FEI Helios NanoLab 600 TEM specimen prep recipe

Nicholas G. Rudawski
ngr@ufl.edu
(352) 392 3077 (office)
(805) 252-4916 (cell)
Last updated: 02/15/21

This recipe is a composite of several methods for preparing relatively soft semiconductor-based specimens for HR-S/TEM analysis; in Si specimens, it can produce lamellas ~20 nm thick with ~2 nm damage layers over a limited area. However, it should by no means be considered universally applicable, and thus only provides a starting point. **Depending on the nature of your particular specimens and your specific TEM analysis needs, substantial deviations from this recipe may be necessary.** You should always consult the scientific literature to see if any FIB-based methods of specimen preparation have been reported for your specific materials/TEM analysis needs and consult staff if you need any recommendations.

1. Ex-situ deposition of a conductive coating prior to FIB

    1.1. If the material is insulating, a conductive coating is required, or stable imaging will not be possible; evaporation coating with a few 10s of nm of amorphous C or Cr (with Cr being preferred) will usually suffice; if the material is conductive, a conductive coating is not needed.

2. Grids and grid loading

    2.1. The best grids for in-situ lift outs are made *specifically* by Omniprobe and are referred to as "Omniprobe" grids. The grids are made of Cu or Mo and have 3 – 5 "fingers" to provide points of attachment for samples. *In general, it is preferable to use Mo rather than Cu as a grid material.* Mo is very stiff, will deform less easily than Cu, and will redeposit less material onto the lamella.

    https://www.tedpella.com/Omniprobe_html/FIB-Omni.htm#liftout

    2.2. When loading a grid into the TEM row holder, it should be loaded so that "Omniprobe" reads forwards as viewed from the top with the row holder inserted into the loading device (clips side facing up).

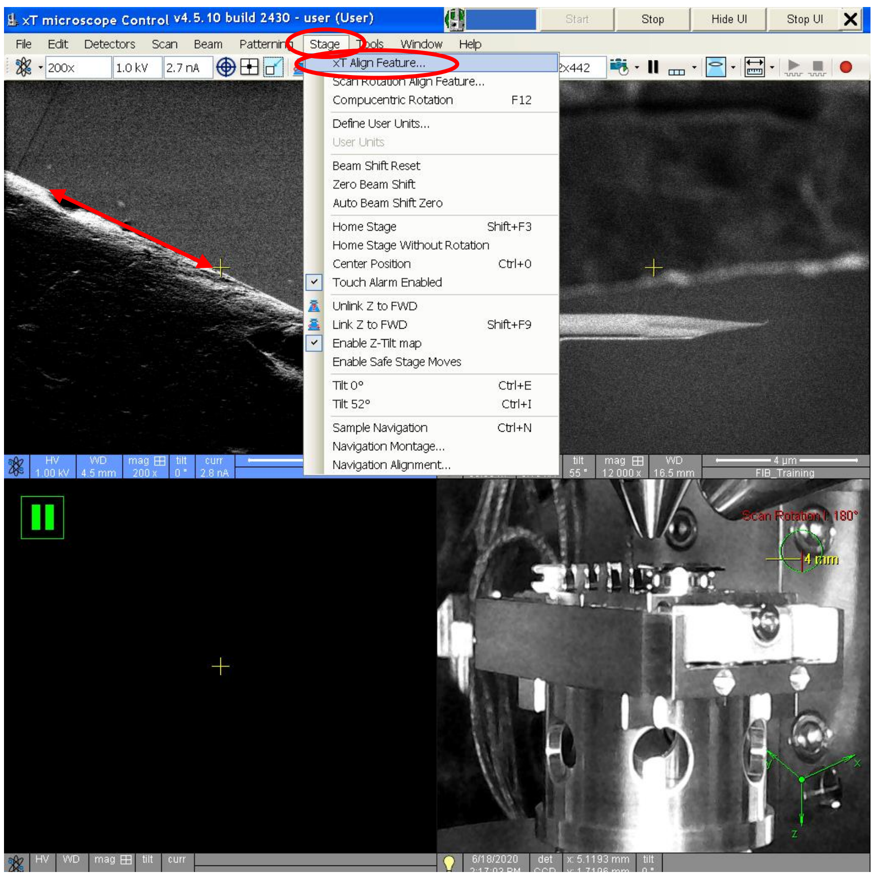
    2.3. After loading the grid into the row holder, it should be examined edge-on using the optical microscope to make sure it is straight. If it appears bent, it should be discarded and replaced with an unbent grid.

    2.4. When loading the TEM row holder into the TEM row holder module in the UMB, orient the row holder so the clips side of the holder faces *towards* the computer desk (this will properly orient the grid when it is time to attach the lamella, assuming you loaded it properly in the previous step).

3. Stage adjustments for specimen

3.1. When the vacuum is sufficient, turn on the beams and Pt heating and start live E-beam imaging.

3.2. If the lamella needs to be prepared so it is aligned with a particular sample direction (e.g. a cleaved edge of a single crystal), navigate over to the feature(s) to be used for alignment in the live E-beam image and move the sample to working distance ~4 mm. Make sure the live image is well focused, select “Stage” from the pull-down menu, then “xT Align Feature”, and follow the instructions; when finished, the stage will rotate to align the feature as specified.



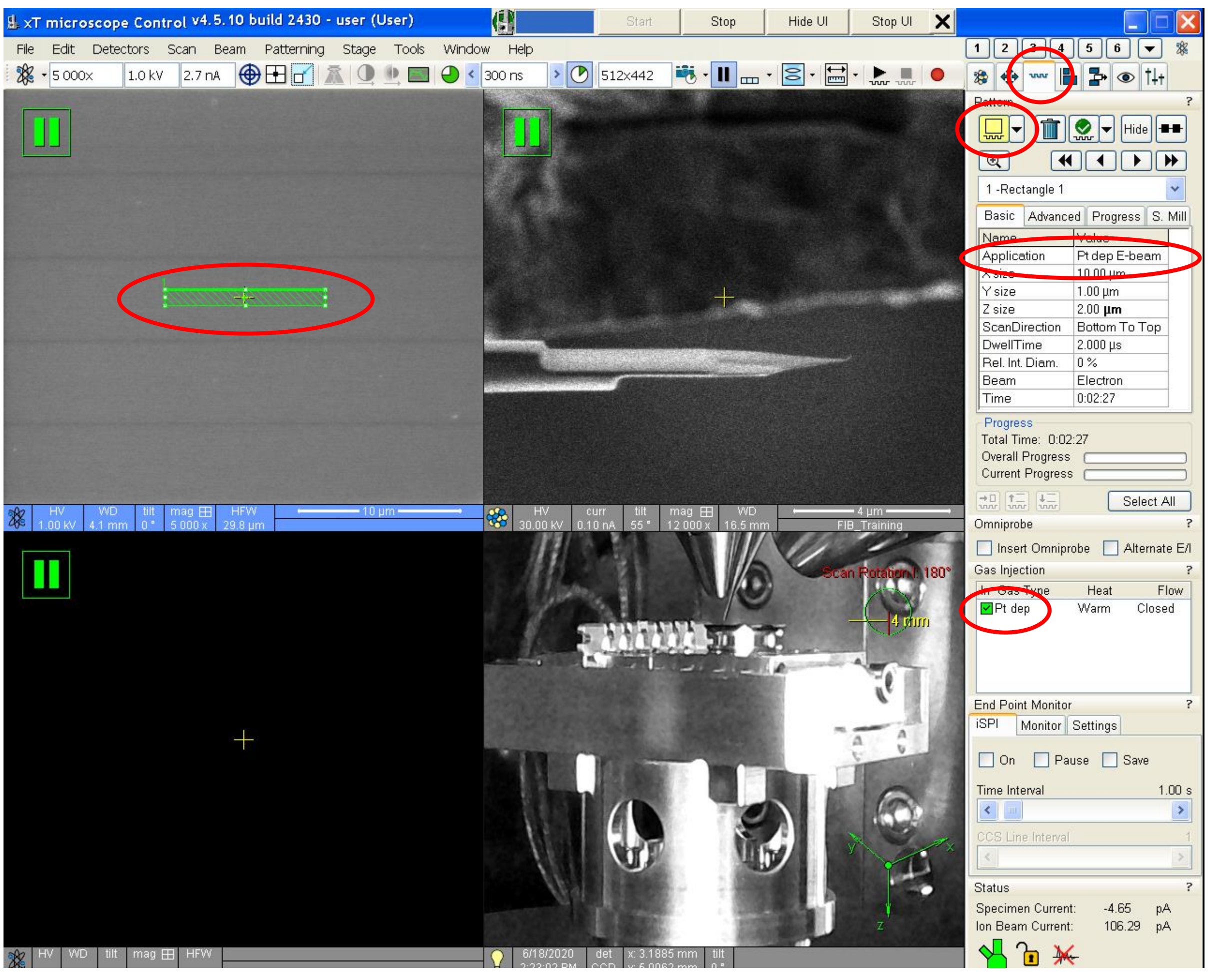
3.3. All subsequent steps of this recipe must be performed with the region of interest properly set at eucentric height and the beams linked (as needed).

4. E-beam Pt strap deposition

Horizontal Field Width = ~30 µm
Stage tilt: T = 0°
E-beam settings = 1 or 2 kV, 2.7 or 5.5 nA
Pattern type: rectangle
Pattern application: “Pt dep E-1kV” or “Pt dep E-2kV” (as appropriate)
Pattern dimensions: X = 10, Y = 1.0, and Z = 0.5 µm

4.1. Perform direct alignments on the electron beam to obtain the best image possible.

4.2. While collecting a live E-beam image, insert the Pt GIS and draw/position the indicated pattern; take a snapshot and then execute the pattern. When the pattern is finished, retract the Pt GIS.

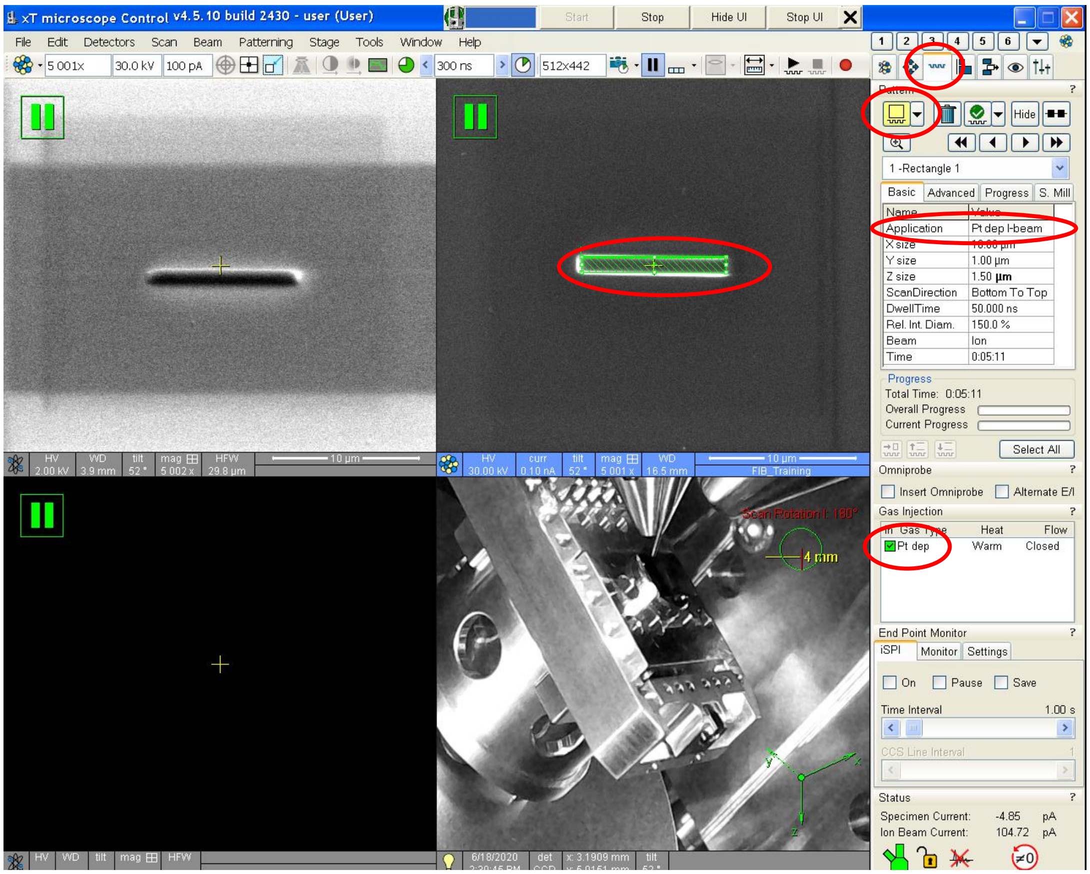


5. I-beam Pt strap deposition

Horizontal Field Width = ~30 µm
Stage tilt: T = 52°
I-beam settings = 30 kV, 100 pA
Pattern type: rectangle
Pattern application: “Pt dep I-beam”
Pattern dimensions: X = 10, Y = 1.0, and Z = 2.0 µm

5.1. Bring the Pt strap just deposited with the E-beam to eucentric height; when finished, leave the stage tilt at T = 52°, and link the beams (making sure the I-beam scan rotation = 180°).

5.2. Insert the Pt GIS, take a snapshot, draw/position the indicated pattern and execute When the pattern is finished, retract the Pt GIS. The total resulting Pt strap thickness after this step should be ~2.0 µm.

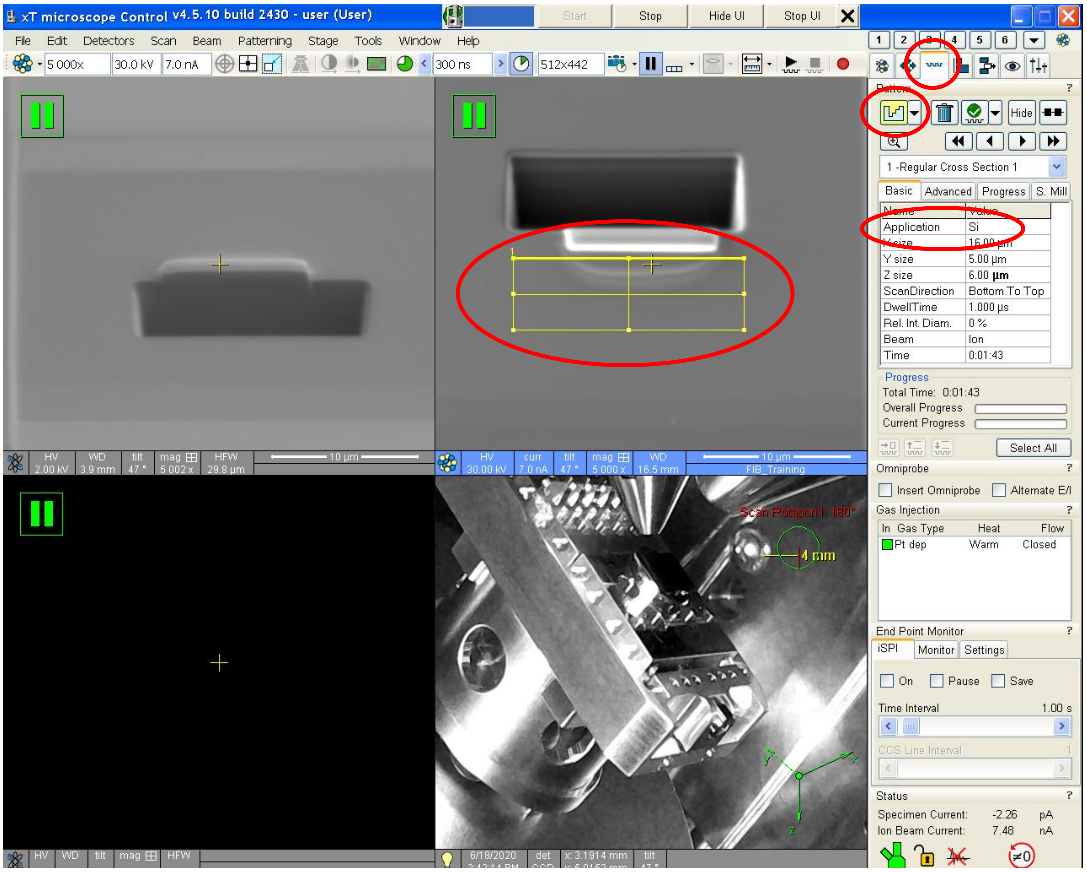


6. Trenches

Horizontal Field Width = ~30 µm
Stage tilt: T = 52 ± 5°
I-beam settings = 30 kV, 7.0 nA
Pattern type: regular cross-section
Pattern rotation = 180° (at T = 57°); 0° (at T = 47°)
Pattern application: “Si”
Pattern dimensions: X = 16, Y = 5.0, and Z = 6.0 µm

6.1. Set T = 57° and take an I-beam snapshot; draw the indicated pattern, set pattern rotation to 180°, and position pattern so the thick line is just above the Pt strap, then execute (not shown).

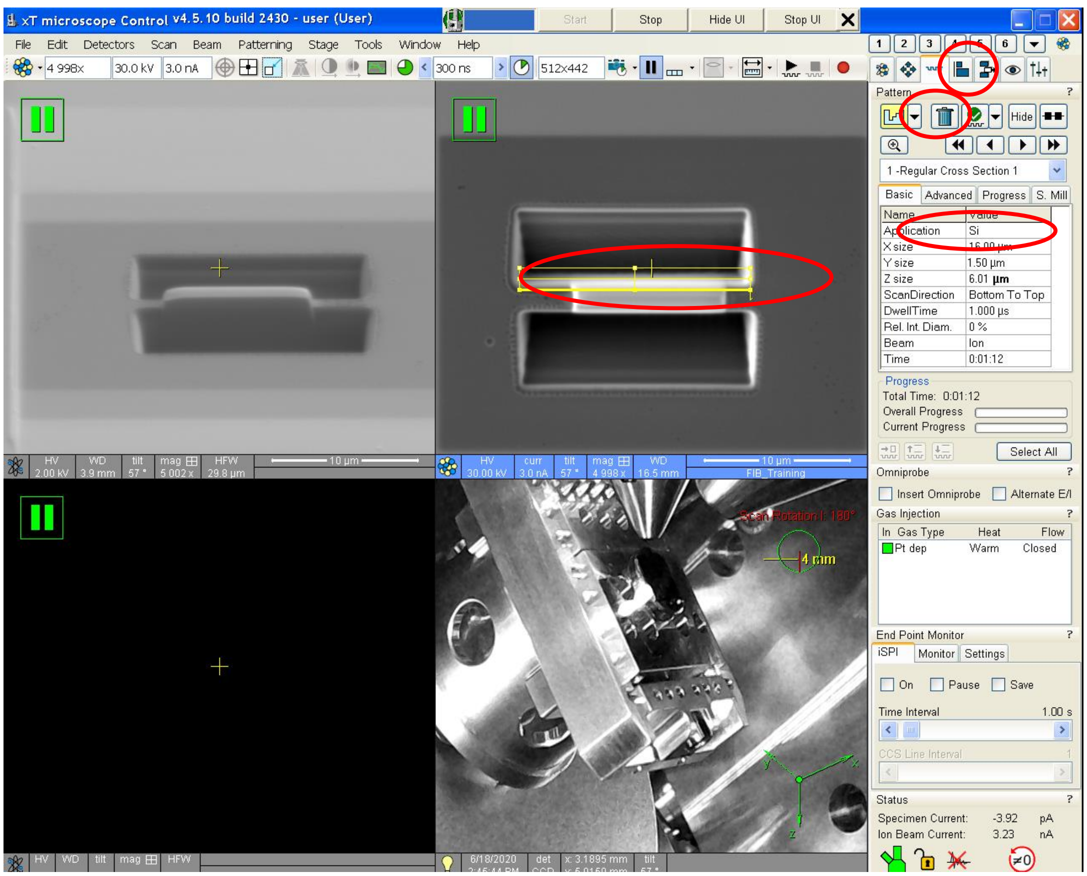
6.2. Set T = 47° and take an I-beam snapshot; set pattern rotation to 0, and position pattern so the thick line is just below the Pt strap, then execute (shown below).



7. Coarse thinning

Horizontal Field Width = ~30 µm
Stage tilt: T = 52 ± 5°
I-beam settings = 30 kV, 3.0 nA
Pattern type: regular cross-section
Pattern rotation = 180° (at T = 57°); 0° (at T = 47°)
Pattern application: “Si”
Pattern dimensions: X = 16.0, Y = 1.5, and Z = 6.0 µm

7.1. Set T = 57° and take an I-beam snapshot; draw the indicated pattern, set pattern rotation to 180°, and position pattern so the thick line is just above the Pt strap, then execute (shown below).



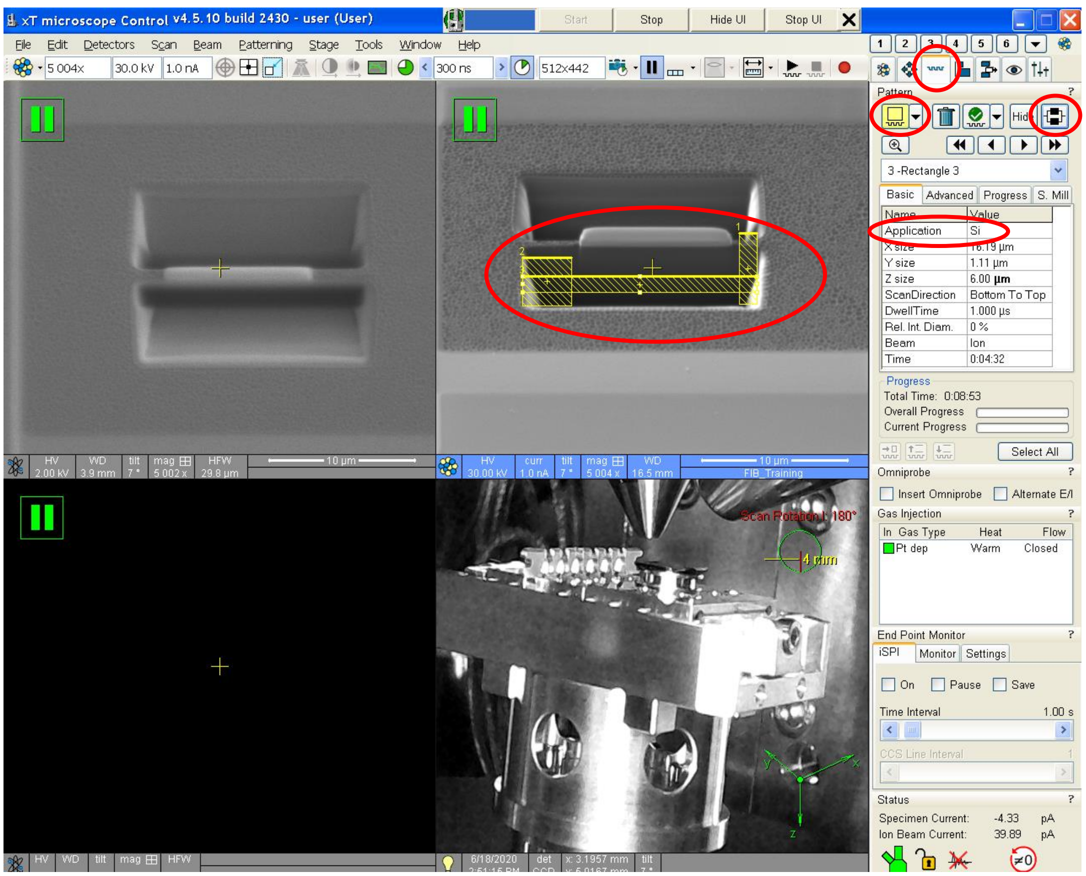
7.2. Set T = 47° and take an I-beam snapshot; set pattern rotation to 0°, and position pattern so the thick line is just below the Pt strap, then execute (not shown).

8. Undercutting

Horizontal Field Width = ~30 µm
Stage tilt: T = 7°
I-beam settings = 30 kV, 1.0 nA
Pattern type: rectangle
Pattern application: “Si”
Pattern dimensions: X = adjust as needed, Y = adjust as needed, and Z = 6.0 µm
Pattern order: parallel

8.1. Shift the I-beam image as needed to bring the lamella back to the center of the image.

8.2. Take an I-beam snapshot and draw/position three rectangle patterns as shown in the image below; the width of each pattern should be >0.7 µm and there should be overlap between the corners.



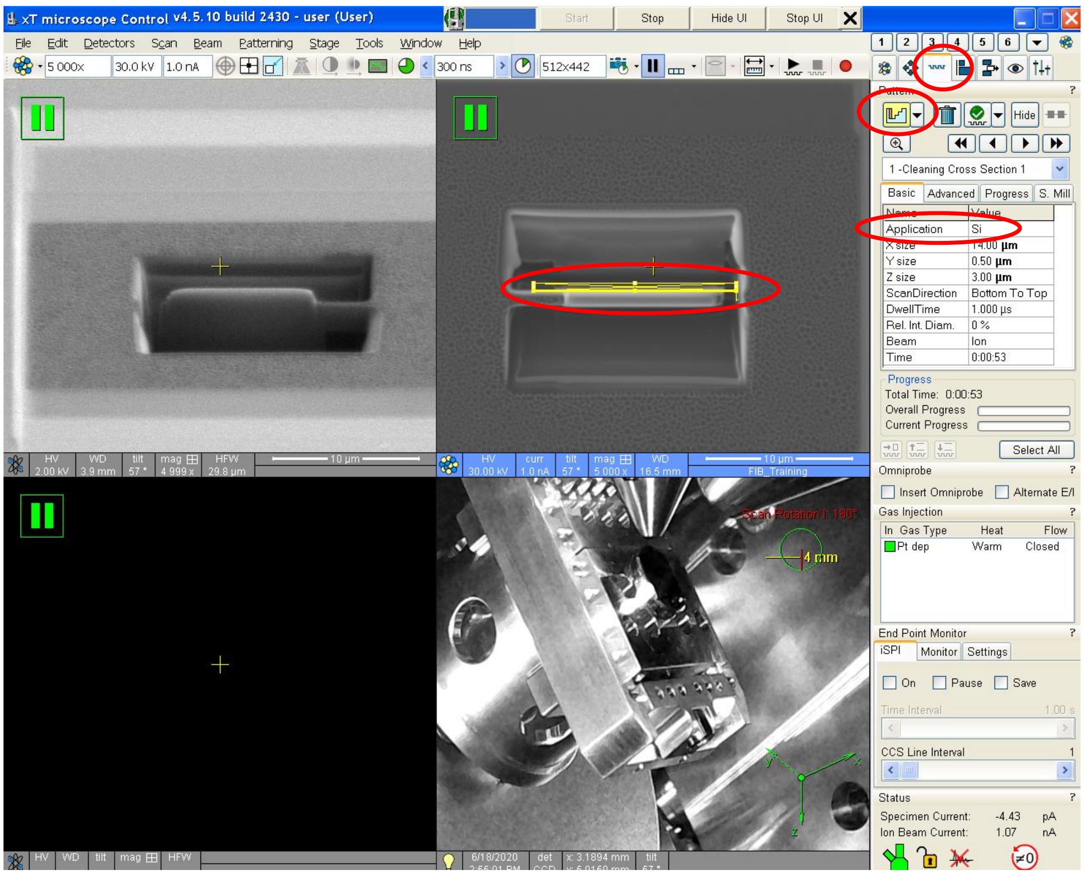
8.3. Execute the pattern and stop once undercutting is complete (live I-beam or E-beam image during milling to verify).

9. Post-undercut cleaning

Horizontal Field Width = ~30 µm
Stage tilt: T = 57°
I-beam settings = 30 kV, 1.0 nA
Pattern type: cleaning cross-section
Pattern rotation = 180°
Pattern application: “Si”
Pattern dimensions: X = 14, Y = adjust as needed, and Z = 3.0 µm

9.1. Take an I-beam snapshot, draw the indicated pattern and position so the thick line is above the Pt strap as indicated.

9.2. Manually adjust Y dimension of the pattern so the “wings” on top the side of lamella are covered and then execute.



10. Approaching the Omniprobe

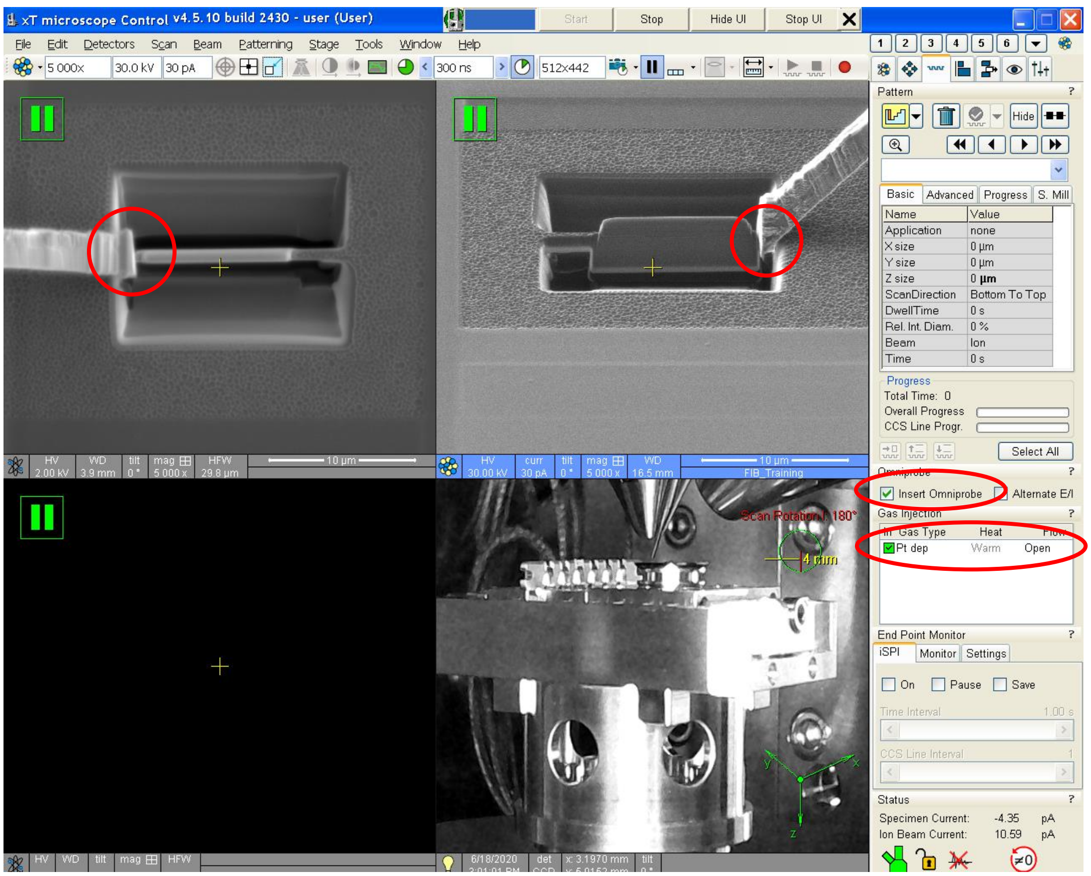
Horizontal Field Width = adjust as needed; final ~30 µm
Stage tilt: T = 0°
I-beam settings = 30 kV, 30 pA

10.1. Start live I-beam imaging and adjust the Y beam shift until the lamella is back within the field of view.

10.2. Insert the Omniprobe and use live E-beam and I-beam imaging to approach it to the lamella

10.3. When the Omniprobe is a few µm away from the lamella, insert the Pt GIS and set Flow = Open.

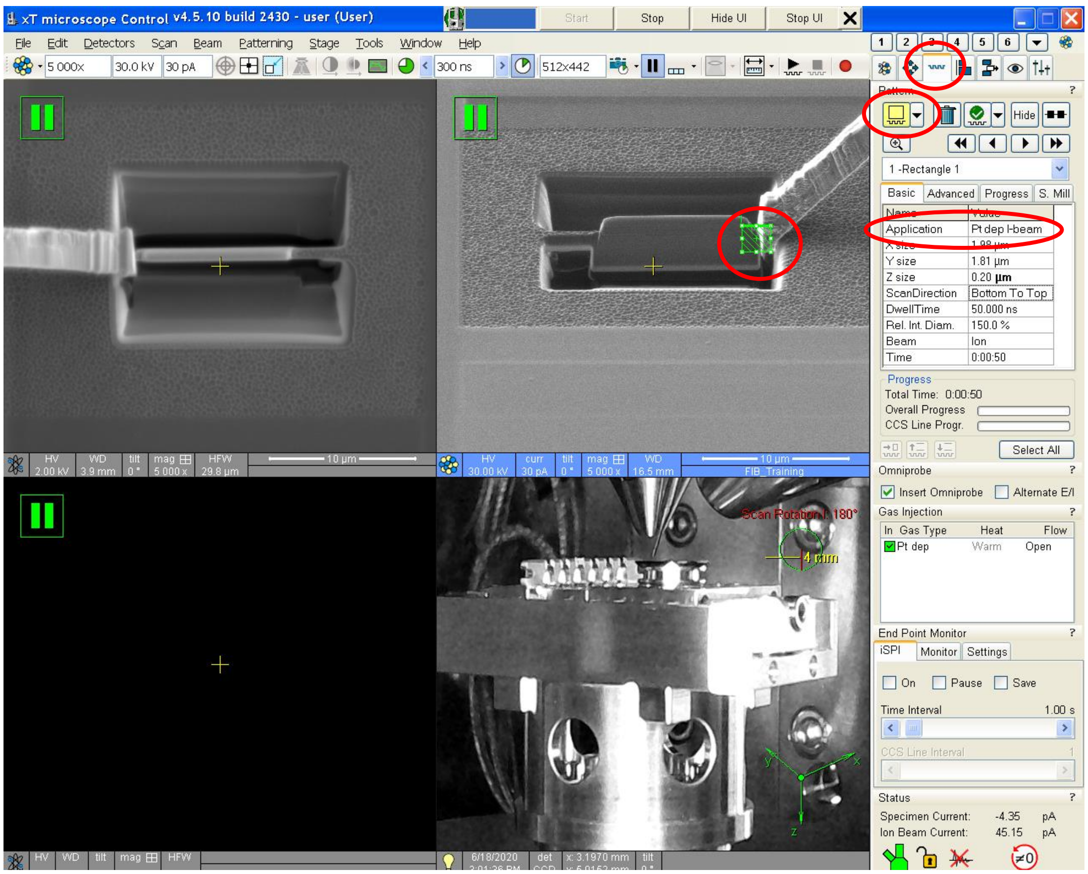
10.4. Position the Omniprobe so it is centered over and lightly touching the free end of the lamella.



11. Attaching Omniprobe to lamella

Horizontal Field Width = ~30 µm
Stage tilt: T = 0°
I-beam settings = 30 kV, 30 pA
Pattern type: rectangle
Pattern application: "Pt dep I-beam"
Pattern dimensions: X ≥ 1.5, Y ≥ 1.5, and Z = 0.2 – 0.4 µm

11.1. Take an I-beam snapshot, draw the indicated pattern and adjust the X and Y dimensions to sufficiently cover the point of contact between the lamella and Omniprobe.

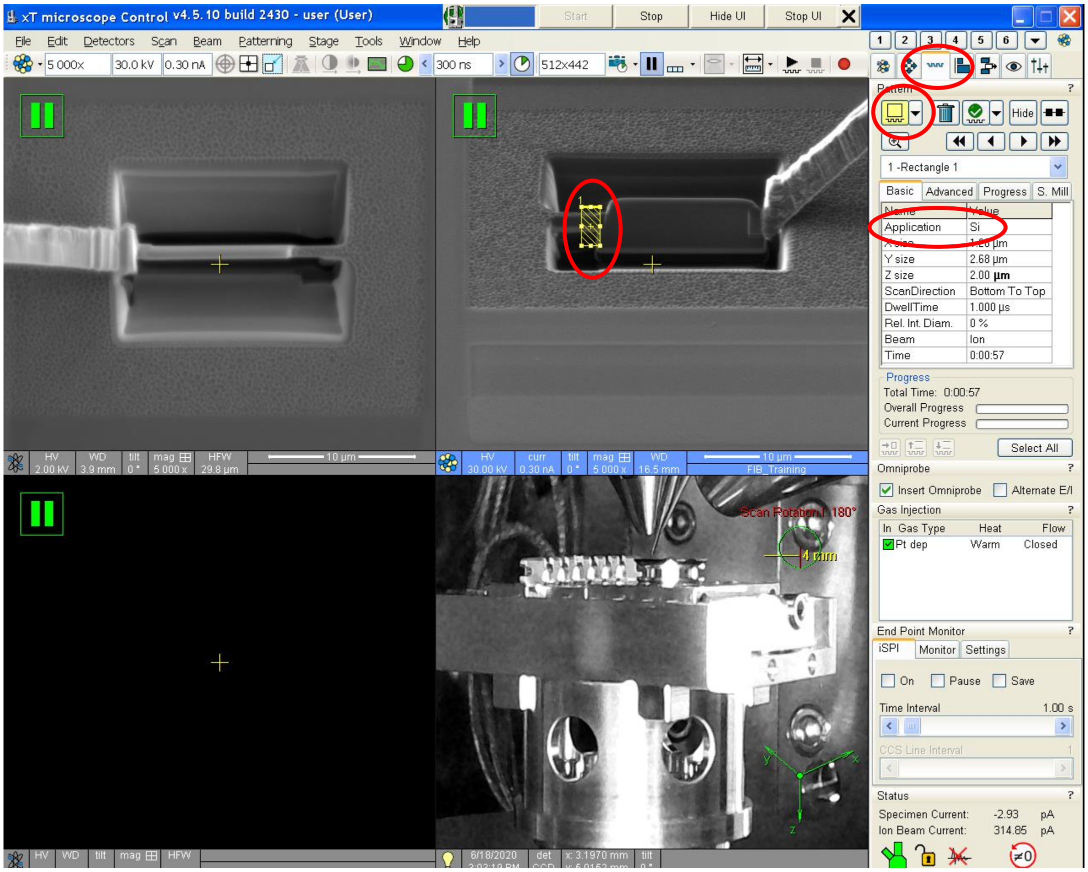


11.2. Execute the pattern; take E-beam snapshots (or live E-beam image) during patterning to observe and verify the Pt deposition.

12. Freeing the lamella

Horizontal Field Width = ~30 µm
Stage tilt: T = 0°
I-beam settings = 30 kV, 0.30 – 0.50 nA
Pattern type: rectangle
Pattern application: “Si”
Pattern dimensions: X = adjust as needed, Y = adjust as needed, and Z = 2.0 µm

12.1. Take an I-beam snapshot, draw/position the indicated pattern and adjust the X and Y dimensions to sufficiently cover the remaining point of contact between the lamella and sample.

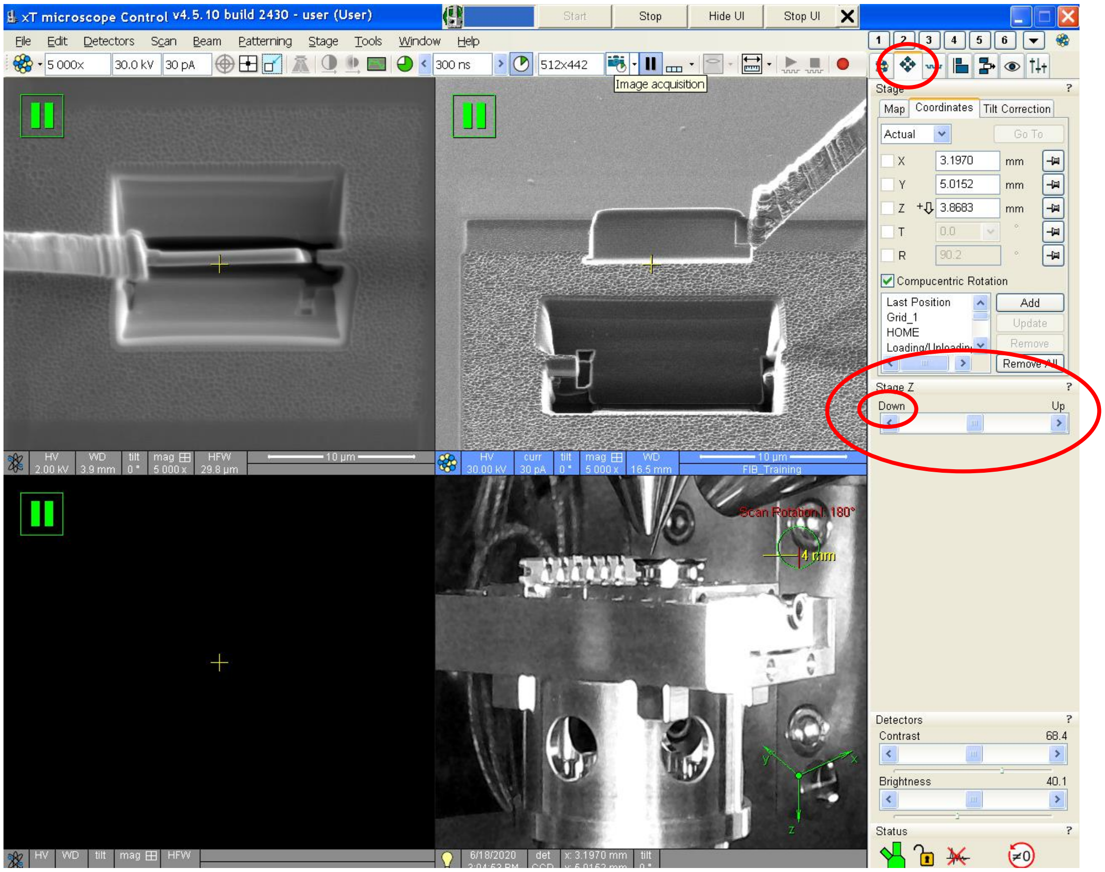


12.2. Execute the pattern; take E-beam snapshots (or live E-beam image) to observe the progress of the pattern and stop the pattern once the lamella is released (the entire Z pattern depth will probably not be needed).

13. Lifting out the lamella

Horizontal Field Width = start at ~30 µm; subsequently adjust as needed
Stage tilt: T = 0°
I-beam settings = 30 kV, 30 pA

13.1. Start live I-beam imaging; slowly lower the Z stage control to pull the specimen away from the lamella (shown below).

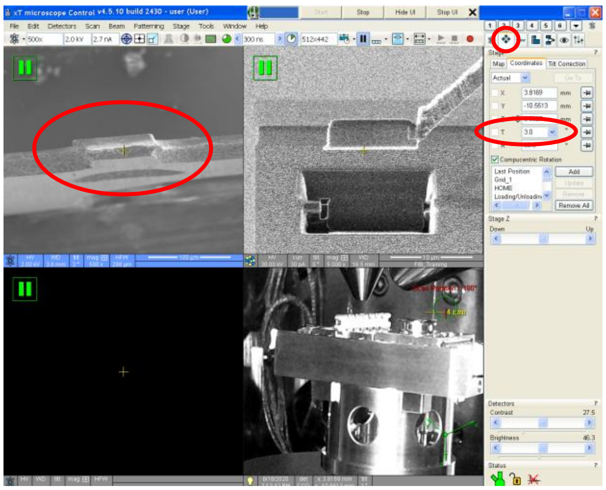


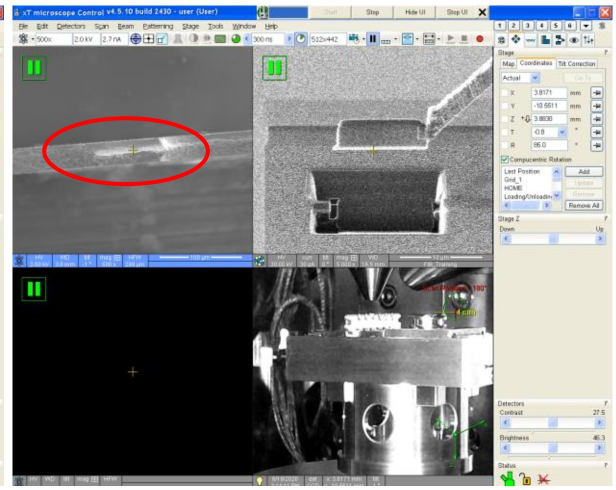
13.2. Once the specimen is <u>sufficiently clear of the specimen surface</u> (a few 10s of µm), retract the Pt GIS; then place the Omniprobe into the "Park" position and retract the Omniprobe (not shown).

14. Grid alignment prior to lamella attachment

    14.1. Navigate over to the position on the grid where the sample is intended to be attached and bring it to eucentric position (notch mount shown here).

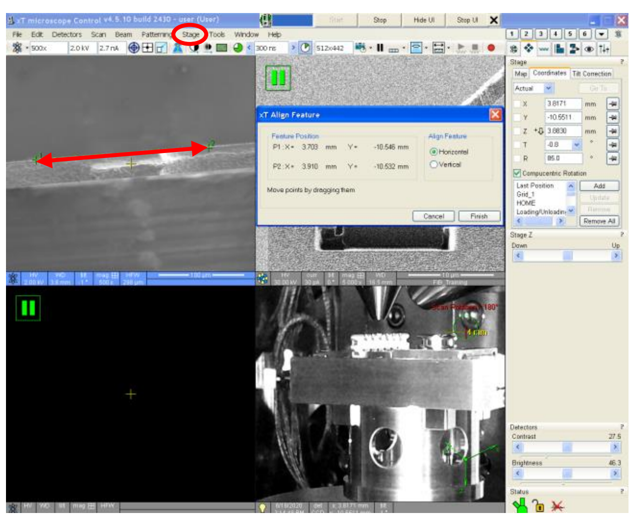
    14.2. Adjust the stage tilt (usually T = –1 to –2°) until the grid is viewed edge on and not tilted; if you have to tilt to T < –2°, leave the tilt at T = –2° (the Omniprobe cannot be inserted for T < –2°).





    14.3. The stage tilt for normal incidence for each beam will be differ by this amount (e.g. –2° + 52° = 50° is now the stage tilt for normal incidence for the I-beam, while –2° + 0° = –2° is normal incidence for the E-beam).

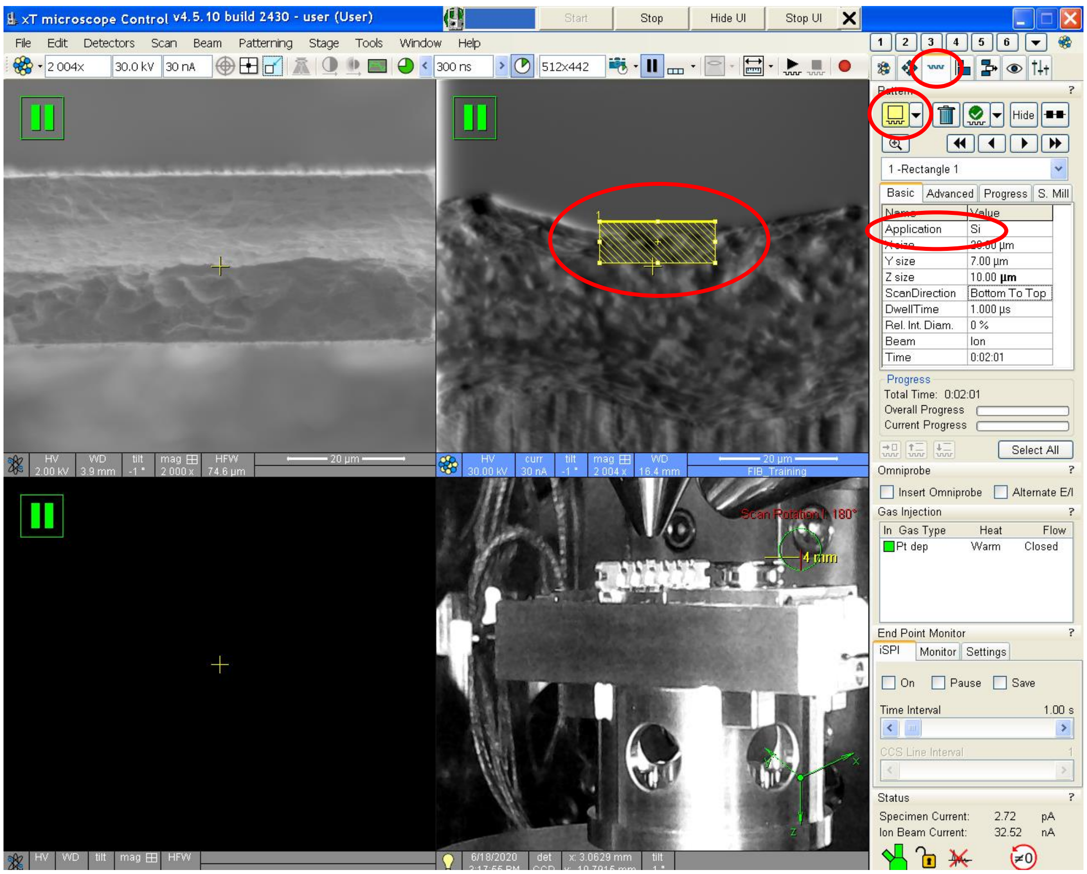
    14.4. The grid should now be rotated so it is horizontal in the E-beam image; select “Stage” from the pull-down menu and then “xT Align Feature” and follow the instructions to rotate the grid (described previously in 3.2).



15. Milling a shelf in the grid

Horizontal Field Width = ~75 µm
Stage tilt: normal incidence for E-beam
I-beam settings = 30 kV, 15 or 30 nA
Pattern type: rectangle
Pattern application: “Si”
Pattern dimensions: X = 20, Y = adjust as needed, and Z = 10.0 µm

15.1. Take an I-beam snapshot, draw/position the indicated pattern in the middle of the notch, manually adjust the Y dimension so the pattern will mill through the projected side of the grid and execute (repeat if necessary).



15.2. The resulting shelf should be flat in the middle with vertical sides.

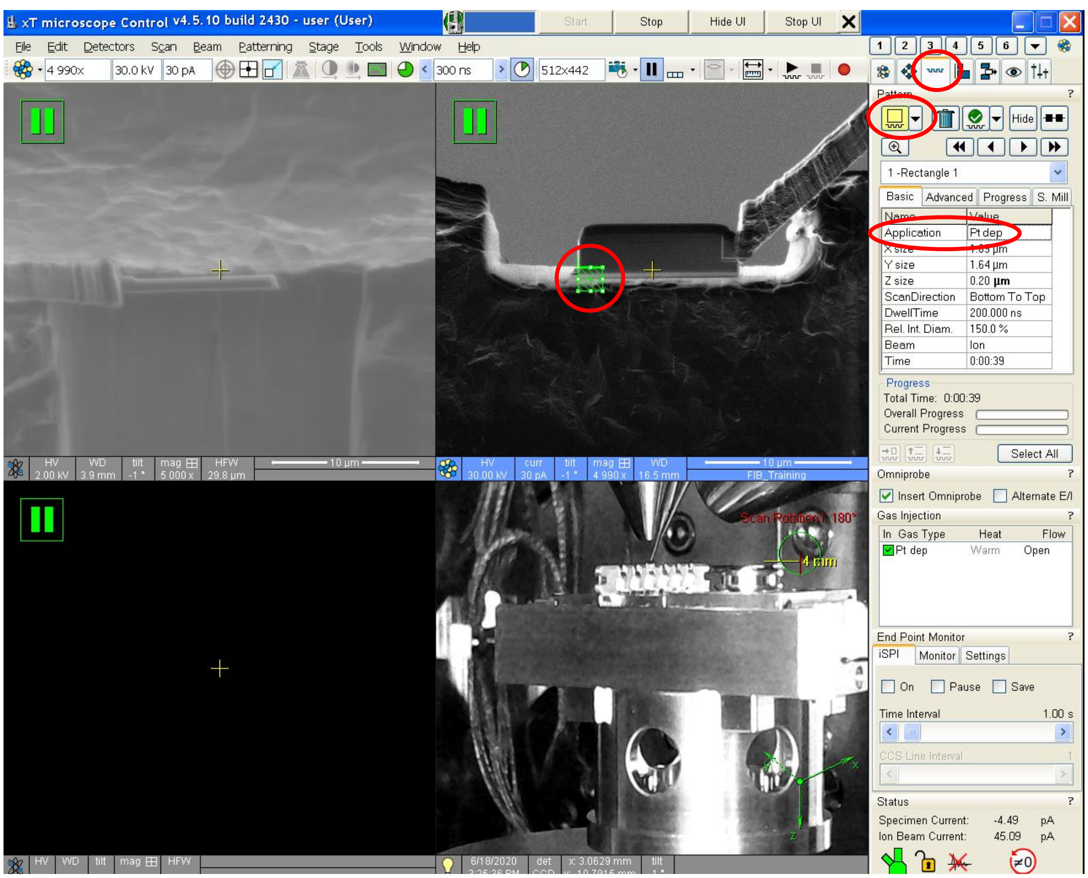
16. Attaching the lamella to the grid

Horizontal Field Width = ~30 µm
Stage tilt: normal incidence for E-beam
I-beam settings = 30 kV, 30 pA
Pattern type: rectangle
Pattern application: “Pt dep I-beam”
Pattern dimensions: X ≥ 1.5, Y ≥ 1.5, and Z = 0.4 µm

16.1. Insert the Omniprobe and use live E-beam and I-beam imaging to approach the lamella to the grid.

16.2. When the lamella is a few µm away from the grid, insert the Pt GIS and set Flow = Open; position the lamella so it is sitting on the edge of the shelf; take an I-beam snapshot

16.3. Draw the indicated pattern with X and Y dimensions sufficient to cover the point of contact between one corner of the lamella and the grid and execute (shown below).

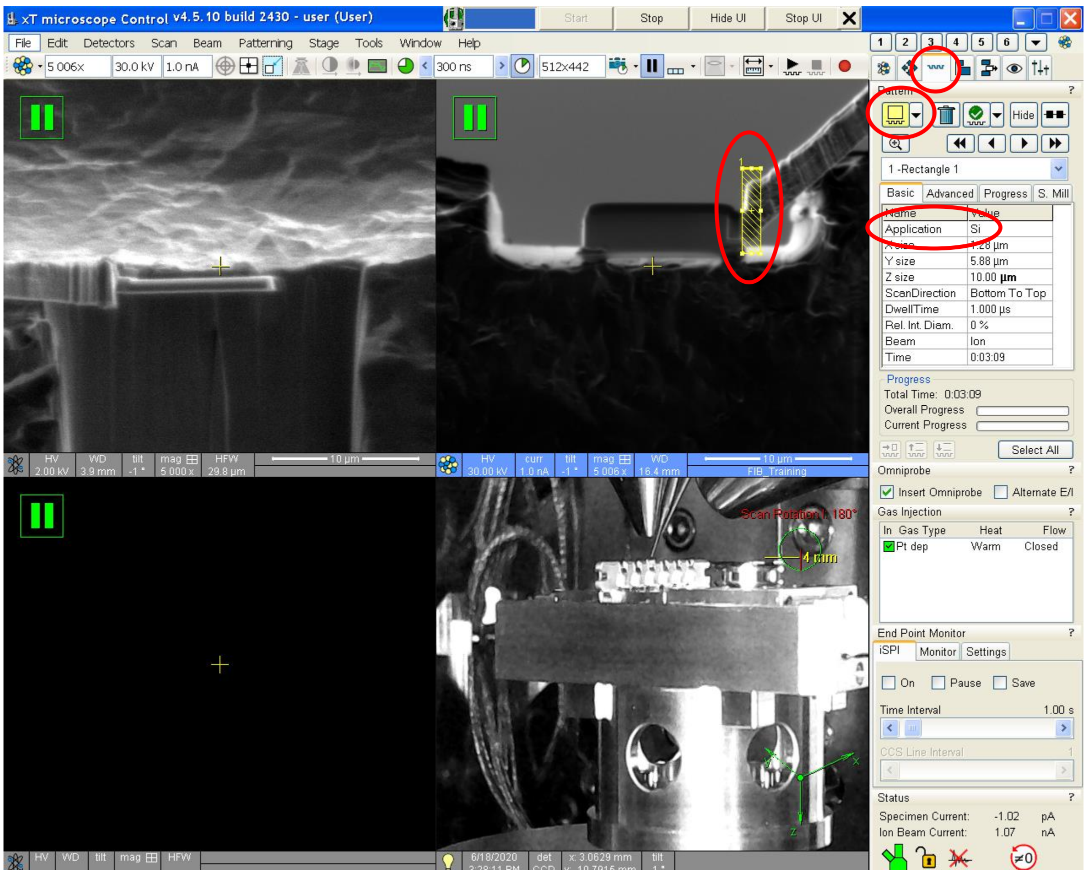


16.4. Repeat this pattern for the other corner of the lamella (not shown).

17. Omniprobe release

Horizontal Field Width = ~30 µm
Stage tilt: normal incidence for E-beam
I-beam settings = 30 kV, 1.0 nA
Pattern type: rectangle
Pattern application: “Si”
Pattern dimensions: X = ≥1, Y = adjust as needed, and Z = 4 µm

17.1. Take an I-beam snapshot, draw/position the indicated pattern and adjust the X and Y dimensions to sufficiently cover the point of attachment between the lamella and Omniprobe (shown below).

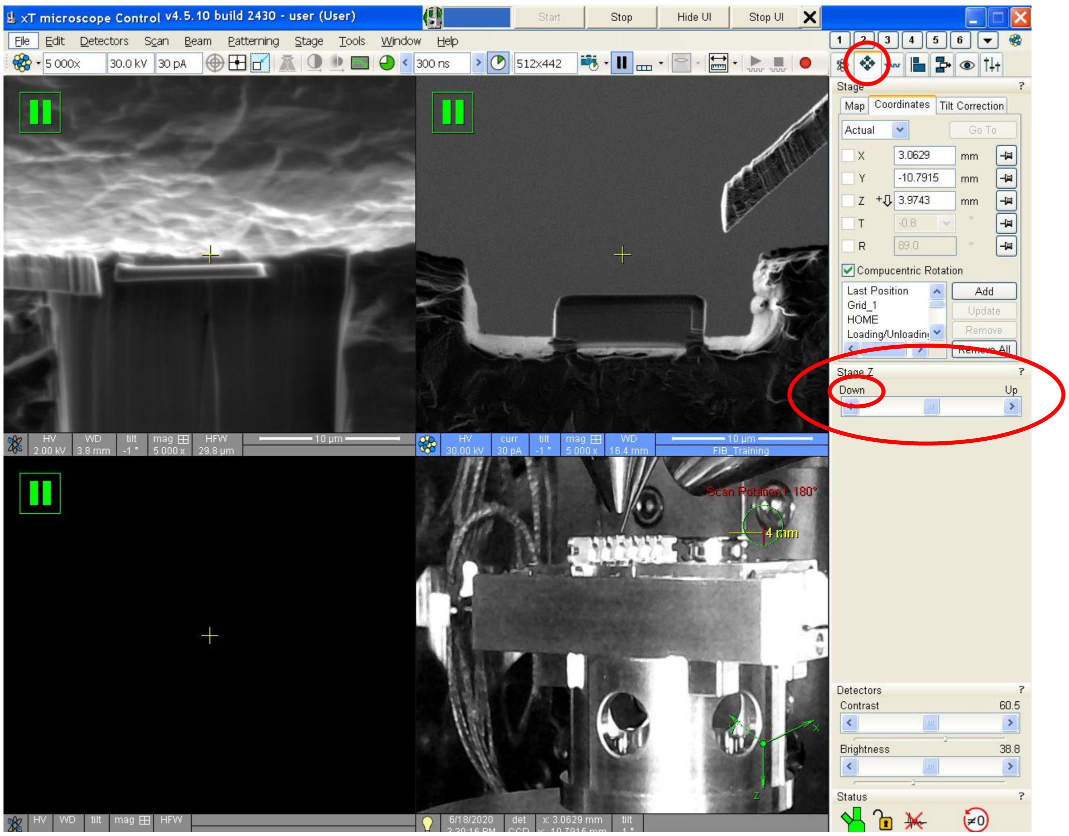


17.2. Execute the pattern; take E-beam snapshots (or live E-beam image) to observe the progress of the pattern and stop the pattern once the Omniprobe is released.

18. Omniprobe and Pt GIS retraction

Horizontal Field Width = ~30 µm
Stage tilt: normal incidence for E-beam
I-beam settings = 30 kV, 30 pA

18.1. Start live I-beam imaging; slowly lower the Z stage control to pull the specimen away from the Omniprobe.



18.2. Once the lamella is <u>sufficiently clear of the Omniprobe</u> (a few 10s of µm), retract the Pt GIS; then place the Omniprobe into the “Park” position and retract the Omniprobe (not shown).

18.3. If you have additional lamellas to prepare, it is best to lift out and attach each lamella to the grid and then final thin all lamellas at once.

19. E-beam imaging during final thinning

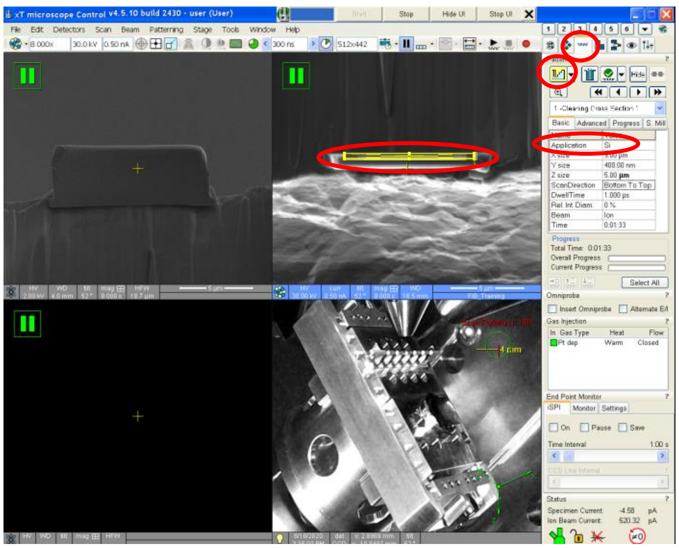
19.1. Reduce the E-beam current (86 pA or 0.17 nA) prior to starting final thinning; Be sure to perform direct E-beam alignments after changing currents.

19.2. You should constantly E-beam image your lamella while final thinning to monitor the progress of each pattern (and to possibly prevent over-milling).

20. Final thinning: 30 kV

Horizontal Field Width = ~20 µm
Stage tilt: ±1° (from normal I-beam incidence)
I-beam settings = 30 kV: 0.50 nA (initial), 0.30 nA, 100 pA, and 50 pA (final)
Pattern type: cleaning cross-section
Pattern rotation = 180° (+1° from normal incidence); 0° (–1° from normal incidence)
Pattern application: “Si”
Pattern dimensions: X = adjust as needed, Y = adjust as needed, and Z = 4.0 µm

20.1. Tilt the stage to relative +1° and take an I-beam snapshot; draw the indicated pattern starting with an X dimension of 8.0 – 9.0 µm, set pattern rotation to 180°, and position over the center of the top side of the lamella; manually adjust the Y dimension to cover the exposed top side of the lamella and execute (shown below).



20.2. Reduce current to 0.30 nA; tilt stage to relative –1° and take I-beam snapshot; reduce X pattern dimension by 1.0 µm, set pattern rotation to 0°, and position over center of bottom side of the lamella; adjust Y dimension to cover exposed bottom side of lamella and execute (not shown).

20.3. Reduce current to 100 pA; tilt stage to relative +1° and take I-beam snapshot; reduce X pattern dimension by 1.0 µm, set pattern rotation to 180°, and position over center of top side of the lamella; adjust Y dimension to cover exposed top side of lamella and execute (not shown).

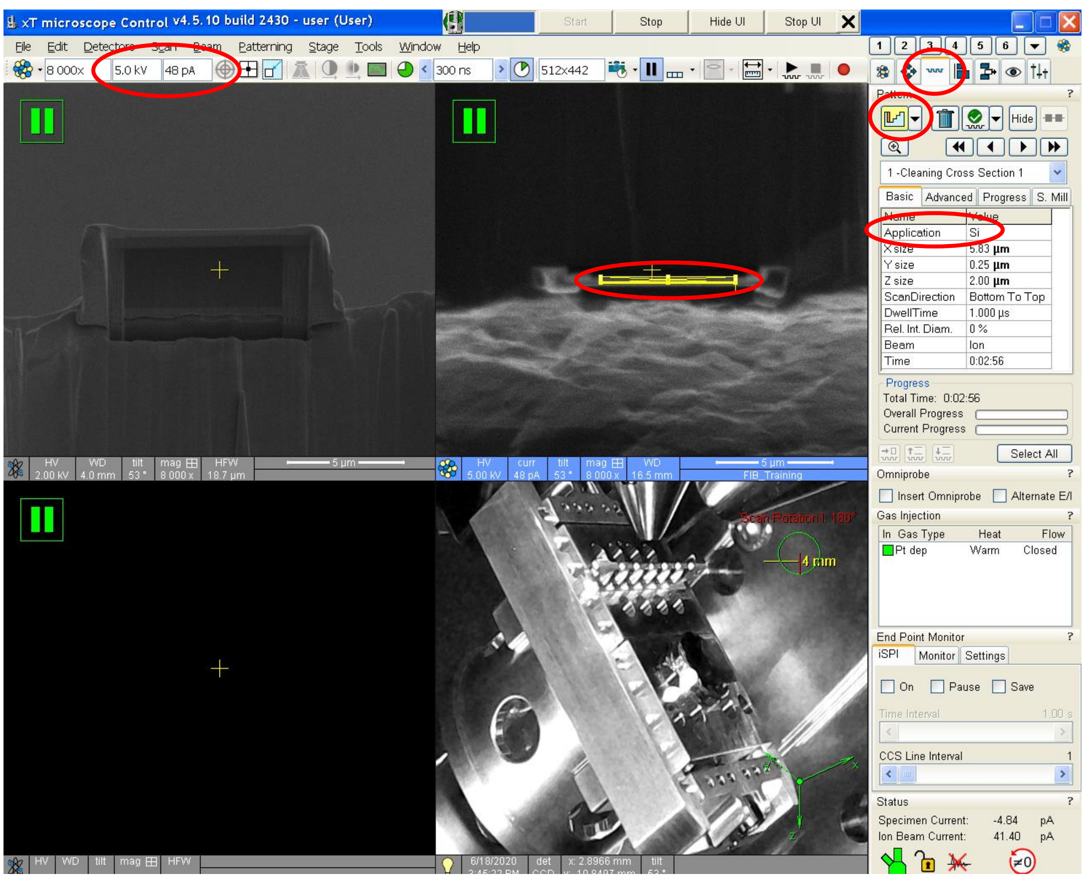
20.4. Reduce current to 50 pA; tilt stage to relative –1° and take I-beam snapshot; reduce X pattern dimension by 1.0 µm, set pattern rotation to 0°, and position over center of bottom side of the lamella; adjust Y dimension to cover exposed bottom side of lamella and execute (not shown); lamella should be ~150 nm thick over a ~5 µm “window” after this step.

21. Final thinning: 5 kV

Horizontal Field Width = ~20 µm
Stage tilt: ±2° from normal I-beam incidence
I-beam settings = 5 kV, 48 or 77 pA
Pattern type: cleaning cross-section
Pattern rotation = 180° (+1° from normal incidence); 0° (–1° from normal incidence);
Pattern application: “Si”
Pattern dimensions: X = 4 – 5 µm, Y = adjust as needed, and Z = 2.0 µm

21.1. Start live I-beam imaging and perform direct I-beam alignments.

21.2. Tilt the stage to relative +2°; take an I-beam snapshot, draw the indicated pattern (with X dimension 0.5 µm smaller than the last pattern executed at 30 kV), set pattern rotation to 180°, and position over the center of the top side of the lamella; adjust the Y dimension to cover the exposed top side and execute (shown below).



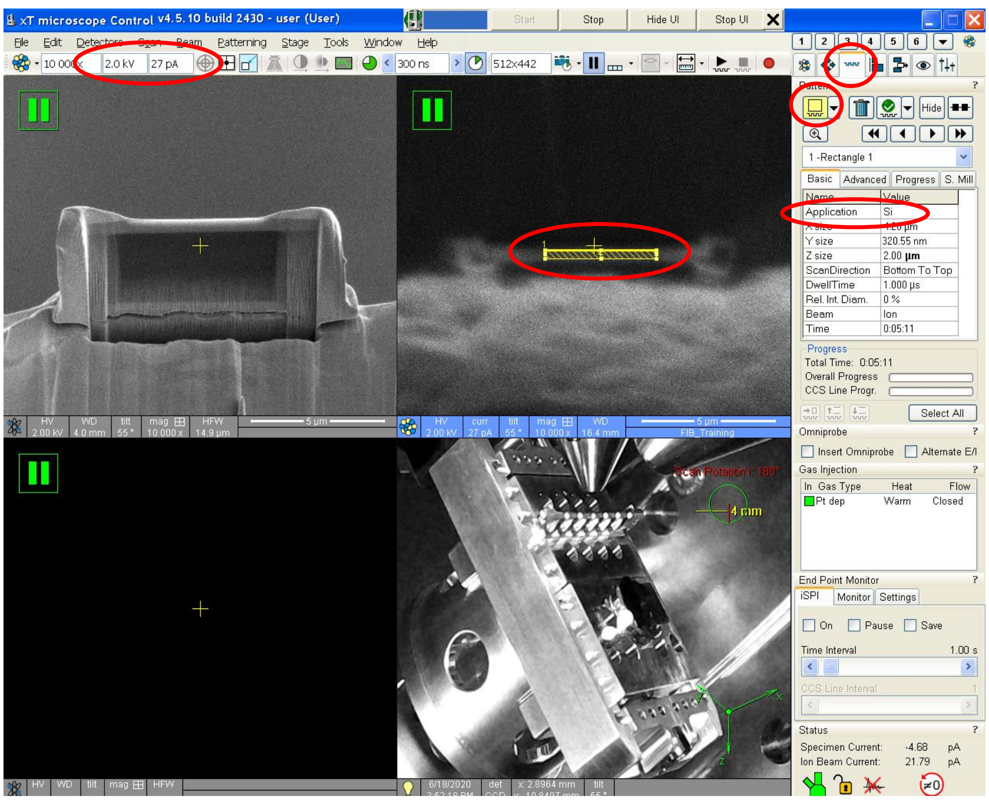
21.3. Tilt the stage to relative –2° and take an I-beam snapshot ; reduce the X dimension of the pattern by 0.5 µm set pattern rotation to 0°, and position over the center of the bottom side of the lamella; adjust the Y dimension to cover the exposed bottom side and execute (not shown).

22. Final thinning: 2 kV

Horizontal Field Width = ~15 µm
Stage tilt: ±4° from normal I-beam incidence
I-beam settings = 2 kV, 27 or 44 pA
Pattern type: rectangle
Pattern application: “Si”
Pattern dimensions: X = 3 – 4 µm, Y = adjust as needed, and Z = 0.5 µm

22.1. Start live I-beam imaging and perform direct I-beam alignments

22.2. Tilt the stage to relative +4°;take an I-beam snapshot, draw the indicated pattern (with X dimension 0.5 µm smaller than the last pattern executed at 5 kV) and position over the center of the top side of the lamella; adjust the Y dimension to cover the whole exposed top side and execute (shown below).



22.3. sTilt the stage to relative –4° and take an I-beam snapshot; reduce the X dimension of the pattern by another 0.5 µm and position over the center of the bottom side of the lamella; adjust the Y dimension to cover the whole exposed bottom side and execute (not shown); a few 10s of nm of E-beam Pt should remain on the final lamella (see example on next page).

22.4. Do not perform any additional I-beam imaging of the lamella; additionally, minimize (ideally, avoid if possible) further E-beam imaging.

Example of a finished Si lamella

A few 10s of nm of protective E-beam Pt is left on the surface, indicating the near surface region of the material is preserved (often, this is the area of interest on the specimen); the final usable area is ~3 µm wide and ~20 nm thick near the top (indicated in image) with the thickness gradually increasing towards the bottom of the lamella.