FEI Helios PFIB TEM specimen prep recipe

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This recipe is a composite of several lamella prep methods. It can produce lamellas ~20 nm thick with ~2 nm damage layers in both soft semiconductor materials. However, this recipe should by no means be considered universally applicable for all materials. This recipe uses Si as the example material and the associated “Si” pattern application file is calibrated for Si. If working on SiC, a calibrated “SiC” pattern application file also exists and should correspondingly be used. If no pattern application file exists for your material, you should choose “Si” as the default. You may also contact RSC staff and we will work with you to create an application file for your specific material (free of charge). Please also note, depending on the nature of your 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/recipes for specimen preparation exist for your 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 for stable imaging and patterning; 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 (or semiconductive), a conductive coating is not needed.

2. Lift-out grids and grid loading

    2.1. The grids for INLOs are made by Omniprobe or Ted Pella and are referred to as “lift-out” grids. Lift-out grids are manufactured from a few different materials and have 2 – 8 posts to provide points of attachment for samples. Mo is the preferred material for lift-out grids. Mo lift-out grids are very stiff, highly resistant to deformation and bending, and tend to redeposit less grid material onto the lamella during preparation.

    https://www.tedpella.com/SEMmisc_html/FIB_Supplies.aspx

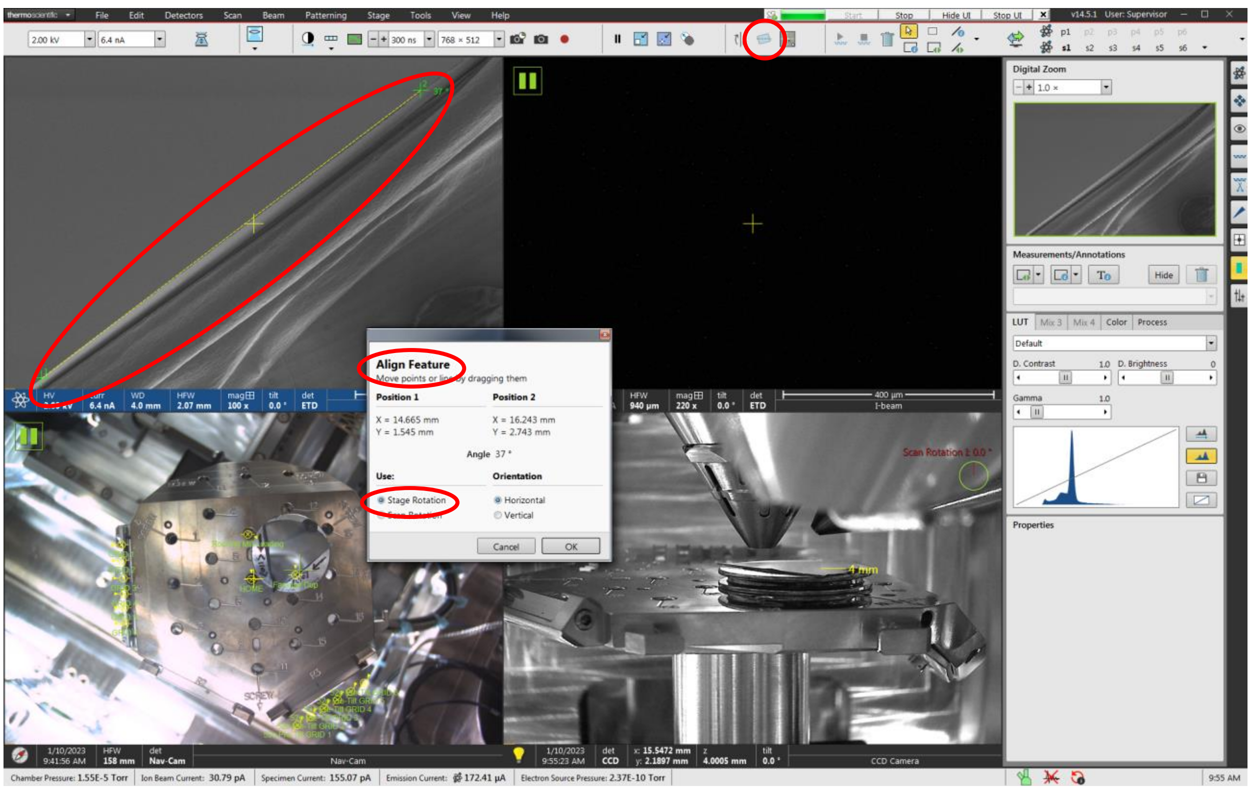
    2.2. Load a grid into the TEM row holder; after loading, inspect the grid visually from the side to make sure it is level in the holder; the grid should also be examined edge-on using an optical microscope to make sure it is straight and not bent/deformed. If the grid appears bent/deformed, it should be discarded and replaced.

3. Stage adjustments for the specimen

3.1. When the vacuum is sufficient, wake up the system 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 "Align Feature" from the toolbar, and follow the instructions in the dialogue box; when finished, the stage will rotate to align the feature as specified.

3.3. In the dialogue box, be sure to select "Stage Rotation"; <u>DO NOT select "Scan Rotation"</u>.



3.4. All subsequent steps of this recipe are performed assuming the following stipulations and conditions:

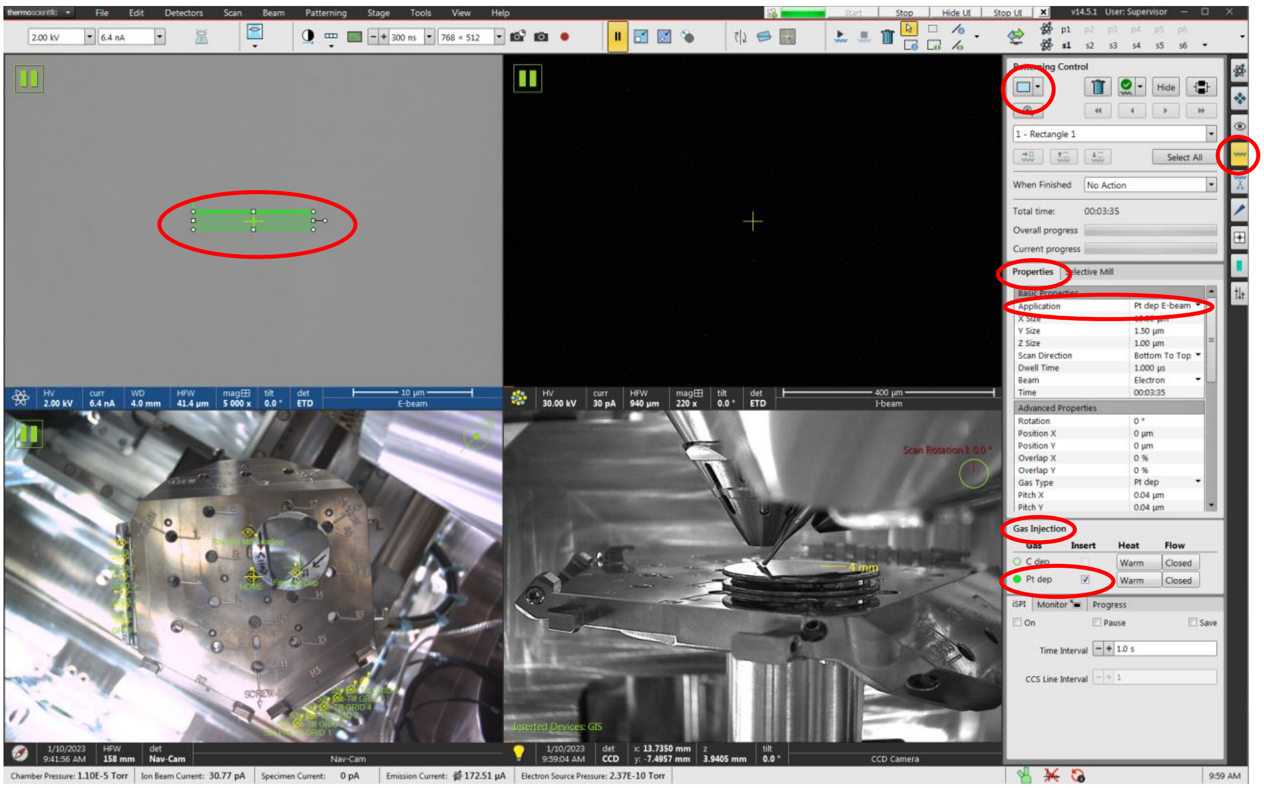
A. The working area is properly set at eucentric height as needed.
B. The beams are properly linked as needed.
C. The I-beam scan rotation is always set = 180°.
D. Milling steps with I-beam current >1 nA are performed using "blind" milling.

4. Protective E-beam Pt strap deposition

Horizontal Field Width = ~40 µm
Stage tilt: T = 0°
E-beam settings = 2 kV, 6.4 nA
Pattern type: rectangle
Pattern application: “Pt dep E-beam”
Pattern dimensions: X = 10, Y = 1.0, and Z = 1.0 µm

4.1. Perform direct alignments on the electron beam to obtain the best image possible (described elsewhere).

4.2. Insert the Pt GIS; take a snapshot; draw/position the indicated pattern; execute the pattern (shown below); retract the Pt GIS when finished.



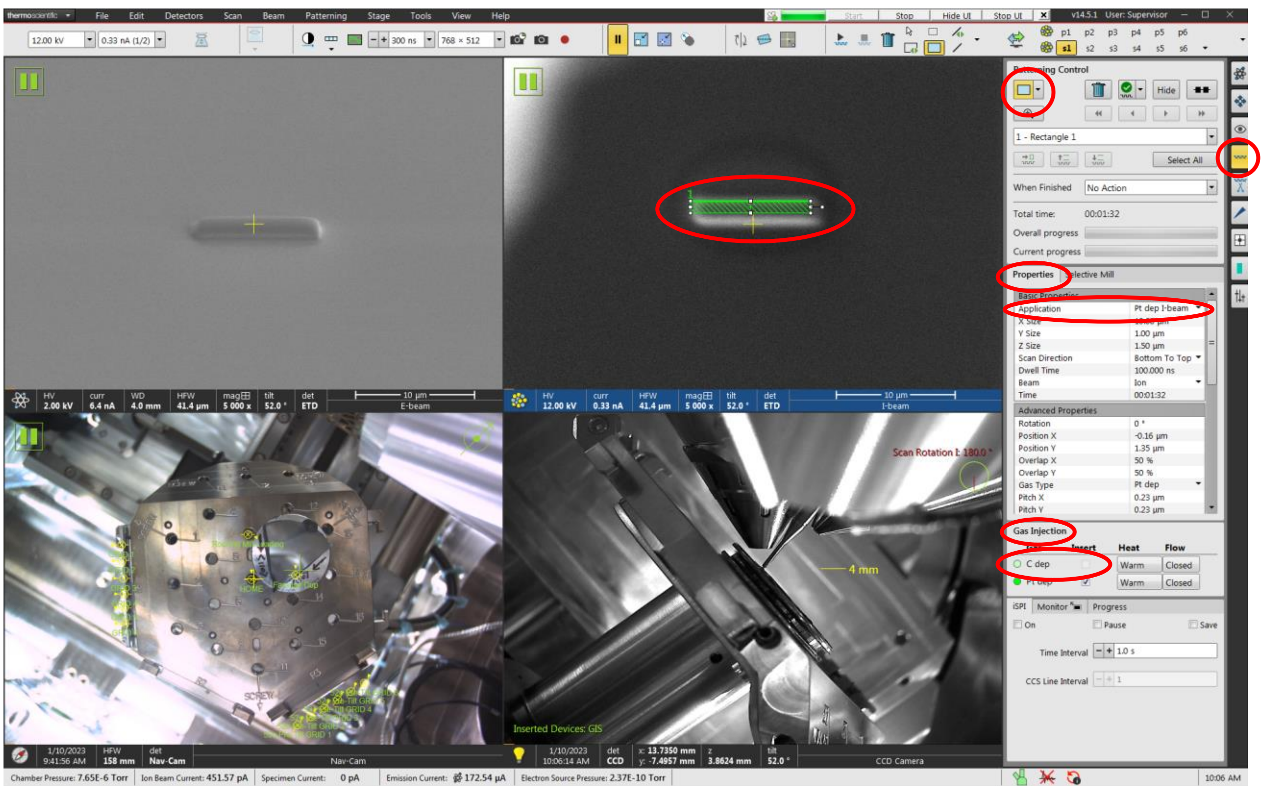
5. Protective I-beam Pt strap deposition

Horizontal Field Width = ~40 µm
Stage tilt: T = 52°
I-beam settings = 12 kV, 0.3 nA (~0.45 nA actual)
Pattern type: rectangle
Pattern application: “Pt dep I-beam”
Pattern dimensions: X = 10, Y = 1.0, and Z = 1.5 µm

5.1. Set protective E-beam Pt strap at eucentric height; set stage tilt T = 52°; link the beams.

5.2. Insert the Pt GIS; take I-beam snapshot; draw/position the indicated pattern; execute the pattern (shown below); retract the Pt GIS when finished.

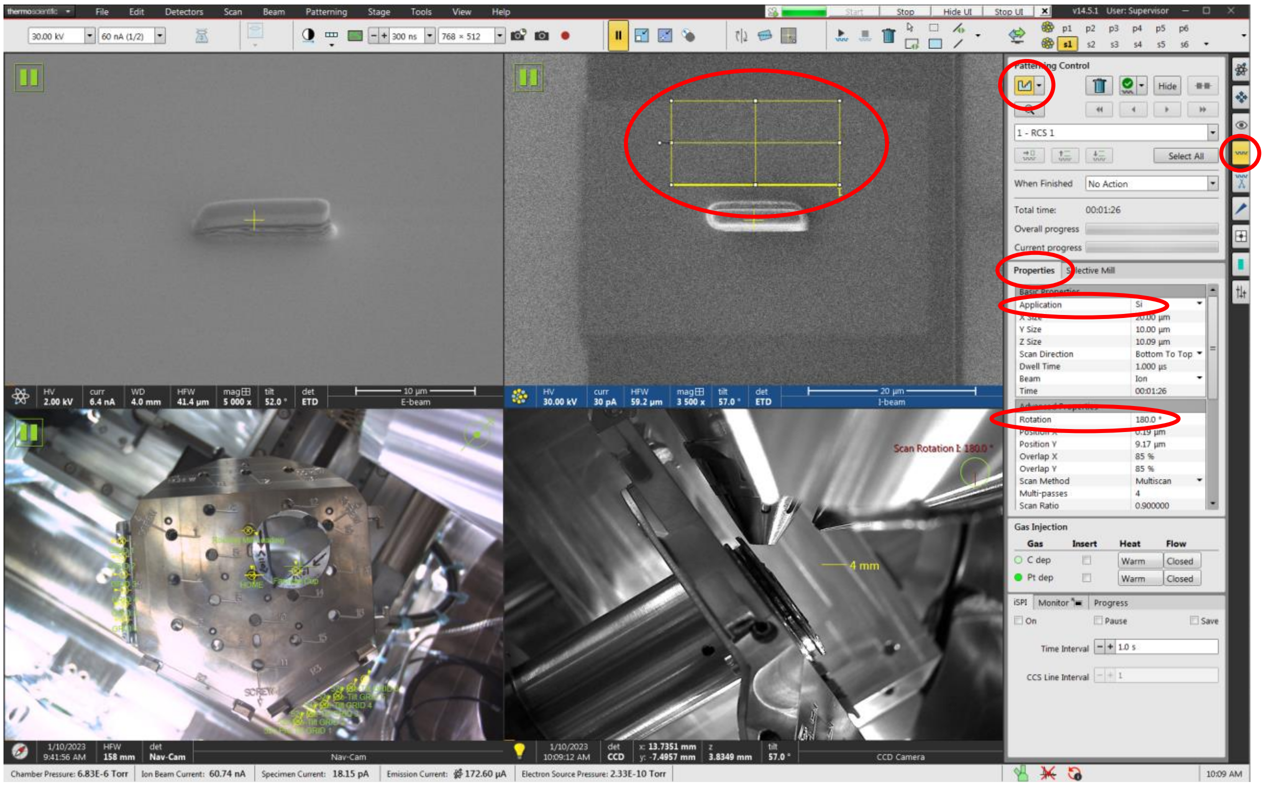
5.3. The total resulting protective Pt strap thickness after this step will now be ~2.5 µm thick.



6. Trenches

Horizontal Field Width = ~60 µm
Stage tilt: T = 52 ± 5°
I-beam settings = 30 kV, 60 nA
Pattern type: regular cross-section
Pattern rotation = 180° (at T = 57°); 0° (at T = 47°)
Pattern application: “Si”
Pattern dimensions: X = 20 Y = 10, and Z = 10 µm

6.1. Set stage tilt T = 57°; take low current I-beam snapshot; draw/position the indicated pattern (~2 µm *above* the protective Pt strap); set pattern rotation to 180°, execute the pattern (shown below).

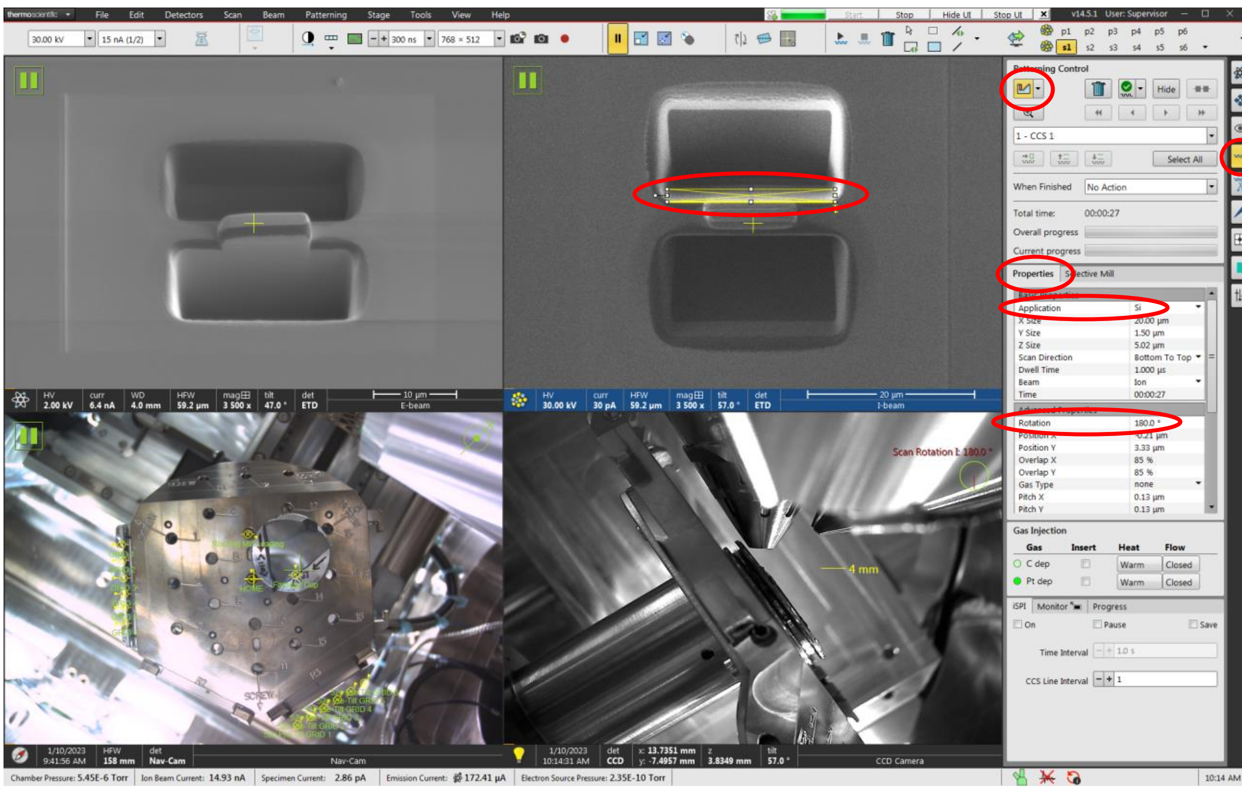


6.2. Set stage tilt T = 47°; take low current I-beam snapshot; draw/position the indicated pattern (~2 µm *below* the protective Pt strap); set pattern rotation to 0°, execute the pattern (not shown).

7. Coarse thinning

Horizontal Field Width = ~60 µm
Stage tilt: T = 52 ± 5°
I-beam settings = 30 kV, 15 nA
Pattern type: cleaning cross-section
Pattern rotation = 180° (at T = 57°); 0° (at T = 47°)
Pattern application: “Si”
Pattern dimensions: X = 20, Y = adjust as needed, and Z = 5 µm

7.1. Set stage tilt T = 57°; take low current I-beam snapshot; draw/position the indicated pattern (just *above* protective Pt strap); set pattern rotation to 180°; execute the pattern (shown below).



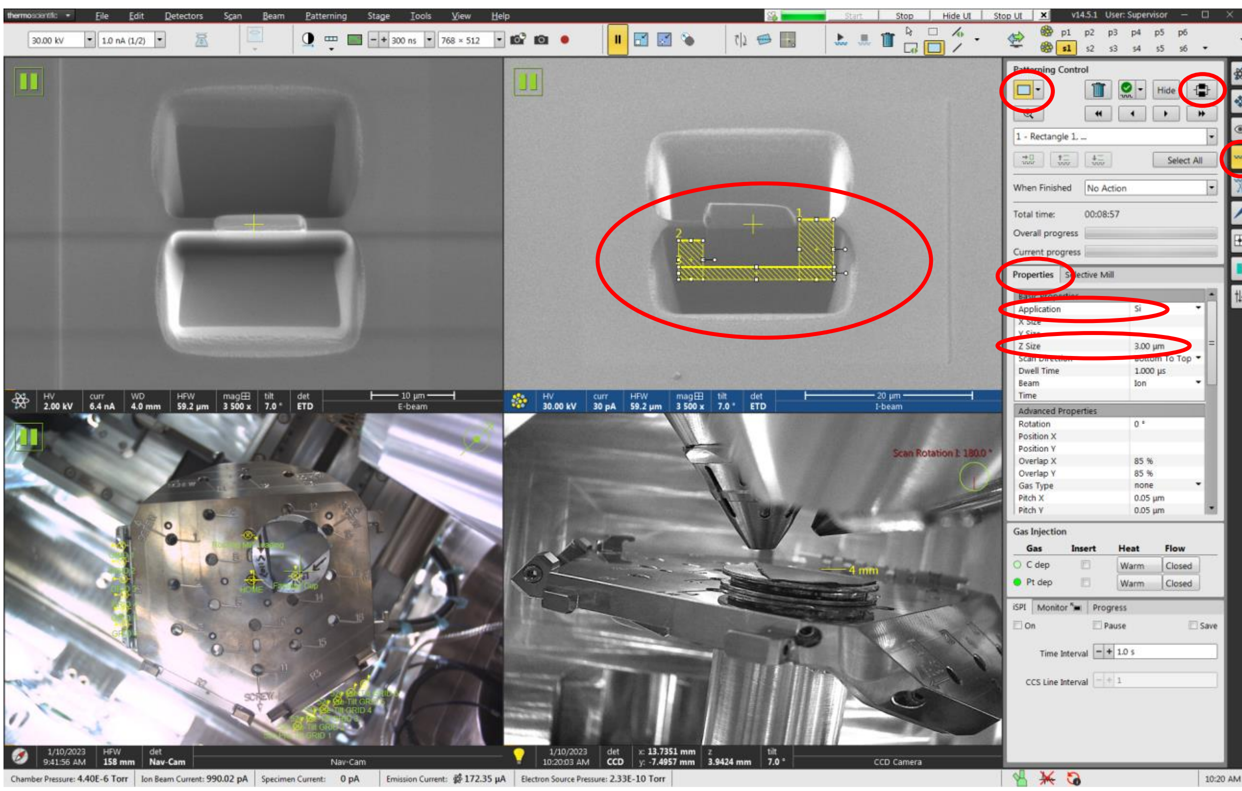
7.2. Set stage tilt T = 47°; take low current I-beam snapshot; draw/position the indicated pattern (just *below* protective Pt strap); set pattern rotation to 0°; execute the pattern (not shown).

8. J-cut (undercut)

Horizontal Field Width = ~60 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 = 10 µm
Pattern order: parallel

8.1. Set stage tilt T = 7°; shift the I-beam image as needed to bring the lamella back to the center of the image.

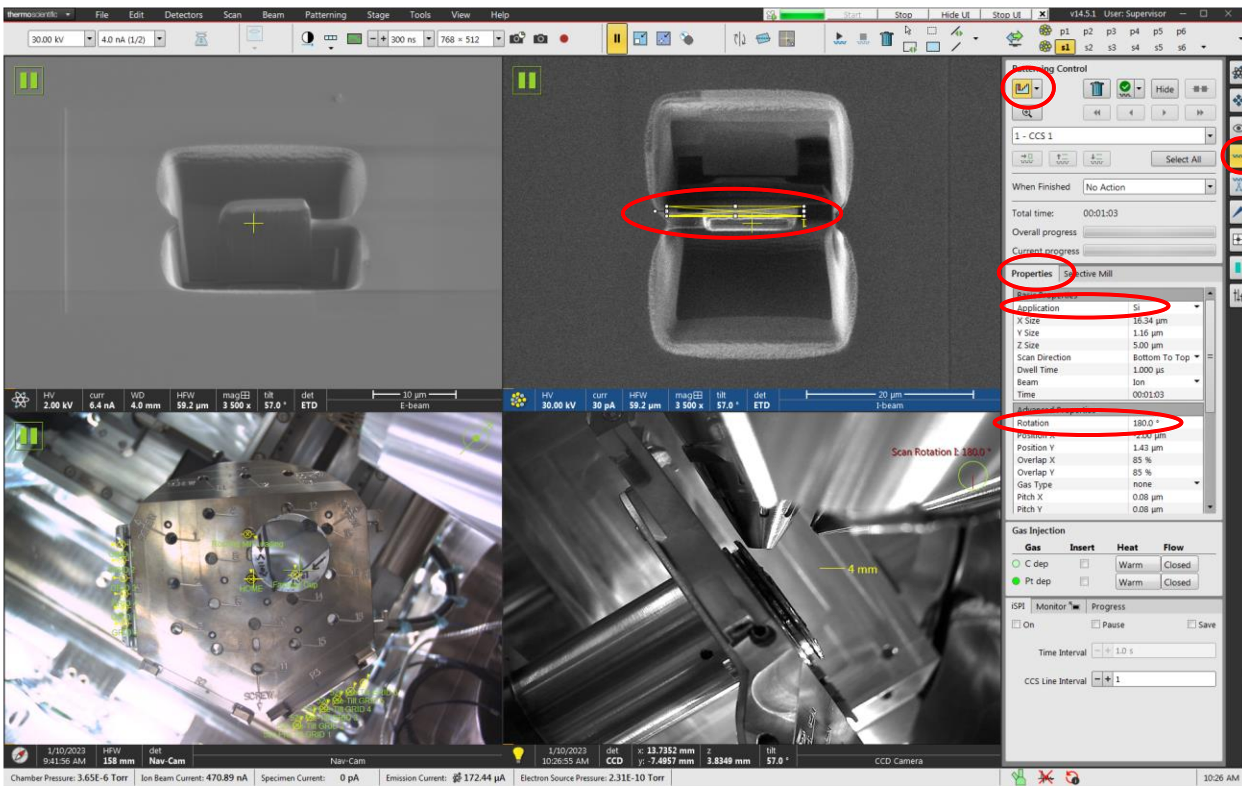
8.2. Take an I-beam snapshot; draw/position three rectangle patterns as shown in the image below (the width of each pattern should be >1.0 µm with complete overlap at the corners); execute the pattern (shown below); stop the pattern once undercutting is complete.



9. Post-undercut cleaning

Horizontal Field Width = ~60 µm
Stage tilt: T = 57°
I-beam settings = 30 kV, 4.0 nA
Pattern type: cleaning cross-section
Pattern rotation = 180°
Pattern application: “Si”
Pattern dimensions: X = adjust as needed, Y = adjust as needed, and Z = 5 µm

9.1. Set stage tilt T = 57°; take low current I-beam snapshot; draw/position the indicated pattern (just *above* protective Pt strap); set pattern rotation to 180°; execute the pattern (shown below).



10. Approaching the EasyLift

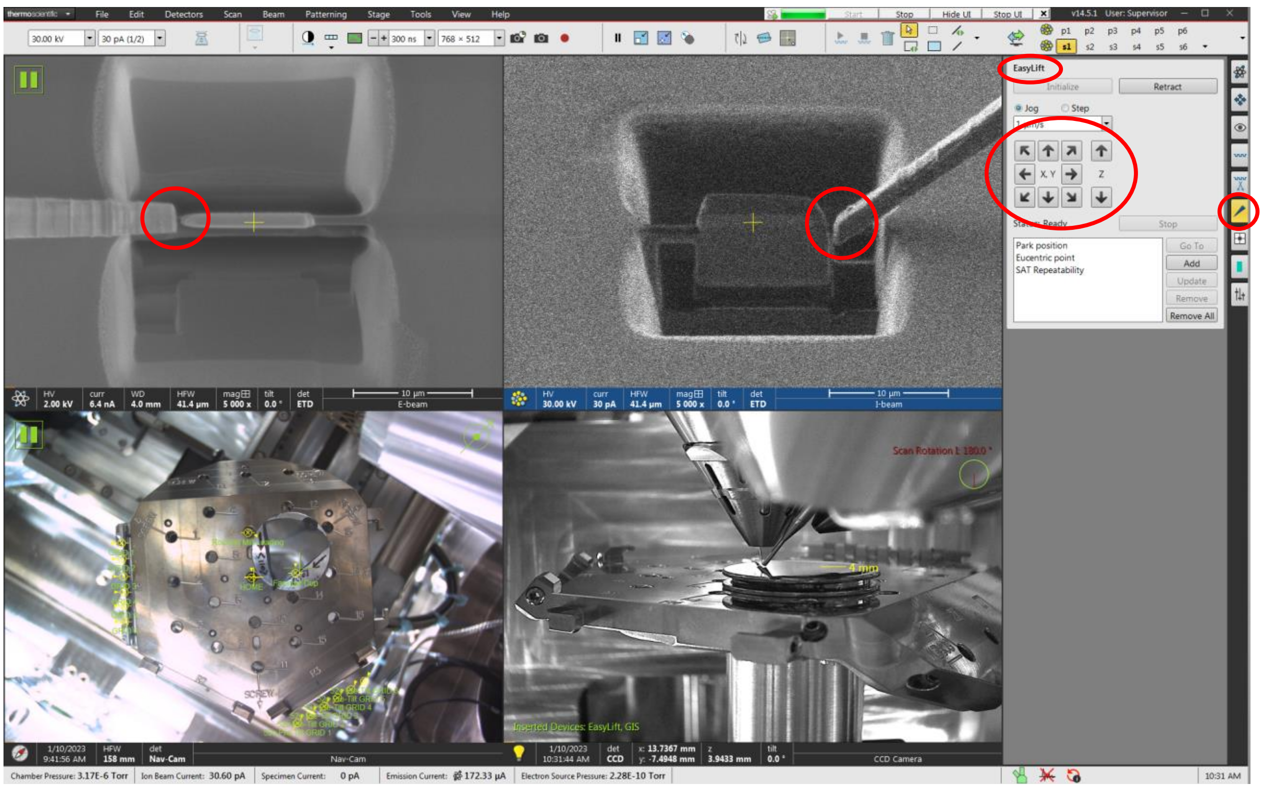
Horizontal Field Width = adjust as needed; final ~40 µm
Stage tilt: T = 0°
I-beam settings = 30 kV, 30 pA

10.1. Set stage tilt T = 57°; start live I-beam imaging; adjust the Y beam shift until the lamella is centered within the field of view.

10.2. Insert the EasyLift; use live E-beam and I-beam imaging to approach it to the lamella (not shown).

10.3. When the EasyLift is a few µm away from the lamella, insert the Pt GIS (not shown).

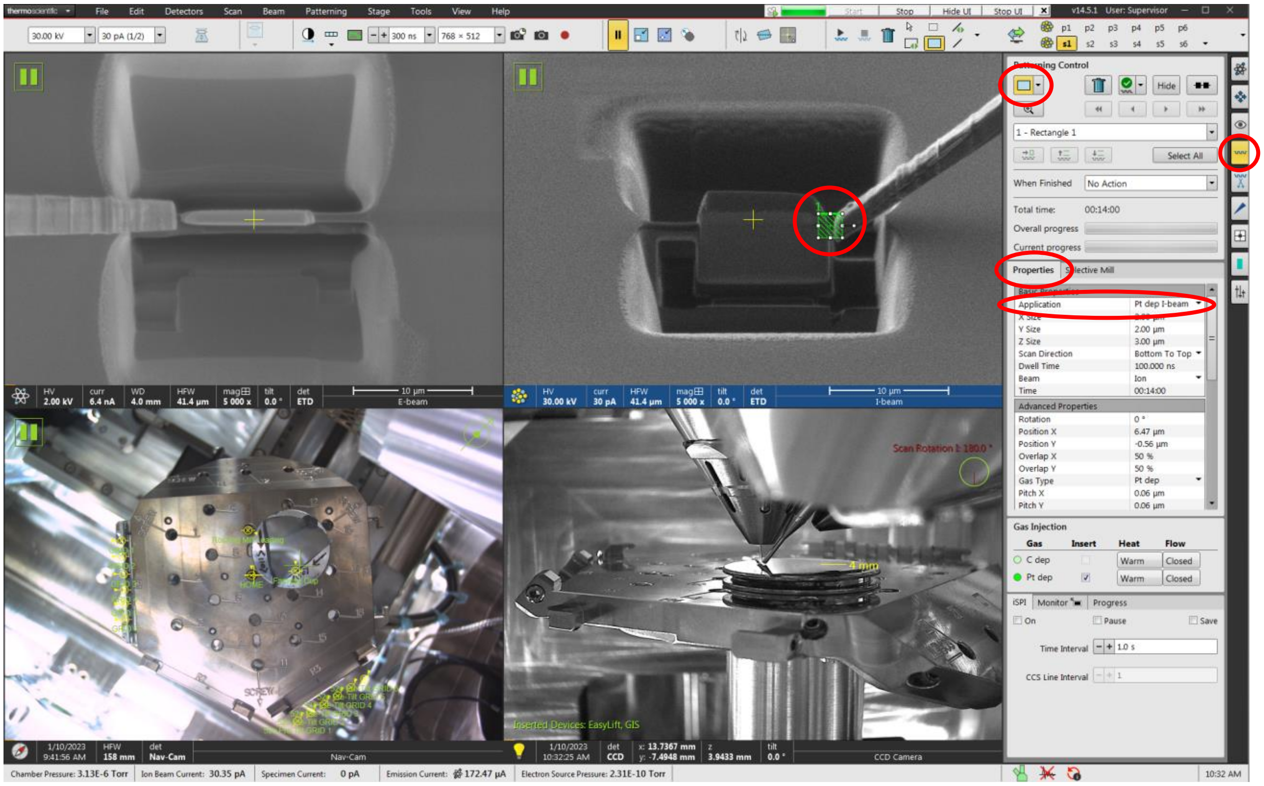
10.4. Position the EasyLift so it is centered next to and nearly touching the free edge of the lamella (shown below).



11. Attaching EasyLift to lamella

Horizontal Field Width = ~40 µm
Stage tilt: T = 0°
I-beam settings = 30 kV, 30 pA
Pattern type: rectangle
Pattern application: "Pt dep I-beam"
Pattern dimensions: X = 2, Y = 2, and Z = 1.0 µm

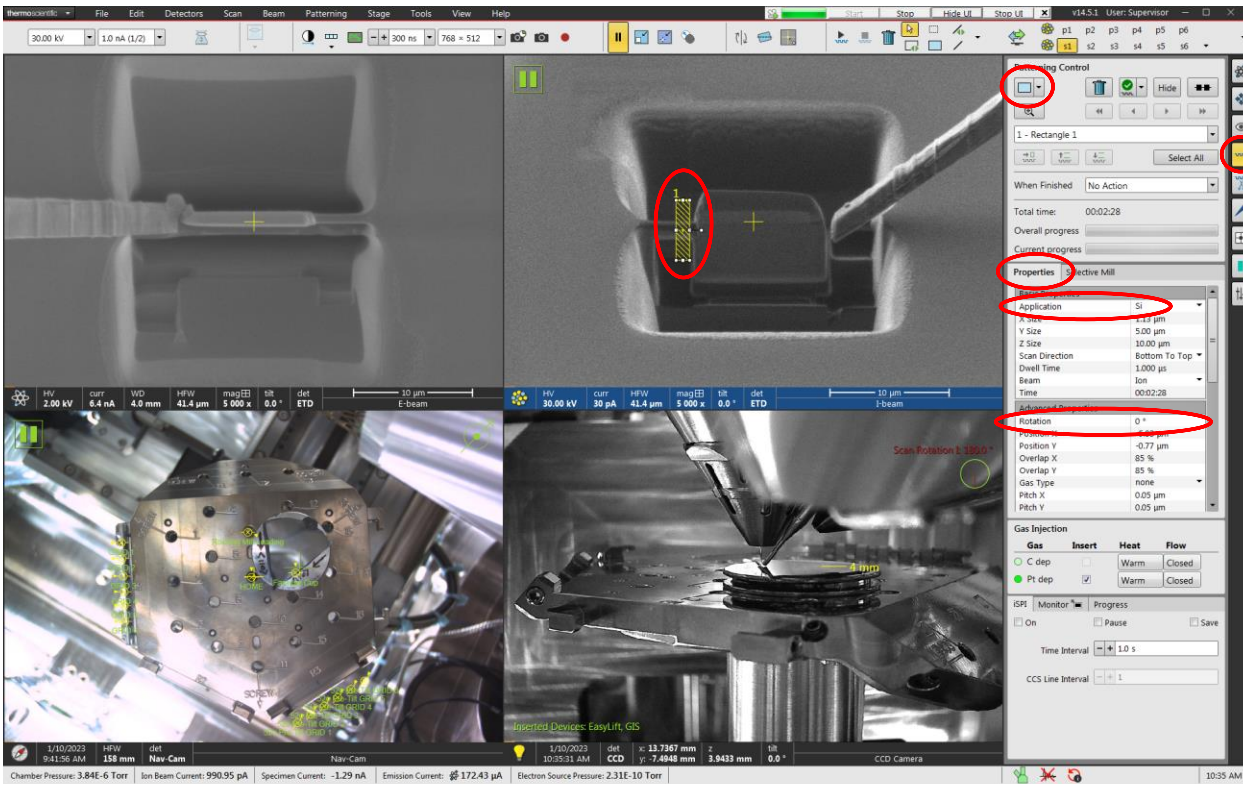
11.1. Take an I-beam snapshot; draw/position the indicated pattern; execute the pattern; stop the pattern once a sufficient weld is obtained.



12. Freeing the lamella

Horizontal Field Width = ~40 µm
Stage tilt: T = 0°
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 = 5.0 µm

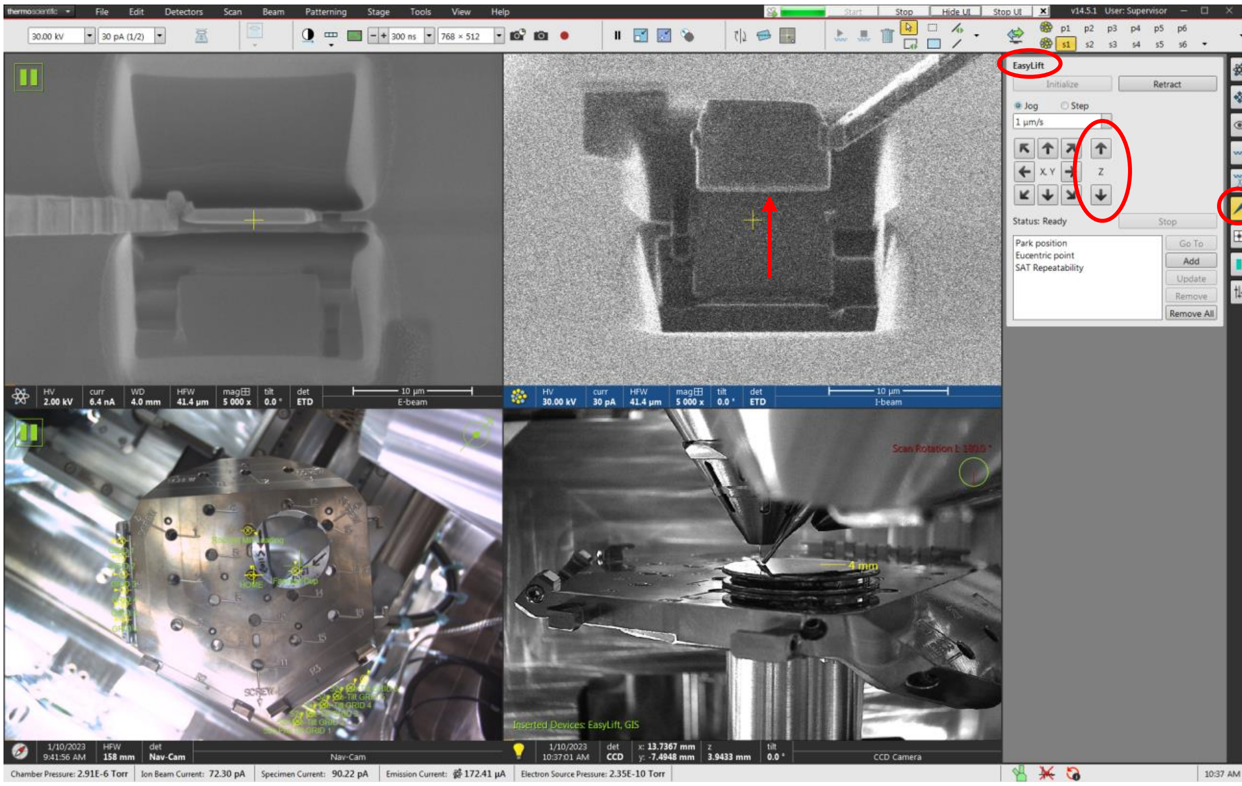
12.1. Take an I-beam snapshot (optional), draw/position the indicated pattern (adjust the X and Y dimensions to sufficiently remove the remaining point of attachment); execute the pattern; stop the pattern immediately once the lamella is released.



13. Lifting out the lamella

Horizontal Field Width = ~40 µm
Stage tilt: T = 0°
I-beam settings = 30 kV, 30 pA

13.1. Start live I-beam imaging; slowly move the EasyLift (with attached lamella) up and out of the trench (shown below).



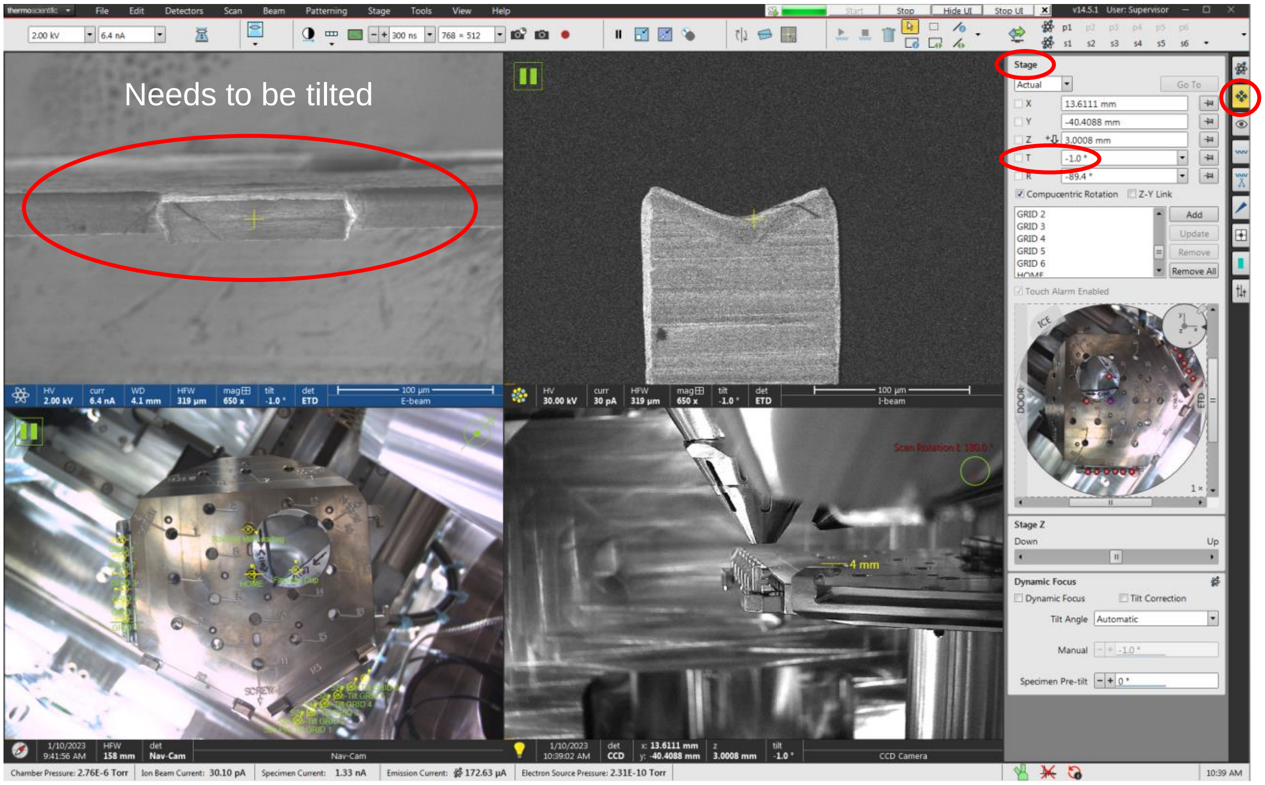
13.2. Once the lamella is ~10 µm clear of the specimen surface, place the EasyLift into the “Park” position and retract the EasyLift (not shown).

13.3. Once the EasyLift is retracted, retract the Pt GIS (not shown).

14. Grid alignment prior to lamella attachment

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

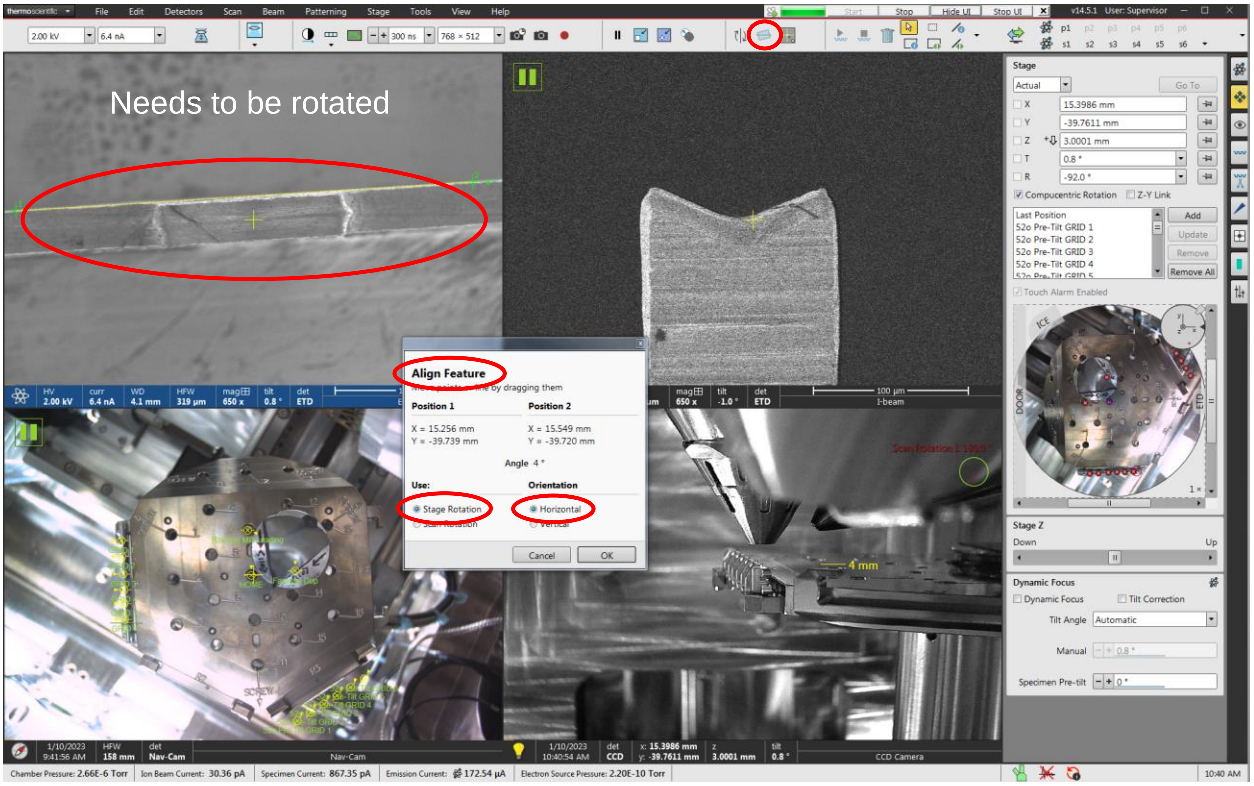
14.2. Using live E-beam imaging, adjust the stage tilt (usually –1° < T < 0°) until the grid is viewed perfectly edge on using the E-beam; if you must tilt to T < –2°, leave the tilt at T = –2° (EasyLift and Pt GIS cannot otherwise be inserted).



14.3. NOTE: the stage tilt for normal incidence for each beam will now 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 “Align Feature” from the toolbar and follow the instructions in the dialogue box to horizontally align the grid.

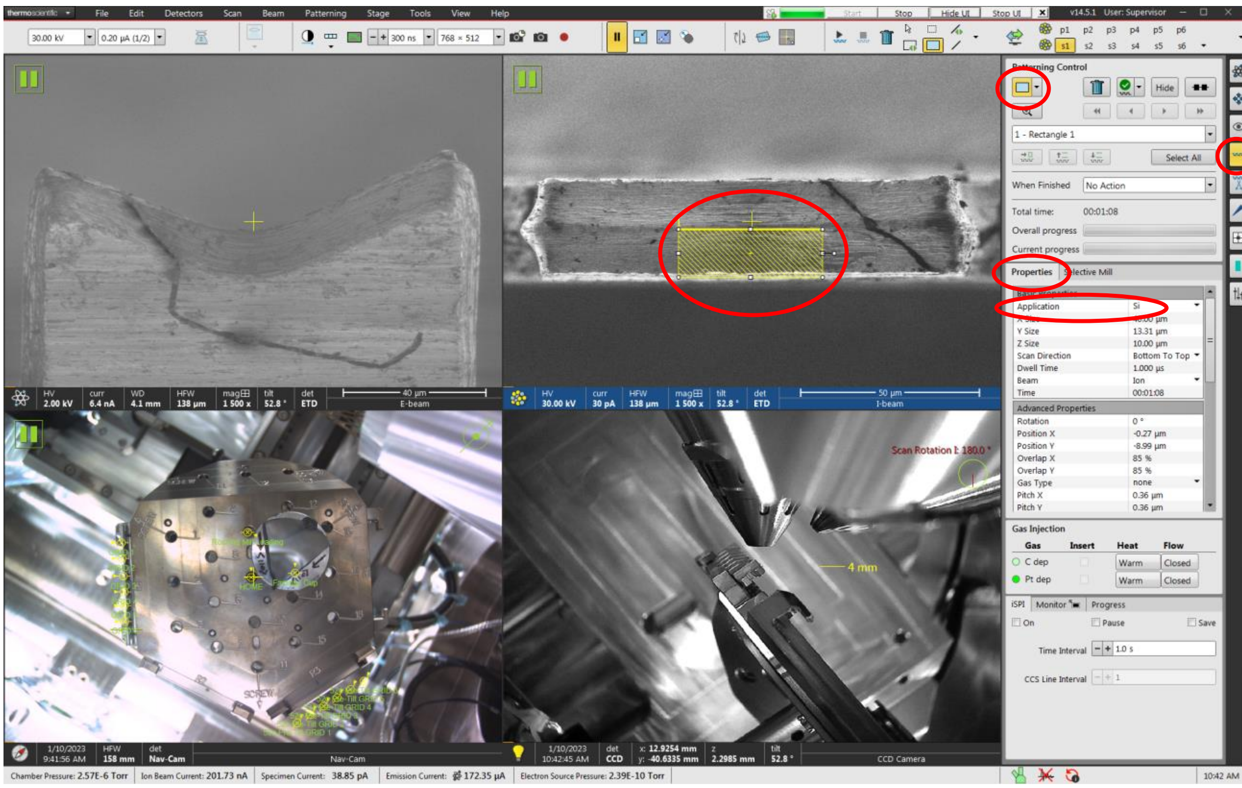
14.5. In the dialogue box, be sure to select “Stage Rotation”; DO NOT select “Scan Rotation”.



15. Removing frontside post material (if needed)

Horizontal Field Width = ~140 µm
Stage tilt: normal incidence for I-beam
I-beam settings = 30 kV, 0.2 µA
Pattern type: rectangle
Pattern application: “Si”
Pattern dimensions: X = 30 – 40, Y = ~half grid thickness, and Z = 10 µm

15.1. Take a low current I-beam snapshot, draw/position the indicated pattern (in the middle of the bottom half of the grid), execute the pattern (shown below).



15.2. This step ensures a clear line of sight for the lamella when performing TEM imaging.

16. Milling a shelf in the post

Horizontal Field Width = ~140 µm
Stage tilt: normal incidence for E-beam
I-beam settings = 30 kV, 60 nA or 0.2 µA
Pattern type: rectangle
Pattern application: “Si”
Pattern dimensions: X = 30 – 40, Y = adjust as needed, and Z = 20 µm

16.1. 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); execute the pattern (stop and manually adjust pattern dimensions and restart as necessary).

16.2. The resulting shelf should be basically flat in the middle as verified using the E-beam (not shown).

17. Attaching the lamella to the grid

Horizontal Field Width = ~40 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 = 2, Y = 2, and Z = 1.0 µm

17.1. Insert the EasyLift and use live E-beam and I-beam imaging to approach the lamella to the grid.

17.2. When the lamella is a few µm away from the grid, insert the Pt GIS; continue approaching the lamella and position so it is nearly sitting on the front edge of the shelf (use both beams to verify the position).

17.3. Take an I-beam snapshot; draw/position the indicated pattern (at the point of contact between one corner of the lamella and the grid);execute the pattern (shown below); the pattern may be stopped once sufficient Pt build up at the corner is obtained.

17.4. Repeat the previous step for the other corner of the lamella (not shown).

18. EasyLift release

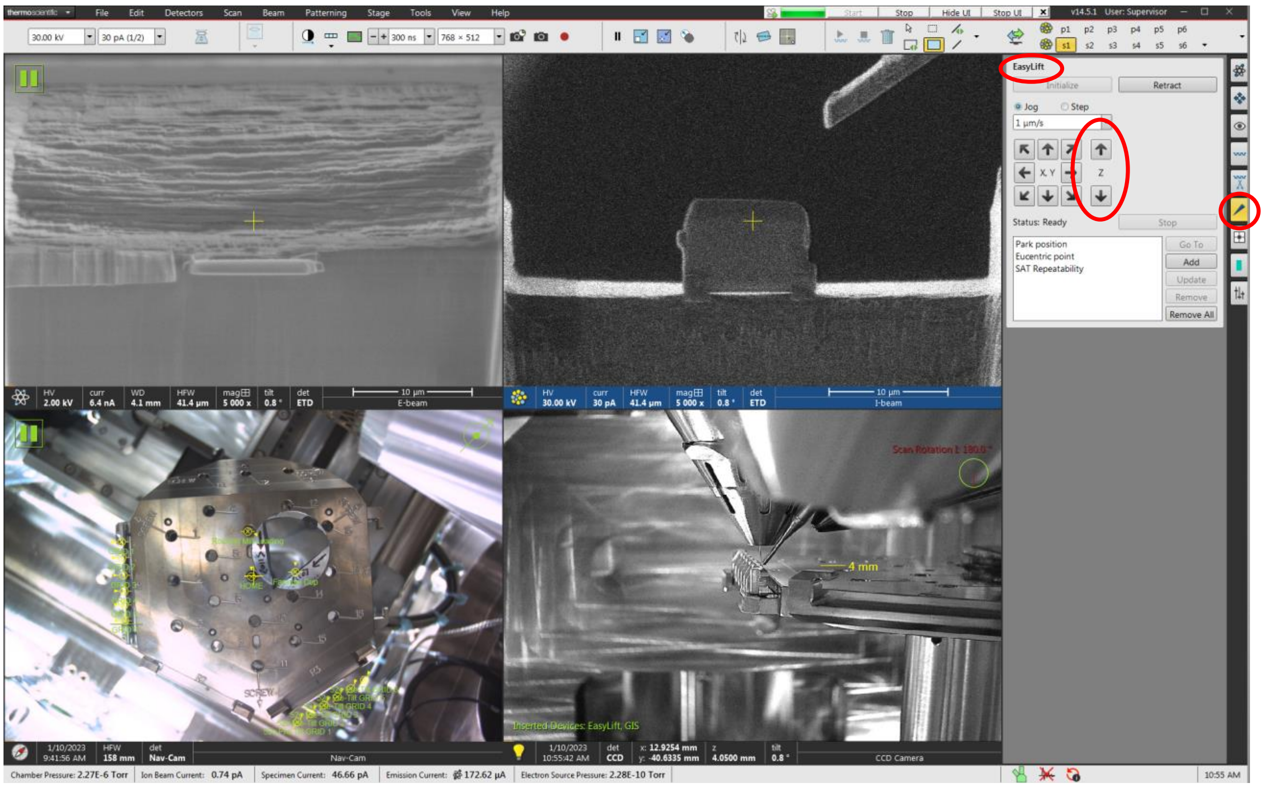
Horizontal Field Width = ~40 µm
Stage tilt: normal incidence for E-beam
I-beam settings = 30 kV, 1.0 nA
Pattern type: rectangle
Pattern application: “Si”
Pattern dimensions: X = ≥1, Y = adjust as needed, and Z = 10 µm

18.1. Take an I-beam snapshot; draw/position the indicated pattern (adjust the Y dimension to sufficiently exceed the thickness of the EasyLift; execute the pattern; stop the pattern once the EasyLift is released and re-sharpened.

19. EasyLift and Pt GIS retraction

Horizontal Field Width = ~40 µm
Stage tilt: normal incidence for E-beam
I-beam settings = 30 kV, 30 pA

19.1. Start live I-beam imaging; slowly move the EasyLift up and away from the lamella (shown below).



19.2. Once the EasyLift is ~10 µm clear of the lamella, place the EasyLift into the “Park” position and retract (not shown).

19.3. Once the EasyLift is retracted, retract the Pt GIS (not shown).

19.4. If there are additional lamellas to prepare, it is best to lift out and attach each lamella to the grid and then final thin all lamellas.

20. E-beam imaging during final thinning

20.1. Reduce the E-beam current (50 – 200 pA) prior to starting final thinning; be sure to perform direct E-beam alignments after changing currents.

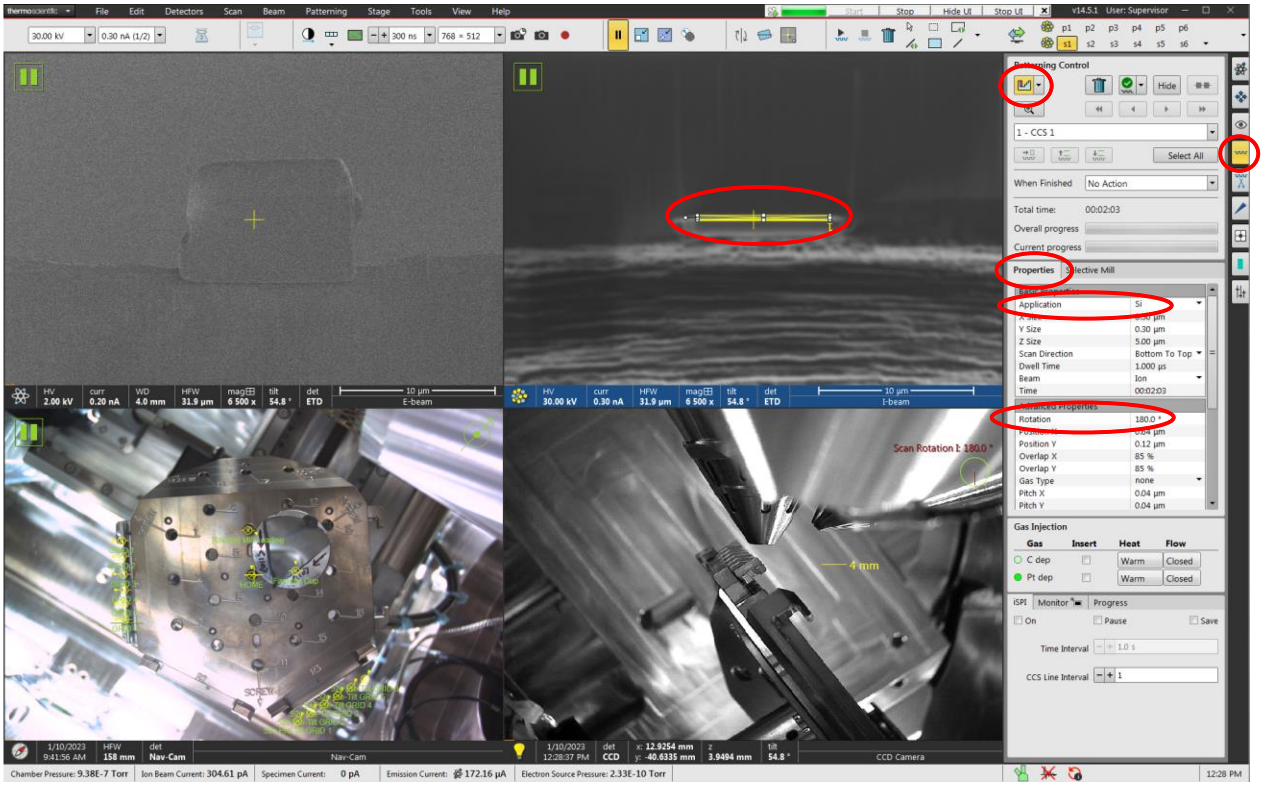
20.2. Constantly E-beam image the lamella while final thinning to monitor the progress of each pattern (and to possibly prevent over-milling).

21. Final thinning: 30 kV

Horizontal Field Width = ~30 µm
Stage tilt: ±1.5° (from normal I-beam incidence)
I-beam settings = 30 kV: 1.0, 0.3, and 0.1 nA
Pattern type: cleaning cross-section
Pattern rotation = 180° (+1.5° from normal incidence); 0° (–1.5° from normal incidence)
Pattern application: “Si”
Pattern dimensions: X = 9 µm (starting) , Y = adjust as needed, and Z = 4 – 5 µm

21.1. If present lamella thickness is >2 µm, start final thinning with 1.0 nA; otherwise, start final thinning with 0.3 nA; stigmate/focus the live I-beam image.

21.2. Tilt the stage to relative +1.5°; take an I-beam snapshot; draw/position the indicated pattern centered and *above* the lamella; set pattern rotation to 180°; manually adjust the Y dimension to cover the projected top sidewall; execute the pattern (shown below).



21.3. Tilt the stage to relative –1.5°; take an I-beam snapshot; reduce the X pattern dimension by 1 µm and position the indicated pattern centered and *below* the lamella; set pattern rotation to 0°, manually adjust the Y dimension to cover the projected bottom sidewall; execute the pattern.

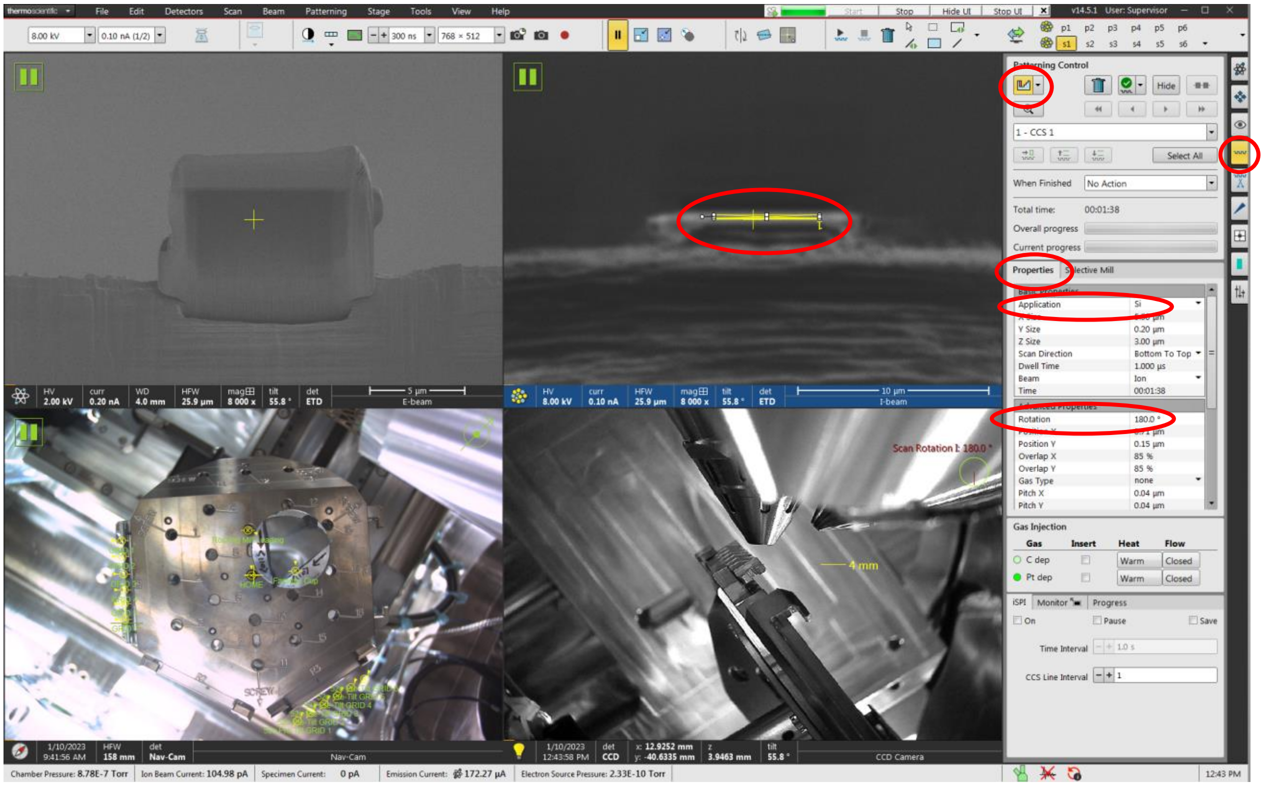
21.4. Repeat the previous two steps at the next lowest I-beam current, each step further reducing the X dimension of the pattern by 1 μm (not shown).

22. Final thinning: 8 kV

Horizontal Field Width = ~30 µm
Stage tilt: ±3.0° from normal I-beam incidence
I-beam settings = 8 kV, 0.1 nA
Pattern type: cleaning cross-section
Pattern rotation = 180° (+3.0° from normal incidence); 0° (–3.0° from normal incidence);
Pattern application: “Si”
Pattern dimensions: X = adjust as needed, Y = adjust as needed, and Z = 3 µm

22.1. Start live I-beam imaging; perform direct I-beam alignments and stigmate/focus to optimize the image (not shown).

22.2. Tilt the stage to relative +3.0°; take an I-beam snapshot, draw/position the indicated pattern centered and *above* the lamella (X dimension 0.5 µm smaller than last pattern executed), set pattern rotation to 180°; adjust the Y dimension to cover the projected top sidewall; execute the pattern (shown below).



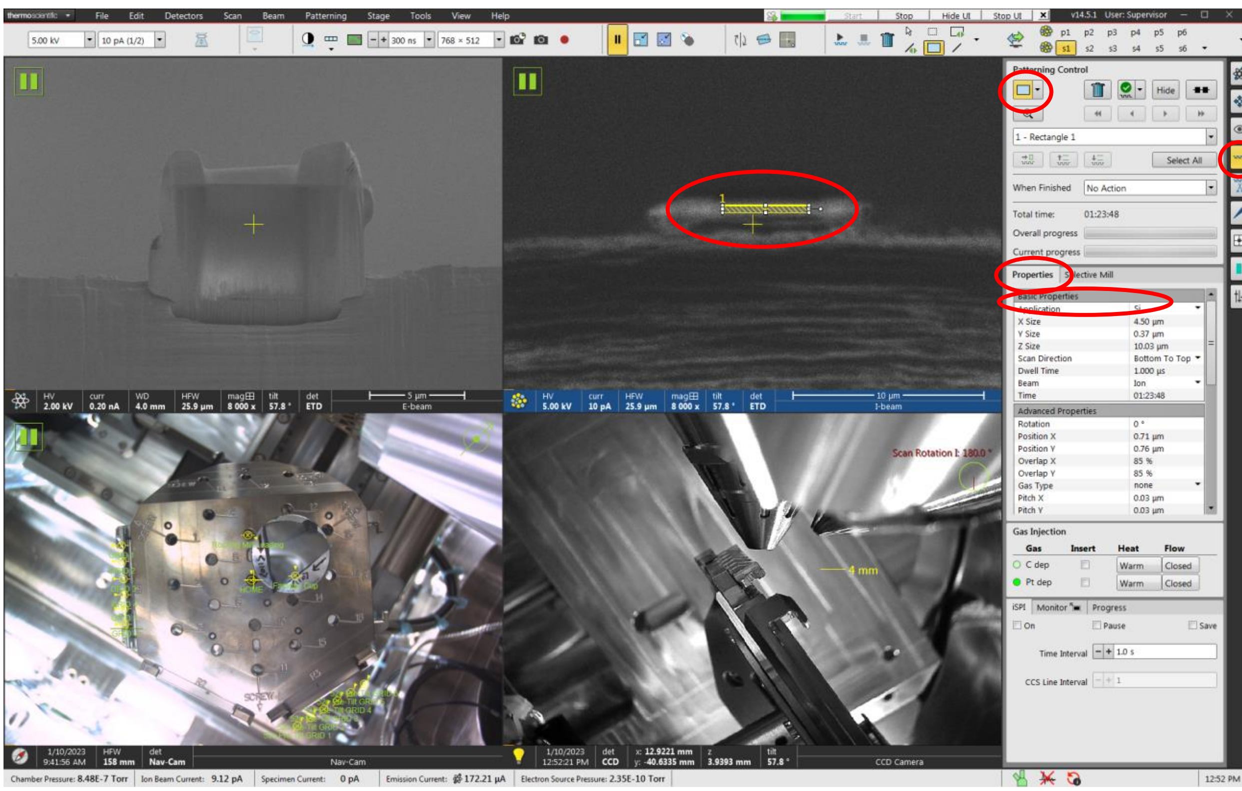
22.3. Tilt the stage to relative –3.0°; take an I-beam snapshot, draw/position the indicated pattern centered and *below* the lamella (X dimension 0.5 µm smaller than last pattern executed), set pattern rotation to 0°; adjust the Y dimension to cover the projected bottom sidewall; execute the pattern (not shown); sufficient protective Pt should remain after this step.

23. Final thinning: 5 kV

Horizontal Field Width = ~30 µm
Stage tilt: ±5° from normal I-beam incidence
I-beam settings = 5 kV, 10 pA
Pattern type: rectangle
Pattern application: “Si”
Pattern dimensions: X = adjust as needed, Y = adjust as needed, and Z = > 1 µm

23.1. Start live I-beam imaging and perform direct I-beam alignments and stigmate/focus to optimize the image (not shown).

23.2. Tilt the stage to relative +5°; take an I-beam snapshot; draw/position the indicated pattern centered and *above* the lamella (X dimension 0.5 µm smaller than last pattern executed); adjust the Y dimension to cover the projected top sidewall; execute the pattern (shown below); stop the pattern once the lamella appearance noticeably changes.



23.3. Tilt the stage to relative –5°; take an I-beam snapshot; draw/position the indicated pattern centered and *below* the lamella (X dimension 0.5 µm smaller than last pattern executed); adjust the Y dimension to cover the projected bottom sidewall; execute the pattern (not shown); stop the pattern when the lamella is satisfactory (some protective Pt should remain).

Example of a finished lamella (Si substrate with Cr thin film)

A think layer 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 ~4 µm wide and ~20 nm thick near the top (indicated in image) with the thickness gradually increasing towards the bottom of the lamella.