



adolphe merkle institute
excellence in pure and applied nanoscience

UNIVERSITY
OF FRIBOURG
SWITZERLAND

Focused ion beam

Introduction

Version 11 – May 2026

PART II –MILLING CONCEPTS



Universal rules

Rule 1: don't touch a control if you are not sure of the outcome of that action

Rule 2: never, ever force anything beyond finger strength

Rule 3: wear gloves when touching anything that goes into the chamber

Rule 4: if in doubt, ask for help

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Demonstration: Log on onto the FIB software**Prerequisites:**

Running xT server

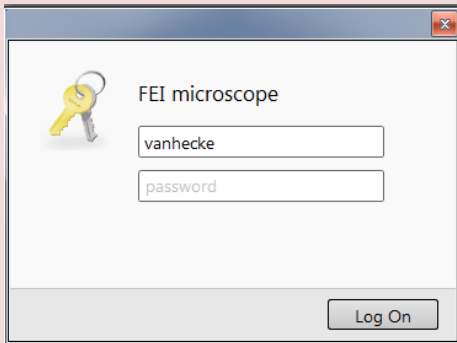
Running UI

Action:

Login using your FIB account

Load the personal settings and history of the user**Experiment**

After startup of the UI, a username and password are requested.



Username: your last name, with capital, no accents, umlauts, etc.

Password: your first name (no capitals, accents, umlauts, etc.)

Click logon

Demonstration: The LMIS (liquid metal ion source)

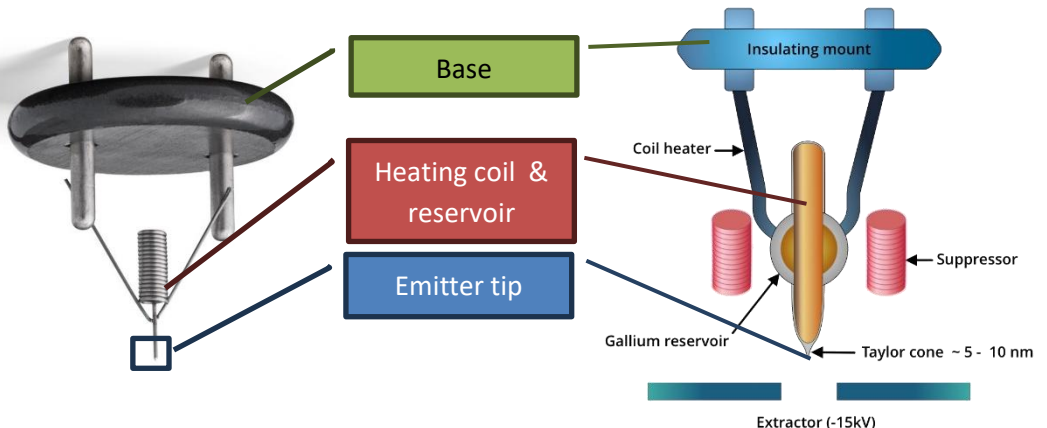
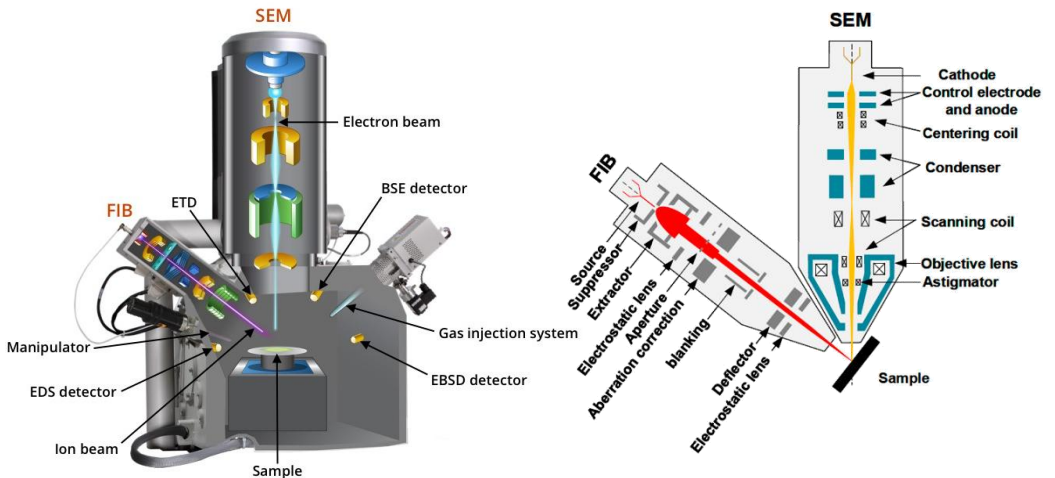
Prerequisites:

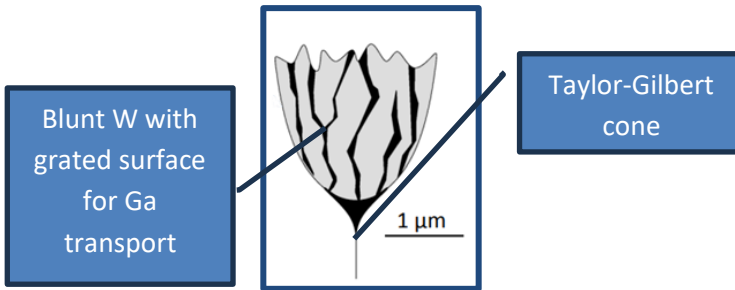
Running xT server

Running UI

Background on the ion beam

System overview





Extractor: Applies high negative voltage (~ -10 to -15 kV) to the tungsten needle tip wetted with liquid Ga, pulling Ga^+ ions from the Taylor cone via field ionization /

evaporation to form the initial ion beam.

Suppressor: Controls emission precisely by fine-tuning voltage around the needle apex, stabilizing total ion current ($\sim 2.2 \mu\text{A}$) and suppressing unwanted ions/electrons.

Together they balance field strength for bright, stable emission and ensure **narrow energy spread** ($\sim 1\text{-}2$ eV FWHM) by electrostatic filtering during ion formation.

Why Ga ions?

- $M_p = 29.8^\circ\text{C}$ (liquid at RT)
- Low Vapor pressure (10^{-10} Pa @ 20°C , i.e. remains liquid without evaporation in high vacuum)
- Narrow energy distribution (near monochromatic beam)

Comparison between Ga ions and electrons

Electrons	Ions
very small	Big -> outer shell reactions (no x-rays)
inner shell reactions	High interaction probability
High penetration depth	less penetration depth
Low mass -> higher speed for given energy	High mass -> slow speed but high Momentum -> milling !!! Ions can remain trapped -> doping
Negatively charged	Positively charged
Magnetic lens (Lorentz force)	Electrostatic lenses

Demonstration: Switch the beams on**Prerequisites:**

Sample loaded

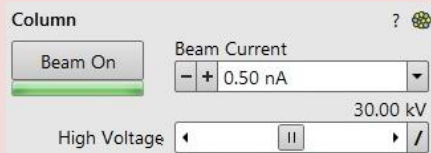
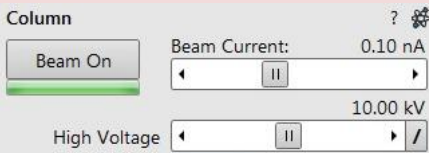
Learn to switch e-beam and ion beam on

If the system is in standby, you will find a **green** bar under the “Beam On”**Experiment**

Select the electron beam quadrant (top left)

In the Beam control  > Column. Check the bar under the button “Beam On”.

Repeat for the ion beam: first click on the ion beam quadrant (top right)

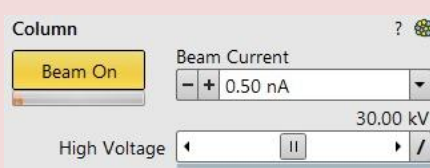
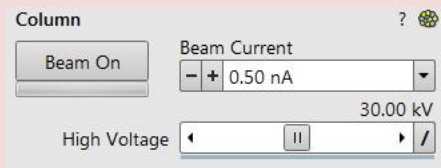
Electron beam and ion beam both with green progress bars: **standby**.

Click both Beam On to start the FIB

If the system is in sleep mode, you will find a **gray** bar under the ion “Beam On”**Experiment**

Electron beam: same as standby

Ion beam: the progress bar will be gray. Click the Beam On.



The gray progress bar will turn red then orange then yellow, while progressing.

It will take about 15 minutes to startup the ion beam.

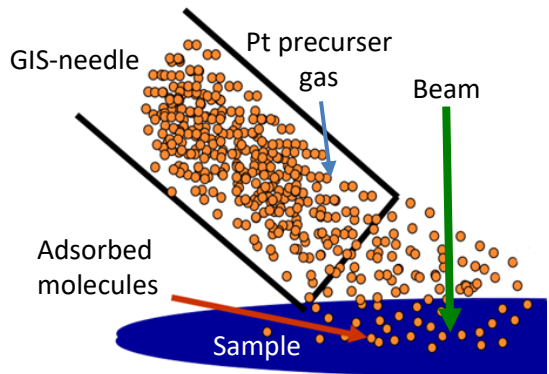
Demonstration: e-beam Pt deposition**Prerequisites:**

Sample loaded

Electron beam on

Eucentric height, focused, WD=7mm**Learn to deposit Pt using the e-beam**

Pt deposition using the e-beam can be used to ensure a proper beam coincidence point setting if no landmarks are available.



IT IS CRITICAL TO BE AT 7 mm WD, IN FOCUS at eucentric height

Settings and preparation**Patterning settings:**

- XYZ: $5\mu\text{m} \times 5\mu\text{m} \times 0.5\mu\text{m}$
- 15 μs dwell time
- Application Pt e-dep surface (e beam induced deposition)

Microscopy settings:

- might slightly vary on your sample):
- Standard mode
 - Magnification: 2000 X or higher
 - Acceleration voltage: 5 KV
 - Beam current: set until time is 3-5 minutes.


Make sure your sample is at eucentric height, focused and the WD=7 mm. Hit CTRL+f

 Pt deposition

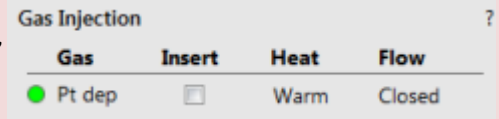
Experiment

Under Patterning > chose a rectangle. Set e.g. 5 μm x 5 μm , deposit using the Pt application (Pt e-dep surface). The rectangle has a green border

Insert the GIS needle

In the patterning tab (), near the bottom, Find the Gas injection menu¹.

- Assure the camera view is live
- Double check if you really are at 7 mm!!
- Then tick “insert” box




You hear a soft “tick” sound and in the camera quadrant you see the needle

Adjust the contrast and brightness

The GIS needle absorbs a significant part of the SE signal and will darken your image.

- Press F9 to adjust Brightness and contrast
- Optional: zoom out in the SEM quadrant (below 1000X) to the the needle

Start patterning

In the top menu, click the start patterning icon 

During patterning, you can pause or stop the patterning

Finishing the patterning

When finished:

- retract the GIS needle.
- Hit F9 to update the brightness and contrast
- Select the pattern in the quadrant and remove it (press DEL)

Note: e- beam deposition is soft and slow, iSPI is not possible

¹ The icon is green, Heat = warm and the flow is closed.

Demonstration: Beam coincidence point**Prerequisites:**

Sample loaded

Electron beam on

Eucentric height, focused, WD=7mm

Set the beam coincidence point

A proper beam coincidence point is crucial to use the FIB beam. A proper beam coincidence point assures that the FIB beam is focused on the SEM image.

Experiment**Reset beam shift**

- Beam > beam shift > right click > reset

Landmark definition

Search for a landmark in your SEM image and center it at the yellow cross (if there is none, get one in the overlay, see below)

- Focus, link, WD = 7mm, CTRL + f to set to eucentric height

- Magnification: around 5000 X, 5 kV, ~0.4 nA

Activate the overlay crosshair: View > center cross (or shift + F5)

Tilt the stage

- Tilt the stage to about 5°. Watch the landmark move up or down. Bring it back to its central position, either by:

→ In the CCD quadrant (Bottom right): hold the middle mouse button.

→ move the mouse down/up to move the landmark accordingly.

- Iterate over 5-10° steps until you reach 52°. Keep an eye on the stage in the chamber

view: **do not touch the pole piece with the sample!**

The intensity of the signal will increase as tilt increases. Also: try CTRL + e and CTRL + i

Demonstration: Aligning electron and ion beam**Prerequisites:**

Sample loaded

Electron beam on

Eucentric height, beam focused, 7mm working distance

Coincidence point set

Find where the ion beam and the electron beam meet

Important notice:

The ion beam will destroy your sample surface (unless it is protected by layers of Pt).

Do not continuously image with an ion beam!

Use low currents for imaging! (30 pA or lower)

Use single image only

Experiment**Align the FIB image**

- Image with the Ga ion beam:

- use a low beam current (10 pA or about), 30 kV
- Zoom out to a magnification below what you had in the electron beam.
- Press CTRL + f. Assure the working distance is **19 mm**
- press F9
- Assure you have low current, dwell times below 1 μ s, live camera settings. Then press F6 and press F6 immediately again (will record 1 image)
- Find an object that is present in both the electron image and the ion image.

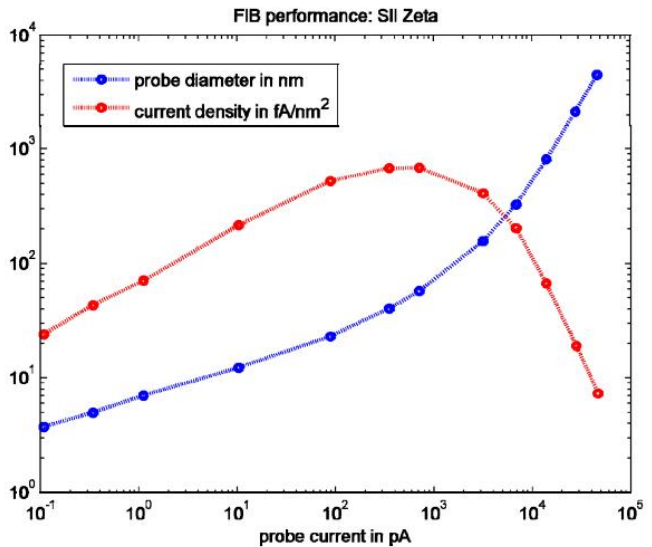
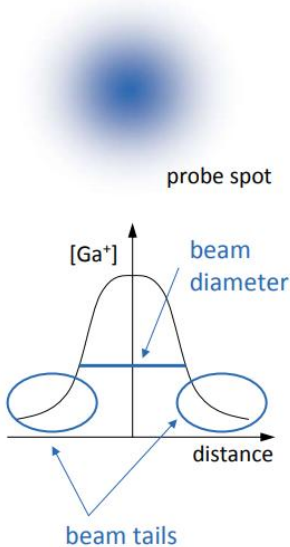
If the same object is not in the middle (use the center cross):

- Use beam shift XY to put it in the middle of the Ion beam image:

1. Open the beam control. Watch the Beam shift module
2. Grab the object / landmark with LMB while holding shift. The mouse icon will change into a hand with a blue sleeve.
3. Move the mouse (holding shift and LMB) to the center cross. The marker in the beam shift module should not reach the borders of the control. The image will not change.
4. release the mouse

Alternatively, use the shift XY buttons are on the physical control panel below the central screen. Note that here you will need a live ion image. switch off the FIB imaging as soon as the landmark is aligned (you are milling away your sample).

Setting: couple magnifications to OFF



Small current → narrow beam

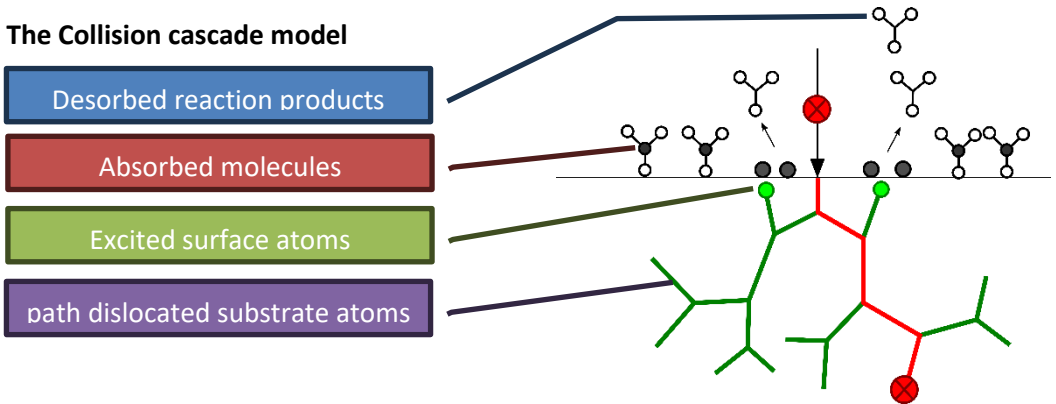
Demonstration: Pt deposition with the ion beam

Prerequisites:

- Sample loaded
- Electron beam on
- Eucentric height, beam focused, 7mm working distance
- Coincidence point set
- Stage tilt: 52°

Deposit Pt with the ion beam

The Collision cascade model



Experiment

Patterning settings

- Choose a rectangle (easiest)
- XYZ: XY, usually < 20, Z usually between 0.1 and 1 μm
- Application: Pt dep

Microscope settings:

- Magnification: 2000 X or higher (Standard mode, ETD detector)
- Ion acceleration voltage: 30 KV
- Beam current: **CALCULATE!** Using this formula:

$$\text{Current (pA)} = X(\mu\text{m}) \cdot Y(\mu\text{m}) \cdot 6 \left(\frac{\text{pA}}{\mu\text{m}^2} \right)$$

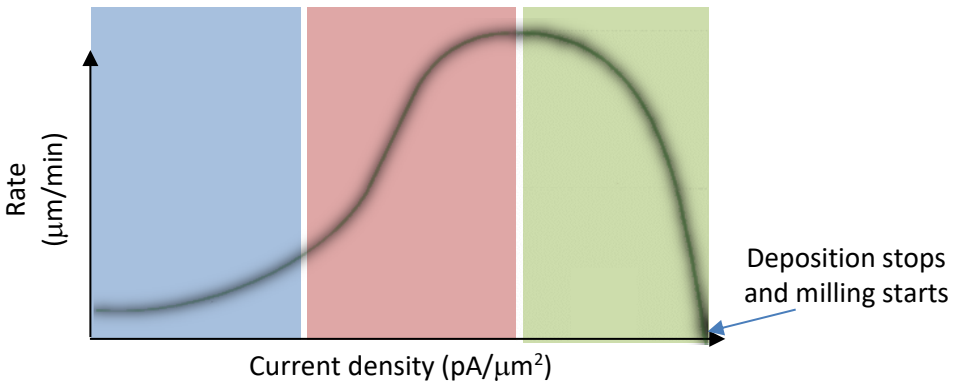
Where X = width of the pattern in μm , Y = height of the pattern in μm .

Choose the ion beam current to be as close as possible to the calculated value

Example: a $20\ \mu\text{m} \times 20\ \mu\text{m}$ rectangle $\rightarrow 6 \times 20 \times 20 = 2400\ \text{pA}$ (actual value: $3\ \text{nA}$)

Example: a $2\ \mu\text{m} \times 3\ \mu\text{m}$ rectangle $\rightarrow 6 \times 2 \times 3 = 36\ \text{pA}$ (actual value: $30\ \text{pA}$)

Failing to choose the correct beam current will either ruin your vacuum or create a hole in your object.



High-efficiency deposition per ion

- Ion dose on each pass does not decompose all gas
- Slow layer growth rate, Long deposition time
- gas may affect vacuum

**Lower beam current
Larger patterns**

High-efficiency growth rate

- Each beam scan uses up nearly all precursor gas
- Fastest layer growth rate

**(2-)6 pA/ μm^2
Standard patterns**

Milling effects

- All gas is used up by only part of ion dose
- Remaining ions sputter / mill the surface

**High beam current
Tiny patterns**

Demonstration: Patterning types

Prerequisites:

Sample loaded

Electron beam on

Eucentric height, focused, WD=7mm, either at 0° or 52°

Information of the different patterning types

Beam control

Navigation

Detectors

Patterning

Patterning Control

Pattern type selector (see Toolbar icons also):
 Rectangle / Line / Circle /
 Cleaning Cross Section /
 Regular Cross Section /

Time indicator

Pattern size

Array settings, passes, ...

Pt Deposition

Basic Properties	
Application	Pt_EBID
X Size	5.00 µm
Y Size	5.00 µm
Z Size	0.49 µm
Scan Direction	Bottom To Top
Dwell Time	15.000 µs
Beam	Electron
Time	00:02:32

Advanced Properties	
Rotation	0 °
Position X	0.08 µm
Position Y	0.08 µm
Overlap X	75 %
Overlap Y	75 %
Gas Type	Pt dep
Pitch X	0.01 µm
Pitch Y	0.01 µm
Area Calculation	Pattern

Gas Injection

Gas	Insert	Heat	Flow
<input checked="" type="checkbox"/> Pt dep	<input checked="" type="checkbox"/>	Warm	Opened

iSPI Monitor Drift Suppression

On Pause Save

Time Interval

CCS Line Interval

Pattern types

Patterns are automatically assigned to one or more particular processes, distinguishable by a different cross-hatch.

- Rectangle / Line / Circle / Polygon pattern: both milling and deposition.
- Cleaning Cross Section: milling line by line (each line with set number of passes).
- Regular Cross Section: has two possibilities selectable in the Property editor:
 - Multipass – processes entire pattern and starts again (with set number of passes)
 - Stairstep – the pattern is created as a compilation of five rectangles with specified overlap between them. Each one is processed with the set number of passes.
- The Bitmap pattern enables importing bitmaps as a pattern. It must be 24 bit RGB bitmap, each pixel consists of:
 - Red component – actually not used
 - Green component – determines if the beam is blanked.
 - Any value other than 0 activates the beam
 - Blue component – determines the dwell time per pixel:

Serial vs parallel patterning



This is the default patterning mode. All patterns defined on the screen are processed consecutively; milling / deposition is completed on one pattern before moving to the next one. Serial patterning is always used with cleaning cross section milling.



All patterns defined on the screen are processed concurrently, one pass of the beam is completed on all patterns before moving to the second pass. Parallel patterning is typically used to avoid a redeposition of material on the adjacent areas.

Pattern properties

A pattern can have many associated parameters, which can be set via the Property module:

Properties		Selective Mill	?
Basic Properties			
Application	none		
Outer Diameter	91.15 μm		
Inner Diameter	0 μm		
Z Size	1.00 μm		
Scan Direction	Inner To Outer		
Dwell Time	1.000 μs		
Beam	Electron		
Time	316.7 ms		
Advanced Properties			
Position X	279.95 μm		
Position Y	386.72 μm		
Rotation	0 °		
Gas Type	none		
Overlap X	50 %		
Overlap Y	50 %		
Pitch X	1.49 nm		
Pitch Y	1.49 nm		
Overlap T	50 %		
Overlap R	50 %		
Pitch T	1.49 nm		
Pitch R	1.49 nm		
Area Calculation	Pattern		
Dose	3.68 nC/ μm^2		
Volume per Dose	2.700E-1 $\mu\text{m}^3/\text{nC}$		
Saturation Sputter Rate	0.000000		
Refresh Time	0 s		
Loop Time	3.9 ms		
Area	6524.74 μm^2		
Scan Type	Circular		
Fill Style	Solid		
Passes	82		
Defocus	0 μm		
Blur	0 μm		
Interaction Diameter	0 μm		
Total Diameter	3.0 nm		
Maximum Dose per Area	0 nC/ μm^2		
Saturation Current Density	1.000E-18 A/ nm^2		
Total Volume Sputter Rate	2.700E+7 nm^3/s		
Selective Milling Enabled	<input type="checkbox"/>		
Selective Milling Time Interv	2.000000		
Min Contrast Threshold	0.000000		
Max Contrast Threshold	1.000000		

Application

clicking on the value slot enables a down arrow bringing a drop down list of applications. Choosing the required one sets the subsequent properties.

X / Y / Z size

Dimensions of the pattern

Scan Direction

Scan movement direction (Bottom to Top; Top to Bottom)

Dwell Time

A time the beam spends on a single pixel per pass (rounded to a multiple of 25 ns).

Beam

The beam used for patterning

Time

required to process this pattern. Calculated from the different parameters

Rotation

Rotation of the patterns (the positive direction is clockwise)

Position X / Y

Position of the pattern relative to the origin (the display center)

Overlap X / Y

Sets the beam diameter overlap. The value of the overlap can be positive (=array) or negative (=overlapping) depending on a particular application. The overlap parameter influences the Area Calculation and the Dose.

Gas Type

the gas to be used to process the pattern (or None if no gas is to be used). This determines the pattern color onscreen (yellow for milling, green for Pt deposition).

Pitch X / Y

Sets the pitch between two spots. Alternative to overlap.

Area Calculation

Defines how the patterning area will be calculated in order to get the most accurate value of the Dose. This value is related with the OverlapX/Y. The Pattern (default) / Array are set for positive / negative overlaps.

Volume per Dose

The volume of material that is removed per charge

Saturation Sputter Rate

The maximum linear sputter rate for a given gas. For Gas = None this is 0 (actually not used).

Refresh Time

The minimum loop time that must at least elapse before the next pass, so that the adsorbed gas can be refreshed

Loop Time

The time required for a single pass (read only)

Area

The surface area of the pattern (read only)

ScanType

the Serpentine means the beam proceeds from left to right and back from right to left, while the Raster scans from left to right, then the beam returns to the left starting point

Fill Style

One can choose either to mill a solid (area) or just a frame (box and circular types only)

Passes

The number of the beam scans over the pattern

Defocus of the beam (WD change)

Influences the Total Diameter and Area Calculation. It allows focusing above / below (negative / positive value) the sample surface

Blur

Like Defocus, but specifying the (additional) diameter of the blurred spot

Interaction Diameter for an infinitely small beam

Influences the Total diameter

Total Diameter

the combination of the beam diameter and interaction diameter influences the

Selective Milling Enabled / Selective Milling Time Interval / Min Contrast Threshold / Max Contrast Threshold items

Correspond to the Selective Mill tab module elements: Enabled check box / Interval adjuster / left / right border of the grey level to be processed for the selected pattern.

A note on Overlap vs Pitch

Overlap and **Pitch (X,Y)** are two different ways of describing the same spot-to-spot spacing, but from opposite directions.

- **Overlap** is a percentage-based setting. It tells the system how much consecutive beam spots should overlap relative to the beam diameter.
- **Pitch** is a distance-based setting. It tells the system the physical center-to-center spacing between adjacent spots in X and Y.

OverlapX/Y and PitchX/Y values (read only)

Maximum Dose per Area

describes the adsorbed gas layer, allowing a certain dose to be deposited at a higher rate than the saturation current density, allowing a temporary higher rate (actually not used)

Saturation Current Density

The current at which 63% of the saturation sputter rate is reached (actually not used)

Total Volume Sputter Rate – the speed at which material is removed or deposited (actually not used)

If the beam diameter is d and the pitch is s , then:

$$\text{Overlap} = \frac{d-s}{d} \times 100\% \quad \text{Pitch} = d \cdot (1 - \text{Overlap})$$

- **0% overlap** means pitch equals one beam diameter.
- **Positive overlap** means spots partially cover each other.
- **Negative overlap** means there is a gap between spots.

For example, if the effective beam diameter were 10 nm:

Pitch = 10 nm = 0% overlap.

Pitch = 5 nm = 50% overlap.

Pitch = 12 nm = -20% overlap.

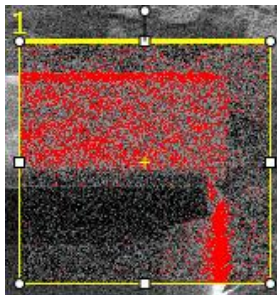
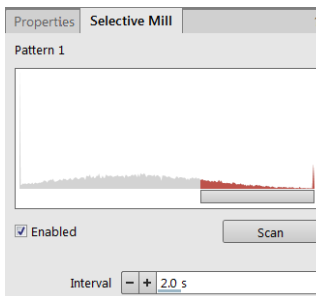
If you keep the same beam current and thus roughly the same effective beam diameter:

- Increasing **Overlap** makes the pattern more continuous, with smoother milling or deposition, but it also increases dose per area.
- Increasing **Pitch** spreads spots farther apart, reducing dose per area and making the pattern less dense.

The software lets you choose whichever is more convenient:

- **Overlap** is intuitive when you think in terms of “how much should spots cover each other?”
- **Pitch** is intuitive when you think in terms of “what spacing do I want between passes?”

Selective milling



Selects to be milled pixels based on their grey level. The scan button reads the pattern area grey level histogram (only ion image).

Demonstration: Basic milling concepts**Prerequisites:**

Sample loaded

Electron beam on

Eucentric height, focused, WD=7mm

Beam coincidence point set

Milling practicalities**Ion version electron**

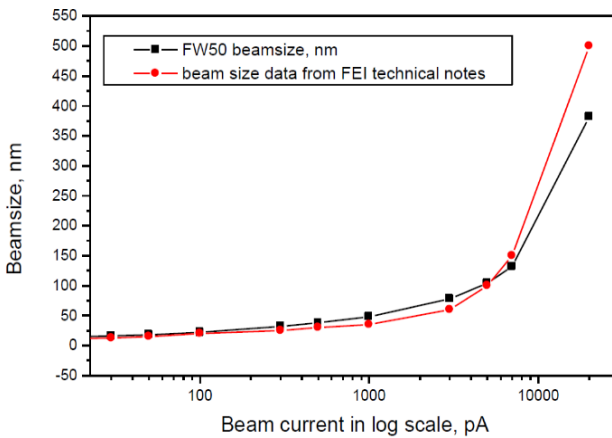
Particle	ion	electron	ratio
Type	Ga ⁺ ion	electron	
Elementary charge	+1	-1	
Particle size	0.2 nm	0.00001 nm	20 000
Mass	$1.2 \cdot 10^{-25}$ kg	$9.1 \cdot 10^{-31}$ kg	130 000
Velocity at 30 kV	$2.8 \cdot 10^5$ m/s	$1.0 \cdot 10^8$ m/s	0.0028
Velocity at 2 kV	$7.3 \cdot 10^4$ m/s	$2.6 \cdot 10^7$ m/s	0.0028
Momentum at 30 kV	$3.4 \cdot 10^{-20}$ kgm/s	$9.1 \cdot 10^{-23}$ kgm/s	370
Momentum at 2 kV	$8.8 \cdot 10^{-21}$ kgm/s	$2.4 \cdot 10^{-23}$ kgm/s	370

Beam	ion	electron
Size	nm range	nm range
Energy	Up to 30 kV	Up to 30 kV
Current	OpA to nA range	OpA to μ A range

Penetration depth	ion	electron
In Polymer @ 30 kV	60 nm	12 000 nm
In Polymer @ 2 kV	12 nm	100 nm
In Iron @ 30 kV	20 nm	1 800 nm
In Iron @ 2 kV	4 nm	25 nm

Average electron signal per 100 particles @ 20 kV	ion	electron
Secondary electrons	100-200	50-75
Backscattered electrons	0	30-50
Substrate atom	500	0
Secondary ion	30	0
X-ray	0	0.7

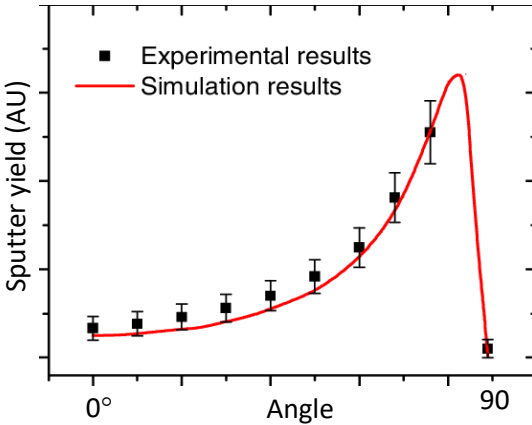
- Ion beam morphology



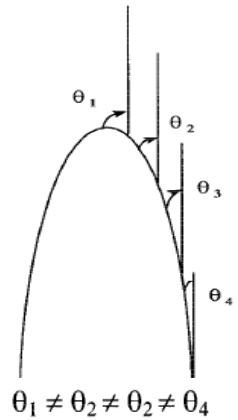
The higher the ion current, the wider and larger the ion beam.

Ion Beam Current (pA)	Aperture Diameter (μm)	Beam Diameter (nm)
1.5	0.008	7.0
10	0.016	13.0
30	0.025	17.0
50	0.032	19.0
100	0.042	24.0
300	0.068	31.0
500	0.087	35.0
1000	0.118	44.0
3000	0.198	66.0
5000	0.250	85.0
7000	0.294	102.0
15000	0.420	182.0
30000	0.600	210.0
50000	0.750	300.0
65000	1.000	400.0

- Sputter yield depends on sputter angle

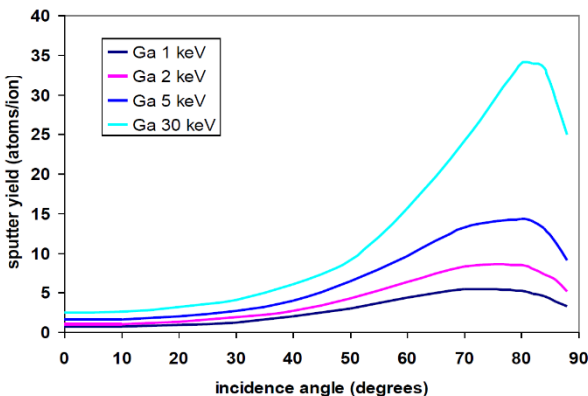


The milling efficiency is a function of the local curvature of the sample. **Milling works most efficiently between 75-85°.** At 90°, the sputter yield is near zero. Hence, do not mill samples that are not flat: you will end up with a preferentially milled object.



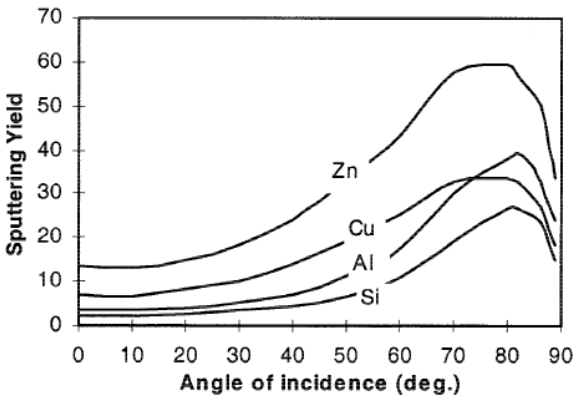
Asperities on a surface will be FIB milled at different rates due to topographic effects on milling. The topographic effects will grow and exacerbate as FIB milling continues. This is why surfaces are “never” FIB milled from the top down; rather, they are created by FIB milling at high incident angles.

- Sputter yield depends on ion acceleration voltage



30 keV is the maximum voltage of the ion gun. Always use 30 kV, unless clearly mentioned otherwise.

- Sputter yield depends on target material



Similar to the Mohs scale of hardness of solids (graphene = soft, diamond is hard), each material has a tendency to get sputtered differently by the Ga ion beam. The sputter rates are not a function of the Mohs scale.

Crystalline structures will cause channeling of the ions depending on the Bragg conditions. Hence,

crystalline structures will not mill to a flat surface.

Material	Sputter rate ($\mu\text{m}^3 / \text{nC}$)
Si	0.27
TEOS	0.24
Al	0.3
Al_2O_3	0.08
GaAs	0.61
InP	1.20
Au	1.50
TiN	0.15
Si_3N_4	0.20
C	0.18
Ti	0.37
Cr	0.10

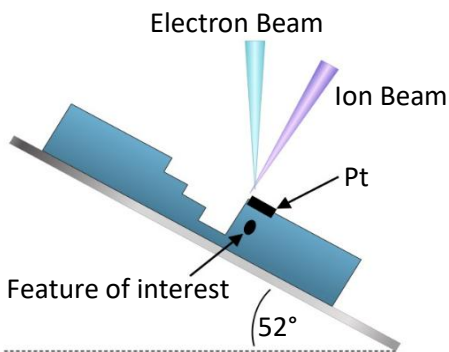
Material	Sputter rate ($\mu\text{m}^3 / \text{nC}$)
Fe	0.29
Ni	0.14
Cu	0.25
Mo	0.12
Ta	0.32
W	0.12
MgO	0.15
TiO	0.15
Fe_2O_3	0.25
Pt	0.23
PMMA	0.40

Demonstration: Making a cross section

Prerequisites:

Eucentric height, beam focused, 7mm working distance
Coincidence point set
Ion beam and e-beam aligned

Produce a cross section



First, a protective Pt layer is deposited over the region of interest using the electron or ion beam. Then, the focused ion beam mills a trench from the side to expose a clean cross-section while the SEM images it *in situ*. Final polishing refines the surface for high-resolution analysis

- Preparation

In the electron beam (0°)

- Proper imaging setting, magnification: sufficient (1500+ X)
- Focus the region
- Link the stage
- Go to 7 mm WD (at 0 degree)
- Set eucentric (CTRL+F)

Make 100% sure you are in focus, eucentric and at 7mm. Incorrect settings will damage the instrument

-
- Electron beam induced Pt deposition (optional)
-

See page 10 for full description. Electron beam Pt deposition (optional, less damaging) protects beam-sensitive samples before milling; use ion beam Pt deposition (see below) instead for higher efficiency.

(Optional) experiment: protect the ROI using e-beam Pt deposition

- Insert the GIS needle
 - it drops a shadow on your image: Press F9
 - If the needle is visible: increase magnification
- Select a rectangle and draw a pattern **in the e-beam quadrant**. As application, choose Pt_EBID (E beam induced deposition). The rectangle should be **green**

- Microscope settings

- Patterning settings

(will vary depending on the sample)

Standard mode

set X, Y: 20 μ m x 2 μ m

2000 X (or higher)

Z = 500 nm

2 KV

- Electron beam current: set to get a time of 5-7 minutes
- Click start patterning

Note: e- beam deposition is slow, iSPI is not possible

- When finished: retract the GIS needle. Hit F9. remove the overlay pattern.
-

- Setup tilting and beam-coincidence point
-

See page 12 for full description. A proper beam coincidence point ensures FIB and SEM beams intersect precisely at the sample surface, enabling simultaneous milling and imaging of the exact same location. Without it, FIB misses the intended target.

A proper beam coincidence point is crucial to use the FIB beam. A proper beam coincidence point assures that the FIB beam is focused on the SEM image.

Experiment: setup beam-coincidence point

Set the BCP. You may use the deposition from the step before as a marker.

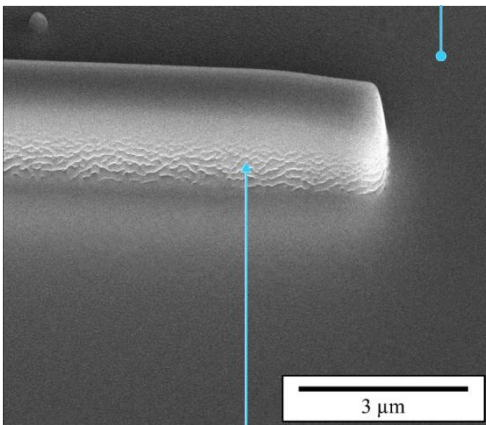
CTRL + i (will tilt to 52° – ion beam)

CTRL + e (will tilt to 0° – electron beam)

Use the ion beam shift to align the e-beam to the ion beam precisely, as described on page 13.

- Protecting your object of interest

Sample surface (very flat example)



Pt deposit (covers and protects the underlying structures)

More detailed explanation on page 15. Pt deposition is crucial for cross-sections, as it protects the surface of interest from ion-beam damage and sputtering artifacts, creating a clean milling mask. Without it, the region smears or curtains during trenching.

For cross sections, it is usually sufficient to make the deposition as wide as you want your section (e.g. X = 15 μm), with a Y value of about 1 μm and Z of 500nm. You can deposit Pt on non-flat surfaces and even on large spherical objects (e.g. 10 μm spheres)

Experiment: ion beam Pt deposition (MANDATORY)

Pt deposition with the Ga ion beam (in the **Ga ion image**)

- First glance: Do not make an image with the Ga ion beam (you may destroy the surface)!
- Draw a rectangle in the Ga ion image with the patterning tool (e.g. 20μm x 1μm).
- Z = 0.5 to 1 μm.

for a cross section of a flat specimen, 0.5 μm is usually enough. For porous or non-flat samples, you can increase Z.

- Select Pt dep (not Si) in the application. The rectangle is **green**
- Calculate the Ga current required using the magic number 6 ($\text{pA}/\mu\text{m}^2$).

$$\text{Current (pA)} = X(\mu\text{m}) \cdot Y(\mu\text{m}) \cdot 6 \left(\frac{\text{pA}}{\mu\text{m}^2} \right)$$

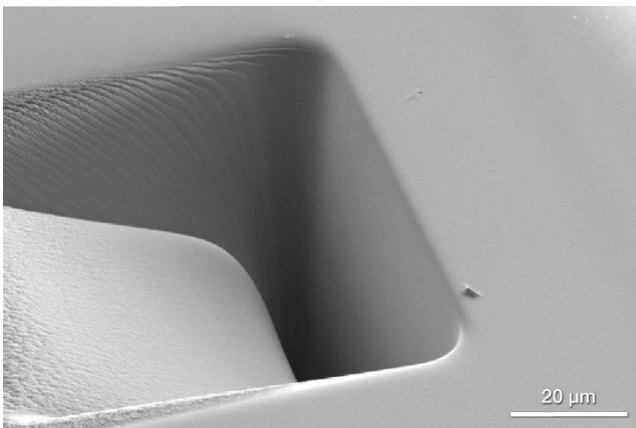
* e.g. 25 μm x 1.5 μm x 6 = 225 pA

* Choose the closest current for the Ga beam (300 pA)

too much current and you will mill instead of deposit
Too less current will destroy your vacuum (see page 16)

- You should get a time around 4-6 minutes for Z = 1 μm , or half that for Z = 500nm
- Insert the Pt GIS
- Press F9 in the ion image (this will contrast/brightness correct and take a snapshot).
Make sure you have the ETD selected
- Check the position of the rectangle, overlay the e-beam deposited marker.
- Run the deposition
- retract the GIS needle

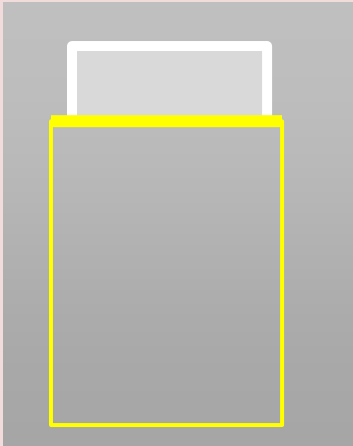
- Bulk Milling (sloped trench)



With FIB perpendicular to the sample, milling removes material fastest from the SEM-distant end (higher effective angle due to pattern geometry and stage view), creating a **sloped trench**.

Experiment: Bulk mill

- Use the **regular cross section** (RCS) pattern.



- Position the yellow RCS pattern just below the Pt deposition you have just made, and a bit wider than the Pt deposition pad (about 10%), exactly touching the Pt above it. The thick line is up. The pattern is **yellow**.

- Application: Si multipass (4 passes)

- Determine / decide on the depth (e.g. 5 μm)

- Calculate the Y, with at least $Y > 2$ times Z. If you intend to do EDX, a factor of 3 is at least needed (see page 38).

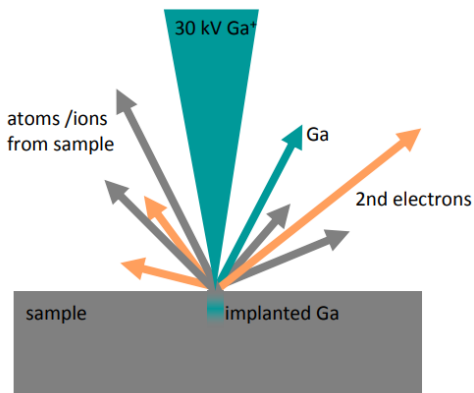
- Pick a Ga ion beam current to mill between 2-5 minutes

- Check your SEM image before you start the milling. F9 to adjust brightness contrast

- Start the patterning ()

Note: iSPI is possible. Use the brightness / contrast buttons on the physical control panel to adjust B/C, not F9

- Note: Ga⁺ ion / sample interaction



Ga⁺ beam hitting a substrate has the following effects:

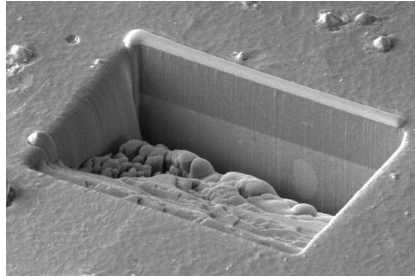
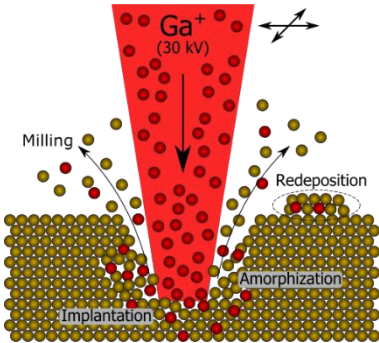
- Secondary electron emission (Imaging)
- Sputtering atoms and ions (Milling) and redeposition
- Implantation of Ga (Deposition)
- Amorphization/recrystallization

But this all happens simultaneously!

By adjusting ion current & atmosphere, we

can pronounce one aspect of the total effect.

There's no true "non-milling" mode (Impinging Ga⁺ ions always mill a little bit)

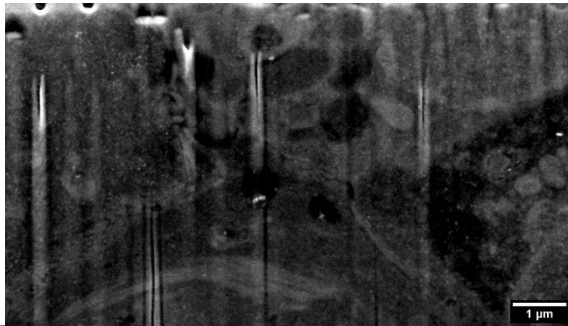
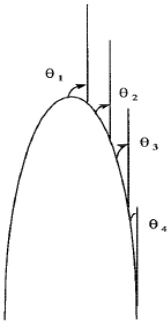


Redeposition in FIB-SEM cross-sections occurs when sputtered material atoms/ions, ejected by the

Ga^+ beam, don't escape ballistically but instead stick to nearby surfaces, typically **bottom of the**

trench and cross-section sidewall.

- Curtaining effect



The sputtering yield depends on the incident angle. Topographic features and voids (e.g. porous material or inclusions of different material)

within the sample create different incident angles for the ion beam, and hence different sputtering yields. Increase in surface roughness will lead to an artefact called **curtaining** which produces vertical lines at the cross-section surface due to the different milling speeds for different angles.

How to reduce/remove:

- Use Pt deposition of smooth layers on the sample surface
- Use lower ion beam currents
- Polishing cross-sections (ie at lower ion beam currents)
- Program polishing from different angles.

- Commercial rocking stages tilt the sample during the cross-sectioning process and remove curtaining most efficiently.
 - In porous samples: embed the sample in resin, or infiltrate with e.g. Pt.
 - When imaging: use T1 instead of ETD
-
- Polishing
-

Polishing is done at a lower ion beam current than the bulk mill. Typically, you start the polishing at 2 steps down the ion beam current list. E.g. if the bulk milling was done at 5nA, you start the polishing at 1 nA (jumping over 3 nA).

You may need to do the polishing iteratively (e.g. 1 nA, 0.3 nA, 50 pA).

Experiment: Polishing

- Set the ion beam current 2 steps lower (in the list) compared to bulk milling
- Place a **cleaning cross-section between the Pt deposition and the edge (or a little bit over it) of the hole that** the bulk milling made before.
- Height: place it just a little bit into the Pt layer. Assure that the pattern starts outside the Pt pad, and ends (=thick yellow line) inside the pad.
- Width of the section: about as wide as the Pt deposition
- Depth: same as the bulk milling setting



- Start the patterning

Repeat at the same or lower beam currents (down to 50 pA), if needed.

Use a rocking stage to improve the polishing. The same concept of lowering beam currents in 2 steps applies.

Experiment: advanced polishing with beam shape correction

Undertilt

- Change the stage tilt angle for 0.5-1.5° (i.e. between 50.5° and 51.5° absolute angle). The higher the ion beam current, the higher the correction angle
- refresh the ion beam image (F9). X and Y as above.
- Z: 25% of the previous setting.
- run the patterning

What is happens? **Less tilt toward the SEM**, directing FIB to hit the **lower part** shallower first, removing the **lower/bottom** preferentially

Overtilt

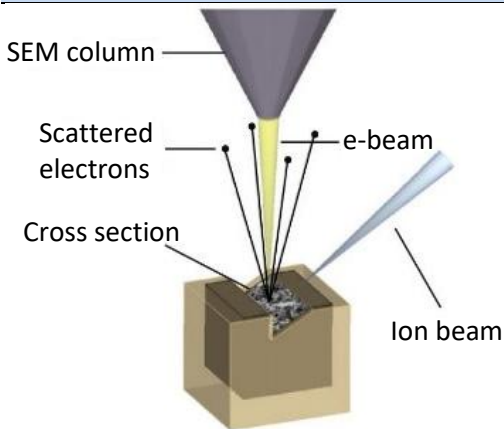
Repeat the patterning (do not change the settings), but overtilt the stage for the same factor (e.g. 53.5° if you used 50.5° before). Proceed carefully and do not evoke a pole touch when tilting!

What is happening? Tilts the stage **more toward the SEM** than nominal, making the FIB beam strike the **upper part** of the cross-section at a **shallower angle** first. This preferentially mills the **upper part** more aggressively, thinning or eroding the top while the **lower part** of the cross section experiences less removal (due to higher incidence angle)

- You can use the iSPY: this will temporarily stop the patterning, make a SEM image and continue

Demonstration: imaging a cross section**Prerequisites:**

- Eucentric height, beam focused, 7mm working distance
- Coincidence point set
- Ion beam and e-beam aligned
- Cross section made

Image the result of your cross section with the e beam**Tilt angle**

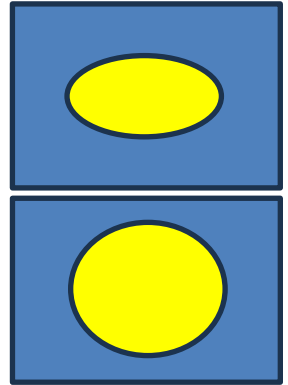
Due to the tilt angle, the SEM image is not seen in an orthogonal, planar dimension.

Hence: you have to compensate for the distorted aspect ratio.

Experiment: Imaging

- Go to a very low ion beam current (10 pA)
- Switch to OptiTilt and use T1 and T2.
- Press F9 and focus/stigmat
- Curtaining issues: Do not use the ETD, since curtaining is the strongest in that detector.
- Lower beam currents: more focused Beam, but more curtaining.

At an angle of 52° , there is a vertical compression that is related to the cosine of that angle (61,6%). For a perfectly round object (top: tilted image at 52° , bottom: original at 0°):



To circumvent this effect, and allow quantification (surface, diameter, length ...) of objects, correct for the tilting angle,

Experiment: Correct for the tilting angle

In Beam control, find Tilt correction & tick Tilt correction

Tilt Angle: Automatic

The correction is set for the current tilt angle (and possible specimen pre-tilts)

Tilt Angle: Automatic (cross section)

The correction is set for a 52° tilt angle (typical cross section angle)

Tilt Angle: Manual

The correction can be set for any tilt angle between $+90^\circ$ and -90°

Dynamic focus

The focus will change as the beam scans from top to bottom, compensating for the working distance in out-of-focus parts of the sample due to the tilt.

Tilt Correction ?

Dynamic Focus Tilt Correction

Tilt Angle

Manual

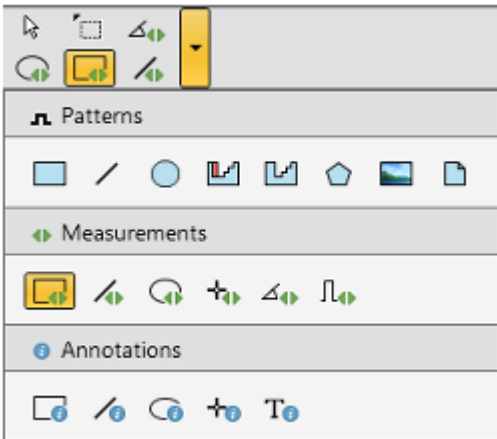
Specimen Pre-tilt

Demonstration: Measuring and making annotations

Prerequisites:

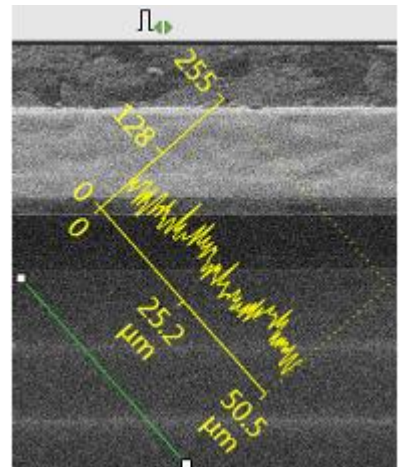
- Eucentric height, beam focused, 7mm working distance
- Coincidence point set
- Ion beam and e-beam aligned

Measure lengths, surfaces, on the image using the Patterns / Measurements / Annotations



Click the down arrow to access the Patterns / Measurements / Annotations tool. The numerical values of linear distances, diameters, angles, or areas of the image are updated while drawing and shown alongside or within the finished measured item.

The Measurement tool dimensions scales with the image;



when changing magnification, the shown tools change their size accordingly.

On the contrary, the Annotation shapes and texts have their sizes fixed relatively to the display. The Measurements / Intensity profile delineates the imaging profile across a freely drawn line.

Note: to save, tick “With overlay” and save as JPG (24 bit) to keep the colours.

Demonstration: Making a cross section using EDX

Prerequisites:

- Eucentric height, beam focused, 7mm working distance
- Coincidence point set
- Ion beam and e-beam aligned

Produce a cross section

Experiment: EDX on a cross section

- Adjust the formula for Y: $Y > 3 \times Z$ (or more). Because BSEs produce X rays in the gap.
- make a second bulk milling on the right side of your ROI to avoid shadowing. You will end up with an L-shaped gap (below and on the right of the ROI).
- Be careful with the interpretation: the imaging is from under an angle, which means that shallow layers may overlap.

EDX Cross section interpretation pitfalls

Ga Implantation Artifacts

FIB milling implants Ga atoms 10-30 nm deep, creating false peaks in EDX maps. Ga L-lines overlap with others (e.g., Zn); standardless analysis has $\pm 5-50\%$ errors on rough surfaces. Ignore <0.5 wt% without confirmation; report semi-quantitative data.

Rough Surface Topography

Cross-sections are never perfectly flat: curtaining, taper, or redeposition causes variable take-off angles, shadowing X-rays from deeper features and biasing low-Z signals.

Beam Damage and Charging

Ion beam amorphizes/etches organics or insulators, altering chemistry and causing charging that deflects electrons, suppressing X-ray generation.

Interaction Volume Overlap

EDX volume ($\sim 0.5-2 \mu\text{m}$ at 5-15 kV) smears signals across layers in nm-scale FIB sections, averaging compositions falsely. Use low kV (3-5 kV).

Demonstration: STEM imaging Demonstration

Prerequisites:

Eucentric height, beam focused, 7mm working distance

Coincidence point set

Stage tilt: 52°

Ion beam and e-beam aligned

Use the STEM detector

Experiment: Link the stage (CRUCIAL)

In the electron beam (0°)

- Proper imaging setting, magnification: sufficient (>1500 X)
- Focus an area of interest
- Link the stage
- Go to 7 mm WD (at 0 degree)
- Set eucentric (CTRL+F)

1. Rotate the stage 180 degrees:

The screenshot shows a 'Stage' control panel with the following fields and controls:

- Actual** dropdown menu
- Go To** button
- X**: -0.4153 mm
- Y**: 2.9500 mm
- Z**: +↓ 6.9994 mm
- T**: 0.0 °
- R**: 180.0 °
- Compucentric Rotation
- Z-Y Link

Each coordinate field has a corresponding 'Go To' button with a double-headed arrow icon.

2. Insert the STEM detector

Doublecheck in the chamber view if the path below the stage is free.

Doublecheck you are in focus, at 7mm

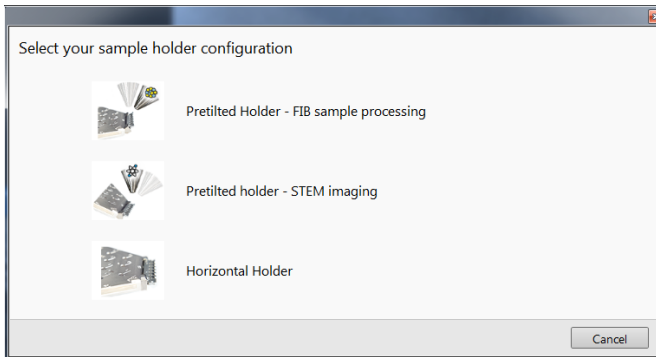
Experiment: insert the STEM detector

In the menu Detectors:

- Select the STEM3 detector from the dropdown list
- Click 'Insert'

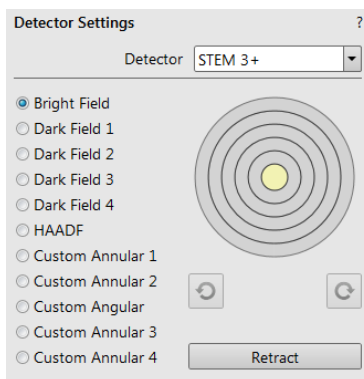
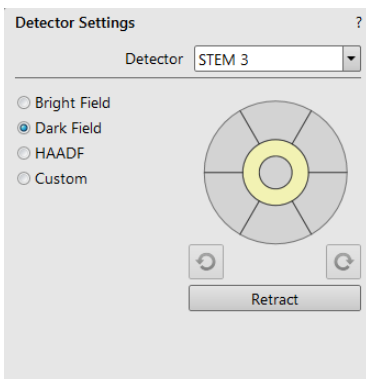


A window will appear:



Select the relevant option, i.e. the situation which describes the stage and the rowbar.

STEM 3 and STE3M+ allow the selection of different parts of the detector to be active.


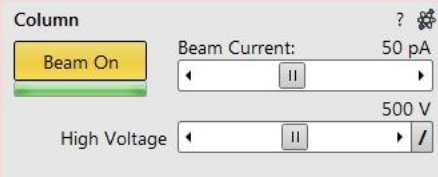


Demonstration: Finishing your session**Prerequisites:**

Data recorded

Concepts of shutdown and standby*As a rule of thumb***SHUTDOWN:** when FIB is not in use for 2 nights or more (36h)**STANDBY:** when the FIB will be used again within 36h**Experiment: Standby****1. Electron beam**


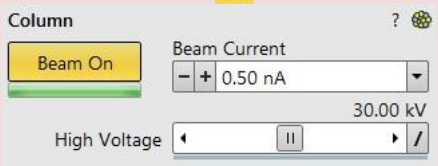
Select the electron beam quadrant (top left)

In the Beam control  > Column. Click the yellow button “Beam On”

← This icon symbolizes electrons

2. Ion beam

Select the ion beam quadrant (top right)

In the Beam control  > Column. Click the yellow button “Beam On”

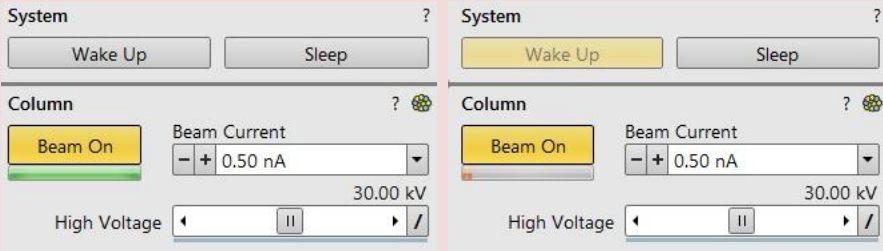
← This icon symbolizes ions

Experiment: Shutdown

1. Electron beam and ion beam: same procedure as standby (see above)

2. Sleep

Click the sleep button in the System menu, just above the Column window.



The green bar below the Beam On button of the ion beam turns yellow, then red and reduces in size.

Notes:



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