Guide to operate the FEI Scios FIB/SEM equipped with EDAX EDS/EBSD --- Cross section and TEM specimen preparation

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Specimen Preparation and Handling

The sample material must be able to withstand a high vacuum environment without outgassing. It must be clean and conductive. Oil, dust, or other materials may cause sample charging or contaminate the chamber, which could hinder or even prevent evacuation.

> ***Note:* 1) *Always wear lint- / powder-free clean room gloves when manipulating inside the specimen chamber to minimize oils, dust, or other contaminants pollution of the chamber environment.***

Caution! *Store samples and sample holders in a dry storage cabinet. Dust on samples can get drawn into the electron / ion column, degrading performance*

Mounting Specimen on Holder

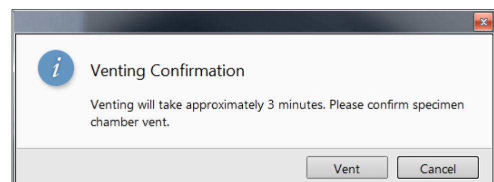
Attach the specimen to the specimen holder using any suitable SEM vacuum-quality adhesive, preferably carbon or silver paint. The specimen must be electrically grounded to the sample holder to minimize specimen charging.

Operation Pre-Check

To ensure correct operation check the following list before continuing.

Adjustment	Electron Beam Setting	Ion Beam Setting
Column Use Case	Standard	Standard
Accelerating Voltage	Select voltage relative to specimen type (1 – 30 kV): - low kV for surface imaging, beam-sensitive samples and slightly charging samples - high voltage for conductors, high resolution, composite info (BSE, X-ray)	30 kV for imaging, milling, depositing 2-5 kV for cleaning 5–10 kV for large field of view
Beam Current & Spot size	100 pA at 30 kV, *Spot size*: 5 – 6	100 pA at 30 kV
Scan rate	fast scan (dwell time 0.1 - 0.3 µs)	Fast scan
Working Distance (FWD)	Set the highest specimen point to approximately 7 mm, tilt to 0° (Yellow mark in an optical imaging display) and press **Ctrl + F** (set FWD to 7 mm function).	Set the stage into the eucentric position and ***tilt to 52°.***
Eucentric Position	7 mm	(*19 mm based on electron beam*)
Magnification	Set to lowest – from 20× to 200×	Set to lowest – about 210×
Detector	ETD (SE) or ICE (SE)	ETD (SE) / ICE (SE)
Filtering	Live or Average (2-4 frames for fast scans)	Live
Contrast and Brightness	With contrast at minimum value adjust brightness to just show a change in intensity to the screen. Increase the contrast to produce a reasonable imaging. Increasing brightness and decreasing contrast produce softer imaging and vice versa.	See Electron beam setting

Inserting / Exchanging Specimen



1. Click on the ***Vacuum*** module / ***Vent*** button. The confirmation dialog appears. After switching the **High Voltage** off the vacuum system switches off the pumps and

opens appropriate valves to vent the system. After a specified venting time (~3 minutes) the venting valve will close.

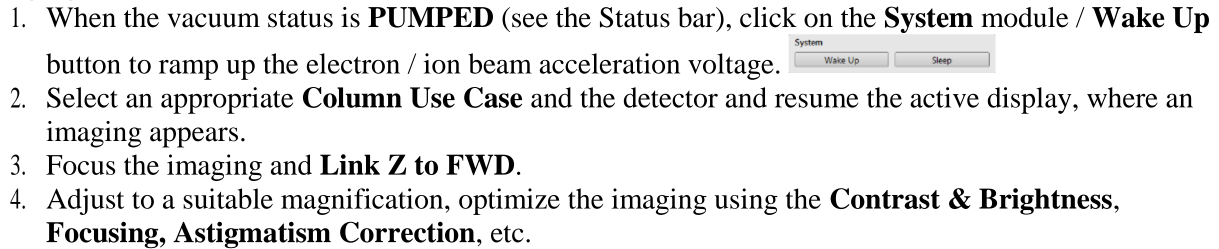
Note :

If the venting valve closes before the chamber is at the atmospheric pressure (the door is not possible to open), click on the Vent button once more to open it again.

2. When vented, slowly pull the specimen chamber open and, using lint-free gloves or tweezers, place a specimen into the specimen holder.
3. Slowly close the specimen chamber door and click on the ***Vacuum*** module / ***Pump*** button.
4. While pumping, choose the highest specimen point and bring it to the **7 mm** Working Distance (yellow line in CCD Quad).

Imaging Onscreen

1. When the vacuum status is **PUMPED** (see the Status bar), click on the **System** module / **Wake Up** button to ramp up the electron / ion beam acceleration voltage.



2. Select an appropriate **Column Use Case** and the detector and resume the active display, where an imaging appears.
3. Focus the imaging and **Link Z to FWD**.
4. Adjust to a suitable magnification, optimize the imaging using the **Contrast & Brightness**, **Focusing, Astigmatism Correction**, etc.

Cross Section Preparation

I. Setting up eucentric position

Note: To enable safe performance of the microscope, do not insert samples of considerably different height in one load. Some of them could collide with the pole piece.

a. Select the *Column Use Case*: **Standard** with High Voltage: **5 kV**, Probe Current: **0.10 nA**.
b. Find the area of interest with the electron beam.
c. Choose a distinguished feature, center it on the display, focus and stigmata the image (use Reduced Area for easier adjustment).
d. **Lin Z** to the Free Working Distance (FWD).
e. Set Stage to **15º** - Position of the selected feature will move up.
f. Adjust stage Z to bring the feature to the center of the field of view.
g. Tilt the stage back to **0º** and then focus the feature and link Z to FWD.
h. Switch to imaging with ion beam at High Voltage: 30 kV, Probe Current: 50 pA,
i. Set Stage to **52º**.
j. Adjust stage Z to bring the feature to the center of the field of view.
k. Switch to imaging with ion beam at High Voltage: 30 kV, Probe Current: 50 pA,
l. Use **Beam Shift** to center the feature.

II. Deposition of surface protection layer

a. Tilt the stage to **52º**.
b. **Insert GIS** needle for Pt deposition.
c. Set the ion beam current to **0.5 nA**.
d. Adjust brightness and contrast if necessary.
e. Draw a 20x5x2 um rectangle pattern with the application being set to '***Pt Dep***'.
f. **Start** the deposition.
g. **Retract GIS** after the deposition is complete.

III. Rough cross section

a. Draw a 25x12x6 um regular cross section pattern in front of the Pt layer.
b. Set the ion beam current to **15 nA**.
   i. Note: with such high ion current direct observation of the sample is not desirable, as the protective layer and sample surface are rapidly destroyed. Instead, make a snapshot, only one scan of the viewed area will be performed in this case.
c. Use the Auto contrast and brightness and focus if necessary.

d. Adjust the position of the RCS pattern leaving few hundreds of nm to the Pt layer.
e. **Start** patterning.

IV. Cleaning cross section

a. Tilt the stage to 53º.
b. Set the ion beam current to 3 nA. Make a snapshot.
c. Use Auto Contrast and brightness, Auto focus and auto stigmator if necessary.
d. Draw a 20 x 2 x 6 um Cleaning Cross Section pattern. Place it so that its bottom edge covers the front side of the cross section and its upper edge slightly overlaps with the Pt layer. Modify X and Y dimensions if necessary.
e. Start patterning.
f. Optionally, repeat the cross section cleaning with 1 nA probe current.
g. After milling is complete, the cross section surface has to be smooth, without curtains.

V. High resolution imaging of the cross section

a. In the Navigation control page select Tilt Correction: Automatic (Cross Section).
b. Set microscopy parameters for high resolution SEM imaging:
    i. Change the use case to OptiTilt.
    ii. Set High Voltage to 2 kV, Probe Current to 0.1 nA.
    iii. Set the electron beam to displays:
        1. Display 1: ETD detector
        2. Display 2: T1 detector
        3. Display 3: T2 detector
c. Start scanning the beam over the sample with image resolution = 768, dwell time = 100 ns, averaging = 4.
d. Use Auto contrast and brightness for all appropriate displays.
e. Adjust the image by auto focusing, stigmation, and lens alignment.
f. Turn the Videoscope on for all appropriate displays and tune the brightness and contrast if necessary.
g. Take the final image with the following scanning parameters: image resolution = 1536, dwell time = 5 us, line integration = 10.
h. For further improvement of image quality:
    i. Gradually decrease the working distance.
    ii. Decrease beam current to 25 pA and increase the number of line integration.

TEM sample preparation

I. Setting up eucentric position

Note: To enable safe performance of the microscope, do not insert samples of considerably different height in one load. Some of them could collide with the pole piece.

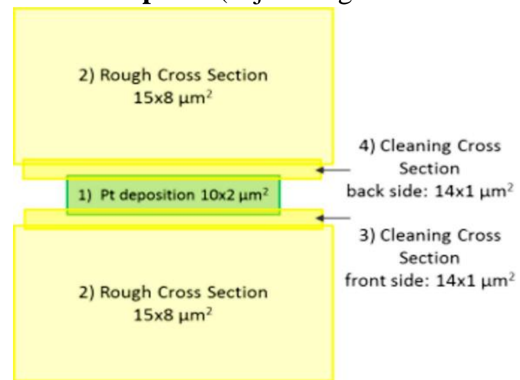
a. Select *Column Use Case*: **Standard** with High Voltage: **5 kV**, Probe Current: **0.10 nA**.
b. Find the area of interest with the electron beam.
c. Choose a distinguished feature, center it on the display, focus and stigmata the image (use Reduced Area for easier adjustment).
d. **Lin Z** to the Free Working Distance (FWD).
e. Set Stage **Z** position to **7 mm.** (use this shortcut only if you have Linked Z to FWD in the previous step!)
f. Set Stage to **15º** - Position of the selected feature will move up.
g. Adjust stage Z to bring the feature to the center of the field of view.
h. Tilt the stage back to **0º** and then focus the feature and link Z to FWD.
i. Tilt the Stage to **52º**.
j. Adjust stage Z to bring the feature to the center of the field of view.
k. Switch to imaging with ion beam at High Voltage: 30 kV, Probe Current: 50 pA or lower,
l. Use **Beam Shift** to center the feature.

II. Deposition of the surface protection layer

a. Electron beam induced deposition of the protective layer (optional)
    i. Tilt the stage to 0 deg.

ii. Set the acceleration voltage of the electron beam to 2 kV, probe current to 0.8 nA.
iii. Insert GIS needle for Pt deposition.
iv. In the SEM display draw a 10 x 2 um rectangle pattern over the area of interest. Set the application to ‘Pt dep’, beam: electron, dwell time: 5 us, time of deposition: 300 s.
v. Start the deposition.
vi. Retract GIS after the deposition is complete.

b. Ion beam induced deposition of the protective layer
   i. Tilt the stage to **52 deg**.
   ii. **Insert GIS** needle for Pt deposition.
   iii. Set the ion beam current to **300 pA**. Make a **snapshot** (adjust brightness and contrast if necessary).
   iv. Draw a 10 x 2 x 1.5 um rectangle pattern over the electron beam deposited area. Set the application to ‘Pt dep’.
   v. **Start** the deposition.
   vi. **Retract GIS** after the deposition is complete.

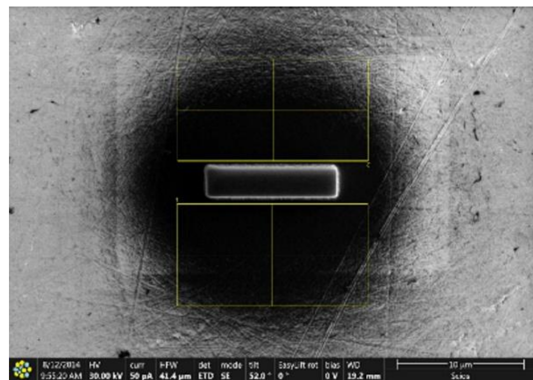


III. Rough cut and cutout

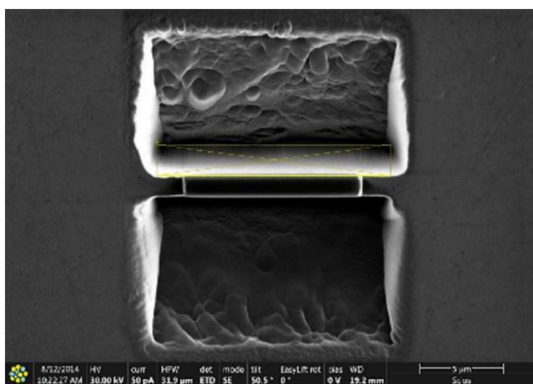
a. Rough cut

Schematic view of the sequence of steps to prepare the lamella for cutout and placement of the milling patterns:

1. Set the ion beam current to **15 nA**.
   Note: with such high current direct observation of the sample is not desirable, as the protective layer and sample surface are rapidly destroyed by the ion beam. Instead, make a **snapshot** only
2. Adjust **Brightness and Contrast** if necessary.
3. Draw *Regular Cross Section* patterns on bottom and top sides of the deposited Pt layer. Select pattern sizes depending on the desired dimensions of the lamella. Use **15 x 8 x 15 µm** for a 5 µm high lamella
4. **Start** patterning. Observe the progress with real time monitor.
5. Repeat patterning if not all material was removed.
6. To clean the front side (visible in SEM) tilt to **53.5°**. Set the ion beam current to **3 nA**. Make a **snapshot**. Use **Auto brightness and contrast** if necessary.
7. **Draw 14 x 1 x 10 µm** Cleaning Cross Section pattern slightly overlapping the deposited Pt layer. **Start** patterning.
8. To clean the back side tilt to **50.5°**. Make a **snapshot**.
9. **Draw 14 x 1 x 10 µm** *Cleaning Cross Section* pattern slightly overlapping the deposited Pt layer. **Start** patterning.



FIB image of the deposited Pt protection layer with the placed regular cross section patterns for the rough cut.

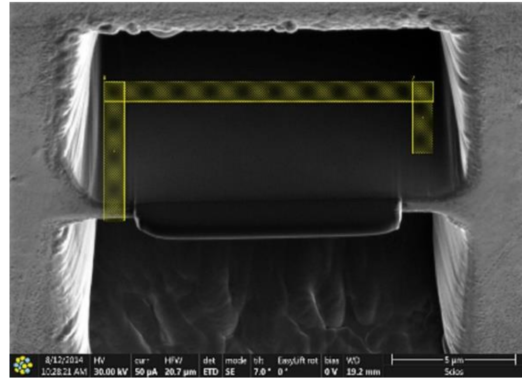


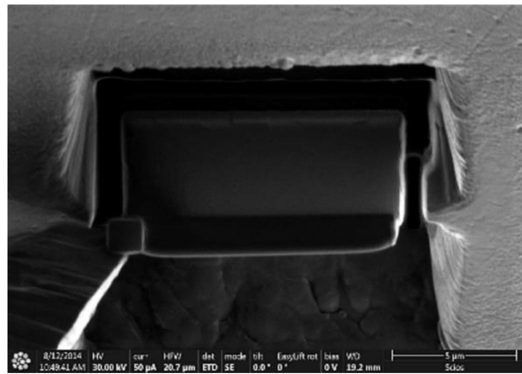
FIB image taken after the rough cut with the cleaning cross section pattern placed on the lamella back side.

Note: direction of patterning always has to be towards the lamella.
Note: it might be necessary to adjust position and dimensions of the patterns.

b. **Under cut**

1. Tilt the sample to **7**°.
2. Set the ion beam current to **3 nA**. Make a **snapshot**. Use Auto brightness and contrast if necessary.
3. For the ***J-cut*** or ***U-cut*** define three rectangular patterns and set them to ***parallel milling***. Place the bottom cut at about 1/3 of the lamella height. Set patterning time to about ***15 min***.
4. Observe the progress with real time monitor. **Stop** patterning when the foil is completely cut out. Check by making a snapshot with the electron beam.





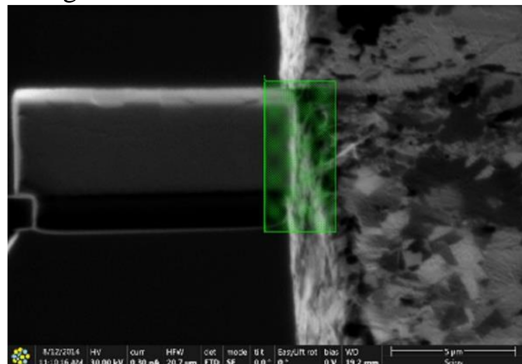
IV. In situ lift out using EasyLift

Note: **EasyLift** needle has to be sharp for the lift out.

1. Tilt the stage to **0**°.
2. Set ion beam current to **50 pA**. Adjust SEM and FIB images so that the lamella is well centered on the display.
3. **Insert the EasyLift needle to the <u>Park position</u>**.
4. **Insert GIS** needle for ***Pt*** deposition.
5. Bring the tip of the **EasyLift** needle to the edge of the lamella. ***Control needle X, Y position in SEM view*** and ***X and height in FIB view***.

Note: To avoid unwilling movements during the lift out stage could be **locked** now.
Note: Needle should be placed as close to lamella as possible, however, without touching or pushing it.

6. Set ion beam current to **50 pA**. Draw a small, typically *1.2 x 1.2 x 0.7 µm*, rectangle over the tip area. **Start** the deposition.
7. Wait that vacuum recovers after the deposition. Set ion beam current to **1 nA**. Make a **snapshot**.
8. Draw a rectangular pattern to disconnect the lamella from the bulk: typically 0.5 x 3 µm. Observe the progress with the real time monitor. **Stop** milling immediately when lamella is released.
9. Lift the lamella above sample surface using needle **Z** motion.
10. **Retract Pt GIS**. **Retract EasyLift** needle.
11. **Unlock** the stage if it was locked earlier.



FIB image of the lamella ready to be attached to the holder with the pattern for Pt deposition.

V. Transfer to TEM grid

1. Tilt the stage to **0**°.
2. Set ion beam current to **50 pA**.
3. Bring the TEM grid in the field of view. **Set up the eucentric position** as explained before.

4. Adjust the TEM grid position so that the area of contact with the lamella is well centered both in SEM and FIB view.
5. **Insert the EasyLift needle** with the attached lamella **to the park position**. **Insert Pt GIS needle**.
6. Drive the EasyLift needle close to the TEM grid. Check Z and X position of the lamella with FIB and X, Y position with SEM. Use successively slower motion of the needle while approaching the grid. In the end, lamella has to be aligned with the post of the TEM grid, practically touching it.
7. Set ion beam current to **300 pA**. Make a **snapshot**.
8. Define a rectangular pattern, typically **2 x 6 x 1 µm**, to attach the lamella to the grid.
9. Place the pattern so that it overlaps both the lamella and the grid. **Start** depositing.
10. Wait till vacuum recovers after the deposition.
11. Set ion beam current to 1 nA. Make a snapshot.
12. Draw 4 x 0.5 µm rectangle over the EasyLift tip and mill until the needle is completely detached from the lamella. Observe the progress with the real time monitor.
13. Manually drive the EasyLift needle away from the lamella, then **retract** it.
14. **Retract GIS**.

SEM image of the lamella welded to the holder and attached to the EasyLift needle.

VI. Final Thinning

1. Thinning is performed from both back and front sides with successively decreasing ion currents until the desired thickness is reached (see table below).
   Note: that tilt angles for final thinning depend on the material. So higher tilt angles should be used for harder materials.
2. Typically, **Cleaning Cross Section pattern** is used. For Z dimension 1/2 of the original lamella height should be enough.
   Note: For both sides direction of milling always has to be towards the lamella.
3. Front side of the lamella could be directly observed with SEM during milling.
4. Watch for the protection layer. Stop thinning before Pt has disappeared.
   Note: Transparency of the lamella to 5 kV electrons, when imaged with ETD SE detector, is an indication that it is thin enough to proceed with final polishing.

Placement of the milling patterns for thinning the back side of the lamella.

Ion beam current	Tilt angle/Milling side	Thickness after milling
1 nA	50.5° /back 53.5° /front	800 nm
0.50 nA	50.5° /back 53.5° /front	400 nm
0.10 nA	51.5° /back 52.5° /front	150 nm
50 pA	51.5° /back 52.5° /front	<100 nm

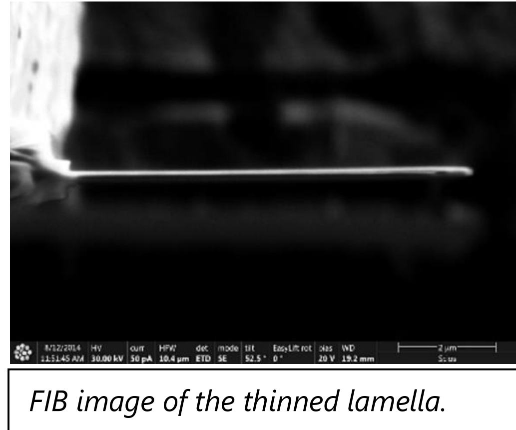
Note: Given tilt angles are valid for Si. Different tilt angles may have to be applied to other materials

VII. Low kV cleaning

Low kV cleaning is a must for high quality TEM samples.
At this step viewing the lamella with both FIB and SEM should be minimized.

A. 5 kV polishing

1. Tilt the stage to **55°** to clean the front side.
2. Set ion beam acceleration voltage to **5 kV**, beam current to **48 pA**. Adjust **brightness and contrast** if necessary.
3. Place a **rectangular pattern** over the lamella side, mill for about 1 min.
4. Tilt to **49°** to clean the back side.
5. Place a **rectangular pattern** over the lamella side, mill for about 1 min.



FIB image of the thinned lamella.

B. 2 kV polishing

1. Tilt to **57°** to clean the front side.
2. Set ion beam acceleration voltage to **2 kV**, beam current to **27 pA**. Adjust **brightness and contrast** if necessary.
3. Place a **rectangular pattern** over the lamella side, mill for 20 sec.
4. Tilt to **47°** to clean the back side.
5. Place a **rectangular pattern** over the lamella side, mill for 20 sec.

VIII. After specimen preparation is done

a. Tilt the stage back to **0°**.
b. **Vent** specimen chamber, and then remove and store your specimen.
c. Close chamber door and **Pump** the chamber till reaching the desired vacuum.
d. Click **Sleep** to put the system in sleep mode if you are the last one using the system of the day; *otherwise,*
   i. *Close both gun valves of the SEM and FIB beams.*
e. Log out of your FIB/SEM session.