Focused ion beam

Introduction

Version 3 – April 2022

IN SITU LAMELLAE / AUTO TEM

D Vanhecke | Adolphe Merkle Institute | University of Fribourg | Switzerland

Notes

- Lamella from beam sensitive / soft samples

- Lower the beam current (500pA and below) -> less melting -> more efficient milling
- Lower ion dose
- Use negative OL -150% and faster DT
- Don’t use e-beam while patterning
- Sputter coat the sample
- Total process will take longer; use autoTEM4 to prepare a chunk

NOTE: Temperature increase during milling; conductive material a few degrees (<10K), non-conductive material (low thermal conductivity) high temperature increases were measured (SiO_2 ± 230K)

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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

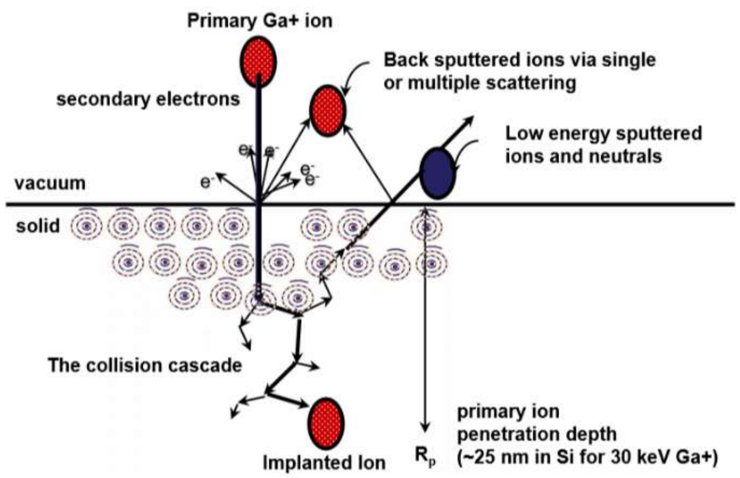
Experiment: Final thinning

- For e-beam end-pointing use < 30kV: typically, end with 5 kV in the ion beam (this is the only time you do not use 30 kV in the ion beam).

- At the same time, increase the tilt angles:
  - 5 kV, 48 pA, +/- 5°
  - 2 kV, 27 pA, +/- 7°

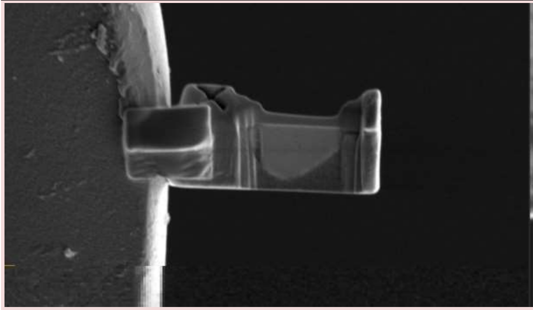
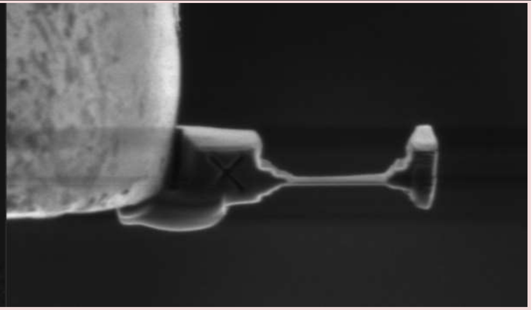
Low kV clean is always needed for crystalline material[3]

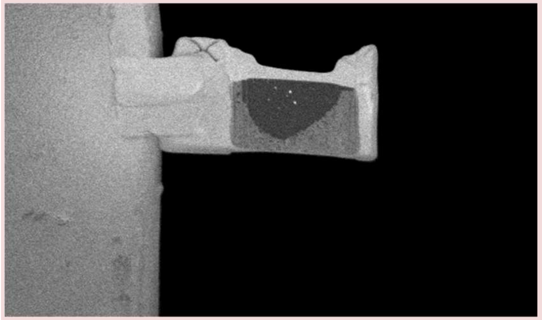
The Pt cover will melt away, but should never be entirely removed. If this tends to be the case: tilt to higher angles and or position your CCS farther away from the center of the lamella.



For good quality TEM lamellae the amorphous layers need to be minimized

[3] Milling @ 30kV: amorphous layer (in Si) = 23nm; @ 5kV: 6 nm; @ 2kV: 2 nm

 2kV transparency in STEM

Thickness: less than 300nm thick.

Step 3 (CCS, 100pA, 30 kV) Repeat at +/- 1.2° tilt

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Demonstration: Loading the halfmoon grids

Prerequisites:

Running xT server

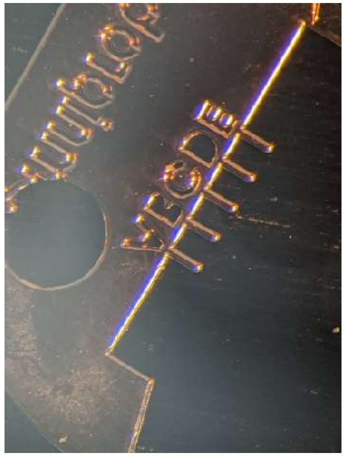
Running UI

Stage vented

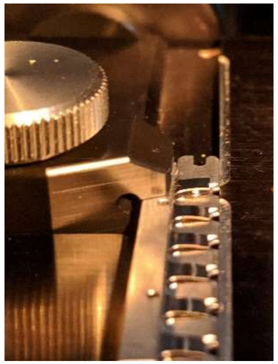
Action:

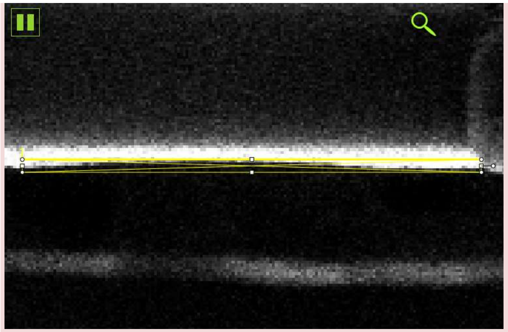
Loading the grids on a row bar

Halfmoon grids are the adapter between SEM and TEM





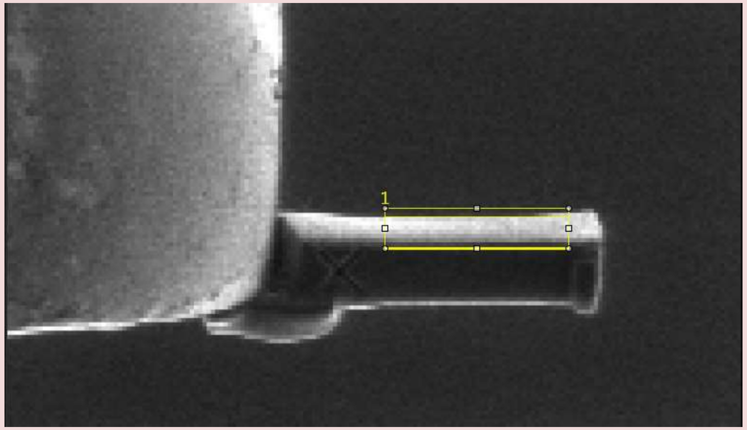





Basic Properties	
Application	Si-ccs
X Size	5.49 µm
Y Size	0.15 µm
Z Size	10.02 µm
Scan Direction	Bottom To Top
Dwell Time	1.000 µs
Beam	Ion
Time	00:00:40

Top

- Tilt to -2 degrees
- Place a Cleaning cross section on top of the chunk. Use otherwise similar settings as above.
- You are removing material on the other side of the chunk. This is the side you cannot see by iSPY in the electron beam.



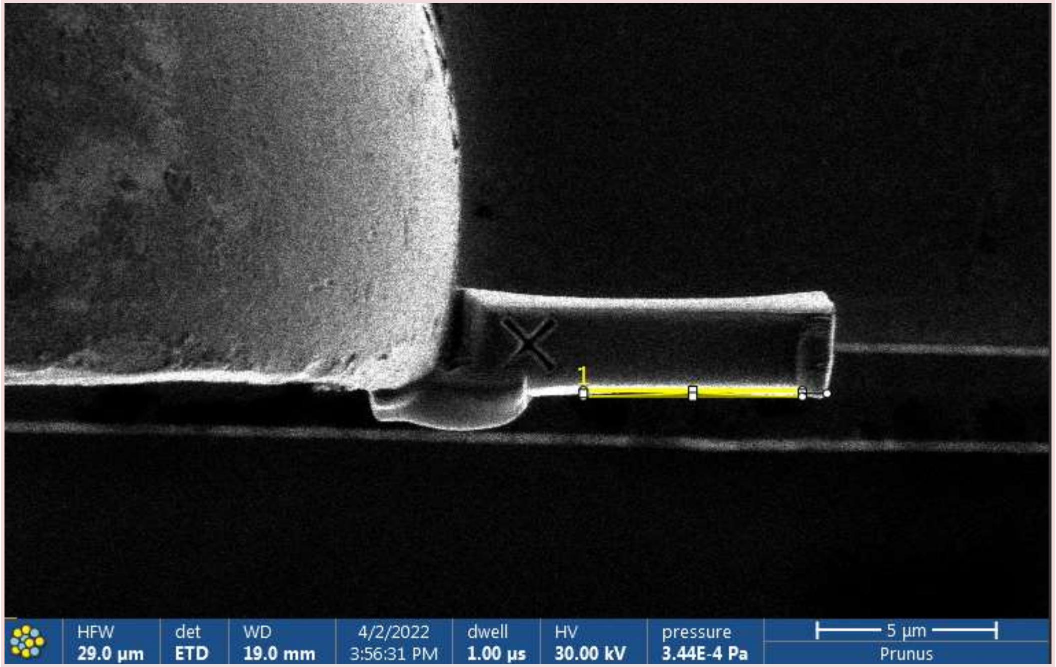
Iterations

Repeat the bottom/top steps above, thereby thinning the chunk. Increasingly make the pattern less wide. Also, decrease the tilt range (2, 1.5, 1.2 degrees) as you go.

Experiment Medium Thinning

Bottom

- Tilt to +2 degrees
- in the ion beam, place a RCS pattern at the bottom of the chunk. It should:
  - Start in the void, end in the chunk
  - Be wide, but not until the end or beginning of the chunk
  - Milling from bottom to top (thick yellow line on top)
  - Z should be sufficient to mill through the entire chunk height. Usually 5-10 µm
  - Current: below 0.5 nA, use 30 kV



The halfmoon grids have two sides:

- Matt side (=should be up). You can read the word “Omniprobe” and ABCDE.
- Shiny side (=should be down). ABCDE and the word “Omniprobe” are mirrored.

The row bars also have two sides:

- Bottom side: with 6 holes at each clamp
- Upper side: with flat clamps

Experiment: load a halfmoon grid in the row bar

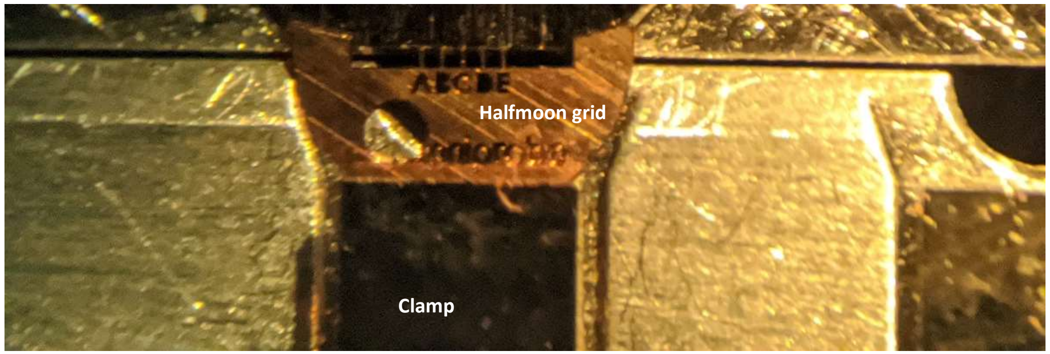
- Place 2-3 halfmoon grids on the stage, matt side up

- Place the row bar with one hole over the stage pin. The upper side should align with the edge of the front panel of the stage

- Place the stage screw over the row bar and tighten it. This will lift the clamp of the row bar.

- gently guide the halfmoon grid towards the position with the opened clamp. Make sure the shiny side is down (= You can read Omniprobe). Steer the round side of the grid under the clamp.

- Release the screw. The clamp releases and holds the grid. Move to the row bar to the next clamp if needed .



Demonstration: mounting the row bar

Prerequisites:

Running xT server
Running UI
Stage vented
Row bar with half moon grids

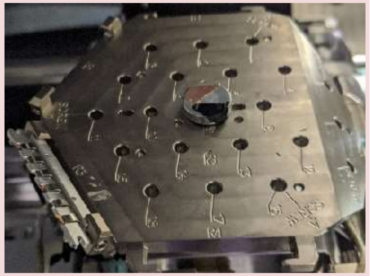
Action:

Mounting the row bar on the FIB SEM stage

The correct mounting of the row bar is critical

Put on gloves when handling the row bar.

Experiment



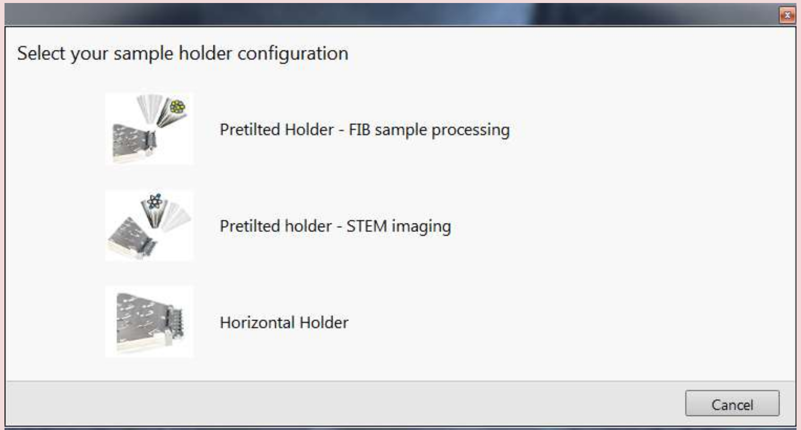
- Rotate the stage (using UI) 90°
- Open the stage and open the side screw of R3. This is the pre-tilted slot (to 38°).
- Place the row bar in R3 with the **clamps facing** up. Then gently tighten the screw.
- Load your sample. Place only one stub in the middle of the stage. The sample must be flat and < 2 cm high.
- Pump the stage and reset the stage rotation to 0°

The pre-tilted row bar will assure that the FIB-SEM beam is parallel to the sides of the chunk.

Large samples (> 1.5 cm tall) will damage the system and cannot be used! Only use ½ inch stubs!

Experiment: Setup the SEM detector

It is very difficult to measure the thickness of the lamella. The best chance is to observe it in STEM. Transparancy in STEM at 2-5 kV is going to be thin enough.
- Quadrant bottom left: STEM 3 detector using bright field (setup in detectors window)
- Start Optitilt mode. Insert the STEM detector, BF mode.
- Press Insert a window pops up



- Choose **Pretilted holder – FIB sample processing**
- In the bottom left quadrant: check if the detector was inserted

Use 30 KV SEM to start with. Adjust to lower KV as the lamella becomes transparent in the STEM image.

Demonstration: Thinning

Prerequisites:

Lamella welded to the half moon grid

Ion beam looking perpendicular onto the lamella

Action:

Reduce the thickness from 1-1.5 µm to 50-100 nm

Know your material!

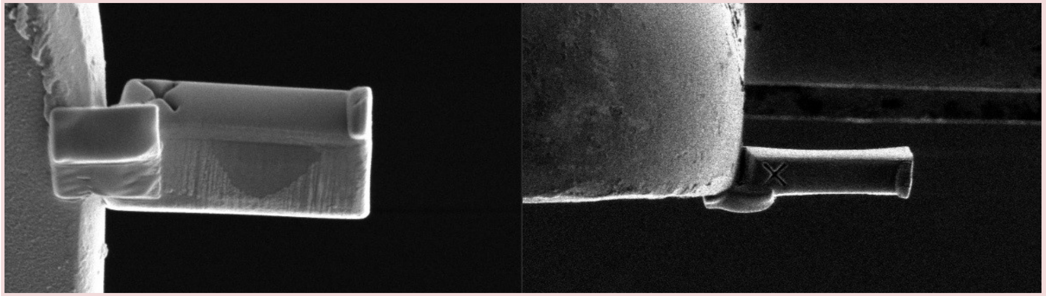
In all cases: use

- cleaning cross sections
- Tilt angle: 0 degrees (=38 degrees due to pretilt).
- Electron beam: 30 kV, ion beam 30 kV.

Note: the automated routines have never worked. Here, everything is manual.

Experiment: Move to thinning position

Tilt to 0 degrees and rotate the stage to -150 degrees. Now, you see the chunk under an angle (with the baby fiducial). In the ion beam, the flat side of the finger is down.



Demonstration: UI setup

Prerequisites:

Running xT server

Running UI

Stage vented

Row bar with half moon grids

Action:

Setup the software for autoTEM

Do not skip these steps!

To run a successful AutoTEM project it is important to:

Experiment: setup the system

- During pumping: home the stage

- When the vacuum allows, move the stage up, **focus and link**. Repeat until the sample (not the half moon grids) is at **7 mm eucentric height**. Set eucentric height (CTRL+f) and repeat the 7mm setting.

- Take a **nav-cam picture**. Reset all the **beam shifts**.

- Save the position of your region of interest on your sample.

Demonstration: Sharpen the easyLift tip

Prerequisites:

Stage pumped
Samples loaded and coincidence point set
Stage at 0° tilt
e- beam and Ga ion beam on

Action:

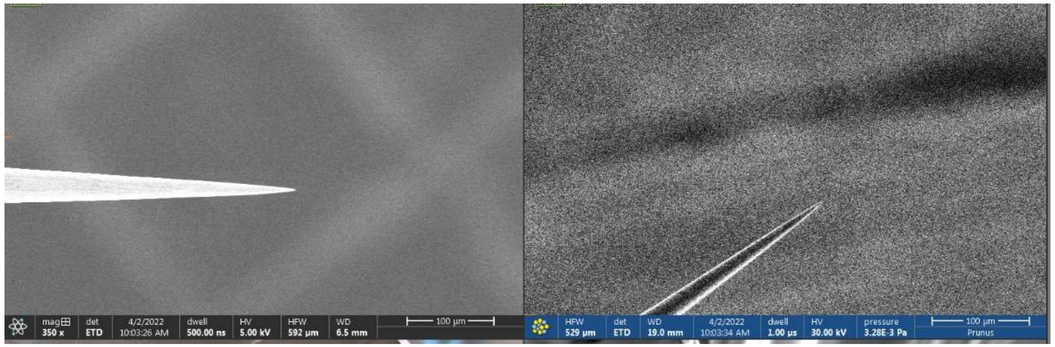
Create a proper easyLift tip

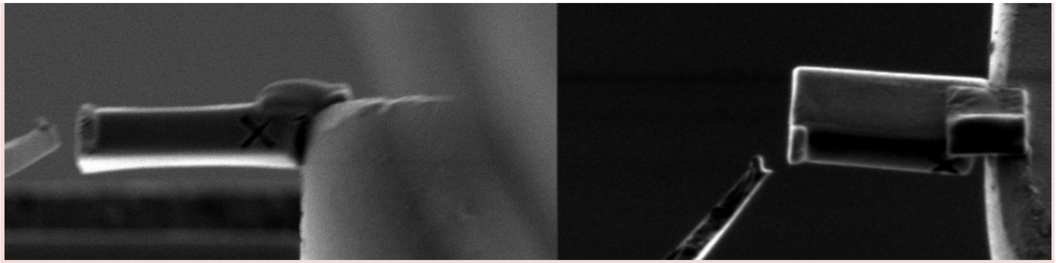
Part of the tip will not survive

Assure the stage is linked and LOWERED (15-20 mm)

Experiment: Image the EasyLift needle

- Make sure you are at 7mm, eucentric and in focus. **Lower the stage** to 20 mm.
- Find the EasyLift menu. Insert the easy lift needle by clicking insert (no parking position). Zoom out (200-300X) and image the needle (you may need to focus) using the electron beam.
- Image the needle with the Ga-ion beam (10 pA, 200X) and zoom in on the tip. Save position of the tip.





Usually, the procedure breaks down during one of the last steps and error appear. Just perform these steps manually.

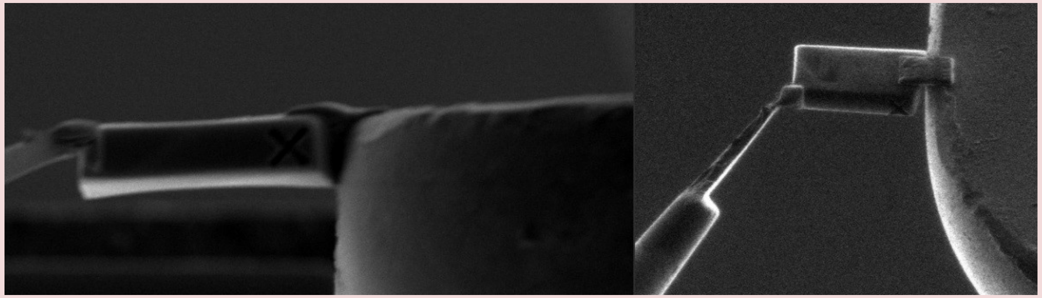
When finished: retract the EasyLift needle and the Pt deposition needle

This was the easy part 😊 Now come the more difficult steps.

The idea is the mill part of the Cu grid and use the redeposition of the Cu to attach the lamella. Usually, this works very well.

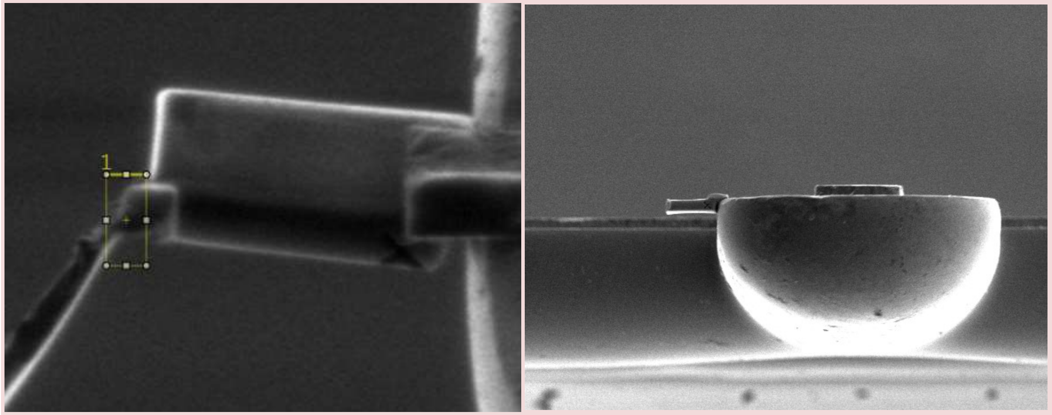
Weld chunk

- A bridge from Pt will be created between the chunk and the finger. Repeat if you consider the bridge not strong enough.



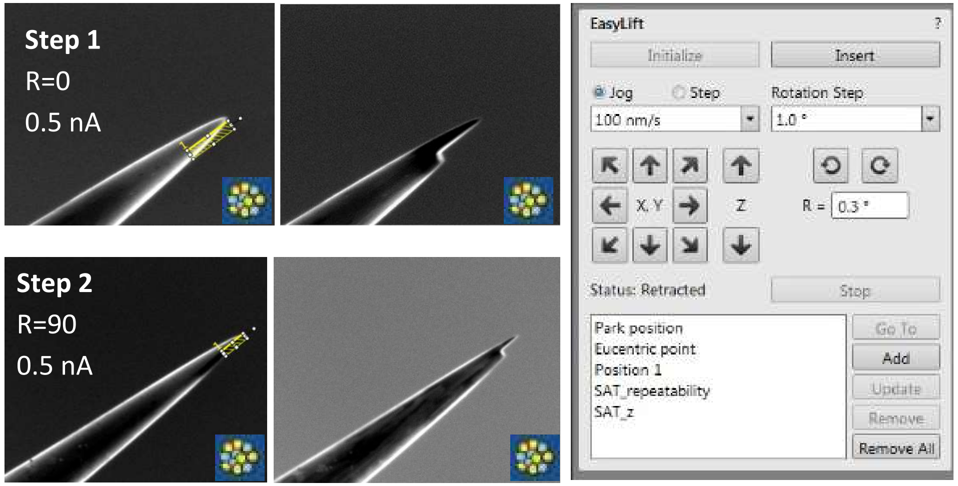
Cut off

The needle is cut off by a rectangle milling step. Make sure you remove all Pt. In this way, the needle is ready to pick up a next chunk.



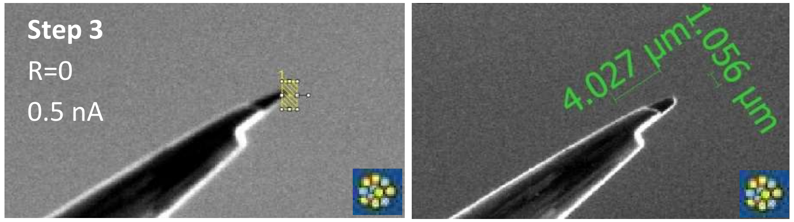
Experiment: sharpen the EasyLift tip

- Goal: to have a sharp tip of about **2 µm width and 2 µm** height.
- In the ion beam image, place a milling pattern at the bottom of the tip needle, perpendicular with the upper edge of the needle (see step 1 below).
    - Pattern: Standard rectangle
    - Z: 2-3 µm (depending on how far inside the needle you operate)
    - EasyLift: Rotation (R)=0 (or as close as possible)
    - Current: 0.5 – 1 nA (few minutes)
    - Magnification: 3000X-5000X
- Check if all material is removed. If not, repeat with the same pattern.
- Rotate the needle 90° (in the EasyLift window).
- Change the ion beam current to 10 pA. Lower the magnification
- Use the Step and Jog functions in the EasyLift window to position the tip in the middle of the ion beam image (you might need a few seconds of continuous imaging). Use the ion beam shift (hold shift, grab with left mouse) to optimize.
- Repeat the milling as described above.



Experiment: Create the flat dock

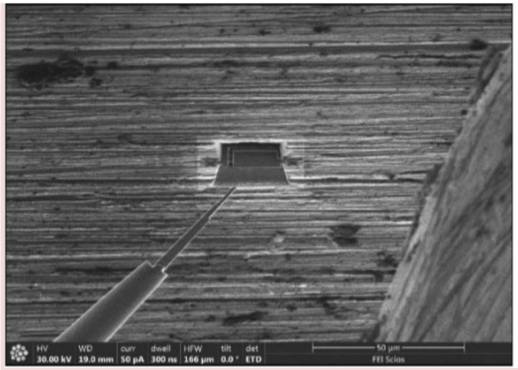
- Goal: To create a flat dock at the tip of the needle
- Rotate the EasyLift needle back to 0 degrees and center it in the ion beam image.
- Zoom in digitally onto the tip. Place a rectangular milling pattern perpendicular with the image axes.
    - Pattern: Standard rectangle
    - Z: 1-2 µm (depending on how far inside the needle you operate)
    - EasyLift: Rotation (R)=0 (or as close as possible)
    - Current: 0.5 – 1 nA (< 1 minute)
- **Retract the needle**

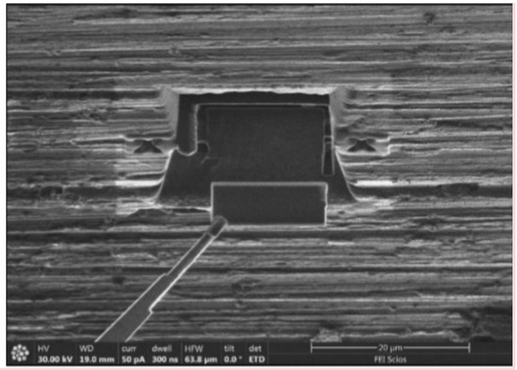


Goal: length of the tip: 2-4 µm. Width: around 2 µm, height: around 2 µm. If the tip is still longer than 2-3 µm, you only need to create the dock.

Experiment: get ready!

- retract the EasyLift needle
- Start e-beam imaging and set the stage back to 7mm
- Perform eucentric height and adjust to 7mm
- Perform beam coincidence alignment and ion-beam shift to image the same ROI between electron and ion beam

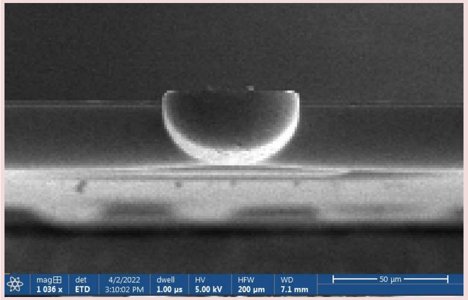


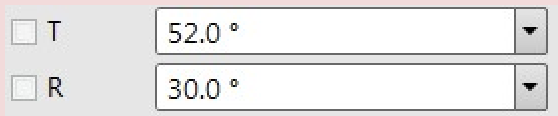


Drive to grid

- Place the green overlay over the top right corner of the chunk. Don't bother about the overlay of the EasyLift needle.

In the electron beam, rotate the stage to +30 degrees, and tilt to 52 degrees. In the electron beam, you should see the tip of the finger face-on





- Place the overlay in such a way, that is is just touching the left side of the finger (as in the wizard image). Do not place it at or on the finger, leave some space for error.

Weld chunk (GIS free)

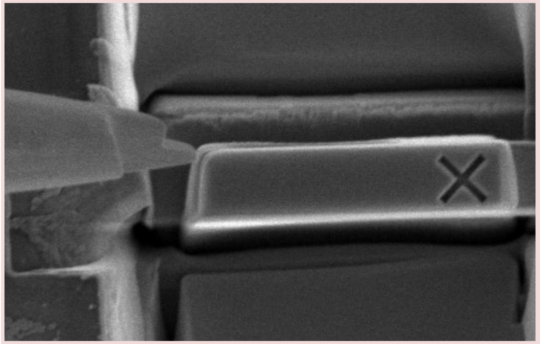
- Use 4 strips of 4x1 µm at about 1 nA.
- This step can be run only once: if it fails, do not repeat it.

Attach needle

The stage is moved back to eucentric/7mm. The EasyLift tip is inserted very close to the lamellae. Follow the wizard and align the tip as shown.

- Read carefully what is expected!

- Align the tip on the left side of the lamella. Do not use "jog" unless you are sure what you are doing!



- Carefully align the tip as shown in the steps. You want to align it to the upper part of the lamella and get as close as possible in Z.

Connect with Pt

- Follow the on-screen wizard and attach the needle with a Pt layer to the lamella.

Extract chunk

- Follow the on-screen wizard and cut off the neck of the chunk with a milling step. Afterwards, the Chunk, now connected to the needle, is retracted.

Demonstration: Start the AutoTEM software

Prerequisites:

Running xT server

Running UI

Sample and half moon grid loaded

Stage linked and focused

Action:

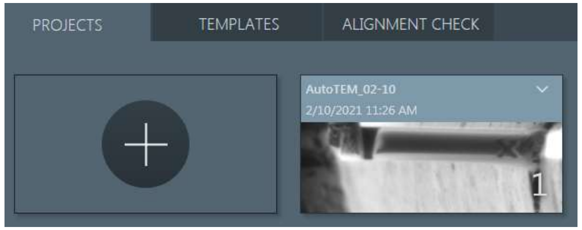
Starting the AutoTEM

AutoTEM is the software that automates the lamellae making

Using the FEI menu, select AutoTEM 4.1

AutoTEM 4 has 3 tabs at the startup:

Projects	Projects from the past are saved here
Templates	Allows to save templates and change settings.
Alignment	For technicians only



Note: the software can take some load of the procedure, but is not always very robust. Manual steps will be needed. However, it helps to structure your steps.

Demonstration: Creating a new project

Prerequisites:

AutoTEM 4 running

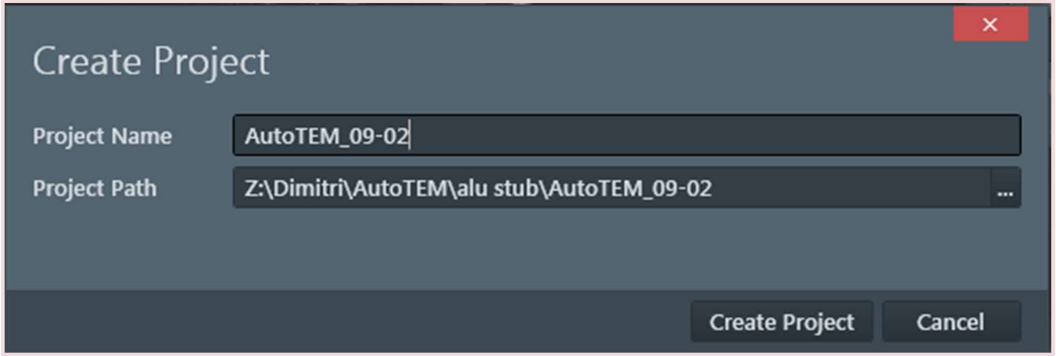
Action:

Creating a AutoTEM project

Make sure you save on SharedData

Experiment: create a new project

- Click the big + in the tab projects.



- In the project path: make sure you save your data on Z (the shared data folder).
- Click create project

You enter the project dashboard.

Prerequisites:

Chunk created

Half moon grid in row bar R3, matt side up

EasyLift needle sharpened

Action:

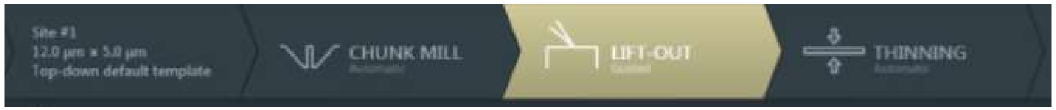
Take the chunk out and move connect it to the half moon grid

The tensest steps of the entire procedure

These steps are semi-automated: at the bottom right, a visual will appear to ask you to check if the desired situation is matched.

Usually, the automated routine will have an error at the start of this process.

Check your half moon grid finger: in ion beam imaging at 52 degrees, it should appear from top, you should see the bar on the upper side and at 0° stage tilting, you should look at the finger face on. Lower the stage to 10mm or more to allow space for the needle. The needle is visible in SEM and ion image at lower magnifications.



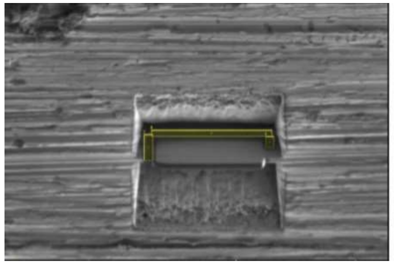
Experiment: connect EasyLift needle

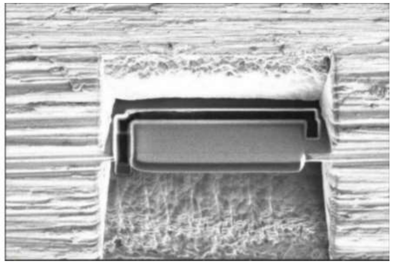
Drive to chunk

-The stage will lower a few mm and the EasyLift tip is going to be inserted.

- You will be presented a series of images (e-beam and ion-beam). Use the mouse to place the green overlay exactly on the tip of the needle.

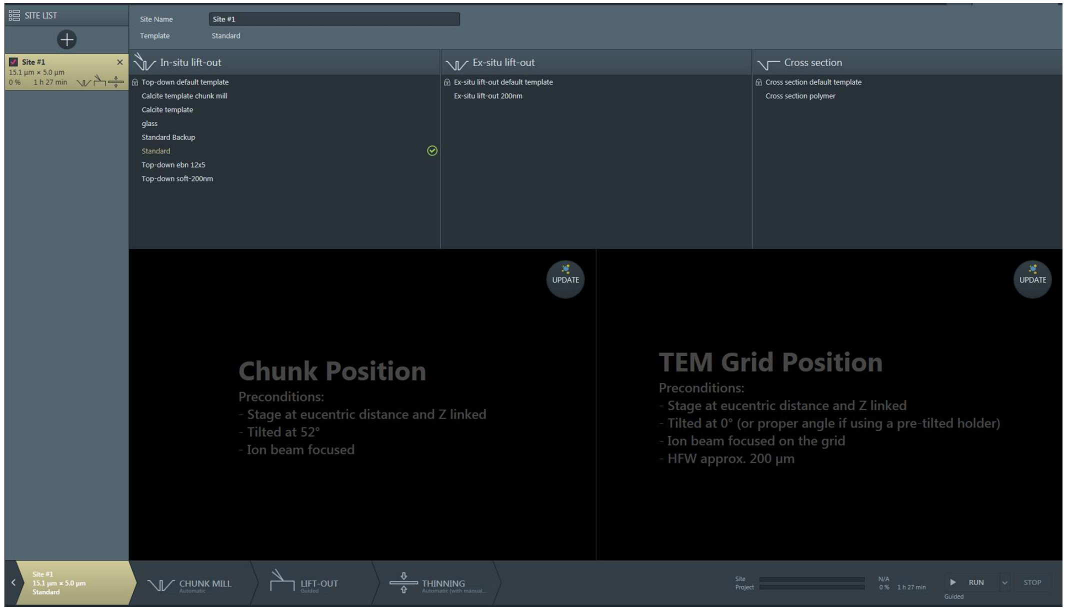
Milling settings:
- Choose parallel milling (right top patterning page) this avoids redeposition. Z=2 µm
- voids redeposition- z=2um (about the lamellae thickness)





Make absolutely sure the J cut has passed through the entire lamella and is only attached with the bridge

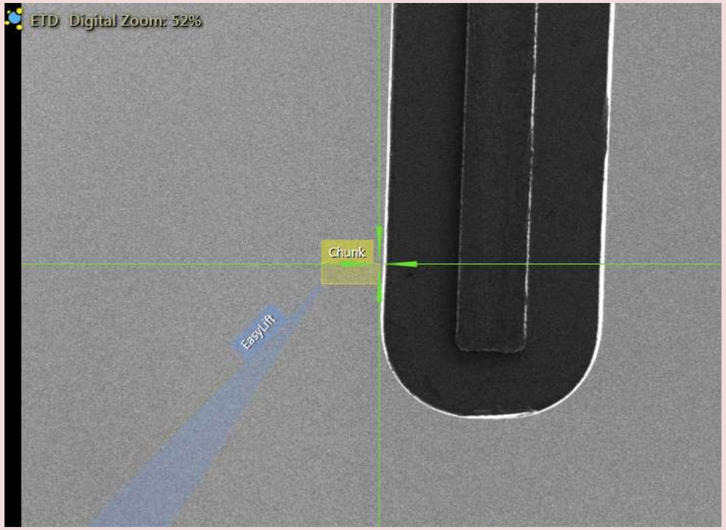
Demonstration: Lift out



Experiment: setup the project

Site list
You can batch create lamellae sites. Add more if needed. The sites will be created in series.
In-situ lift-out
This means the creating of lamellae. Here, you can select the relevant templates. (ex-situ lift-out and cross section are irrelevant)
TEM grid position
- **This part is absolutely critical**
- Place the AutoTEM on the right screen. Go back to the user interface (left screen).
- Tilt to 0 degrees
- Rotate the stage 30 degrees
- E-beam: Zoom out and goto the position of the finger of the half moon grid. Use the electron beam. The word "Omniprobe" and the letters ABCDE are upside down, but not mirrored.

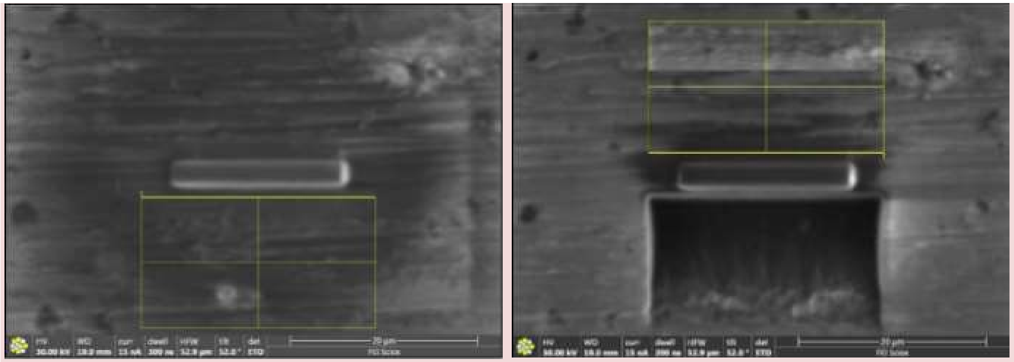
- Setup eucentric height for the half moon grid as it usually is not at the same height as the sample (7mm, in focus).
- In the SEM image, you see the finger top down (at 38 pretilt). The surface of the finger is flat, with a ledge in the middle. The ion beam looks perpendicular (90 degrees).
- Focus, Link and set 7mm for the tip of a finger.
- Perform beam coincidence between e-beam and ion beam (ion beam shift)
- Image the finger with the ion beam (10 pA). Set the magnification to a HFW of about 200 µm.
- In the TEM grid position, the imaging is done with the **Ga ion beam**. Click update.



The finger must come from top, be flat (not rounded) and have a ledge in the middle. The chunk must be placed on the left side of the finger.

Chunk position

Here, the position of the area of interest will be set. The chunk is a rough lamella.
- Move back to the region of interest of your sample
- Tilt to 52 degrees. Perform the beam coincidence procedure. Shift the ion beam to image the same object as the electron beam.
- Imaging is done with the **Ga ion beam** / ETD dectector



Medium milling

apply an extra tilt (in + and -) using the cleaning cross section.
- Z = ½ of the bulk milled depth
- Apply to the front and backside

The final lamella thickness: 1-1.5 µm

Needle trench

The needle trench is a small gap, roughly 5 x 5 µm on the left side of the Pt cover (about 1 µm away) to allow space for the EasyLift needle. About 1 um deep.

The overcut or J cut

This will disconnect almost the entire chunk, leaving it connected to the bulk with only a small bridge.

Cut-out: Tilt to 7 degrees (make sure your beam coincidence is set perfectly).
First rectangle: at the bottom (3/4 from top).
- Width of bottom cut at about 1 µm width.
- Do not put this rectangle too deep (at about ¾ from top). It defines the width of your final lamellae
Second rectangle: at the EasyLift side (usually left side)
- This side is to be cut out completely
Third rectangle: at right side
- Leave a part of the lamella attached to the bulk.

Demonstration: Chunk mill – manual procedure

Prerequisites:

AutoTEM 4 running

Project created and templates assigned

Action:

Manually milling a chunk

Try the multi site option!

Experiment: Manual procedure

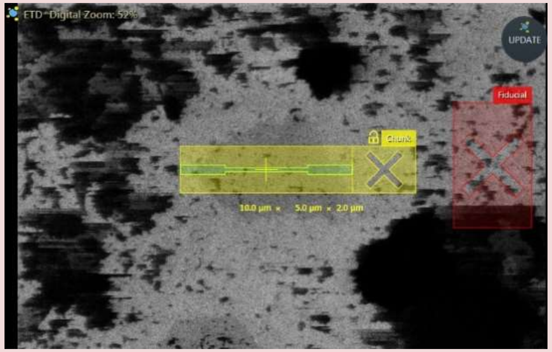
Preparation

- Find the area of interest
- Set coincidence point using e-beam + Z-adjustment (start with: zero beam shift and uncheck Z-Y link)
- Optional: e-beam deposition
- Tilt to 52° and cover the area of interest with Pt by Ga ion.

Rough milling

- Rough cut/bulk milling using Regular Cross Section + Si multipass:
    - Regular Cross Section. Size of pattern: X slightly wider as Pt layer width
    - Set Z to required depth , Y=2 times Z.
    - Choose an ion beam current according to size and material (usually 5-50nA).
    - Put the pattern not too close to the Pt layer, not overlapping with the Pt layer
    - Start to mill front side first.
- Repeat from the other (back) side
    - Rotate pattern 180°
    - NOTE: end point (where patterning stops = the thick yellow line) is always the Pt layer

- In the ion beam, press F9 to auto C/B. Then Click update in the AutoTEM chunk position window. An ion beam image appears.
- **You cannot set the magnification**! Magnification is calculated (and applied) based on the chunk size you want to use. Change the chunk size to change the magnification.
- Unlock the yellow lock sign on the top right of the yellow chunk overlay.
- Reposition and adjust the size of the chunk over your ROI.
- Place the red fiducial on the right.
- Leave space between the chunk And the fiducial
- In the UI: update the position of Your ROI.



Experiment

Ready to start.

Go back to your sample. Setup eucentric height, focus at 7 mm.

Demonstration: Chunk mill

Prerequisites:

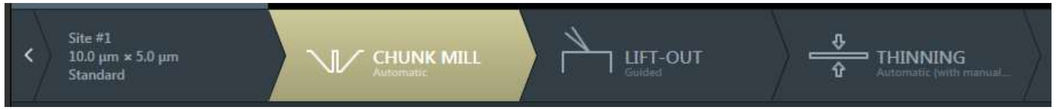
AutoTEM 4 running

Project created and templates assigned

Action:

Automatically digging a chunk

Try the multi site option!

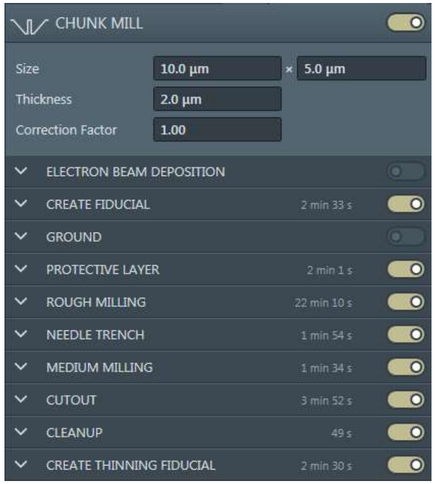


Click "chunk mill" at the bottom. This algorithm will automatically:

1	A fiducial marker is created	very high chance of success
2	Create a protective Pt layer over your region of interest/chunk (future lamellae)	Very high chance of success
3	Rough milling: create two trenches on both sides of the chunk.	High chance of success
4	Create needle trench: on the left side of the chunk, material is removed to facilitate the needle attachment	High chance of success
5	Medium milling: thinning of the trench at lower currents.	OK chance of success[1]
6	Cutout: creating a side and undercut. After this step, the chunk is only attached on the right side with a short bridge	OK chance of success[2]
7	Cleanup: overtilting the chunk to remove debris at the lower end of the chunk	OK chance of success
8	Create a thinning fiducial (aka Baby fiducial)	High chance of success

[1] High milling currents cause rather blurry images, lowering the chance to recognize the fiducial

[2] Long tilting ranges can cause the software to loose the ROI, especially if the beam coincidence is poorly set.



This entire procedure should run automatically and takes about 30 minutes of time per site.

There is a very high chance of running this automatically without errors.

The process failed.. what to do:

1. Cut a deeper X in the fiducial
2. User lower milling currents (1nA instead of 3 nA)