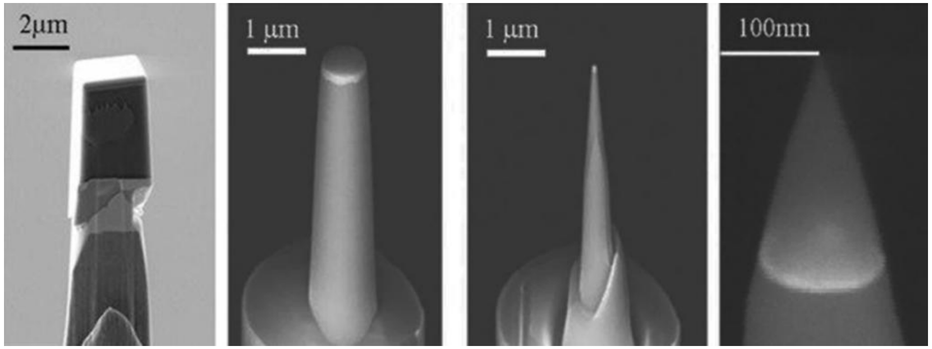




G.C. Gazzadi et al., APL 89, 173112 (2006)







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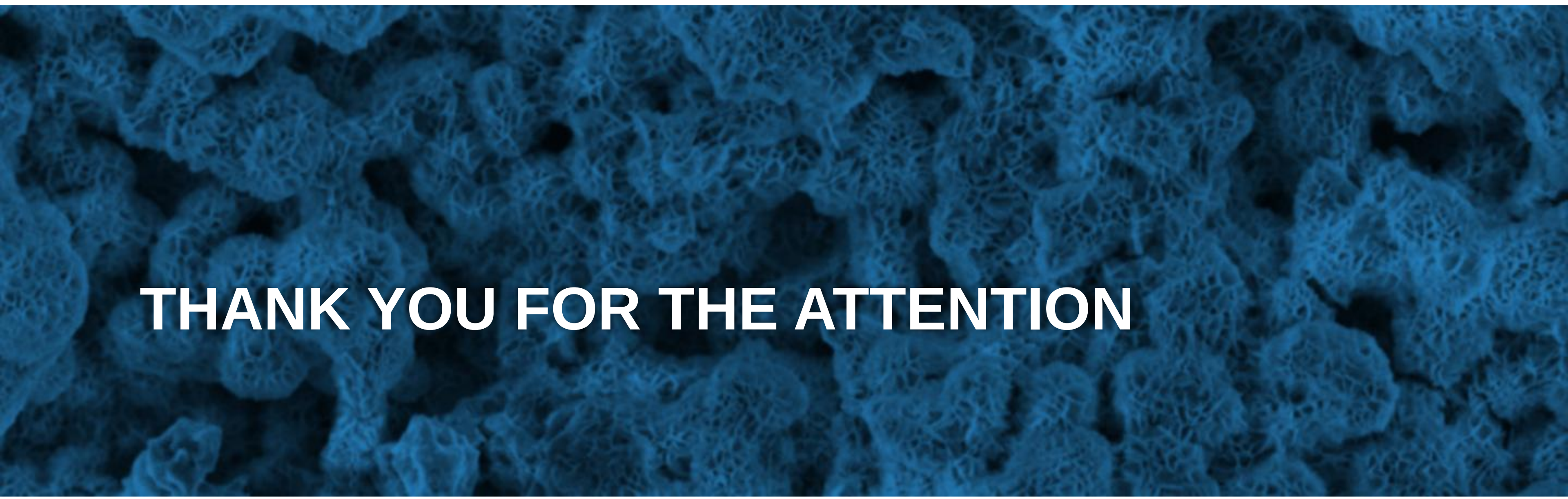

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