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Focused-Ion Beam (FIB) Transmission Electron Microscopy (TEM) Sample Preparation

by Wendy L. Sarney, B. Chad Hornbuckle, and Asher C. Leff

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14. ABSTRACT This report explains the basics of focused-ion beam (FIB) sample preparation, and its advantages for fabricating certain types of materials and device structures for scanning transmission electron microscopy (TEM). The FIB used is the Thermo–Fischer Helios 5 at Aberdeen Proving Ground, Maryland. This report has detailed schematic illustrations, pictures, and instructions of the complex steps needed for making high-quality TEM samples with FIB.					
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Contents

List of Figures	v
1. Introduction	1
1.1 Transmission Electron Microscopy Research and Sample Preparation at ARL	1
1.2 Brief Introduction to the FIB Technique	4
1.3 Pre-FIB Sample Preparation	5
1.4 Mount and Load Grids and Samples	6
2. FIB Geometry and Computer Interface	8
2.1 FIB Geometry	8
2.2 Computer Interface	9
3. Startup Procedure	11
4. Align the SEM image	15
5. Platinum (Pt) Deposition and Initial Milling	16
5.1 Pt Deposition	16
5.1.1 e^- Beam Pt Deposition	16
5.1.2 Tilt for i^+ Beam Pt Deposition: Set the Eucentric Height	17
5.1.3 i^+ Beam Pt Deposition	18
5.2 Mill, Planarize, and Undercut Sample	20
5.2.1 Mill 1st (Lower) Trench	20
5.2.2 Mill 2nd (Upper) Trench	20
5.2.3 Mill Side Trench	21
5.2.4 Planarize the Front Face	21
5.2.5 Planarize the Back Face	22
5.2.6 Tilt and Mill for Undercuts	23
6. Lift-Out Procedure	24
6.1 Attach Probe to Sample	24

6.1.1	Insert the Lift-Out Probe	24
6.1.2	Attach the Probe with Pt Deposition	26
6.2	Detach Sample	27
6.2.1	Cut Right Side of Lift-Out to Detach	27
6.2.2	Lift the Sample Out of The Trench	28
6.2.3	Retract the Probe	28
6.3	Attach the Sample to the Grid	28
6.3.1	Move to Grid Location	28
6.3.2	Clean the Mo Post Edge with the i^+ Beam	30
6.3.3	Bring Lift-Out to the Position Next to the Post	30
6.3.4	Attach Lift-Out to Post using i^+ Beam Deposition	32
6.3.5	Cut the Lift-Out Free from the Probe	32
7.	Final Thinning	33
7.1	Planarize Sample	33
7.1.1	Tilt to Front Face Milling Angle and Planarize	33
7.1.2	Rotate 180° and Tilt to Reversed Face Milling Angle, Planarize	35
7.2	Lower-Energy Thinning	37
7.2.1	Thin Reversed Face at 5 kV	37
7.2.2	Rotate 180° and Tilt to Front Face Milling Angle	38
7.2.3	Thin the Front Face at 5 kV	39
7.2.4	Clean Front Face at 2 kV	39
7.2.5	Tilt and Clean Back Face at 2 kV	40
8.	Finished Sample and Shutdown	41
8.1	Finished Sample	41
8.2	Shutdown	41
9.	Summary/Conclusion	41
10.	References	42
	List of Symbols, Abbreviations, and Acronyms	47
	Distribution List	49

List of Figures

Figure 1.	Device structure prepared by FIB and imaged with high-angle annular dark-field STEM.	3
Figure 2.	Bi on WSe ₂ flakes on an oxidized Si substrate. (a) Optical microscope image, and (b) cross-sectional TEM image of an FIB lift-out taken from the largest flake seen in (a).....	4
Figure 3.	Sputter coating (a) stub mount, (b) Leica EM ACE600 Sputter Coater, (c) control touch screen, and (d) manual.	6
Figure 4.	FIB grids and clamping arrangement (a) Mo three-post Omniprobe grid, (b) TEM FIB grid clamp, (c) side and (d) top-down view of grid clamp.....	7
Figure 5.	Four-vane multi-stub holder. (a) Top view, (b) side view showing adjustable height piston, and (c) side view showing the height reference tool.	7
Figure 6.	Geometry of FIB beams and sample. (a) e ⁻ and (b) i ⁺ beam images of a sample undergoing FIB, and (c–f) schematics from multiple perspectives to show the geometry of the two beams and sample stage.	9
Figure 7.	Main display for FIB showing the e ⁻ and i ⁺ beam images, the Nav-Cam view, and the chamber camera.	10
Figure 8.	Labeled main and right-hand menu bar.	10
Figure 9.	Menus needed for loading the sample stage: (a) navigation pane, (b) sample exchange window, (c) vacuum control, and (d) vacuum indicator.	11
Figure 10.	Initial beam conditions (a) scan rotation and (b) beam shift.	12
Figure 11.	Detector indicator window.....	13
Figure 12.	More initial beam settings (a) live image selection/averaging deselection, (b) magnification coupling, and (c) set i ⁺ beam dwell time.	14
Figure 13.	(a) Wake up the beam, and (b) take a Nav-Cam image.	14
Figure 14.	Chamber camera view is live.....	15
Figure 15.	Schematic of the (a) geometry and the (b) e ⁻ beam image after Pt deposition.....	16
Figure 16.	Adjust the eucentric height. (a) instrument geometry schematic with accompanying (a) e ⁻ and (b) i ⁺ beam images.....	18
Figure 17.	(a) e ⁻ and (b) i ⁺ beam images after Pt deposition.....	19
Figure 18.	(a) e ⁻ and (b) i ⁺ beam images after the first trench dug.	20
Figure 19.	(a) e ⁻ and (b) i ⁺ beam images after the second trench dug.....	21

Figure 20. (a) e^- and (b) i^+ beam images after side trench dug.	21
Figure 21. (a) Geometry during front face planarization. (b) e^- and (c) i^+ beam images after the side trench is constructed.	22
Figure 22. (a) geometry during and (b) i^+ beam image after the backside face planarization.	23
Figure 23. Undercut (a) geometry, (b) e^- beam image, and (c) i^+ beam image. The red arrows point to the sample's backside. It is just barely visible in (b) due to the -5° stage tilt. Do not cut where the orange arrow is pointing in (c).	23
Figure 24. (a) The undercut indicated by the red arrow in the e^- beam image should connect all the way across the bottom of the trench. (b) The sample should clearly be completely detached everywhere in the i^+ beam image after undercutting except at the point shown by the orange arrow.	24
Figure 25. EasyLift control window.	25
Figure 26. (a) Sample geometry during lift-out probe insertion, (b) e^- beam image, and (c) the i^+ beam image after lift-out probe insertion.	26
Figure 27. (a) e^- beam image and (b) i^+ beam image after probe attachment with Pt.	27
Figure 28. (a) e^- beam image and (b) i^+ beam image after cutting right side of lift-out.	27
Figure 29. e^- beam image after the sample is lifted out of the trench.	28
Figure 30. Trench after we lift out the sample (a) e^- beam image and (b) i^+ beam image.	28
Figure 31. (a) Sample geometry and (b) e^- beam image and (c) i^+ beam image of the grid posts with the peaks and valleys labeled.	29
Figure 32. Higher magnification images of the (a) e^- beam image and (b) i^+ beam image of the selected grid post. The i^+ beam image is upside down, so move Z opposite of the direction that you want.	30
Figure 33. (a) e^- beam image and (b) i^+ beam image after cleaning the post edge.	30
Figure 34. (a) e^- beam image and (b) i^+ beam image of the probe holding the lift-out as it approaches the post.	31
Figure 35. (a) e^- beam image and (b) i^+ beam image attaching the lift-out to the post.	32
Figure 36. (a) e^- beam image and (b) i^+ beam image after freeing the probe.	33
Figure 37. (a) e^- beam image and (b) i^+ beam image after tilting to the front face milling angle.	34
Figure 38. (a) e^- beam image and (b) i^+ beam image after planarizing and thinning the front face.	35

Figure 39. (a) e^- beam image and (b) i^+ beam image after switching to the back face.	36
Figure 40. (a) e^- beam image and (b) i^+ beam image after thinning the back face.	36
Figure 41. (a) e^- beam image and (b) i^+ beam image after low-energy thinning of back face.	37
Figure 42. (a) e^- beam and (b) i^+ beam image after orienting for front face milling at 5 kV.	38
Figure 43. (a) e^- beam and (b) i^+ beam image after thinning the front face at 5 kV.	39
Figure 44. Watch the e^- beam image (a) where the red arrow is pointing, mill for 0.1 μm of Z or until half of remaining Pt is gone, whichever occurs first. (b) rectangle indicating cleaned area in the i^+ beam image.	39
Figure 45. (a) e^- beam image and (b) i^+ beam image after cleaning the back face at 2 kV.	40
Figure 46. (a) Geometry and (b) e^- beam image of the sample at the end of the procedure.	41

1. Introduction

1.1 Transmission Electron Microscopy Research and Sample Preparation at ARL

Scanning transmission electron microscopy (STEM) is integral to materials and device research, unlocking specifics of a material's structural and chemical morphology. At the U.S. Army Combat Capabilities Development Command Army Research Laboratory's Adelphi Laboratory Center (ALC), Maryland, we use the JEOL ARM 200F, and we use a JEOL 2100F at Aberdeen Proving Ground, Maryland. Both instruments are field emission STEMs. The ARM 200F has a spherical-aberration corrector before the objective lens, which further improves the practical resolution of the microscope. Preparing samples requires as much care, expertise, and time as using the STEM, and high-end imaging tools and techniques cannot overcome poor sample fabrication. The state-of-the-art features of an expensive STEM are not useful without equally high-quality samples.

Microscopists often use the STEM and transmission electron microscopy (TEM) acronyms interchangeably, but for clarification, ARL's instruments are capable of both. The generalization of the terms is simply due to TEM being around longer (1930s vs. 1970s). The TEM acronym can refer to all microscopy involving transmitting electrons through a sample to form an image, which includes STEM. Most, but not all, modern microscopes can operate in both the scanning and transmission mode. One technique is not universally "better" than the other, and often we use both modes for the same sample. The focused-ion beam (FIB) technique for preparing samples is the same regardless of which mode you use. See the online book by Liao¹ for more information.

STEM data is foundational for DEVCOM Army Research Laboratory's materials research, some of which we documented in highly cited papers over the last few decades.²⁻¹¹ We used these results to inform the design and growth of materials and devices across numerous ARL programs. A non-exhaustive survey over the last few years includes studies of III-V semiconductors including proximity superconductivity materials,¹²⁻¹⁴ nitrides for micro-electromechanical systems (MEMS),¹⁵ indium arsenide antimonide for long wavelength infrared detector applications¹⁶ and wide bandgap semiconductors for ultraviolet emitters/absorber.^{17,18} In addition, we examined oxides such as hafnium oxide (HfO₂) compounds for neuromorphic applications,¹⁹⁻²³ and indium tin oxides for investigating epsilon-near-zero behavior.²⁴⁻²⁶ Finally, we studied many energetic materials²⁷⁻²⁹ along with metal alloys.³⁰⁻³²

The sample must be thin enough to be electron transparent (<100 nm). It is impossible for the sample to be completely unaffected by the sample preparation process. The thinning process can leave artifacts, making image interpretation difficult, or even destroying a sample. Surface amorphization, preferential milling by material and/or along certain directions, and sample heating introduce damage or reduce the thin area available for observation.

Until about 20 years ago, microscopists did not widely use FIB tools for TEM sample preparation. Since their introduction, FIBs have significantly improved in ease of use and performance. One limiting issue was that computers were simply not fast enough and did not have the memory needed to run the software needed to make FIB practical.

We described the long-time sample preparation procedure at ALC in a prior technical report.³² It is based on tripod polishing,³³ and we use it today for samples that are not site-specific. We enhanced the procedure in recent years with the purchase of a Gatan precision ion polishing system (PIPS) II ion mill. This allows milling at lower energies and temperatures, and at shallower angles compared with the retired ion mill. This reduced the argon ion (Ar^+) beam-induced heating damage to the samples.

The manual tripod polishing and ion milling technique can produce high-quality samples, but it is a very tedious process and difficult to learn. It also takes a minimum of 3 days for each sample, since epoxies must cure at room temperature at two different points in the process (for heat-sensitive samples). An ARL contractor developed a small-angle cleavage (SAC) technique during his prior employment. This technique eliminates the need for ion milling and limits the number of hot plate exposures.^{34,35} Preparing SAC samples requires precise manual labor and samples that easily cleave. The manual polishing/ion milling and SAC techniques both produce electron transparent areas, but of limited size, and do not allow for targeted thinning of specific sites. It does, however, have the advantage of exposing the sample to less ion beam energy than the FIB. The tripod polishing/PIPS technique also has the advantage of sample rotation during milling, which reduces curtaining effects³⁶ due to different grains/materials milling at different rates. Curtaining is one of the drawbacks of FIB that can never be completely avoided, although it can be reduced.³⁷

ARL has a Thermo Scientific Helios G4 UX DualBeam System. This FIB uses low accelerating voltages (as low as 500 V), which minimizes ion beam damage to the thin TEM cross sections. The procedure for creating TEM samples with an FIB is nontrivial in terms of difficulty and tedium. It does, however, allow for large-area

samples with site-specific selection. In this report, we document a FIB procedure for TEM sample preparation.

Since the scanning electron microscope (SEM) allows nanoscale imaging resolution, you can exactly select the area that you want to prepare. Recently, for instance, we imaged HfO_2 films grown by molecular beam epitaxy onto 2-D tungsten diselenide (WSe_2) flakes that had been exfoliated onto silicon (Si) substrates.³⁸ The manual sample preparation method would not work for two reasons: 1) the flakes were too small to see with the optical microscope and there would be no way to precisely control Ar^+ ion milling in the PIPS to thin the desired interface, and 2) the WSe_2 material cannot survive mechanical polishing without slipping into its individual 2-D planes.

Figure 1 shows an example of a sample that could not be prepared with manual polishing or cleavage techniques. We needed to image specific layer stacks, which varied laterally across the wafer. Using the SEM in the dual-beam system, we could find the area shown in the center, which has a vertical height difference relative to the adjacent area. We were able to create an FIB lift-out that consisted of the region with the higher-height gold (Au)/nickel (Ni) region along with a portion of the two adjacent stacks containing the Au/bismuth (Bi) regions.

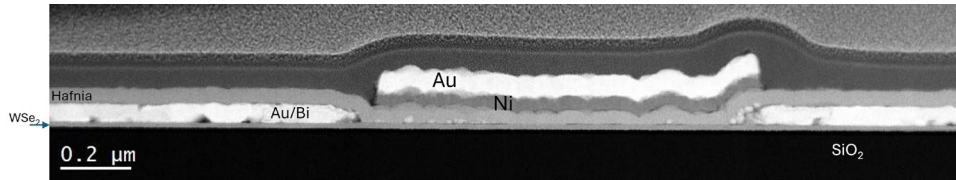


Figure 1. Device structure prepared by FIB and imaged with high-angle annular dark-field STEM.

Figure 2 is another example of a sample that could only be prepared by FIB. ARL researchers exfoliated WSe_2 onto Si coupons and then placed them into a molecular-beam epitaxy chamber for Bi deposition. The light blue and white jagged shapes in the optical microscope image of Figure 2a are the WSe_2 flakes. Since the WSe_2 regions are not continuous, the traditional method of randomly sectioning a 3-mm-length area and ion milling would not likely yield an area that contained a flake. We were able to locate the flakes in the FIB-SEM and section out the flattest possible flake. Figure 2b shows the TEM image.

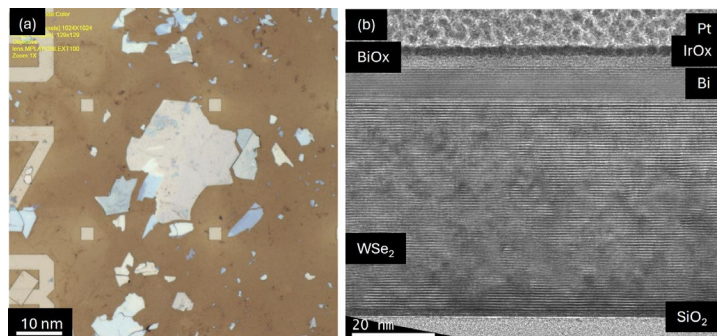


Figure 2. Bi on WSe₂ flakes on an oxidized Si substrate. (a) Optical microscope image, and (b) cross-sectional TEM image of an FIB lift-out taken from the largest flake seen in (a).

Other examples of samples that required FIB were niobium diselenide films that dissolved in acetone when the traditional polishing method was attempted.³⁹ Any material not rigidly attached to its substrate cannot survive mechanical polishing.

While this report discusses a cross-sectional sample, there is a procedure for plan-view samples outlined in a 2023 ARL technical report.⁴⁰

1.2 Brief Introduction to the FIB Technique

Beginning in the 1990s, TEM sample preparation using FIB was accomplished with standalone, single-ion beam tools.³⁷ A dual-beam FIB tool is an enhanced SEM that also has a focused ion (i^+) beam, allowing material deposition and/or ablation in a controlled manner while simultaneously imaging the sample. The introduction of dual-beam instruments increased the ease and advantages of FIB and led to its commonplace adoption for TEM sample preparation. The electron beam (e^-) allows superior imaging with less damage than i^+ beam imaging in single-beam tools.

Gas injection systems, such as the MultiChem (MC4), introduce reactive gases near the vicinity of the electron or ion probe. This permits site-specific material deposition.³⁷ The FIB's micromanipulators achieve precise lift-outs, rotations, and movement of the samples.

The i^+ beam has a liquid metal gallium (Ga) source. Simplistically, when the Ga flows to a tungsten (W) needle, ionization and field emission of the Ga atoms occurs. Depending on the energy of the i^+ beam, it images, mills, or sputters (although i^+ beam exposure always causes some milling of the sample, even during sputtering).

It is important to remember the i^+ beam is destructive and amorphizes the sample's top layer and can implant unwanted Ga into the sample. For this reason, it is critical to minimize the i^+ beam energy, the milling angle, and the beam exposure time. A

low-energy/angle cleanup in the PIPS ion mill can remove residual damage if needed.

This report does not intend to imply that FIB is only for TEM sample preparation. These versatile tools are also used for direct pattern writing,⁴¹ MEMs device fabrication,⁴² and FIB micromachining.⁴³ At ARL, the FIB is extensively used to prepare samples for atom probe tomography.⁴⁴

1.3 Pre-FIB Sample Preparation

We often deposit a thin iridium (Ir) oxide layer onto the sample's surface to improve its conductivity. Au or carbon (C) are also available options. We mount the samples onto SEM stubs with double-sided conducting tape and then place them into a multi-stub holder (Figure 3a), which then goes into the Leica EM ACE600 Sputter Coater (Figure 3b).⁴⁵

The steps are as follows:

- 1) Use the menu on the touch screen (Figure 3c) to turn on the rough pump.
- 2) Open the Ar⁺ cylinder.
- 3) Run the selected deposition program. We typically deposit 1 nm of Ir. The program will not begin running until the system has reached the appropriate process vacuum.
- 4) Vent the chamber. There is a checkbox on the touch screen that allows the system to automatically vent once the deposition is complete.
- 5) Close the Ar⁺ and remove samples once the chamber has reached atmospheric pressure.
- 6) Return the chamber to vacuum.

We keep the manual (Figure 3b) near the sputter coater in the lab.

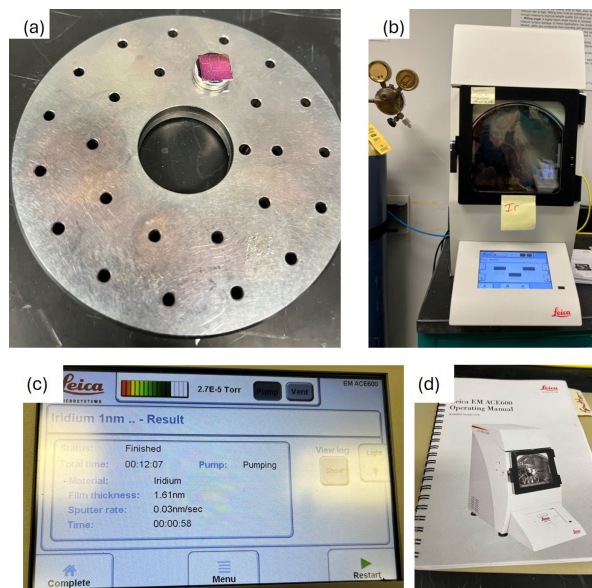


Figure 3. Sputter coating (a) stub mount, (b) Leica EM ACE600 Sputter Coater, (c) control touch screen, and (d) manual.

1.4 Mount and Load Grids and Samples

Copper (Cu), molybdenum (Mo), Si, and more recently, C, lift-out grids are available for purchase from vendors such as Ted Pella, SPI, and Electron Microscopy Sciences. An example of a Mo three-post Omniprobe lift-out grid is shown in Figure 4a.⁴⁶ All grids except for those made of C produce energy-dispersive X-ray spectroscopy (EDS) peaks that can complicate the compositional analysis. For instance, the Cu $K\alpha$ X-ray peak is near the Hf $L\alpha$ X-ray peak. Mo grids are more expensive and have a rougher surface than Cu grids but are more rigid and less likely to bend. Choose the grid that is best for the application. If you know there is no Cu in your samples, or that its $K\alpha$ peak will not complicate your analysis, then you might choose to use Cu grids and just ignore the Cu EDS peaks.

There is a loading clamp on a SEM pin for mounting the TEM grids (Figure 4b). Loosening the screw opens the clamp, and then up to two grids can be slid into position. We labeled the grid positions as L and R. Figure 4c and d shows a top and side view of the clamp with the grids loaded. Take care not to bend the grids and mount them all facing the same direction.

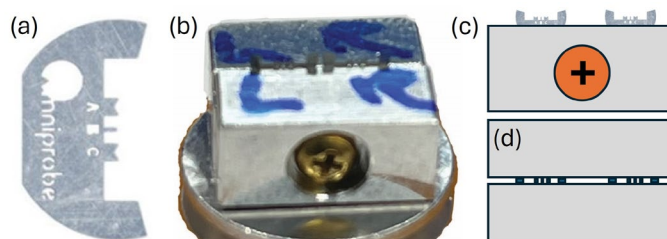


Figure 4. FIB grids and clamping arrangement (a) Mo three-post Omniprobe grid, (b) TEM FIB grid clamp, (c) side and (d) top-down view of grid clamp.

Figure 5 shows one of the stub mounts. In Figure 5a, we see a top-down view that shows five holes that can accept SEM stubs or grid clamps. Figure 5b shows a side view. The upper arrow points to the threads that allow height adjustments and the lower arrow points to the threaded pin that screws into the SEM stage. Figure 5c shows the sample height standard to check that the stubs or grids are not going to hit the pole piece.

(In this procedure, we mark instructions that are pertinent for the health of the FIB tool and the sample in bold italics.)

If the samples are taller than the clamp, place them into the stub mount so the edge parallel to the zone axis is perpendicular to the axis of the clamp. This ensures that tilting the stage to 52° will not cause a collision with the pole piece.

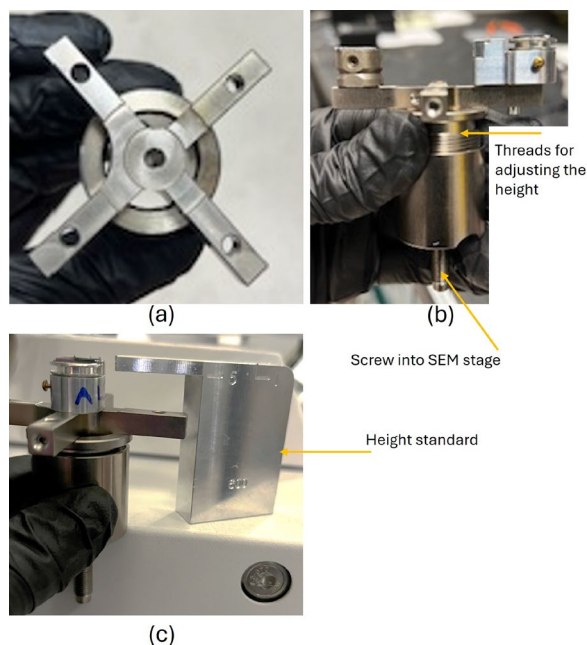


Figure 5. Four-vane multi-stub holder. (a) Top view, (b) side view showing adjustable height piston, and (c) side view showing the height reference tool.

2. FIB Geometry and Computer Interface

2.1 FIB Geometry

It is difficult to visualize the geometry of the sample and the e^- and i^+ beams. Figure 6 shows an example of an (a) e^- and (b) i^+ beam image of an FIB sample in progress. Schematics (Figure 6c–e) further illustrate the arrangement. When the sample stage is at 0° tilt, the e^- beam is perpendicular to the sample's surface and we see a top-down view (Figure 6a). The e^- beam image axes are perpendicular to the sample plane, and the i^+ beam image axes are tilted 52° relative to the e^- beam image. The i^+ beam is 38° to the sample. If you imagine looking at the sample straight on (Figure 6e), the projection of the ion beam is along the sample and perpendicular to the e^- beam, which is going into the page. The top of the image is physically closer to the i^+ beam than the bottom of the image. Figure 6f has the sample with its surface directly facing the viewer.

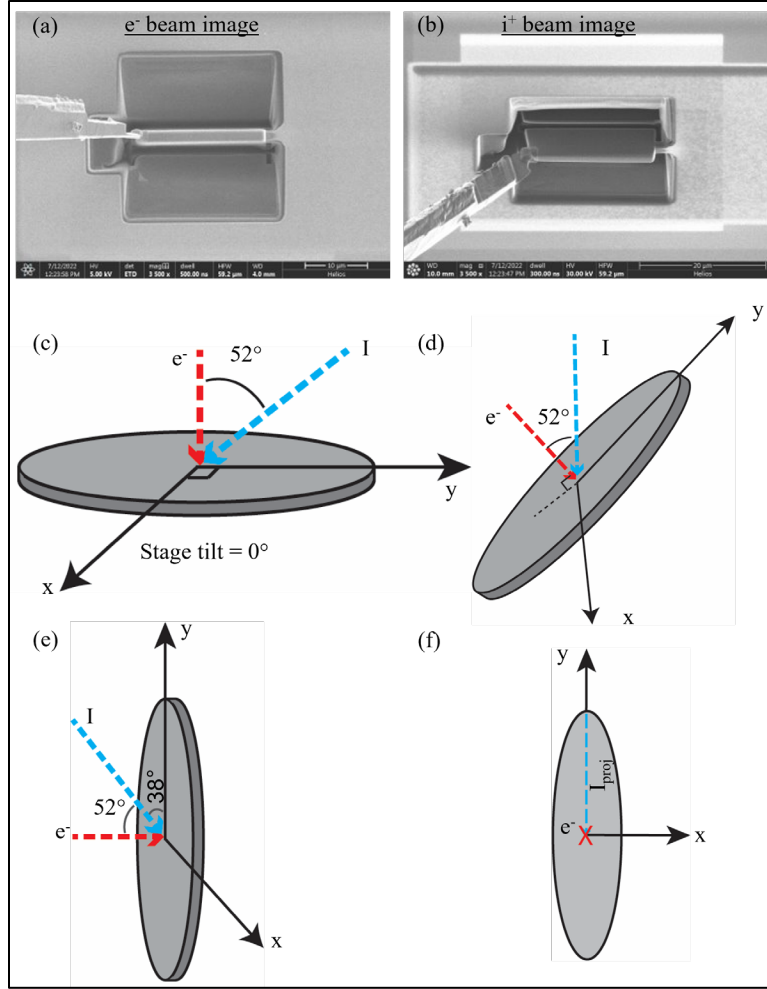


Figure 6. Geometry of FIB beams and sample. (a) e^- and (b) i^+ beam images of a sample undergoing FIB, and (c–f) schematics from multiple perspectives to show the geometry of the two beams and sample stage.

2.2 Computer Interface

One of the two monitors for the FIB computer has the display as shown in Figure 7. Clockwise, from the upper left panel, the view is of the e^- beam image, the i^+ beam image, chamber camera, and the navigation camera (Nav-Cam). Menus and ribbons at the top and left of the display allow the user to control the FIB's functions. The green pause symbols in the upper-left-hand quarter of three of the quadrants means that the image is not live. We can customize the information bar at the bottom of the images by right-clicking the bar.

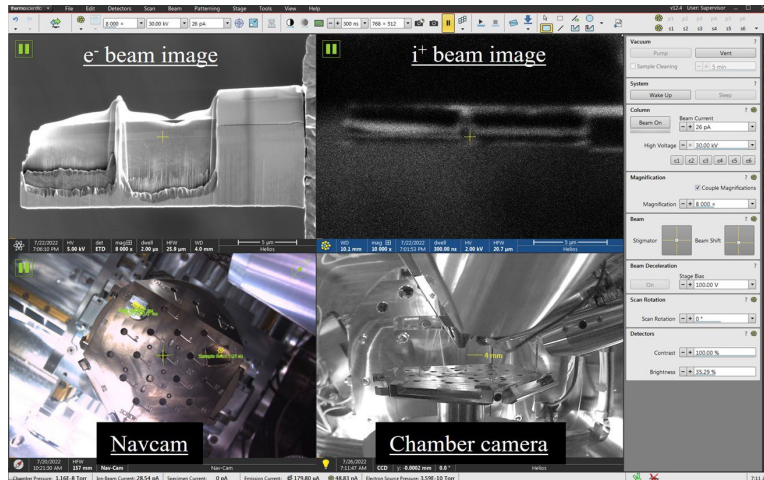


Figure 7. Main display for FIB showing the e^- and i^+ beam images, the Nav-Cam view, and the chamber camera.

Figure 8 is an expansion of the main menu bar and the right-hand menu bar. We labeled the key selection buttons mentioned in this procedure.

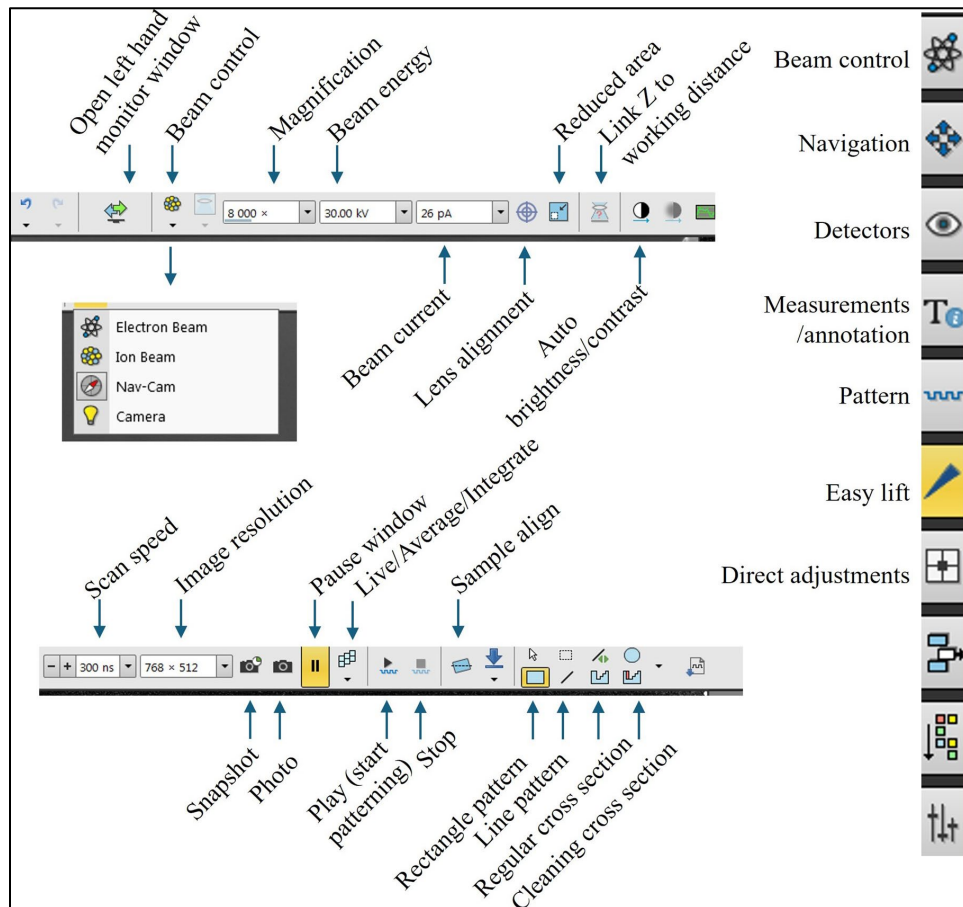


Figure 8. Labeled main and right-hand menu bar.

3. Startup Procedure

The startup procedure includes these steps:

- 1) Go to the navigation icon (yellow highlighted box, Figure 9a) in the right-side ribbon and zero out the stage axes positions X, Y, Z, tilt (T), and rotation (R).

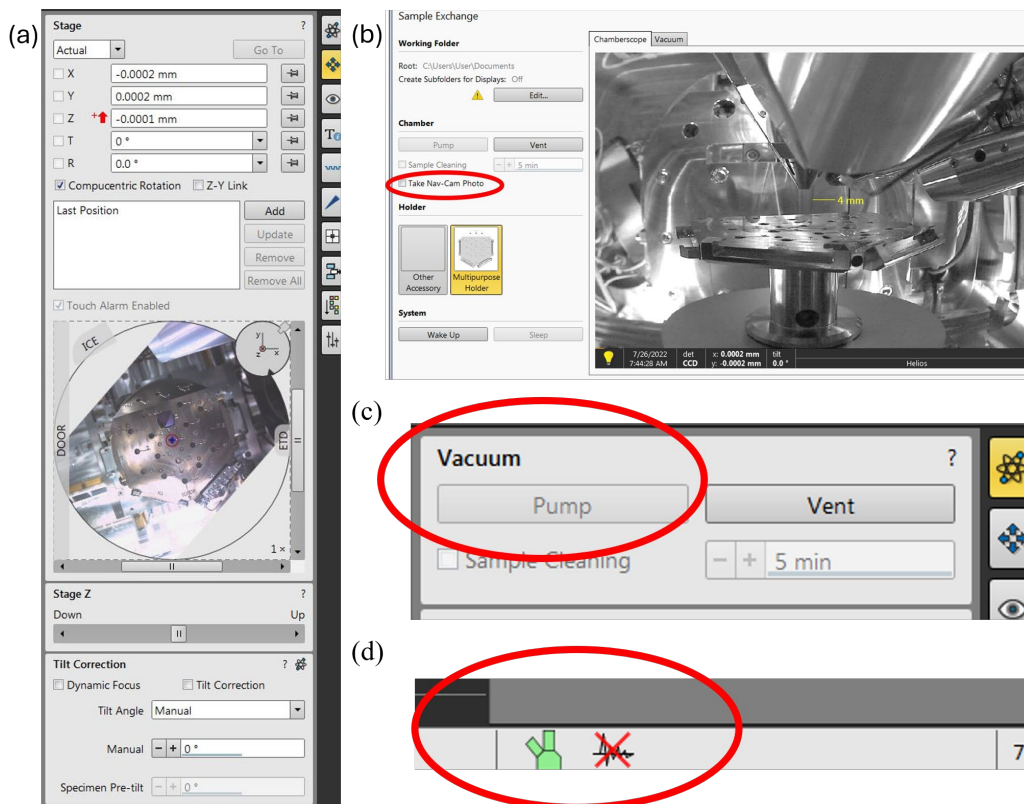


Figure 9. Menus needed for loading the sample stage: (a) navigation pane, (b) sample exchange window, (c) vacuum control, and (d) vacuum indicator.

- 2) Check that the Nav-Cam photo is off. On the camera view panel on the left monitor, deselect the “take Nav-Cam photo” box (Figure 9b). If this window is not open, use the opposing arrow main menu button (Figure 8).
- 3) Close the chamber door and press the PUMP button on the screen (Figure 9c). Wait for the chamber vacuum status to turn green, approximately 3–5 mins (Figure 9d).
- 4) Check that the scan rotation is off for both beams. Under the scan menu, click on 0° for both the e^- and i^+ beam windows, then go back to scan rotation to deselect (Figure 10a).

- 5) Zero the beam shifts for both the e^- and i^+ beam windows. Select beam control button on the right-side ribbon (red circle, Figure 10b). Center the crosshairs by selecting the beam shift control box (yellow circle). Right-click onto the beam shift control box and select “zero” from the pull-down choices.

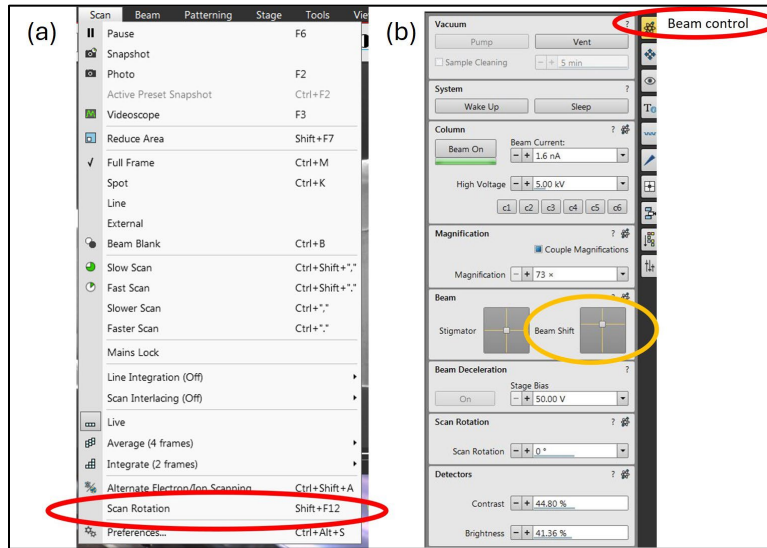


Figure 10. Initial beam conditions (a) scan rotation and (b) beam shift.

- 6) Select the Everhart–Thornley detector (ETD) in both the e^- and i^+ beam windows (Figure 11a). You can also check this in the detector pull-down menu or the detector tab on the right-hand ribbon (Figure 11b).

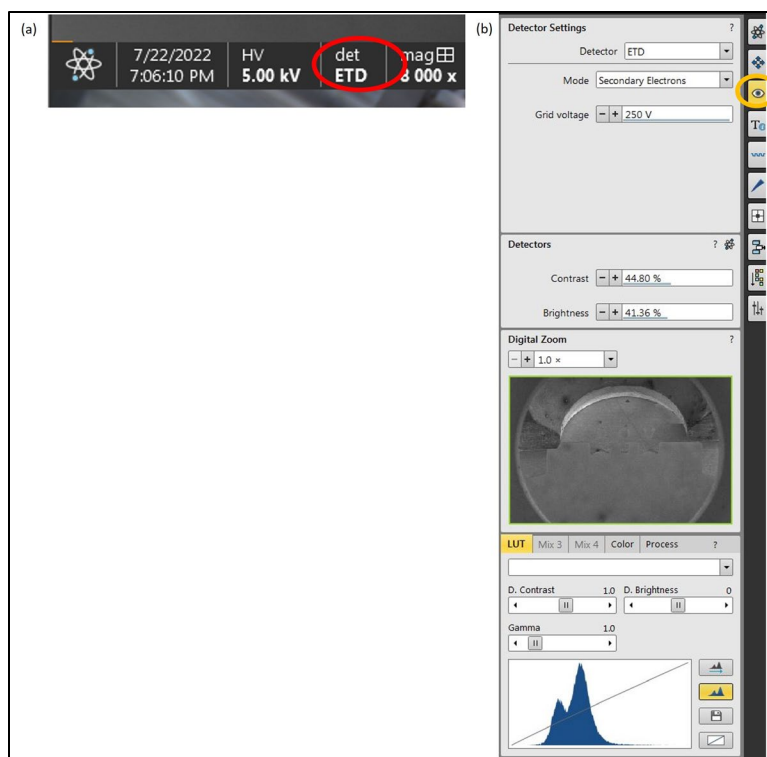


Figure 11. Detector indicator window.

- 7) Ensure that the image averaging is off. Under the scan menu, select the live view for both the e^- and i^+ beam windows (Figure 12a). You can integrate a single frame to check a sample's condition during thinning. (It is basically the same as taking a snapshot). You can average the image instead of the iSPI technique for samples that are not as sensitive to beam damage when getting toward the end of the thinning process (specifically when doing low-kilovolt cleaning at 2 and 5 kV).
- 8) Couple the magnifications between the e^- and i^+ beam windows (Figure 12b).
- 9) Set i^+ beam dwell time to 100 ns (Figure 12c) and the resolution to 1536×1024 . Start with a voltage of 30 kV and a beam current of 26 pA. For the e^- beam, use a voltage of 5–20 kV (depending on material type) and beam current of 0.8–1.6 nA.

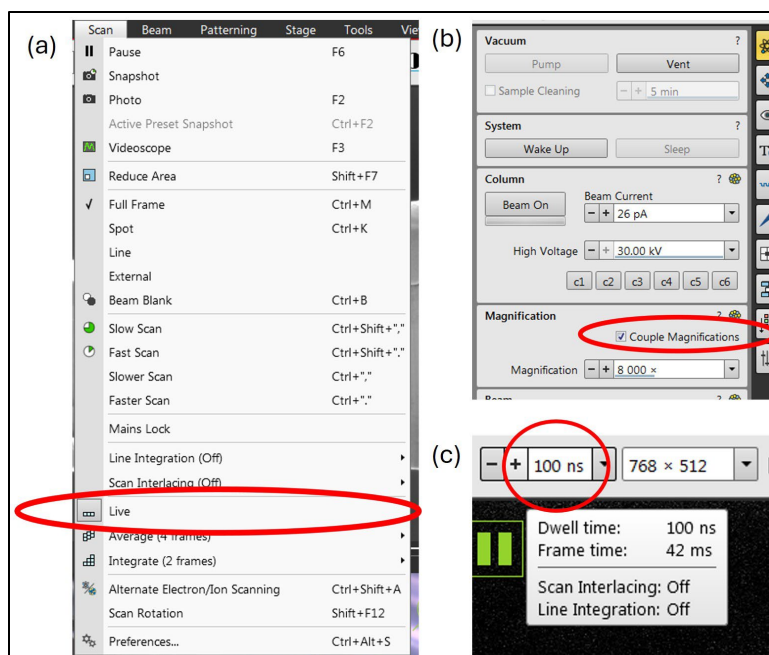


Figure 12. More initial beam settings (a) live image selection/averaging deselection, (b) magnification coupling, and (c) set i^+ beam dwell time.

10) Clicking “Wake Up” brings up both the e^- and i^+ beams (Figure 13a). Make sure that the system is pumped down before doing this step. The rectangle under the “Beam On” button will be completely green, and you will have an emission current once the process is complete.

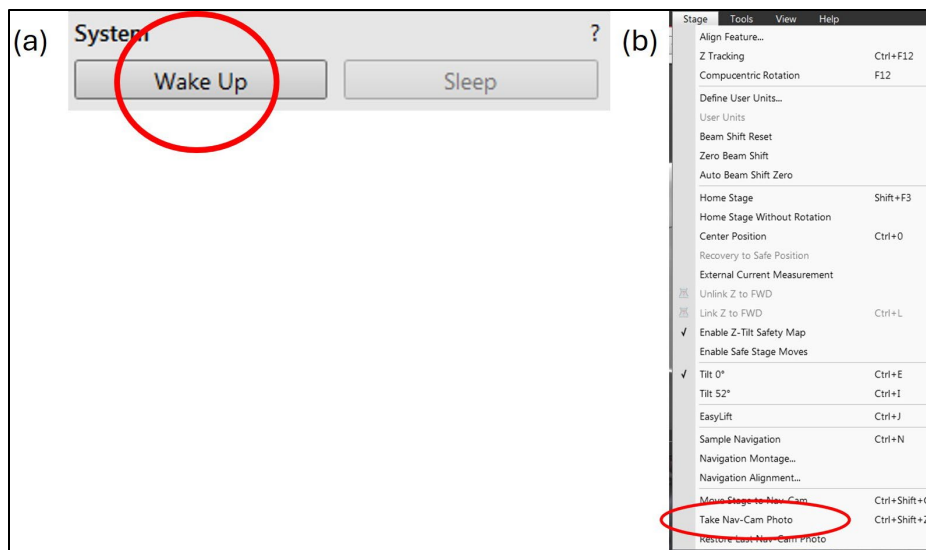


Figure 13. (a) Wake up the beam, and (b) take a Nav-Cam image.

11) Take a Nav-Cam photo. Find this function under the stage menu (Figure 13b).

4. Align the SEM image

General rules:

- 1) *Minimize the time at high magnifications to avoid sample charging.*
- 2) When you change sample heights, reset the free working distance (FWD).
- 3) If you have Cu tape on the sample, check the ion beam image at 30 kV, 26 pA.
- 4) Make sure that the e^- and i^+ views agree.
- 5) The milling box will be yellow, and the deposition box will be orange.
- 6) *Always be mindful to not hit the pole piece!*
- 7) Make sure that the chamber camera view is live (Figure 14, no pause indicator).

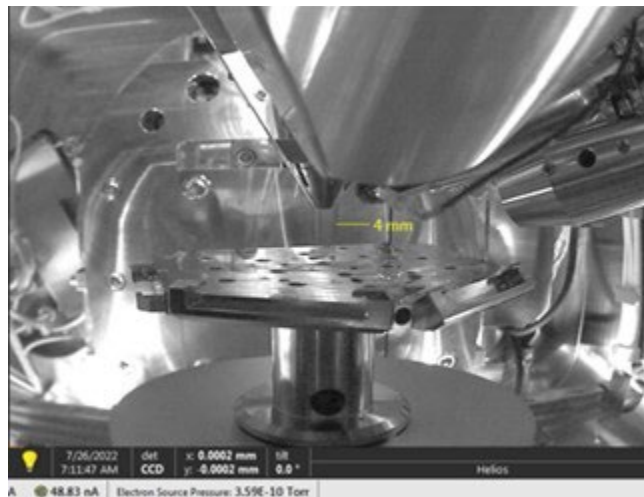


Figure 14. Chamber camera view is live.

- 8) Double-click on a corner of your sample in the Nav-Cam view; this will move the stage and center it on that location for SEM viewing. If there is Cu tape on the sample, focus on that to avoid exposing the sample.
- 9) Set the e^- beam voltage to 5 kV and current to 1.6 nA.
- 10) Focus on the sample at a magnification of around 3000 $\times$, then link z to the FWD.
- 11) Set the e^- beam working distance (WD) to 6 mm, right panel, navigation ribbon (icon has arrows).
- 12) Focus, check the stigmation, link z to FWD again, and reset WD to 4 mm.

- 6) The e^- beam parameters should be 5 kV, 1.6 nA. The time should be approximately 4–6 min.
- 7) Hit start. When you check on the deposition, the e^- beam image will look like Figure 15b.

5.1.2 Tilt for i^+ Beam Pt Deposition: Set the Eucentric Height

General rule: *When at 0° , double-click on the e^- beam image to center. When at 52° , use Z to move up and down.*

- 1) ***Remove the MC4 nozzle. We must always retract it before tilting the stage!***
- 2) Center the rectangular Pt feature in the e^- beam image by double-clicking in the middle of the image.
- 3) Use Control-I to tilt the stage to 52° . Click on the bottom-right stage window. Press and hold the mouse wheel down and move cursor slowly to recenter the feature in the e^- beam window in the camera window. Go the same direction that the e^- beam window indicates.
- 4) Use Ctrl-E to move the stage back to zero. Double-click on the feature to center it in the SEM window.
- 5) Repeat Steps 3 and 4 until the feature is stationary. End at Control-I.

This set of steps leaves the stage tilted so that e^- beam has 38° incidence with specimen surface, but opposite to the way the i^+ beam is oriented when the stage is flat (Figure 16a). This means that the bottom of the image is now closer to the e^- beam and the i^+ beam is perpendicular to the specimen surface. Figure 16b and c shows the e^- and i^+ beam views of the Pt region.

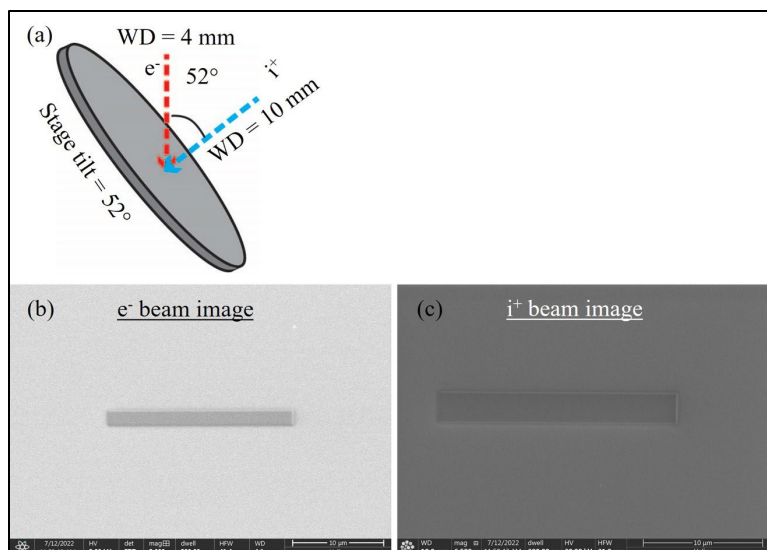


Figure 16. Adjust the eucentric height. (a) instrument geometry schematic with accompanying (a) e^- and (b) i^+ beam images.

5.1.3 i^+ Beam Pt Deposition

Remember that the alignment between the spot sizes is not perfect. If you image in one spot size and then deposit/mill in another, you may not deposit/mill where you intend!

If an area in the i^+ beam image does not match the e^- beam image, center a feature in the e^- beam window by double-clicking, take an i^+ beam image, then hold shift and click and drag the feature to the center.

- 1) While still at 52° tilt, move laterally away from the area of interest. Set the i^+ beam current to 26 pA (30 kV) for imaging. ***The i^+ beam image should not ever be live at currents exceeding 26 pA.*** If we have Cu tape on the sample, we may have done this step already. You may want to switch to a two-frame integrated view.
- 2) Make the i^+ beam view live, adjust the contrast, focus, and stigmatism, pause. Use the e^- beam image to return to area of interest.
- 3) Change the i^+ beam current to 0.26–0.44 nA for deposition, insert the MC4 nozzle, turn up the contrast one-half knob turn (clockwise) (window is “dark”). Click snapshot to take an image.
- 4) Adjust contrast as needed (approximately two good turns up). Make sure you have centered the feature. Hold shift and click and drag feature if needed in e^- beam and adjust the i^+ beam image with the beam shifts. Take a new snapshot.

- 5) Select the rectangle tool (make sure the rectangle button is yellow). Draw deposition box, size $20 \times 2.5 \times 1.5 \mu\text{m}$. Adjust the box position by clicking and dragging the edge while the cursor looks like two bisecting and perpendicular double arrows.
- 6) Make sure the selected application is Pt_M. The drawn box should be orange. The indicated time should be approximately 5–8 min. Hit play.
- 7) The iSPI camera can be turned on to track the sample intermittently. Set the time interval to 15 s. Click the “on” box. Make the e^- beam “live.”

At the end of this step, the Pt protective layer is now approximately $2 \mu\text{m}$ thick and the can sticks up substantially in the inclined e^- beam view (Figure 17a). The i^+ beam view is orthogonal to the Pt surface (Figure 17b).

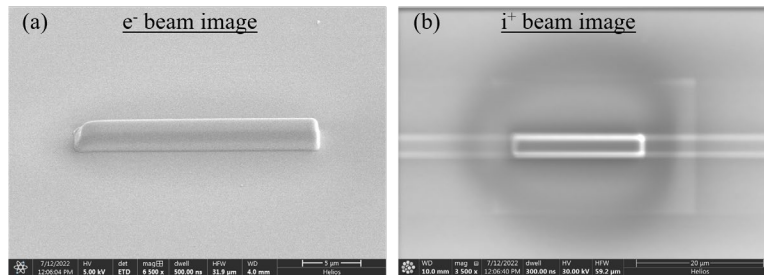


Figure 17. (a) e^- and (b) i^+ beam images after Pt deposition.

The purpose of the e^- beam deposition is to ensure that we never directly expose the region of interest to the i^+ beam. We must remember that the i^+ beam always mills material even as it is depositing. Furthermore, e^- beam deposition creates a protective layer consisting of smaller metal particles than can be accomplished with i^+ beam deposition, leading to smoother milling conditions.³⁷ However, the e^- beam deposition is significantly slower than i^+ beam deposition. Therefore, we take advantage of the gentler e^- beam deposition for the first Pt layers, and then switch to the faster i^+ beam deposition once we are farther away from our area of interest. The goal is to deposit a protective layer sufficiently thick enough that some of it remains intact throughout the milling procedure.

5.2 Mill, Planarize, and Undercut Sample

5.2.1 Mill 1st (Lower) Trench

- 1) *Retract the MC4 nozzle.*
- 2) Zoom the magnification to approximately 3500 $\times$. Set the i^+ beam current to 2.4 nA, turn up the contrast one-half turn, zoom out on the image, take a snapshot, and adjust contrast as needed. The focus may need tiny counterclockwise adjustment.
- 3) Select the regular cross-sectional pattern under the patterning icon in the right-side ribbon and draw a box $24 \times 11 \times 9 \mu\text{m}$ ($14 \mu\text{m}$ for hard samples like Al_2O_3).
- 4) Make sure the scan direction is set to travel from the bottom to the top for the lower trench. Always mill in a direction toward the sample. Make sure to select the Si-multipass application (the rectangle pattern on the i^+ beam image will be yellow). Hit play. Select the e^- beam window, set to average, and set iSpi to 10 s. If you lose Pt, add more in the i^+ beam window.
- 5) The lower trench is now clearly visible (Figure 18).

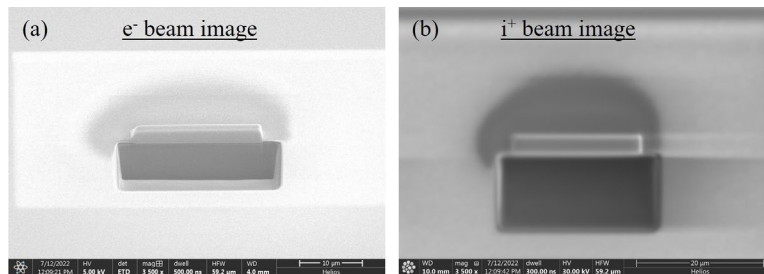


Figure 18. (a) e^- and (b) i^+ beam images after the first trench dug.

5.2.2 Mill 2nd (Upper) Trench

- 1) Take a photo and adjust the contrast as needed.
- 2) Move the milling box to the top above the Pt.
- 3) Repeat procedure 5.2.1, except that the scan direction is from the top to the bottom.
- 4) The top and bottom trenches are now clearly visible (Figure 19).

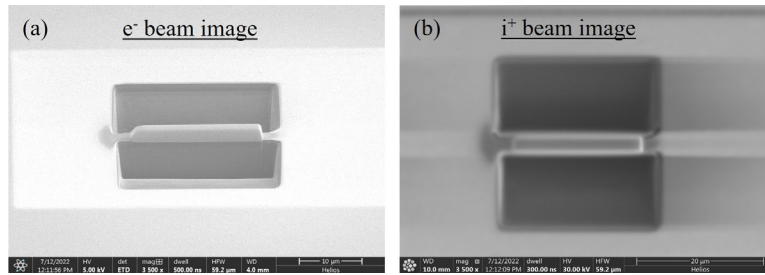


Figure 19. (a) e^- and (b) i^+ beam images after the second trench dug.

5.2.3 Mill Side Trench

Delete the rectangle from the prior step (garbage can button).

- 1) Select and draw a rectangle pattern.
- 2) Set application to Si (yellow multipass).
- 3) Make a side trench on the left-hand side. Draw an approximately square shape using the rectangle pattern ($8 \times 9 \times 4 \mu\text{m}$).
- 4) Set the scan direction to travel from the left to the right. Turn the iSPI time down to approximately 8 s. Hit play.

Ensure that the side trench is as deep as both the lower and upper trenches at the point where it meets the sample (red arrow, Figure 20a). This guarantees that it cuts free later.

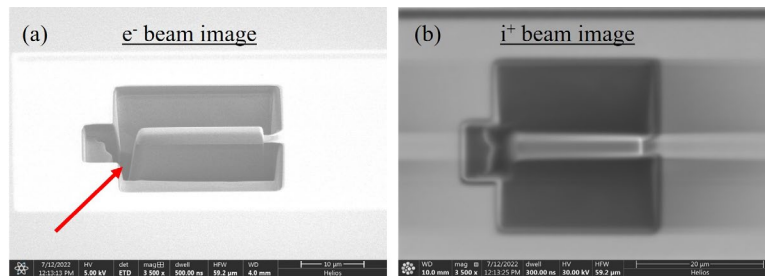


Figure 20. (a) e^- and (b) i^+ beam images after side trench dug.

5.2.4 Planarize the Front Face

- 1) Tilt the stage to 53.7° (Figure 21a). For hard substrates, use 54° . Change the i^+ beam current to 0.75–1.2 nA.

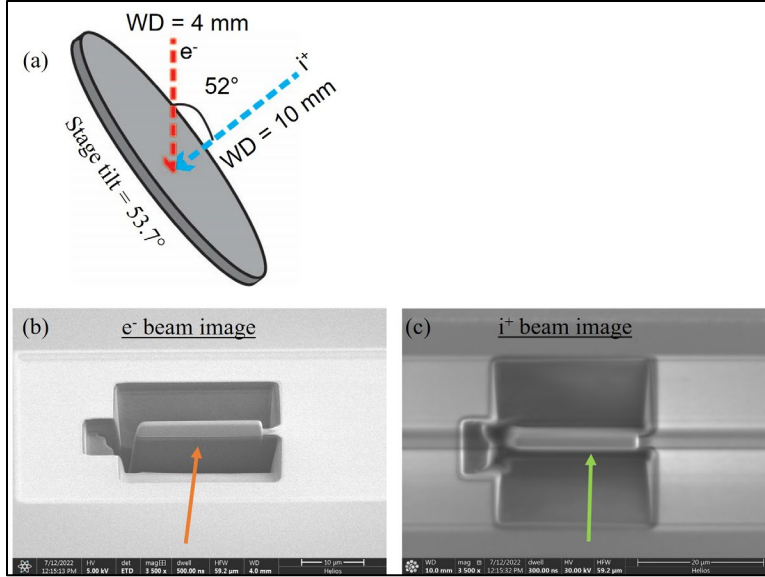


Figure 21. (a) Geometry during front face planarization. (b) e^- and (c) i^+ beam images after the side trench is constructed.

- 2) Turn down the i^+ beam contrast and take an image.
- 3) Clean up the bottom trench. Set the scan direction from bottom to top. Draw a rectangular cross-sectional pattern box ($20 \times 1.5 \times 3 \mu\text{m}$) and position it where the top is aligned to the bottom edge of the Pt (cover the bright area) or the top of the bottom trench. Hit play.

The face of the sample should look flat and clean, and film layers should be visible if applicable (orange arrow, Figure 21b). The sidewall should be sharp in this top-down view (green arrow, Figure 21c), there should be no projected width from the inclination.

5.2.5 Planarize the Back Face

- 1) Tilt the stage to 50.3° (50° for hard samples) (Figure 22a).
- 2) Change scan direction to top to bottom.
- 3) Take an image. Position the selector box and hit play.

There is no e^- beam image in Figure 22 because the back face is not visible. The sidewall should be sharp in the top-down view of the i^+ beam (Figure 22b), meaning there is no projected width from the inclination.

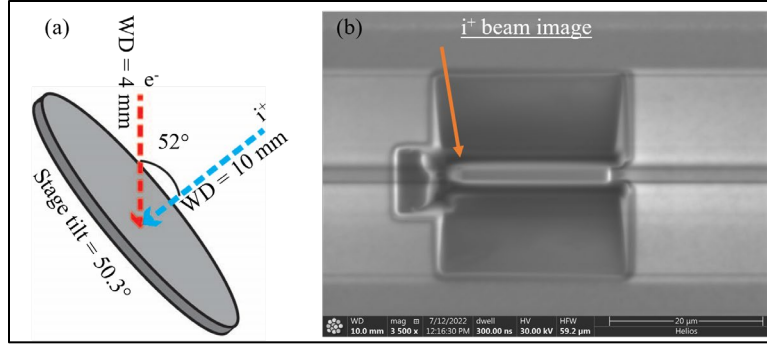


Figure 22. (a) geometry during and (b) i^+ beam image after the backside face planarization.

5.2.6 Tilt and Mill for Undercuts

- 1) Tilt the stage to -5° (Figure 23a).

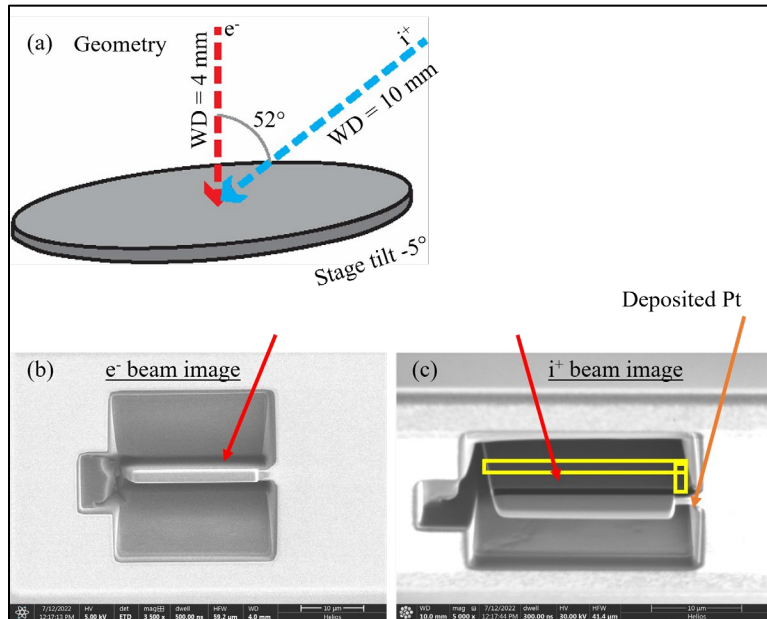


Figure 23. Undercut (a) geometry, (b) e^- beam image, and (c) i^+ beam image. The red arrows point to the sample's backside. It is just barely visible in (b) due to the -5° stage tilt. Do not cut where the orange arrow is pointing in (c).

- 2) Set i^+ beam current to 0.75 nA or lower. The backside of the sample is visible in Figure 23b.
- 3) Decrease the contrast and take snapshot.
- 4) Position the top rectangle to ensure it goes past the end of the sample on the left side (Figure 23c). Set the milling direction to top to bottom.
- 5) Position side rectangle as shown—do not cut all the way through to the top (bottom in i^+ beam view). Set Z for both cuts to around $3\ \mu\text{m}$ and the narrow dimension (X) to $1\ \mu\text{m}$. Switch to parallel milling. Milling direction is right

to left for the vertical box. Hit play and watch in the e^- beam window for the cut to go through.

The cuts should go approximately where the yellow boxes are—***do not make the undercut at the bottom of the trench since the beam mills diagonally downward.*** The sample will now appear as shown in Figure 24.

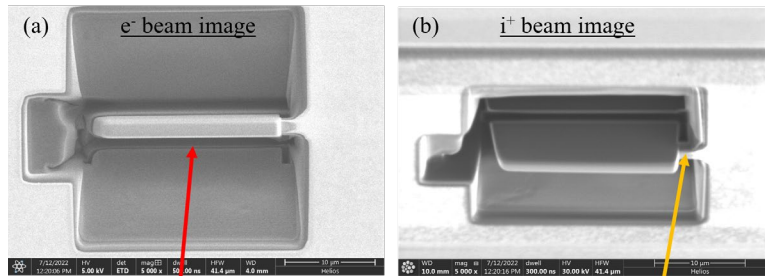


Figure 24. (a) The undercut indicated by the red arrow in the e^- beam image should connect all the way across the bottom of the trench. (b) The sample should clearly be completely detached everywhere in the i^+ beam image after undercutting except at the point shown by the orange arrow.

6. Lift-Out Procedure

6.1 Attach Probe to Sample

6.1.1 Insert the Lift-Out Probe

- 1) Ctrl-E to return to the stage to 0° tilt.
- 2) Set the i^+ beam to 26–41 pA.
- 3) Zoom all the way out.
- 4) Check beam shifts.
- 5) Go to the EasyLift control icon (Figure 25, red box), click park position, and click insert in the pop-up window.

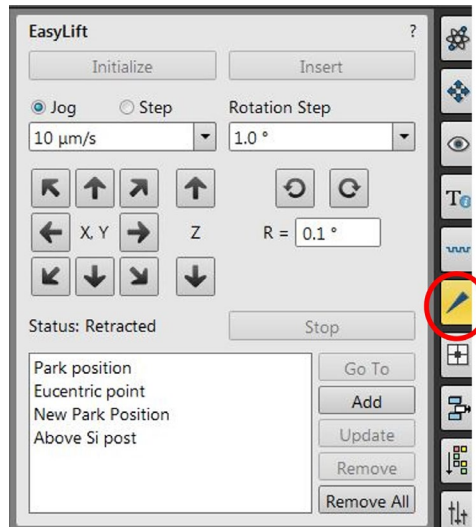


Figure 25. EasyLift control window.

- 6) Set the movement speed to 10 $\mu\text{m/s}$. We set the movement to jog, not step.
- 7) Increase the magnification. Align the probe in X and Y so that it is over the side trench with the e^- beam, and then bring it down in Z using the i^+ beam.
- 8) Use the e^- beam view to adjust the X-Y position of the probe until it aligns over the left trench.
- 9) Use the i^+ beam view at low magnification to bring probe down in Z until just above your sample. Use the down arrow; probe will appear to be going up since it is upside down.
- 10) Turn speed down to 1 $\mu\text{m/s}$. Use the e^- beam to position the probe just left of the sample. You can see a small gap between the probe and the sample, and with the back face of the probe flush with the back face of sample.
- 11) Use i^+ beam view to bring the probe down in Z until the it is at the height of the side of your sample as shown in Figure 26a. Use X to bring it to just barely contacting your sample in Figure 26b.

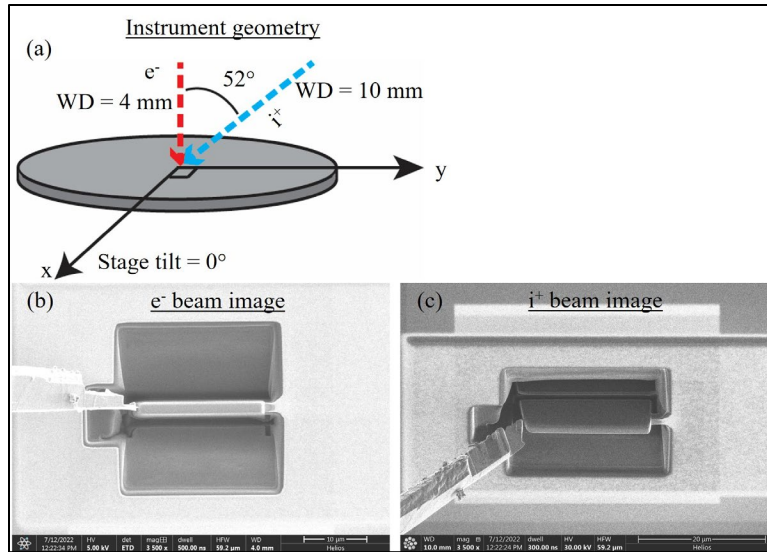


Figure 26. (a) Sample geometry during lift-out probe insertion, (b) e^- beam image, and (c) the i^+ beam image after lift-out probe insertion.

- 12) Finally, bring it close to the sample using both views to ensure that the back of the probe aligns with the back face of the specimen and is just touching.
- 13) Pause the i^+ beam view immediately.

6.1.2 Attach the Probe with Pt Deposition

- 1) Change the i^+ beam current to 90 pA–0.26 nA.
- 2) Insert the MC4 nozzle.
- 3) Turn up the contrast and take an i^+ beam image.
- 4) Draw a small rectangle deposition box just covering the tip of probe and edge of sample.
- 5) Set Z to 0.3 μm . Make sure the application is set to Pt_M_Weld (box will be orange). Should take approximately 6 s.
- 6) **Click start. Take image afterward to check that there has been sufficient deposition, as shown in Figure 27a. This may need to be done twice.**

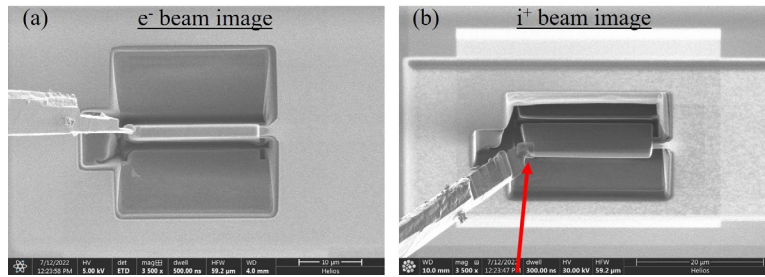


Figure 27. (a) e^- beam image and (b) i^+ beam image after probe attachment with Pt.

- 7) We should see that the probe looks clearly attached with Pt, as shown in Figure 27b. Deposit the minimum amount needed to attach the probe to the sample to avoid later having to cut off a bunch of your sample and/or the probe tip.

6.2 Detach Sample

6.2.1 Cut Right Side of Lift-Out to Detach

- 1) Change the application to Si and change the scan direction to right to left.
- 2) Move/change shape of milling box to be flush with the half cut on the right side of the sample and adjust the height so it goes to the top edge of the sample to ensure a flat edge.
- 3) Set Z to 4 μm . Hit play and watch with e^- beam using the iSPI camera to track the sample intermittently. Set the time interval to 8 s.
- 4) Stop milling when the cut has gone through and the edge looks flat.
- 5) The sample should appear clearly detached (red and orange arrows in Figure 28). If in doubt, drop the i^+ current to 26 pA and take a snapshot.

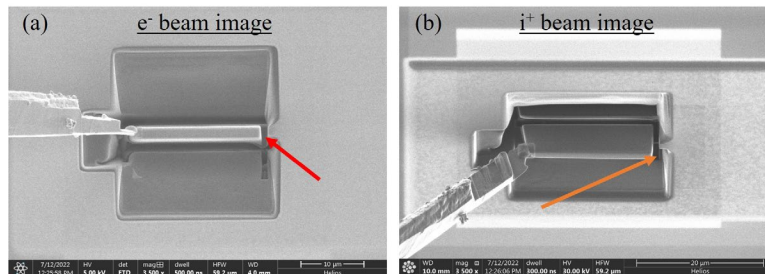


Figure 28. (a) e^- beam image and (b) i^+ beam image after cutting right side of lift-out.

6.2.2 Lift the Sample Out of The Trench

- 1) *Retract the MC4 nozzle.*
- 2) Use the EasyLift Z control buttons to lift the probe straight out of the trench at approximately 200 nm/s. After approximately 5 s, increase the speed up to 10 $\mu\text{m/s}$ and hold up for another 5 s. The sample should have cleared the trench (Figure 29). There is no i^+ beam image for this step because the sample is beyond the e^- - i^+ intersection height (4 mm WD).

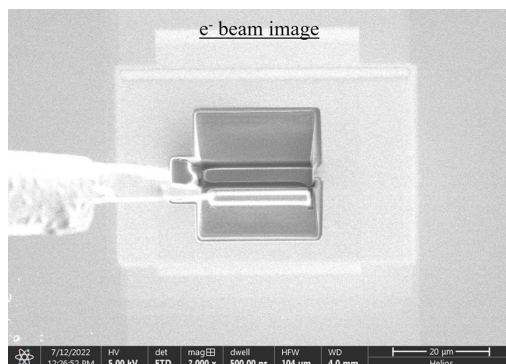


Figure 29. e^- beam image after the sample is lifted out of the trench.

6.2.3 Retract the Probe

- 1) Click park position.
- 2) Click go to, then click retract. Figure 30 shows the empty trench.

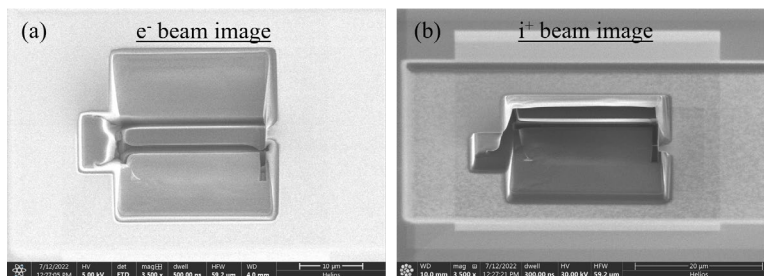


Figure 30. Trench after we lift out the sample (a) e^- beam image and (b) i^+ beam image.

6.3 Attach the Sample to the Grid

6.3.1 Move to Grid Location

- 1) Click on the grid holder in the Nav-Cam window.
- 2) Navigate at low magnification in the e^- beam view until you find the grid. We will use the left-hand side of the post.
- 3) Figure 31 shows the posts and valleys of the grid.

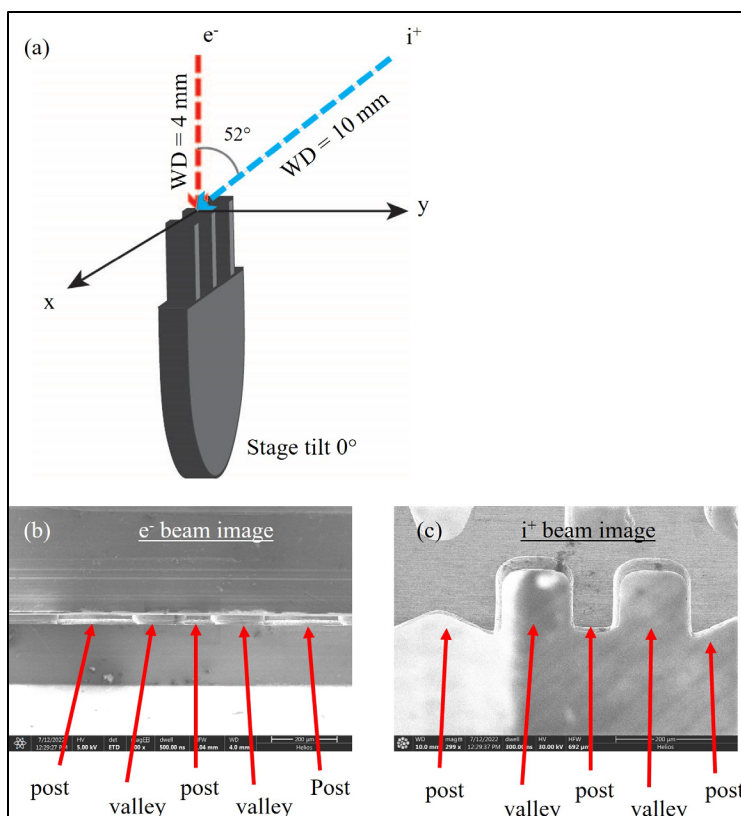


Figure 31. (a) Sample geometry and (b) e^- beam image and (c) i^+ beam image of the grid posts with the peaks and valleys labeled.

- 4) Use the align feature tool to rotate so the grid is horizontal. If ledges on grid are facing the wrong way, rotate the stage 180°.
- 5) Zoom to the top of post, focus, link FWD, set WD to 4 mm, focus, link, and set to 4 mm again.
- 6) Align with the part of grid that is in the middle (front to back) and sticking out so that there is a place to attach with Pt (Figure 32). Adjust Z in the camera window (live in ion beam view), We need 20 μm on one side to provide room for the probe.

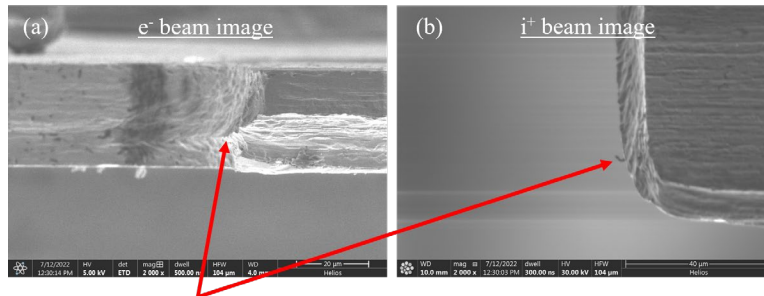


Figure 32. Higher magnification images of the (a) e^- beam image and (b) i^+ beam image of the selected grid post. The i^+ beam image is upside down, so move Z opposite of the direction that you want.

6.3.2 Clean the Mo Post Edge with the i^+ Beam

- 1) We only need this set of steps for Mo grids, since the Cu grids are smoother.
- 2) Set the i^+ beam to 9.1 nA (or 2.4 if aperture is not good). Turn up the contrast. Use a rectangle milling pattern with Y approximately 10 μm to flatten the edge of the grid post so there is a good place to attach the sample. Choose Si, then press play.
- 3) The red arrows in Figure 33 show a small triangle shelf created from milling. We will use that for aligning the sample in the next step.

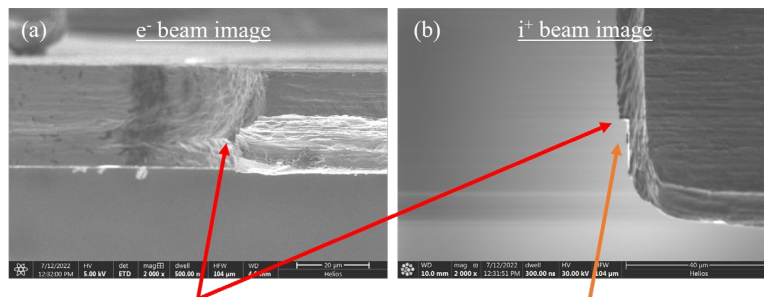


Figure 33. (a) e^- beam image and (b) i^+ beam image after cleaning the post edge.

- 4) Mill the edge of the post to remove just enough material to create a flat surface longer than the height of the lift-out (orange arrow, Figure 33b).

6.3.3 Bring Lift-Out to the Position Next to the Post

- 1) Set the i^+ beam to 26 pA.
- 2) You need 25 μm of space to the left of the post in the e^- beam view for the probe and sample. Zoom in so that the scale bar is approximately 10 μm and move the stage so that enough of the post is visible to attach the lift-out to it on the right side, and there is enough field of view to the left of it to see approximately 25 μm and be able to see the probe, sample, and post edge.

- 3) Check the i^+ beam view and use beam shift (hold shift button and drag feature with mouse) as necessary so that it aligns laterally with the e^- beam positioning. Use the stage Z control to move vertically and center the part of the post where you want to attach the sample. This is important because you cannot move the stage with an EasyLift probe inserted.
- 4) Zoom all the way out.
- 5) Go to the EasyLift icon, click insert, and click park position in the pop-up window.
- 6) Set movement speed to 10 $\mu\text{m/s}$.
- 7) Use e^- beam view to adjust X–Y position of probe until it aligns to the left of the post.
- 8) Use i^+ beam view at low magnification to bring probe down in Z until it is at the height where you want to attach. (Before it gets too close, insert MC4.)
- 9) Turn speed down to 1 $\mu\text{m/s}$ and use e^- beam to position it just left of the post so you can see a small gap between sample and post and with the back face of the sample flush with the back face of the post ledge. Use i^+ beam view to finely adjust the X position.
- 10) Make the back edge of the sample flush with the back of milled, flat area (red arrow, Figure 34a).

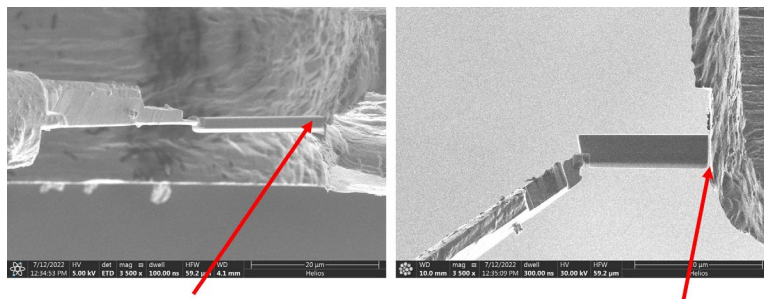


Figure 34. (a) e^- beam image and (b) i^+ beam image of the probe holding the lift-out as it approaches the post.

- 11) Leave a small gap. We will fill this gap with Pt (red arrow, Figure 34b).
- 12) Use the beam shift (hold shift key and click and drag feature with the mouse) to center the sample if needed.
- 13) If you are making another sample later, name this grid position by hitting “add” in the EasyLift menu.

6.3.4 Attach Lift-Out to Post using i^+ Beam Deposition

- 1) Set i^+ beam to 0.26 nA and take an image.
- 2) Turn up the contrast. Use a rectangle deposition pattern with $Z = 1\ \mu\text{m}$ to attach the sample.
- 3) Insert MC4 nozzle and make sure the application is Pt_M Weld. Hit play.
- 4) Narrow the box slightly in Y and extend slightly in X and do an additional $Z = 0.5\ \mu\text{m}$ deposition. The Y value is approximately equal to the height of the sample and the X feature is around $4.5\ \mu\text{m}$. Hit play.
- 5) Ideally, no gap should be visible because we filled it with Pt (red arrow, Figure 35a).

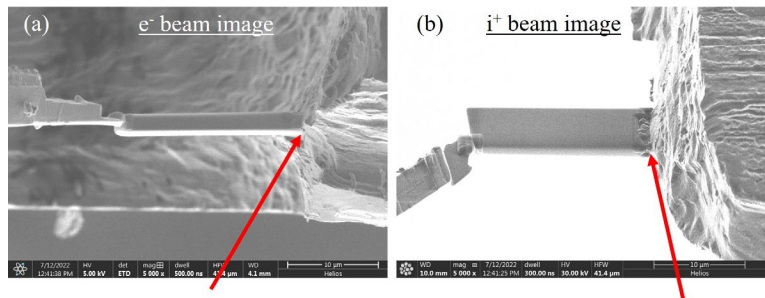


Figure 35. (a) e^- beam image and (b) i^+ beam image attaching the lift-out to the post.

- 6) Use enough Pt to securely attach the sample to the post (red arrow, Figure 35b).

6.3.5 Cut the Lift-Out Free from the Probe

- 1) Draw a milling box that just covers the Pt used to attach the sample to the probe with Z approximately $= 3\ \mu\text{m}$. The smallest X direction is $0.7\ \mu\text{m}$. We mill from left to right. Make sure i^+ beam current is 0.26 nA, and the application is set to Si.
- 2) Use iSPI to watch cutting progress. Stop milling once probe detaches (Figure 36).

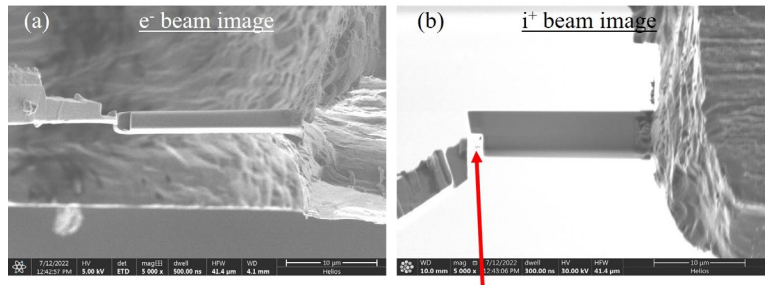


Figure 36. (a) e^- beam image and (b) i^+ beam image after freeing the probe.

- 3) You can take an i^+ beam snapshot while milling to confirm but minimize the number of images since the sample's side faces to the beam.
- 4) Once free, move the probe to the left in X until it is well away from sample, set speed to $10\ \mu\text{m/s}$, bring it up in Z for a few seconds, select park position, go to, and then retract. Watch this happen in the e^- beam window.

Remove as little of sample and probe tip as possible and try to leave probe with flat end for next specimen.

7. Final Thinning

7.1 Planarize Sample

7.1.1 Tilt to Front Face Milling Angle and Planarize

- 1) *Remove MC4 nozzle.*
- 2) Ctrl-I to tilt to 52° .
- 3) Adjust Z by clicking the e^- beam, and clicking and holding the mouse wheel, and dragging the cursor (in the camera view). If it was far off, tilt to 0° (Ctrl-E), recenter specimen, tilt back to 52° , and adjust again as necessary.
- 4) Change i^+ beam to $0.75\ \text{nA}$.
- 5) Take a low magnification i^+ beam image.
- 6) Hold shift and click and drag the specimen to the center crosshair. Tilt to 53.7° . For extremely hard samples, like sapphire, we may use 54° zoom in until specimen fills e^- beam view like below. Take an i^+ beam image. Adjust the focus as needed. (Use beam shifts to center if needed.)
- 7) Bright, visible front face of specimen (Figure 37) shows it is not planar.

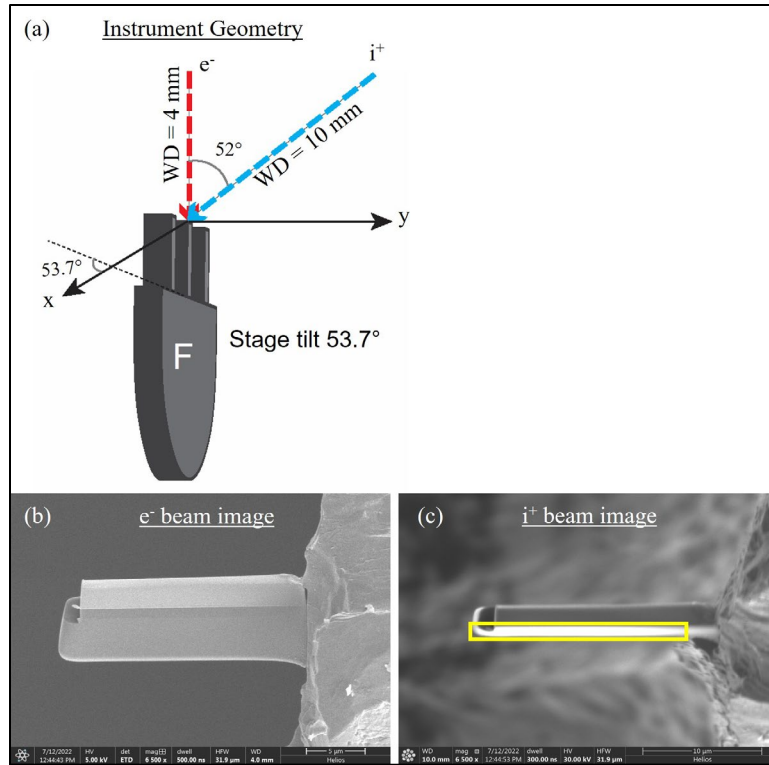


Figure 37. (a) e^- beam image and (b) i^+ beam image after tilting to the front face milling angle.

- 1) Draw a cleaning cross-sectional box and place it like shown in Figure 38b, so that it starts just below the bottom of the sample and removes the visible front face of the specimen. $Z = 9$ is default, increase if the material is harder than Si or lift-out is taller than normal.
- 2) Scan bottom to top. Set iSPI to line interval 1, turn on e^- beam view, and hit play.
- 3) Cut line should appear at the bottom first and move up as lines progress.
- 4) If it does not appear to have cut all the way down the sample after a line finishes, immediately click the “proceed with previous line” button.
- 5) Front face of specimen should be perfectly smooth and specimen features should be visible (grain boundaries, and thin film layers).
- 6) Sample appears to have lost height from the bottom because it is thinner and viewed from a downward angle (Figure 38a, red arrow).

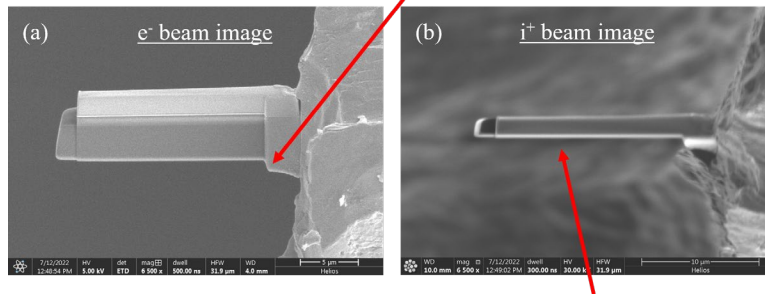


Figure 38. (a) e^- beam image and (b) i^+ beam image after planarizing and thinning the front face.

- 7) If sample does not look like right image (red arrow, Figure 38b), then adjust placement of box and repeat. The front face of the sample should no longer be visible in i^+ beam (Figure 38b). It is now completely planar with plane normal perpendicular to beam direction.

7.1.2 Rotate 180° and Tilt to Reversed Face Milling Angle, Planarize

- 1) Tilt to 0° (Control-E).
- 2) Drop the stage to $Z = 6$ mm, rotate 180° (keyboard shortcut on the left side), center sample, focus, link FWD, set FWD to 4, focus, link, and set to 4 again
- 3) Tilt to 52°, adjust height as necessary, tilt to 53.7° (54° for hard samples), take an i^+ beam image, and use beam shift to center as needed
- 4) We are now viewing what was the back side of specimen, the bright, visible front face of specimen shows it is not planar (Fig. 39).

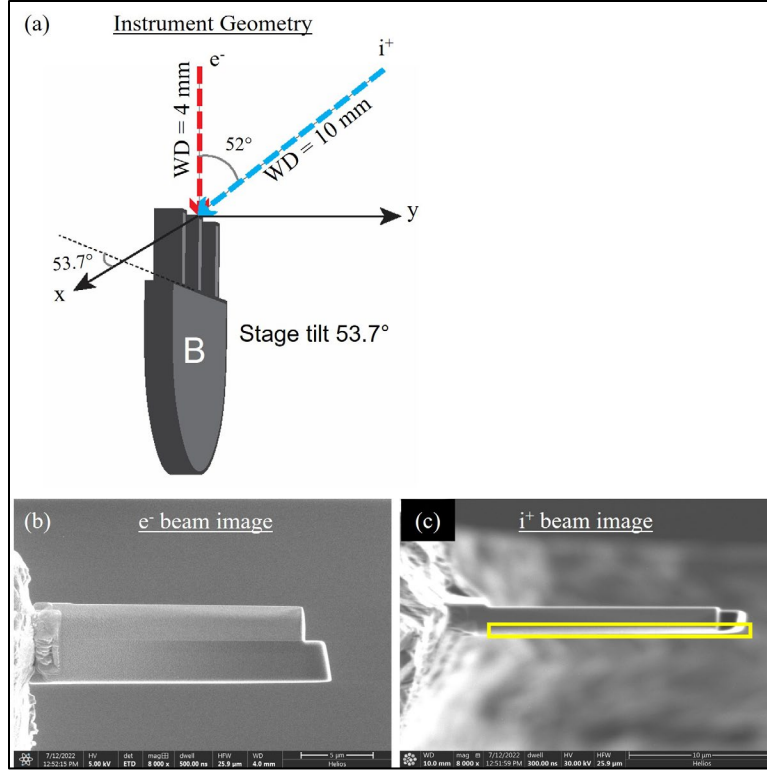


Figure 39. (a) e^- beam image and (b) i^+ beam image after switching to the back face.

We now perform the same procedure as Section 7.1.1, but on what was the back face prior to rotating. Use the same cleaning cross-sectional box as before. Reposition the yellow box and hit play. The sample now appears as shown in Figure 40.

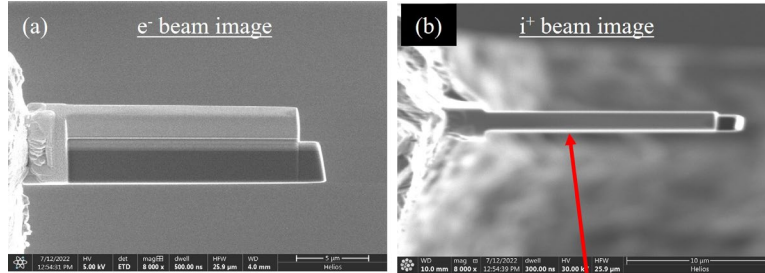


Figure 40. (a) e^- beam image and (b) i^+ beam image after thinning the back face.

7.2 Lower-Energy Thinning

7.2.1 Thin Reversed Face at 5 kV

At this point, minimize imaging the sample with the e^- beam and especially the i^+ beam.

- 1) Change i^+ beam voltage to 5 kV, current = 0.57 nA.
- 2) Increase contrast, take an image, and use beam shift if necessary. Adjust focus slightly, and take a new image, repeat until in focus.
- 3) Use the existing cleaning cross-sectional box. Position bottom at bottom edge of sample and top at halfway through the remaining sample width (Z ~3).
- 4) Cut line will appear at the top of the sample first. If it does not make it all the way down after a given line, immediately click the “proceed with previous line” button.
- 5) As the lines progress, the shelf indicated below will get visibly deeper. If a line/shelf appears horizontally, then the cuts are not going all the way down —back up and increase Z setting before milling the other side.
- 6) If the sample does not look like Figure 41b, with no visible sidewall sticking out, adjust box position and repeat.
- 7) Cross-sectional box should be positioned slightly to the right of previous one, so the new shelf is visible. If shelf does not make it to bottom, back up (Figure 41a). Approximately half of the previous thickness is removed (Figure 41b).

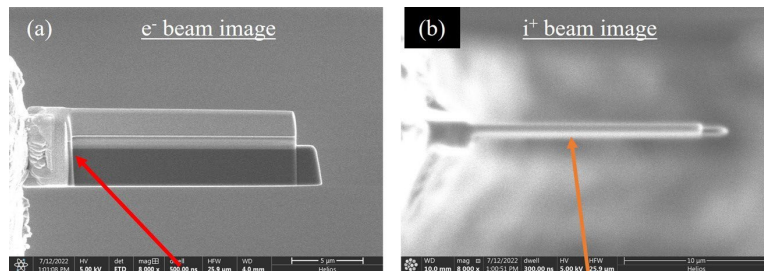


Figure 41. (a) e^- beam image and (b) i^+ beam image after low-energy thinning of back face.

7.2.2 Rotate 180° and Tilt to Front Face Milling Angle

We now return to original thinning orientation to perform 5-kV thinning.

- 1) Tilt to 0°. (Control-E) (Figure 42a).

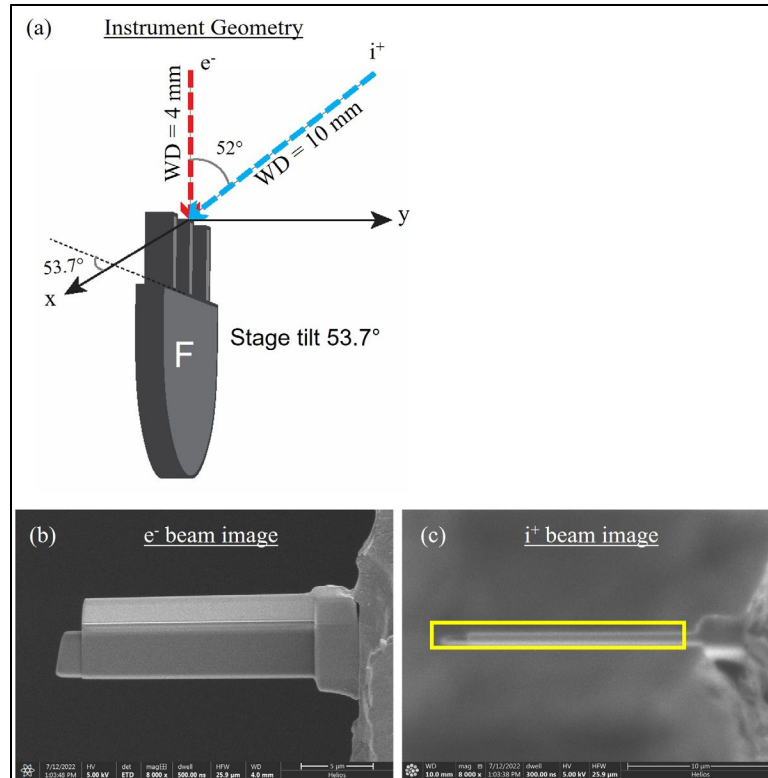


Figure 42. (a) e^- beam and (b) i^+ beam image after orienting for front face milling at 5 kV.

- 2) Lower the stage to $Z = 6$ mm.
- 3) Rotate the stage by 180°, center the sample (double-click on it), focus, link FWD, and set FWD to 4.
- 4) Focus, link, set to 4 again, follow steps from Step 21 (tilt to 52°, adjust height as necessary, tilt to 53.7° [54.0° for hard samples]), take an i^+ beam image, and use beam shift to center as needed.
- 5) Position the cleaning cross section so that it goes past the top of the sample and leaves a new shelf—stop it manually (Figure 42b). The cleaning cross section runs from bottom to top.

7.2.3 Thin the Front Face at 5 kV

This is the same as the procedure outlined in Section 7.2.1, except that the top of the box should go past the top edge of the sample (Figure 43b).

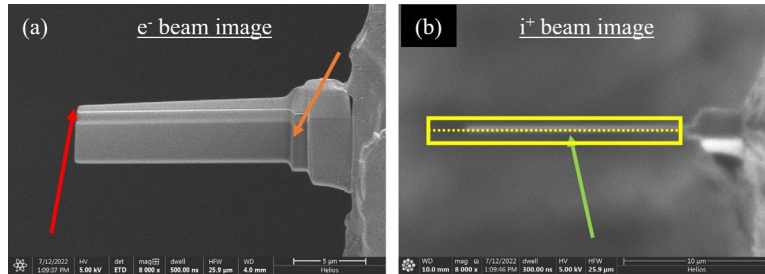


Figure 43. (a) e^- beam and (b) i^+ beam image after thinning the front face at 5 kV.

- 1) Back up if it appears that we are not cutting all the way down, or if Pt is being lost very quickly. Stop when Pt is almost depleted (Figure 43a).
- 2) The shelf indicated by the orange arrow in Figure 43a should always be visible. If it is not, back up a line. Stop milling when Pt is almost gone at end of specimen (red arrow, Figure 43b). Back off a line if Pt is depleting quickly but a lot of the sample is visible above the progression line.
- 3) In Figure 43b, the sample should be barely visible past the dashed line where milling stops. This image was from after thinning and will not have refreshed live, which is why we see the sample above that dashed line.

7.2.4 Clean Front Face at 2 kV

- 1) Change i^+ beam voltage to 2 kV. Turn up contrast and take a snapshot. Adjust the focus if needed.
- 2) Draw a rectangle milling box (Figure 44b), covering the entire thinned region of the specimen.

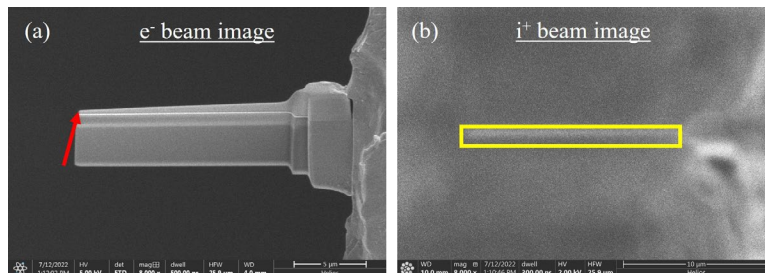


Figure 44. Watch the e^- beam image (a) where the red arrow is pointing, mill for $0.1\ \mu\text{m}$ of Z or until half of remaining Pt is gone, whichever occurs first. (b) rectangle indicating cleaned area in the i^+ beam image.

- 3) Set Z = $0.1\ \mu\text{m}$. Set iSPI to 3 s. Hit start.

- 4) Stop if remaining Pt is approximately 50% depleted.

7.2.5 Tilt and Clean Back Face at 2 kV

- 1) Tilt the stage to 50.3° , or 50° for hard samples (Figure 45a).

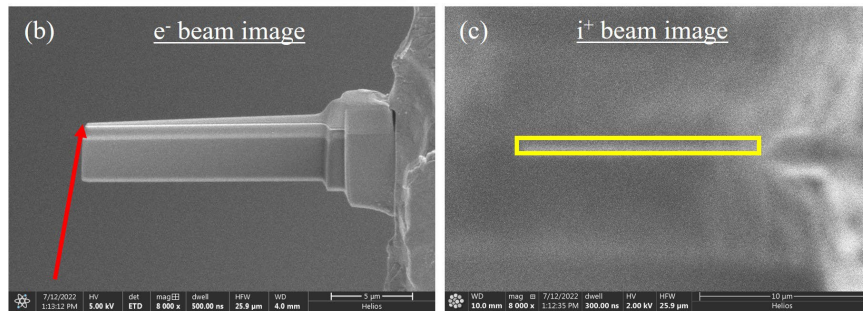
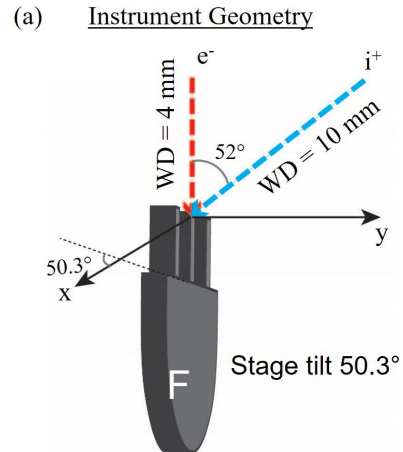


Figure 45. (a) e⁻ beam image and (b) i⁺ beam image after cleaning the back face at 2 kV.

- 2) Note the remaining Pt thickness (Figure 45b).
- 3) Change the scan direction from top to bottom.
- 4) Position the box as shown in Figure 45c. Since 2-kV resolution does not provide good imaging, align the cleaning cross section with bottom edge of the sample. Set Z for 0.1 μm .
- 5) Hit start. ***Stop if the Pt becomes depleted.*** Watch the edge near where the red arrow is pointing in Figure 45b.

8. Finished Sample and Shutdown

8.1 Finished Sample

Figure 46 shows the final sample as oriented in (a) and imaged with the e^- beam (b). There is no i^+ beam image because *we never expose the finished sample face to the i^+ beam.*

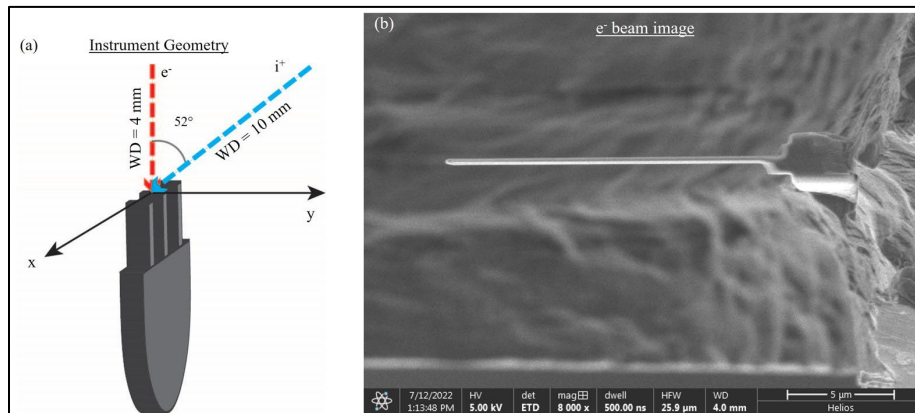


Figure 46. (a) Geometry and (b) e^- beam image of the sample at the end of the procedure.

8.2 Shutdown

- 1) Tilt the stage back to 0°.
- 2) Turn the beam off.
- 3) Vent the specimen chamber.
- 4) Return i^+ beam to 30 kV.
- 5) Zero out any scan rotations, the stigmation, and the beam tilt.
- 6) Remove and store your specimen.
- 7) Close the SEM chamber door and pump the chamber.

9. Summary/Conclusion

At the end of this procedure, you should have a large thin area for TEM examination. If the FIB process left contamination on the sample, then a short low-angle ion cleanup in the PIPS may be necessary. Be sure to use the washer in addition to the clamping plate in the TEM specimen rod.

10. References

1. Liao Y. Practical electron microscopy and database – an online book. GlobalSino; 2006.
2. Balakrishnan G et al. Room-temperature optically pumped (Al)GaSb vertical-cavity surface-emitting laser monolithically grown on an Si(1 0 0) substrate. IEEE J Sel Top Quantum Electron. 2006;12:1636–1641. <https://doi.org/10.1109/jstqe.2006.885342>
3. Belenky G et al. Properties of unrelaxed $\text{InAs}_{1-x}\text{Sb}_x$ alloys grown on compositionally graded buffers. Appl Phys Lett. 2011;99:141116. <https://aip.scitation.org/doi/abs/10.1063/1.3650473>
4. Collins CJ et al. Enhanced room-temperature luminescence efficiency through carrier localization in $\text{Al}_x\text{Ga}_{1-x}\text{N}$ alloys. Appl Phys Lett. 2005;86(3):031916. <https://doi.org/10.1063/1.1856702>
5. Dong H et al. Highly transparent and toughened poly(methyl methacrylate) nanocomposite films containing networks of cellulose nanofibrils. ACS Appl Mater Interfaces. 2015; 7(45):25464–25472. <https://doi.org/10.1021/acsami.5b08317>
6. Svensson SP et al. Molecular beam epitaxy control and photoluminescence properties of InAsBi. J Vacuum Sci Technol B. 2012;30:02B109. <https://doi.org/10.1116/1.3672023>
7. Svensson SP et al. Band gap of $\text{InAs}_{1-x}\text{Sb}_x$ with native lattice constant. Phys Rev B. 2012;86:245205. <https://doi.org/10.1103/PhysRevB.86.245205>
8. Thompson MP et al. Deposition factors and band gap of zinc-blende AlN. J Appl Phys. 2001;89:3331–3336. <https://doi.org/10.1063/1.1346999>
9. Zheleva T et al. Transition layers at the SiO_2/SiC interface. Appl Phys Lett. 2008;93:022108. <https://doi.org/10.1063/1.2949081>
10. Zhu L et al. Engineering topochemical polymerizations using block copolymer templates. J Am Chem Soc. 2014;136(38):13381–13387. <https://doi.org/10.1021/ja507318u>

11. Darling KA et al. Extreme creep resistance in a microstructurally stable nanocrystalline alloy. *Nature*. 2016;537:378–381. <https://doi.org/10.1038/nature19313>
12. Sarney WL et al. Aluminum metallization of III–V semiconductors for the study of proximity superconductivity. *J Vac Sci Technol B*. 2020;38:032212. <https://doi.org/10.1116/1.5145073>
13. Sardashti K et al. Tuning superconductivity in Ge:Ga using Ga⁺ implantation energy. *Phys Rev Mater*. 2021;5(6):064802. <https://doi.org/10.1103/PhysRevMaterials.5.064802>
14. Sardashti K et al. Tailoring superconducting phases observed in hyperdoped Si:Ga for cryogenic circuit applications. *Appl Phys Lett*. 2021;118:073102. <https://doi.org/10.1063/5.0039983>
15. Strnad NA et al. Plasma enhanced atomic layer deposition of textured aluminum nitride on platinized substrates for MEMS. *J Vac Sci Technol A*. 2022;40:042403. <https://doi.org/10.1116/6.0001633>
16. Sarney WL et al. Influence of strain on the InAs_{1-x}Sb_x composition. *J Vac Sci Technol B*. 2020;38(3):032206. <https://doi.org/10.1116/1.5145066>
17. Aqib M et al. Crack-free > 1-μm AlN layer on Si substrate using ductile interlayer for strain modification in epitaxial film. *Appl Phys Lett*. 2024;125(11):112104. <https://doi.org/10.1063/5.0219833>
18. Sarney WL et al. Formation of spontaneous lateral heterostructures in high Al content Al_xGa_{1-x}N alloys grown by high-temperature plasma-assisted molecular beam epitaxy. *J Electron Mater*. 2024;53:2789–2797. <https://doi.org/10.1007/s11664-024-10952-x>
19. Sales MG et al. WSe₂ growth on hafnium zirconium oxide by molecular beam epitaxy: the effect of the WSe₂ growth conditions on the ferroelectric properties of HZO. *2D Mater*. 2021;9:015001. <http://dx.doi.org/10.1088/2053-1583/ac2d3b>
20. Hanrahan B et al. The other model antiferroelectric: PbHfO₃ thin films from ALD precursors. *APL Mater*. 2021;9:021108. <https://doi.org/10.1063/5.0035730>
21. Payne A et al. Dielectric, energy storage, and loss study of antiferroelectric-like Al-doped HfO₂ thin films. *Appl Phys Lett*. 2020;117:221104. <https://doi.org/10.1063/5.0029706>

22. Najmaei S, Ekuma CE, Wilson AA, Leff AC, Dubey M. Dynamically reconfigurable electronic and phononic properties in intercalated HfS₂. *Mater Today*. 2020;39:110–117. <https://doi.org/10.1016/j.mattod.2020.04.030>
23. Sarney WL et al. Monolithic integration and ferroelectric phase evolution of hafnium zirconium oxide in 2D neuromorphic synaptic devices. *Mater Today Nano*. 2023;24:100378. <https://doi.org/10.1016/j.mtnano.2023.100378>
24. Zhou W et al. Developing a chip-scale optical clock. *Opt Eng*. 2021;60(2):027107. <https://doi.org/10.1117/1.OE.60.2.027107>
25. Ni JH, Sarney WL, Leff AC, Cahill JP, Zhou W. Wavelength-thick ENZ ITO metafilm for near IR photonic devices. *Conference on Lasers and Electro-Optics*; 2020. https://doi.org/10.1364/Cleo_Qels.2020.fm1b.3
26. Ni JH, Sarney WL, Leff AC, Cahill JP, Zhou W. Property variation in wavelength-thick epsilon-near-zero ITO metafilm for near IR photonic devices. *Sci Rep*. 2020;10(713):713. <https://doi.org/10.1038/s41598-020-57556-z>
27. Wu C-C, Wen J, Walck SD, Pesce-Rodriguez RA, Arslan I. Advanced nanoscale characterization of aluminum nanoparticles with modified surface morphology via atmospheric helium and carbon monoxide plasmas. *J Appl Phys*. 2021;129(6):063302. <https://aip.scitation.org/doi/abs/10.1063/5.0037637>
28. Malek MI, Wu C-C, Walck SD, Pantoya ML. Hydration of alumina (Al₂O₃) toward advancing aluminum particles for energy generation applications. *Colloids Surf A Physicochem Eng Asp*. 2022;652(5):129740. <https://doi.org/10.1016/j.colsurfa.2022.129740>
29. Hu Y et al. Chemically driven energetic molecular ferroelectrics. *Nat Commun*. 2021;12(5696). <https://doi.org/10.1038/s41467-021-26007-2>
30. Sharma S et al. Investigation of microstructural and thermal stability of Ni-Y-Zr ternary nanocrystalline alloy. *Mater Charact*. 2024;217:114378. <https://doi.org/10.1016/j.matchar.2024.114378>
31. Jannotti P et al. Characterizing stable nanocrystalline Cu-Ta behavior and failure dynamics under extremes of strain rate, strain, temperature and pressure by modified dynamic tensile extrusion. *Acta Materialia*. 2024;263:119476. <https://doi.org/10.1016/j.actamat.2023.119476>
32. Field DM, Magagnosc DJ, Hornbuckle BC, Lloyd JT, Limmer KR. Manipulation of the stacking fault energy of a medium-mn steel through

- temperature and hierarchical compositional variation. *Metall Mater Trans A*. 2024;55:161–172. <https://doi.org/10.1007/s11661-023-07239-x>
33. Li H, Salamanca-Riba L. The concept of high angle wedge polishing and thickness monitoring in TEM sample preparation. *Ultramicroscopy*. 2001;88(3):171–178. [https://doi.org/10.1016/S0304-3991\(01\)00079-1](https://doi.org/10.1016/S0304-3991(01)00079-1)
 34. Walck SD. A Simplified method for modifying TEM copper grids for use with the small angle cleavage technique. *Microscopy Today*. 1996;4(4):12–13. <https://doi.org/10.1017/S1551929500068413>
 35. Walck SD, McCaffrey JP. The small angle cleavage technique: an update. *MRS Online Proc Lib*. 1997;480:149–171. <https://doi.org/10.1557/PROC-480-149>
 36. Stokes DJ, Hayles MF. Methodologies for the preparation of soft materials using cryoFIB SEM. *Proc SPIE Int Soc Opt Eng*. 2009;7378. <https://doi.org/10.1117/12.821834>
 37. Schaffer M, Schaffer B, Ramasse Q. Sample preparation for atomic-resolution STEM at low voltages by FIB. *Ultramicroscopy*. 2012;114:62–71. <https://doi.org/10.1016/j.ultramic.2012.01.005>
 38. Sales MG et al. Effects of atmospheric UV-O₃ exposure of WSe₂ on the properties of HfO₂/WSe₂ interface. *Appl Phys Lett*. 2023;123:123502. <https://doi.org/10.1063/5.0165599>
 39. Litwin PM et al. The growth of self-intercalated Nb_{1+x}Se₂ by molecular beam epitaxy: the effect of processing conditions on the structure and electrical resistivity. *J Vac Sci Technol A*. 2023;41:042707. <https://doi.org/10.1116/6.0002593>
 40. Hirsch SG. Efficacy of a FIB and planar preparation technique for producing TEM lamella of CLS. DEVCOM Army Research Laboratory (US); 2023. Report No.: ARL-TN-1159.
 41. Lee JS, Hill RT, Chilkoti A. Surface patterning. In: Wagner WR, Sakiyama-Elbert SE, Zhang G, Yaszemski MJ, editors. *Biomaterials Science: An Introduction to Materials in Medicine*. 4th ed. Academic Press; 2020. p. 553–573.
 42. Wilhelmi O, Reyntjens S, Van Leer B, Anzalone P, Giannuzzi LA. Focused ion and electron beam techniques. In: Lindroos V, Tilli M, Lehto A, Motooka T, editors. *Handbook of Silicon Based MEMS Materials and Technologies*. William Andrew Publishing; 2010. p. 323–325.

43. Nguyen N-T. Micromixers: fundamentals, design and fabrication. William Andrew Publishing; 2012. p. 113–161.
44. Thompson K et al. In situ site-specific specimen preparation for atom probe tomography. *Ultramicroscopy*. 2007;107(2-3):131–139. <https://doi.org/10.1016/j.ultramic.2006.06.008>
45. Leica EM ACE600 Sputter Coater. Leica Microsystems; c2024 [accessed 2024 Nov 7]. <https://www.leica-microsystems.com/products/sample-preparation-for-electron-microscopy/p/leica-em-ace600/>
46. Ted Pella, Inc.: microscopy products for science and industry. Ted Pella, Inc.; c2024 [accessed 2024 Nov 7]. <https://www.tedpella.com/>

List of Symbols, Abbreviations, and Acronyms

2-D	two-dimensional
ALC	Adelphi Laboratory Center
Ar ⁺	argon ion
ARL	Army Research Laboratory
Au	gold
Bi	bismuth
C	carbon
Cu	copper
DEVCOM	U.S. Army Combat Capabilities Development Command
e ⁻	electron beam
EDS	energy-dispersive X-ray spectroscopy
ETD	Everhart–Thornley detector
FIB	focused-ion beam
FWD	free working distance
Ga	gallium
HfO ₂	hafnium oxide
i ⁺	ion beam
Ir	iridium
iSPI	intermittent SEM imaging and FIB patterning
MC4	MultiChem gas injection system
MEMS	micro-electromechanical systems
Mo	molybdenum
Nav-Cam	navigation camera
Ni	nickel
nm	nanometer
PIPS	precision ion polishing system

pm	picometer
Pt	platinum
R	rotation
SAC	small-angle cleavage
SEM	scanning electron microscope
Si	silicon
STEM	scanning transmission electron microscopy
T	tilt
TEM	transmission electron microscopy
WD	working distance
WSe ₂	tungsten diselenide

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