



**adolphe merkle institute**  
excellence in pure and applied nanoscience

UNIVERSITY  
OF FRIBOURG  
SWITZERLAND

## Focused ion beam

### Introduction

Version 10 – May 2025

PART II – BASIC 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: Login onto the FIB software****Prerequisites:**

Running xT server

Running UI

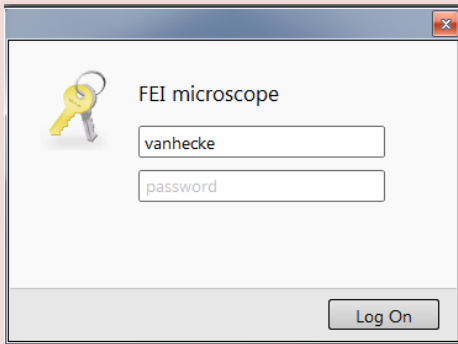
**Action:**

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 Log On

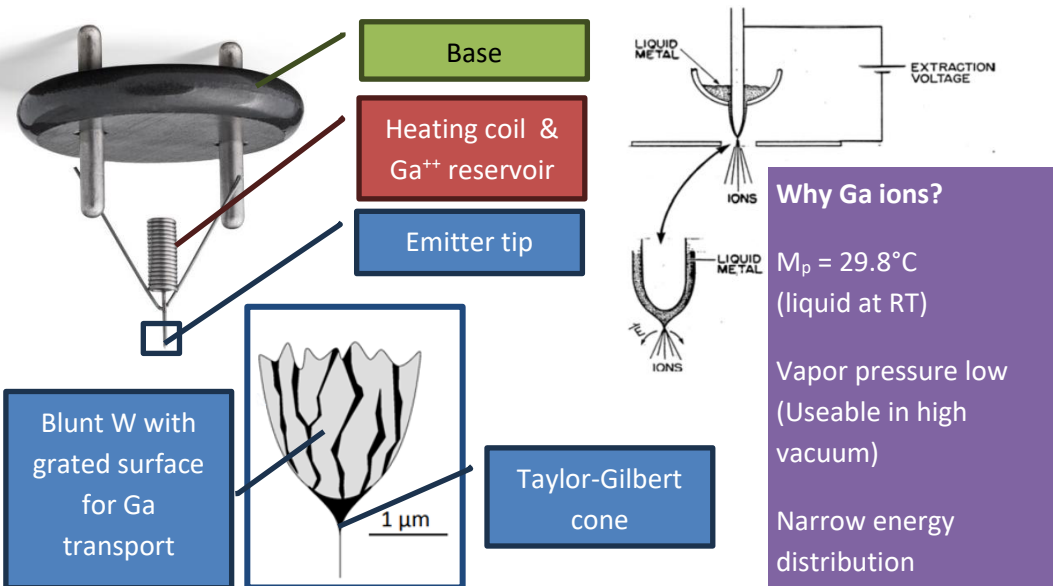
## Demonstration: The LMIS (liquid metal ion source)

### Prerequisites:

Running xT server

Running UI

### Background on the ion beam



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

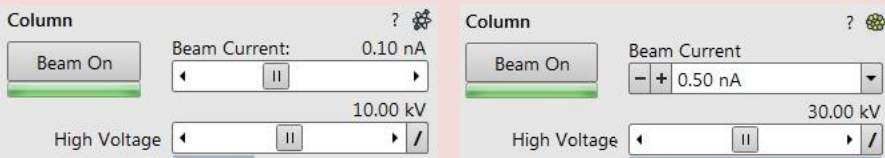
If the system is in standby, you will find a **green** bar under the “Beam On”

### 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**.

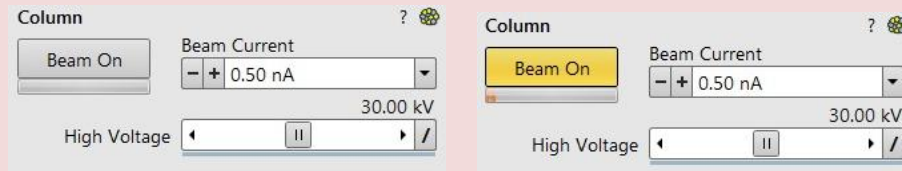
Click both Beam On to start the FIB

If the system is in sleep mode, you will find a **grey** bar under the icon “Beam On”

### Experiment

Electron beam: same as standby

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



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

It will take about 15 minutes to start up the ion beam.



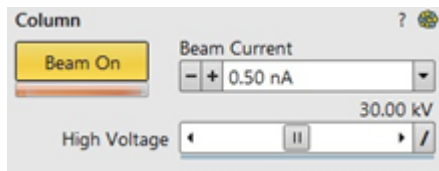
**Demonstration: an empty LMIS?****Prerequisites:**

Sample loaded

**Learn how to recognise an empty LMIS**

There is no sensor for how full the Ga reservoir still is. There is no warning when it is empty. When the LMIS needs to be replaced, the following will happen:

- During heating up of the LMIS, the system remains in the orange phase, never turns green (after waiting 30 minutes or more)
- When you hover the mouse over the orange bar, you will get the lifetime of the current LMIS. An LMIS has a lifetime of 1500 to 2000 hours<sup>1</sup>



If this situation occurs, inform the admin and cancel your session. Generally, an LMIS exchange needs about 4-10 days for exchange (depending on the availability of technical engineers).

<sup>1</sup> With notable ups (2700 hours) and downs (800 hours)

## Demonstration: e-beam Pt deposition

### Prerequisites:

Sample loaded

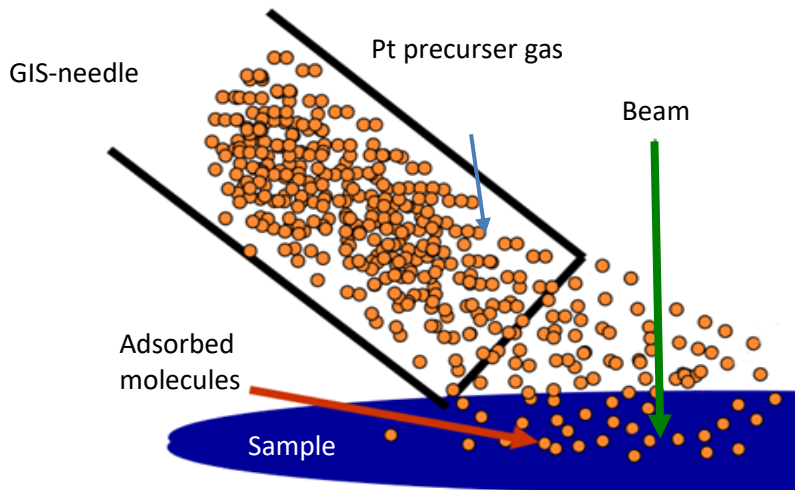
Electron beam on

**Eucentric height, focused, WD=7mm**

Learn to deposit Pt using the e-beam

Pt deposition using the e-beam marks is 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. If not, you risk damaging the system!!**



**Note: Pt deposition is usually done with the ion beam, but can also be performed with the electron beam**

**Experiment**

Make sure your sample is at eucentric height, focused and the WD=7 mm. Hit CTRL+f Under Patterning > chose a rectangle. Set e.g. 5  $\mu\text{m}$  x 5  $\mu\text{m}$ , deposit using the Pt application (Pt e-dep surface). The rectangle has a green border


**Patterning settings**

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

**Microscope Settings** (sample depending:

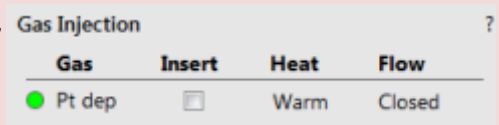
- Case: Standard mode
- Magnification: 2000 X or higher
- Acceleration voltage: 5 KV
- Beam current: set until time is 3-5 minutes.

**Insert the GIS needle**

In the patterning tab () , near the bottom, Find the Gas injection menu<sup>2</sup>.

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

<sup>2</sup> 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 using 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 a single image only (no continuous scans)**

## 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  $\mu$ 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.

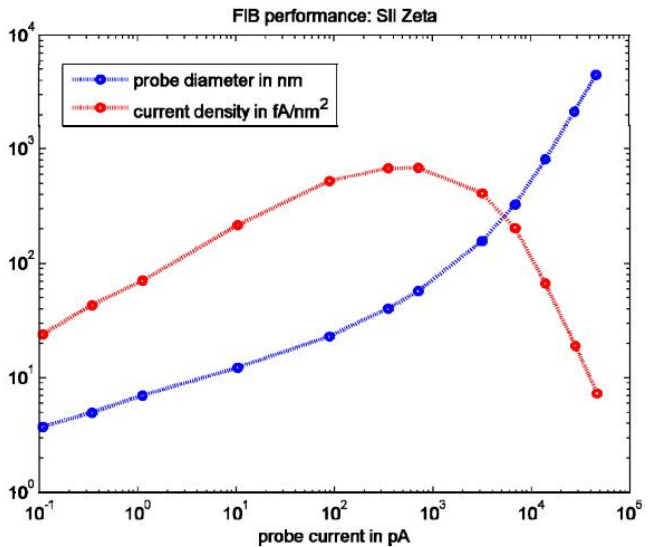
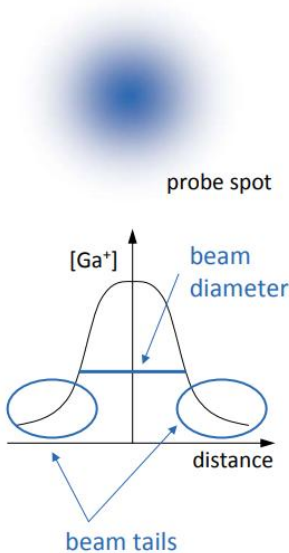
If the same object is not in the middle (use the center cross):

- 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 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**

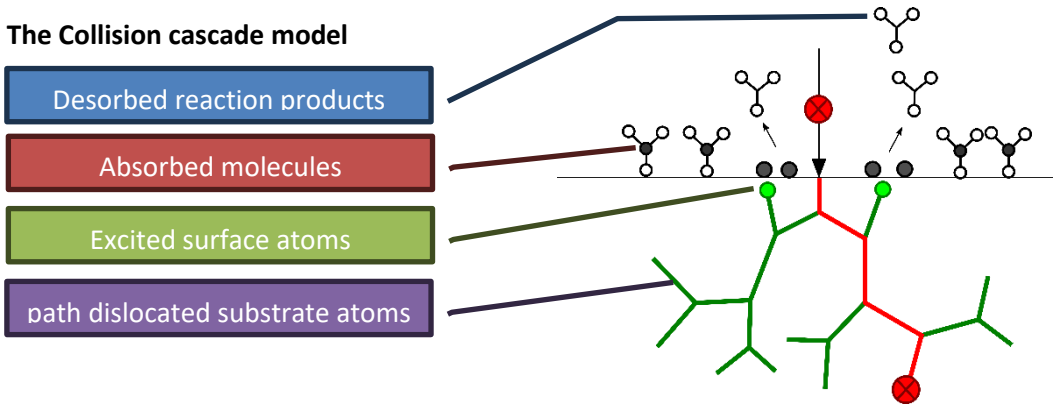
## Demonstration: Pt deposition with the ion beam

### 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  $\mu\text{m}$
- 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:

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

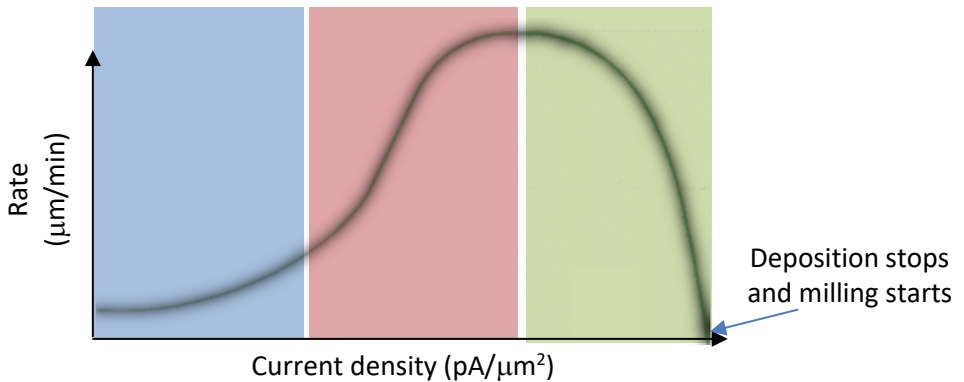
Where X = width of the pattern in  $\mu\text{m}$ , Y = height of the pattern in  $\mu\text{m}$ .

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 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 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
- Excess gas may affect vacuum

#### High-efficiency growth rate

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

**$2\text{-}6\ \text{pA}/\mu\text{m}^2$**

#### Milling effects

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



## Demonstration: Patterning types

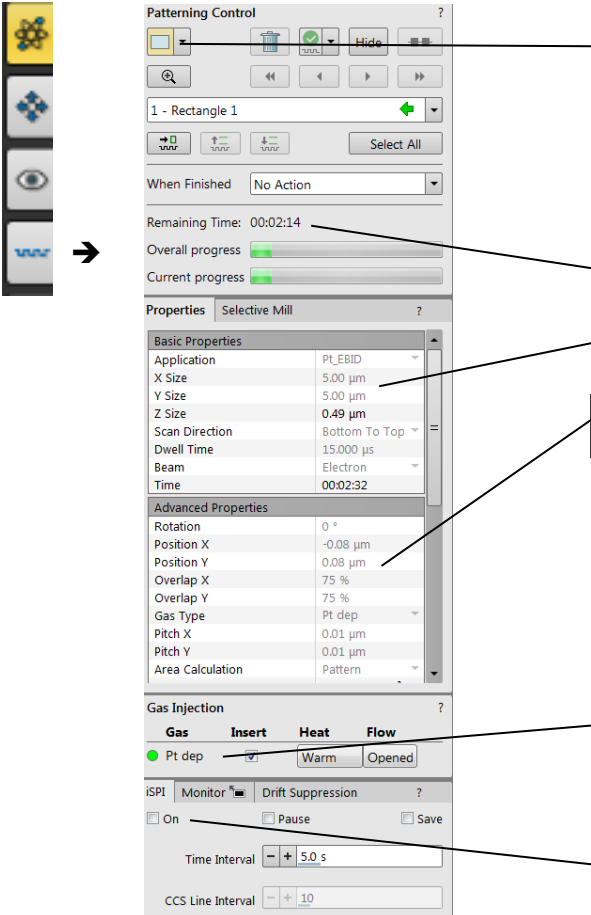
### Prerequisites:

Sample loaded

Electron beam on

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

## Information of the different patterning types



The screenshot shows the Patterning Control software interface. On the left, a vertical toolbar contains icons for various patterning functions. The main window is titled 'Patterning Control' and includes a toolbar with icons for pattern selection, zoom, and navigation. Below the toolbar, a dropdown menu shows '1 - Rectangle 1'. A 'When Finished' dropdown is set to 'No Action'. A 'Remaining Time' field shows '00:02:14'. Two progress bars, 'Overall progress' and 'Current progress', are shown. The 'Properties' tab is active, displaying 'Selective Mill' settings. The 'Basic Properties' section includes fields for 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), and Time (00:02:32). The 'Advanced Properties' section includes fields for 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), and Area Calculation (Pattern). The 'Gas Injection' section has tabs for Gas, Insert, Heat, and Flow. The 'Gas' tab is active, showing 'Pt dep' selected and 'Warm' and 'Opened' buttons. The 'ISPI' section has a 'Monitor' checkbox and a 'Drift Suppression' section with 'On', 'Pause', and 'Save' checkboxes. A 'Time Interval' field is set to '5.0 s'. A 'CCS Line Interval' field is set to '10'.

Annotations point to the following elements:

- Pattern type selector (see Toolbar icons also): Rectangle / Line / Circle / Cleaning Cross Section / Regular Cross Section / Polygon / Bitmap / Stream File
- Time indicator
- Pattern size settings
- Array settings, passes, ...
- Pt Deposition
- iSPI: imaging during deposition

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## Pattern types

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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
  - 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:

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## Serial vs parallel patterning

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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 the 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	?
<b>Basic Properties</b>			
Application	none		
Outer Diameter	91.15 $\mu\text{m}$		
Inner Diameter	0 $\mu\text{m}$		
Z Size	1.00 $\mu\text{m}$		
Scan Direction	Inner To Outer		
Dwell Time	1.000 $\mu\text{s}$		
Beam	Electron		
Time	316.7 ms		
<b>Advanced Properties</b>			
Position X	279.95 $\mu\text{m}$		
Position Y	386.72 $\mu\text{m}$		
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/ $\mu\text{m}^2$		
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 $\mu\text{m}^2$		
Scan Type	Circular		
Fill Style	Solid		
Passes	82		
Defocus	0 $\mu\text{m}$		
Blur	0 $\mu\text{m}$		
Interaction Diameter	0 $\mu\text{m}$		
Total Diameter	3.0 nm		
Maximum Dose per Area	0 nC/ $\mu\text{m}^2$		
Saturation Current Density	1.000E-18 A/ $\text{nm}^2$		
Total Volume Sputter Rate	2.700E+7 $\text{nm}^3/\text{s}$		
Selective Milling Enabled	<input type="checkbox"/>		
Selective Milling Time Interval	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 to the Overlap X/Y. The Pattern (default) / Array is 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 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

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)

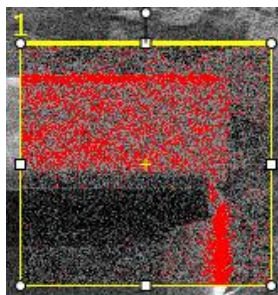
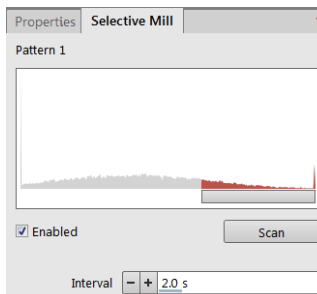
### Selective Milling Enabled / Selective Milling Time Interval / Min Contrast Threshold / Max Contrast Threshold items

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.

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### Selective milling

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

**Demonstration: Basic milling concepts****Prerequisites:**

Sample loaded

Electron beam on

Eucentric height, focused, WD=7mm

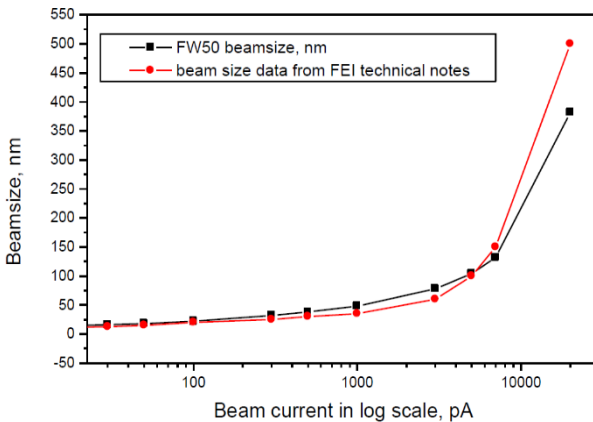
Beam coincidence point set

**Milling practicalities**

- Ions version electrons

		<b>FIB</b>	<b>SEM</b>	<b>Ratio</b>
Particle	type	Ga <sup>+</sup> 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	size	nm range	nm range	
	energy	up to 30 kV	up to 30 kV	
	current	pA to nA range	pA to uA range	
Penetration depth	In polymer at 30 kV	60 nm	12000 nm	
	In polymer at 2 kV	12 nm	100 nm	
	In iron at 30 kV	20 nm	1800 nm	
	In iron at 2 kV	4 nm	25 nm	
Average electrons	secondary electrons	100 - 200	50 - 75	
signal per 100				
particles at 20 kV	back scattered electron	0	30 - 50	
	substrate atom	500	0	
	secondary ion	30	0	
	x-ray	0	0.7	

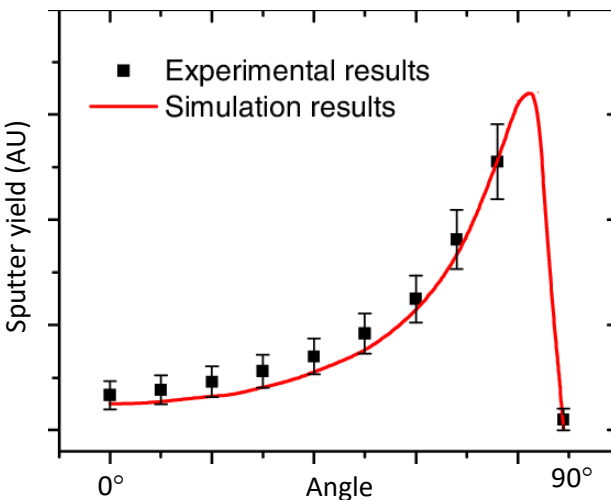
- Ion beam morphology



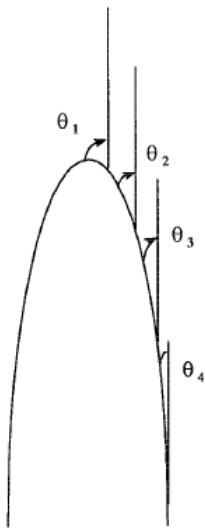
The higher the ion current, the wider, larger the ion beam.

Index	Beam current	Aperture diameter (um)	Beam diameter
0	1.5	0.008	7.0
1	10	0.016	13.0
2	30	0.025	17.0
3	50	0.032	19.0
4	<b>100</b>	0.042	24.0
5	300	0.068	31.0
6	500	0.087	35.0
7	<b>1000</b>	0.118	44.0
8	3000	0.198	66.0
9	5000	0.250	85.0
10	7000	0.294	102.0
11	15000	0.420	182.0
12	30000	0.600	210.0
13	50000	0.750	300.0
14	maximum	1000.000	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.



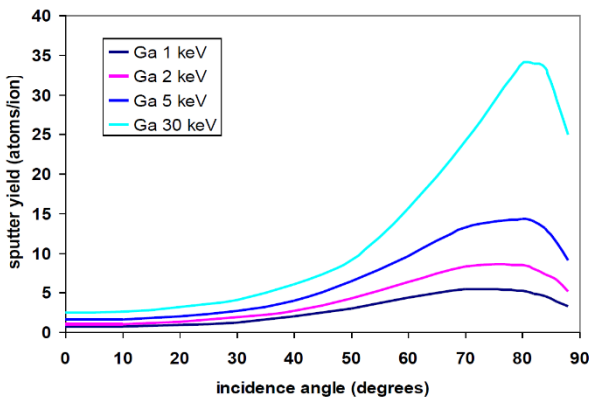
$$\theta_1 \neq \theta_2 \neq \theta_3 \neq \theta_4$$

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 top-down, but rather, are created by FIB milling at high incident angles.

See also page 29, Curtaining effect.

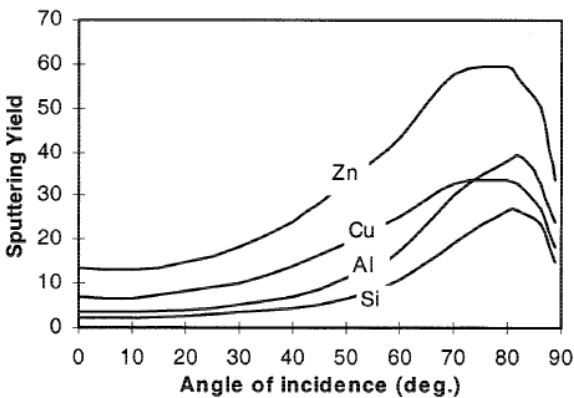
- Sputter yield depends on ion acceleration voltage



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 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
$\text{Al}_2\text{O}_3$	0.08
GaAs	0.61
InP	1.20
Au	1.50
TiN	0.15
$\text{Si}_3\text{N}_4$	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
$\text{Fe}_2\text{O}_3$	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

- Preparation

The settings used here are generic and should be seen as a starting point for developing the settings needed for your specific application and/or sample.

## Experiment: preparation

*In the electron beam (0°)*

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

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

## (Optional) Experiment: Protect the ROI using e-beam Pt deposition

- Insert the GIS needle
  - \* If 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 an application, choose Pt\_ebid (electron beam induced deposition). The rectangle should be green

- Microscope settings (will obviously vary depending on the sample)
  - \* Standard mode
  - \* 2000 X (or higher)
  - \* 2 KV
  - \* 1.6 nA beam current (to start with, see below)
- Patterning settings:
  - \* set X, Y: 20µm x 2µm
  - \* Z = 500 nm
  - \* Set the current to get a estimate time of 5-7 minutes
- Click start patterning

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

- retract the GIS needle. Hit F9. remove the pattern.

**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: set up the 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)

Align the e-beam to the ion beam precisely.

- Protecting your object of interest

#### **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!
- Draw a rectangle in the Ga ion image with the patterning tool (e.g. 20µm x 2µm)
- z= about 1 µm

- select Pt dep (not Si) in the application
- calculate the Ga current required using the magic number 6 (pA/μm<sup>2</sup>).

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

- \* e.g. 10 μm x 4 μm x 6 = 240 pA
- \* Use this value and chose the closest current for the Ga beam

**too much current and you will mill instead of deposit**


**Too less current will destroy your vacuum**

**6 is the magic number!**

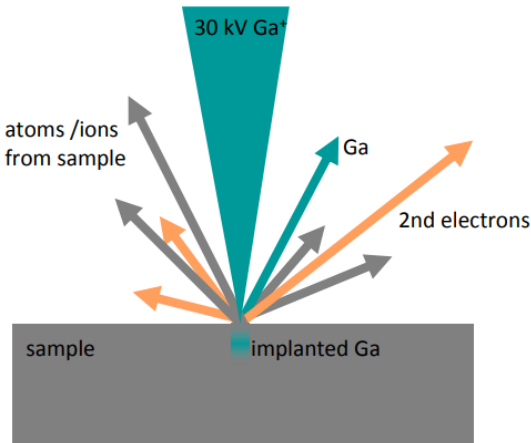
- You should get a time round 3-5 minutes
- Insert the Pt GIS
- Press F9 in the ion image (this will contrast/brightness correct and take a snapshot).  
Make sure you have the ETD selected
- Check the position of the rectangle, overlay the e-beam deposited marker.
- Run the deposition
- retract the GIS needle

- Milling

### Experiment: Bulk mill

- Use the **regular cross-section (RCS)** pattern.
- Position it just below the Pt deposition you just made, and a bit wider than the Pt deposition pad (about 10%), exactly touching the Pt above it. 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.
- 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.



Ga<sup>+</sup> beam hits substrate and **yields**:

- Secondary electrons
- Sputtered atoms and ions
- Implantation of Ga<sup>3</sup>
- Amorphisation/recrystallisation

3 FIBing **modes** happen simultaneously:

- Milling
- Deposition
- imaging

- 
- Curtaining effect
- 

The curtaining effect in FIB milling refers to the formation of vertical streaks or “curtains” on the milled surface. This occurs due to variations in the milling rates of different materials (e.g. porous inclusions) or milling angles within the sample (see page 23). When a sample contains

materials with different sputtering yields, the ion beam removes material at different rates, leading to uneven surfaces. The polishing step reduces the curtaining effect.




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<sup>3</sup> The depth of the damage layer depends on the energy of the Ga<sup>+</sup>

- Polishing

### Which current to use?

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 5 nA, 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.

### Experiment: advanced polishing with beam shape correction

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

### **Undertilt**

- Change the stage tilt angle for a correction factor between 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: 1/4 of the previous setting.
- run the patterning

### **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!

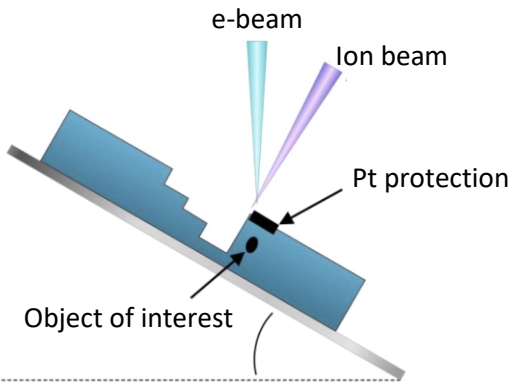
- You can use the iSPY: this will temporarily stop the patterning, make a SEM image and continue

## 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

Now image the result of your cross-section in 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

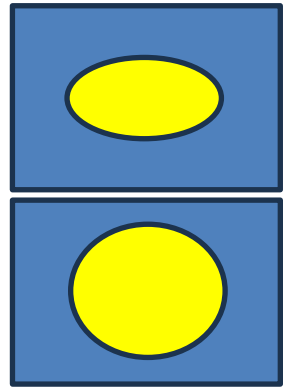
- Go to a very low ion beam current (10 pA)
- Switch to OptiTilt and use T1 and T2.
- Press F9
- Curtaining issues: Do not use the ETD, since curtaining is the strongest in that detector. Use T1.
- Lower beam currents: more focused Beam, but more curtaining.



## Tilt angle correction

At an angle of  $52^\circ$ , 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  $52^\circ$ , bottom: original at  $0^\circ$ ):

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  $52^\circ$  tilt angle (typical cross-section angle)

#### Tilt Angle: Manual

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

#### Dynamic focus

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

#### Tilt Correction

☐ Dynamic Focus

☐ Tilt Correction

Tilt Angle Manual

Manual - + 0.0 °

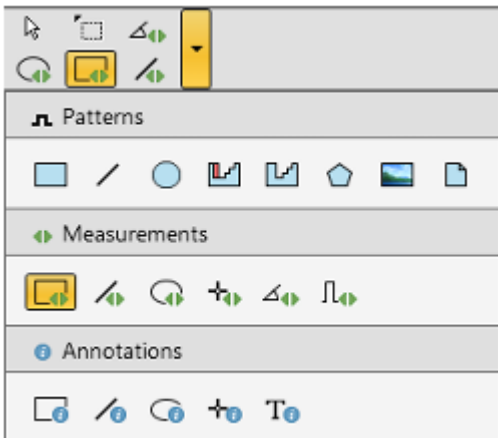
Specimen Pre-tilt - + 0 °

## Demonstration: Measuring and making annotations

### Prerequisites:

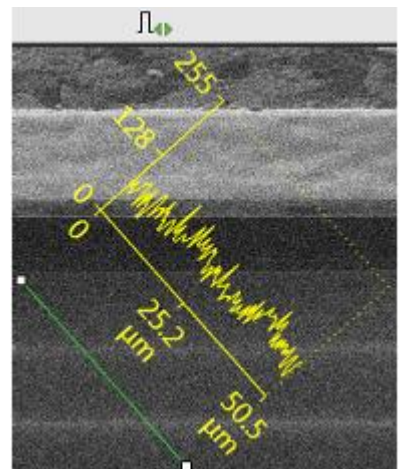
Eucentric height, beam focused, 7mm working distance  
Coincidence point set  
Ion beam and e-beam aligned

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.

## Demonstration: STEM imaging Demonstration

### Prerequisites:

- Eucentric height, beam focused, 7mm working distance
- Coincidence point set
- Stage tilt: 52°
- Ion beam and e-beam aligned

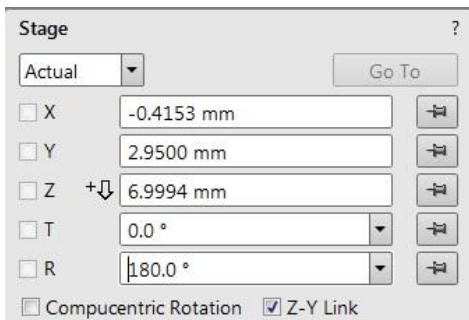
## Use the STEM detector

## Experiment: Link the stage (CRUCIAL)

### *In the electron beam (0°)*

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

### 1. Rotate the stage 180 degrees:



Stage ?

Actual Go To

☐ X -0.4153 mm

☐ Y 2.9500 mm

☐ Z +↓ 6.9994 mm

☐ T 0.0 °

☐ R 180.0 °

☐ Compucentric Rotation ☒ Z-Y Link

## 2. Insert the STEM detector

Double-check in the chamber view if the path below the stage is free.

Double-check you are in focus, at 7mm

### Experiment: insert the STEM detector

In the menu Detectors:

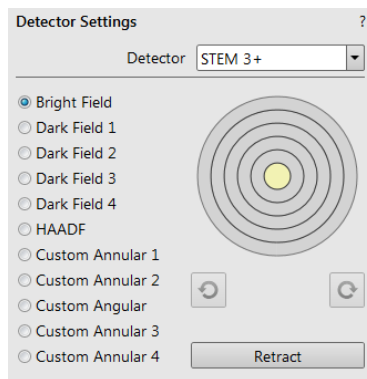
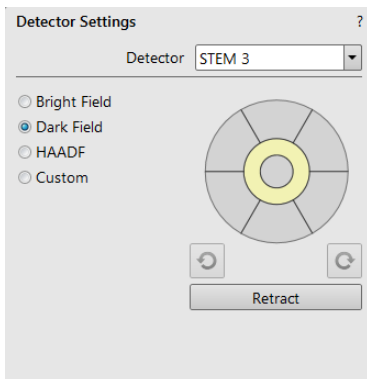
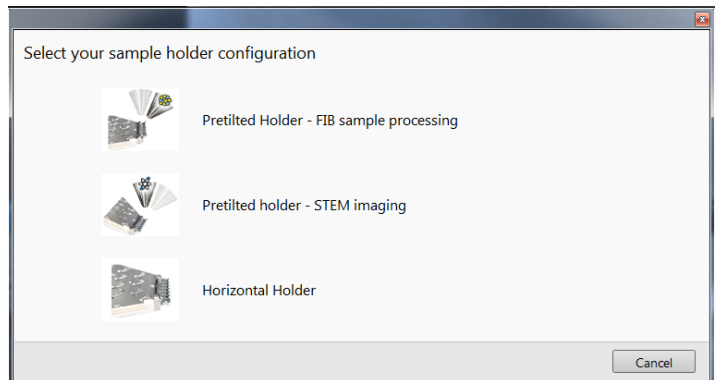
- Select the STEM3 detector from the dropdown list
- Click 'Insert'



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: Correlative microscopy (Maps 3.3)

### Prerequisites:

Sample loaded

Sample set at eucentric height

Electron beam on

### Start the Maps 3.3 software

Select Maps 3.3 from the top left FEI menu:



Or double-click the icon on the left screen:



The software will open and request to open an existing project or start a new project.

Note: each project's name must be unique. Make sure you save on the support PC!

### Experiment: import the nav-cam picture



on the top right, click this button to load the nav-cam picture from the xT software.

Note: the precision of this image is not very great. Expect mismatches of several mm.

### Experiment: create a tile-scan

**Step 1:** Set up a single image in the xT software (see above)

Calculate the resolution =  $\text{HFW} / \text{pixels along the x-axis}$

**Step 2:** Click the tile scan icon in Maps

There are 3 types of tile scans:



A square tile set

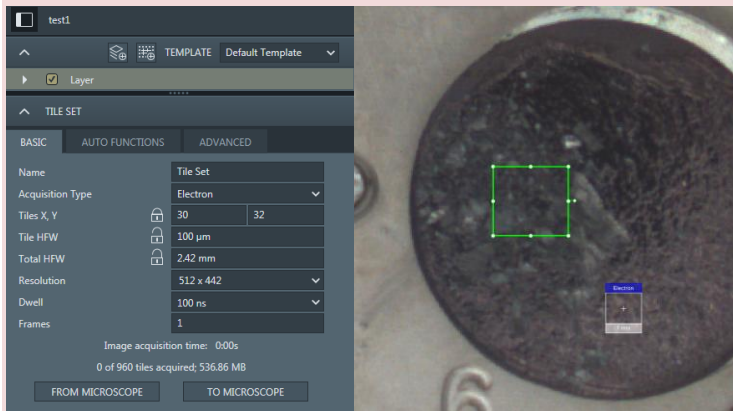


A freehand tile set

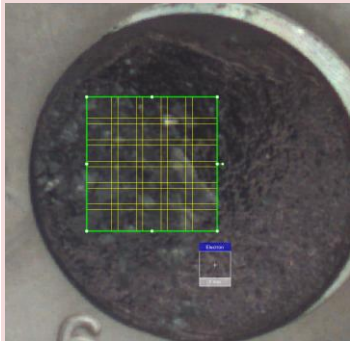


A circular tile set

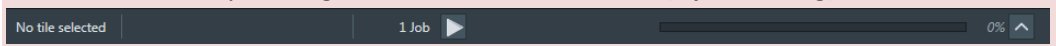
Use the appropriate pattern and draw on the region of interest. Then, from the left menu, click **'From microscope'**



This reads the settings of the microscope and calculates the number of tiles that are required. The image is updated:



Run the tile scan by clicking the run icon at the bottom (1 job waiting):



## Demonstration: saving data in Maps 3.3

### Prerequisites:

Sample loaded  
Sample at eucentric height  
Electron beam on  
Maps software started

## Saving images

After a tile scan was recorded, it can be saved.

### Experiment: save tile scan

Click the 'Save image to file button'



Select the region you want to export



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