Pacific Northwest
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PNNL-29451

Preparing FIB Lift Outs for Flash Electropolishing

October 2019

Alan L. Schemer-Kohrn
Steven R. Spurgeon

U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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The following instruction was adapted from a procedure used by Steven R. Spurgeon of PNNL.

Acronyms and Abbreviations

E-Beam – Electron beam
FIB/SEM – Focused Ion Beam/Scanning Electron Microscope
FEP – Flash Electrochemical Polishing
I-Beam – Ion beam

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1.0 Introduction

The process described below is written for the Focused Ion Beam/Scanning Electron Microscope (FIB/SEM) novice. For more experienced operators, please use the following guidelines when preparing samples for flash electrochemical polishing (FEP)

1.1 Guidelines

- Samples need to be mounted on a gold grid. A standard 100 mesh gold TEM grid that has been cut in half works well. Other mesh sizes are acceptable.
- Mark one side of the grid in some way to establish a fiduciary mark. A small triangle cut off the right-hand side works well.
- Since the samples will be handled repeatedly, securely attach the sample to the grid with a strong Pt weld.
- Samples work best as a 10µm square. Other geometries will work as well if required for the region of interest.
- Final thickness should be no less than 200nm, with a preferred target thickness of 250nm for Fe-Cr alloys, <300 nm for W alloys. Materials that are sensitive to ion implantation may be left thicker.
- A U-shaped support structure is required to provide resistance to buckling due to internal stresses in the sample. The thinning method described toward the end of this procedure works well to provide that structure.
- Final thinning should be done at 5-8keV. Final cleaning at 2keV on each side is optional.

1.2 General Tips and Notes

- Make sure the sources are heated and on.
- Zero the beam shifts and check the alignments.
- In general, the steps listed below are written for the FEI Quanta 3D.
- For materials that are not iron-based or that will use a NaOH solution for electropolishing, omit the carbon cap described below, and deposit ~1µm of platinum using the e-beam.

2.0 Process Instructions

2.1 Preparation

- Cut the gold grid as shown in the cut diagram image *(Figure 1)*. Working under a low-power stereo microscope, use a clean razor blade to make each cut.
- With the ridged edge of the grid facing up, the reference cut will be to the right.
- Mount the cut grid into the grid holder so that the ridged edge is facing up.
- Coat sample if needed to prevent charging. Conductive samples should not require this step.
- Mount sample securely, using copper tape over two opposite top corners to hold it and provide a good sample ground.
- Insert the sample into the chamber and bring into focus. Align the Focus/WD link.
- Translate the stage to Eucentric working distance (4mm for the Helios, 10mm for the Quanta). Align the FWD at 5-8kX on the sample
- Image at 5keV / 4.0nA (Quanta) or 5keV / 0.34nA (Helios).
- Align the edge of the sample using Stage → XT Align.
- Find eucentric height.

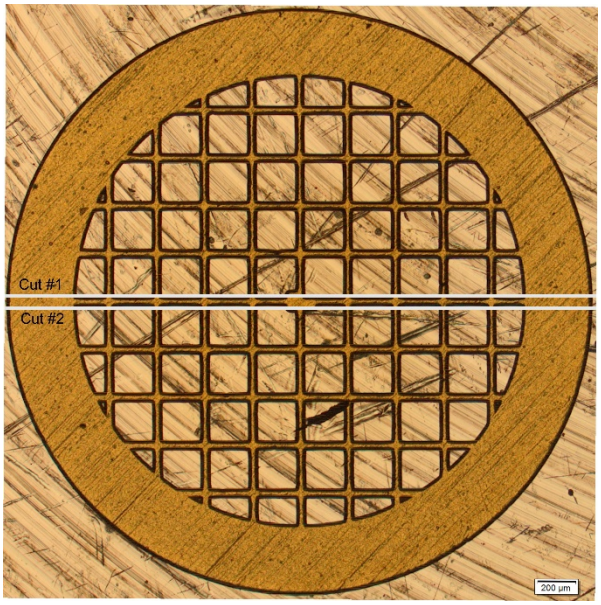

Figure 1. Gold Grid Cut Diagram

2.2 Surface Preparation

- Stage tilt = 0°
- Electron beam *(E-Beam)* = 5keV @ 4.0nA
- Insert Pt source GIS. (If desired, lay down 500nm C, then 500nm Pt.)
- Pattern = Rectangle 15µm x 5µm x 500nm. Mode = Pt E-beam Sur.
- Deposit ~500nm of E-Beam Pt. This may take multiple iterations or a greater Z dimension on the pattern.
- Remove the Pt GIS.

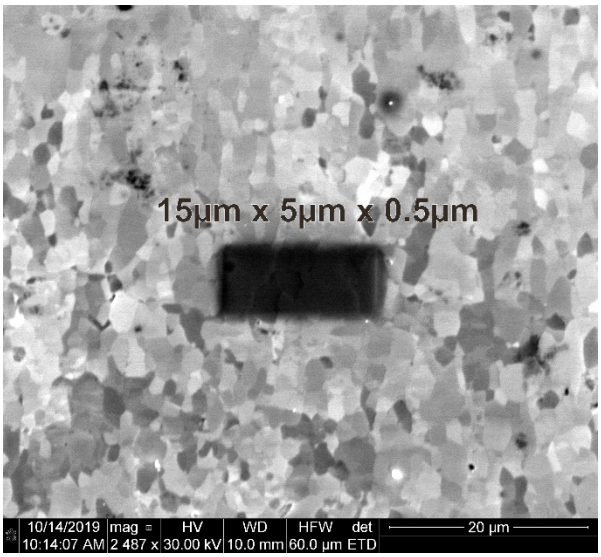

Figure 2. Electron Beam Caps

- Stage tilt = 52°
- Align beam shifts with I-beam. 30keV @ 30pA
- Insert C source GIS.
- Pattern = Rectangle 11µm x 2µm x 1.5µm. Mode = C Dep.
- Deposit ~1.5µm of carbon.
- Retract C GIS, turn off the C heating and insert Pt GIS and change mode to Pt Dep.
- Using the same pattern, deposit ~1.5µm of platinum.
- Retract the Pt GIS

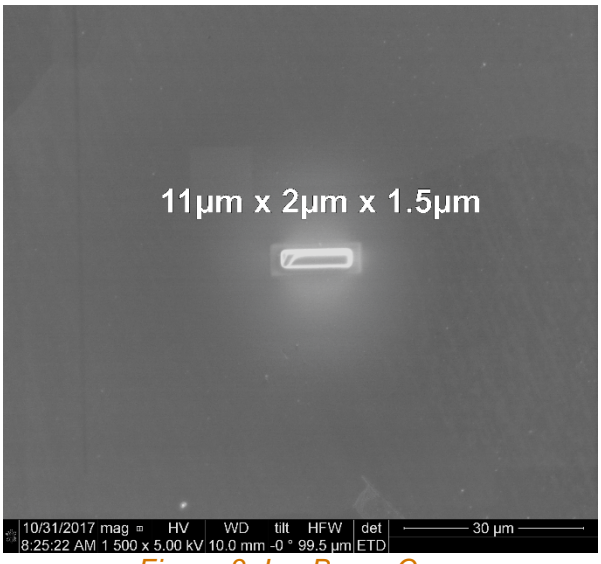

Figure 3. Ion Beam Caps

2.3 Trench Cuts

- Stage tilt = 52°

- I-Beam = 30keV @ 15nA (9nA in Helios)
- Pattern = *Regular* cross section 15µm x 15µm x 10µm. Mode = Si.
    - Advanced Options: Multiscan; # of Passes = 5; Scan Ratio =0.4
    - Place the heavy line of the pattern against the cap.
- Trench with I-Beam. The target is 14+µm deep. Measure in E-Beam using cross-section measuring tool.
- Do one of the two options below
    - 1) Move pattern to other side of cap, set pattern rotation to 180° **or**
    - 2) Return to 0° tilt, Select Eucentric rotation, Relative and rotate 180° and Check Eucentric position.
- Repeat trench cut above.

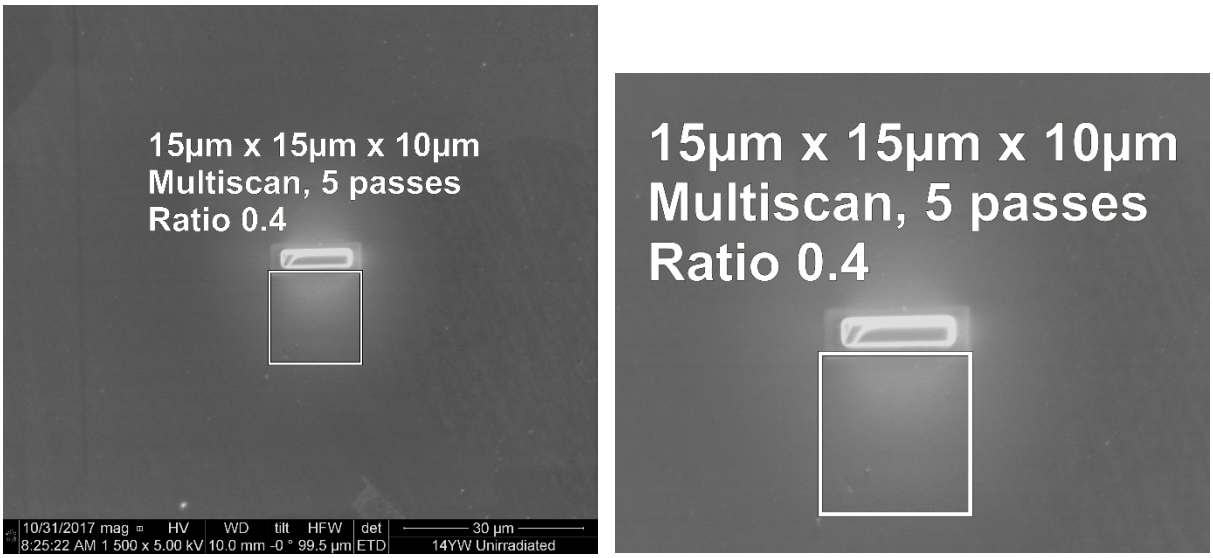

Figure 4. Trench Cut

- Stage tilt = 53.5°.
- I-Beam = 30keV @ 7nA. Check alignment.
- Pattern = Cleaning cross-section. 15µm x 3µm x 6µm. Mode = Si.
    - Heavy line faces the cap.
- Cut with I-Beam to flatten off the bottom face. Set Y dimension ~1µm wider than the slanted surface of the trench face.
- Stage tilt = 50.5°.
- I-Beam = 30keV @ 7nA. Check alignment.
- Pattern = Cleaning cross-section. 15µm x 3µm x 6µm. Mode = Si.
    - Heavy line faces the cap.
- Cut with I-Beam to flatten off the top face. Set Y dimension ~1µm wider than the slanted surface of the trench face.

Figure 5. Trench Cleaning Cut

2.4 Under Cut

- Stage tilt = 7°.
- I-Beam = 30keV @ 3nA. Set scan rotation to 180° (Shift-F12 on the Quanta).
- Pattern = Rectangle.
- With the measuring tool in cross-section mode, measure 10µm from the bottom of the cap into the trench.
- Draw a rectangle pattern 15µm x 1.5µm x 4µm. Place the top of the box 10µm below the sample surface.
- Draw two more rectangle patterns 1.5µm x 8µm x 4µm. Place the inside edge of the boxes even with the edges of the cap and overlapping the bottom box.
- Select simultaneous pattern.
- Cut while monitoring in I-Beam. Stop as soon as the cuts are all the way through. Periodically check in E-Beam during cut.
- Tilt = 53.5°. Cleaning cross-section to clean up top face.

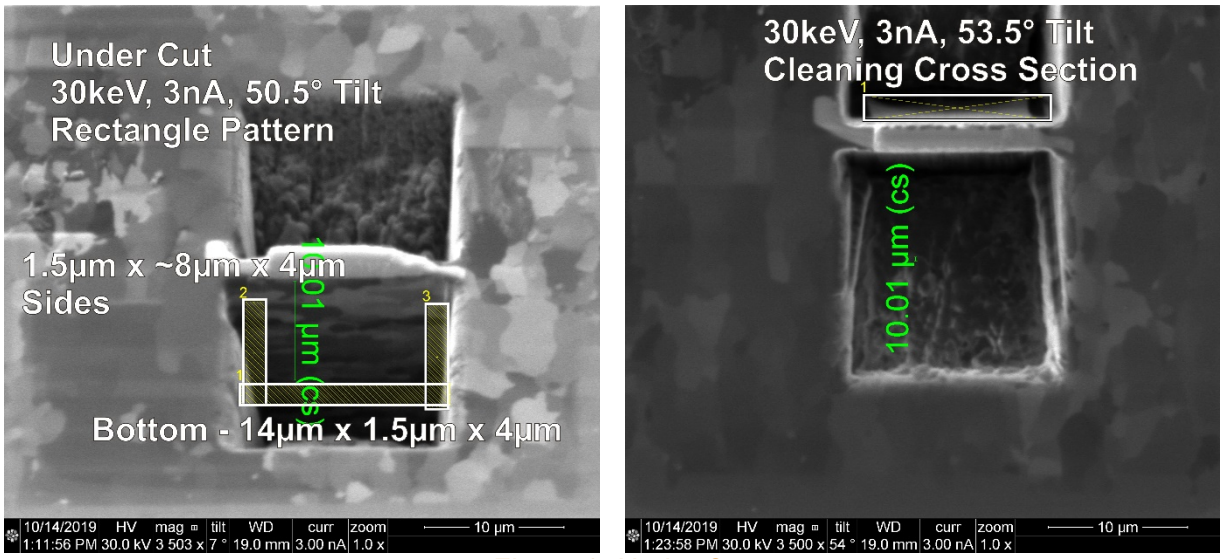

Figure 6. Under Cut

2.5 Attaching the Omniprobe Manipulator

- Stage Tilt = 0°.
- I-Beam = 30keV @ 30-45pA. Align I-Beam and E-Beam shifts.
- Pattern = Rectangle. Mode = Pt Dep. 2µm x 2µm x 1µm
- Insert Pt GIS needle
- Insert Omniprobe.
- Using AutoProbe (or appropriate driving) software, drive the Omniprobe from Park to Eucentric High.
- Lower Omniprobe tip to ~10µm of the sample surface and check the focus and position.
- Lower Omniprobe tip to ~5µm and check focus and position.
- Switch to E-Beam image. Align tip over the sample such that the top edge of the tip lines up with the top edge of the sample as seen in the E-Beam image,
- Switch to I-Beam image. Align the tip over the sample such that the left edge of the tip is 0.5-1.0µm inside the left edge of the cap.
- Draw the rectangular pattern and place such that the lower edge of the pattern overlaps the top edge of the cap at the desired attachment point.
- Lower the tip until it is inside the rectangular deposition pattern. Leave 0.5-1.0µm separation between the top of the cap and bottom of the Omniprobe tip.
- Deposit 1µm of platinum to attach the Omniprobe tip to the top of the cap.

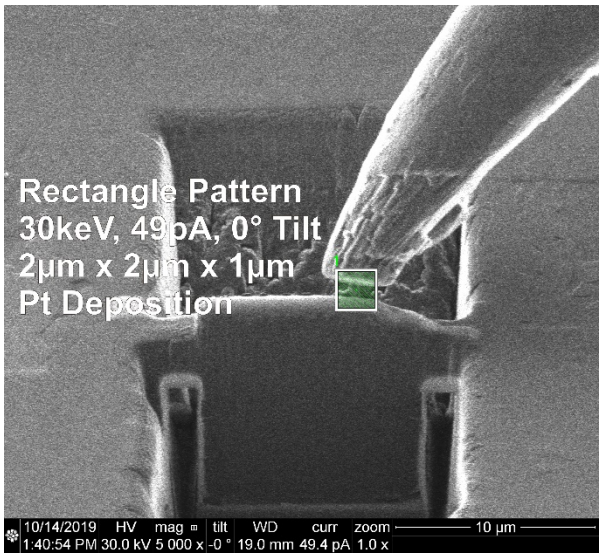

Figure 7. Attach the Omniprobe

2.6 Extracting the Sample

- Stage tilt = 0°.
- I-Beam 30keV @ 1.0nA (Increase current in steps while monitoring in E-Beam. Check to ensure that the weld between the sample and the tip does not break.
- Pattern set 1= Rectangle, 1.5µm x Bridge width +2µm x 4µm, Bottom-to-Top.
- Pattern set 2= Rectangle, 1.5µm x Bridge width +2µm x 4µm, Top-to-Bottom.
- Cut the bridges with two rectangular pattern sets. 1.5µm x 2µm longer than the width of the bridge x 4µm. Overlap the two sets.
- Carefully raise the Omniprobe out of the trench.
- Using a rectangular pattern, clean the left and bottom edges of the lamella to create flat mounting surfaces.
- Measure the left edge of the sample, including the cap.
- Drive the Omniprobe to Eucentric high → then to Park.
- Retract Omniprobe. Retract Pt GIS.

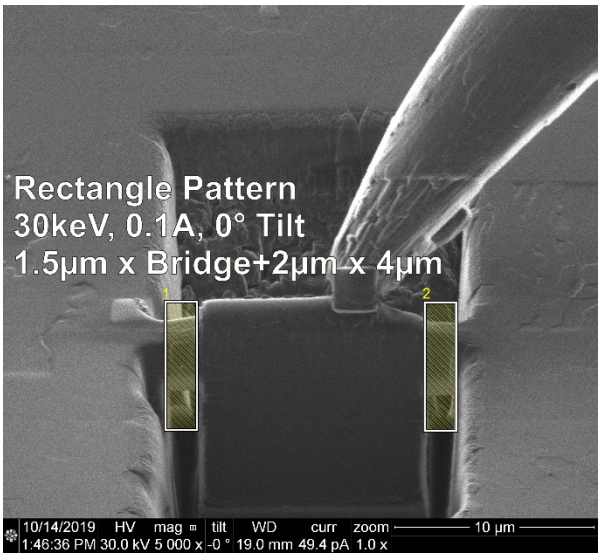

Figure 8. Extraction

2.7 Preparing the Sample Grid

- Stage tilt = 0°
- Move to the grid position. Rotate the grid so that the ridged side is pointing up in the E-Beam image.
- Check stage eucentric height.
- Select the post, typically the post to the right or left of the center. We are going to mount to the left side of the post in the E-beam.
- I-Beam = 30keV @ 15-30nA. Remove the top section of the post so that the top of the post is even with edge.
- I-Beam = 30keV @ 7nA. Remove a rectangle equal in height to the measurement in the previous step and 1µm wide. 1µm x Left Edge x 1-2µm.

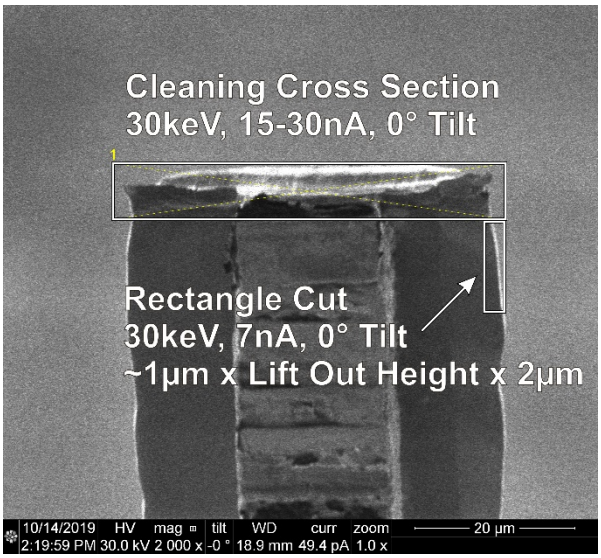

Figure 9. Prepare the Grid

2.8 Mounting the Lift Out on the Sample Grid

- Make sure shifts are aligned at I-Beam = 30keV @ 45pA.
- Insert Pt GIS needle.
- Insert Omniprobe, move to eucentric high and lower to within ~10µm of the grid.
- Align X and Y.
- Move to almost touch the grid post (bring down in Z first, then slide over last with X).
- Pattern = Rectangle, 2µm x 2µm x 1µm. Mode = Pt Dep. (Figure 10)
- Weld at top and bottom of sample to connect sample to the grid.
- I-Beam = 30keV @ 1nA (Increase carefully so as not to break any of the welds).
- Pattern = Rectangle. Rotate the rectangle clockwise 1-2°. Mode = Si.
- Cut the square connecting the sample to the Omniprobe tip. Use a thin rectangle that just touches the actual tip of the Omniprobe and passes over the top of the sample.
- Move the Omniprobe up in Z until it is clear of the sample. Drive to eucentric high → Park → then retract.
- I-Beam = 30keV @ 0.1nA.
- Pattern = Rectangle 2µm x Sample Height x 1µm. Mode = Pt Dep.
- Add a final weld to the sample/grid interface covering the two existing welds and all space in between.
- Retract Pt GIS.
- Rotate sample 135° clockwise in the E-Beam image.
- Insert Pt GIS.
- I-Beam = 30keV @ 0.1nA.
- Pattern 1 = Rectangle 2µm x Sample Height x 1µm, Pattern 2 = Rectangle 2µm x 2µm x 1µm.
- Mode = Pt Dep.
- Place 1 rectangle of pattern 1 at the top of the sample where it meets the grid. Rotate the pattern so that the edge of the pattern is parallel to the edge of the sample.
- Place a second rectangle of pattern 1 at the bottom of the sample where it meets the grid.
- Add the welds along the back face to finish securing the sample to the grid.
- Retract the Pt GIS.
- Rotate the sample to horizontal again.
- State tilt = 52°.
- Insert the Pt GIS.
- Pattern = Rectangle 12µm x 1µm x 1.5µm.
- I-Beam = 30keV @ 0.1nA.
- Center the pattern over the sample and align it so that it stretches from the end of the sample to ~1µm onto the grid. Lay down 1-1.5µm of platinum.
- Retract Pt GIS.

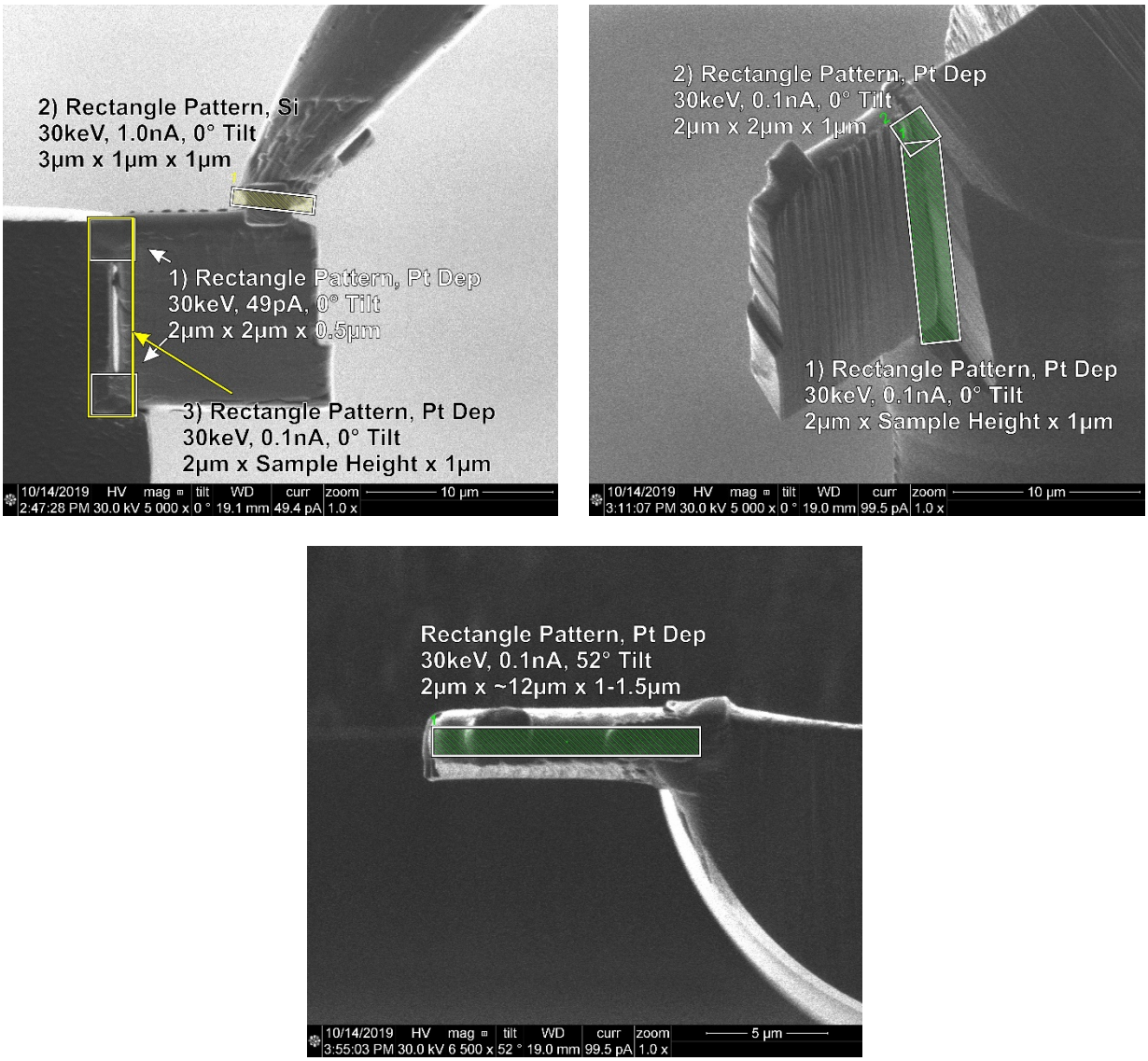

Figure 10. Attach Lift Out to Grid

2.9 Coarse Thinning of the Lamella

- Stage tilt = 0°
- Align sample horizontally and mark position.
- Rotate 180° and mark position of the other side of the sample.
- Use the average option on the SE detector while cutting to get better contrast.

When thinning windows, make sure not to clip the edge of the sample with the ion beam rectangle or you will amorphize the surface. See below for proper positioning of the window

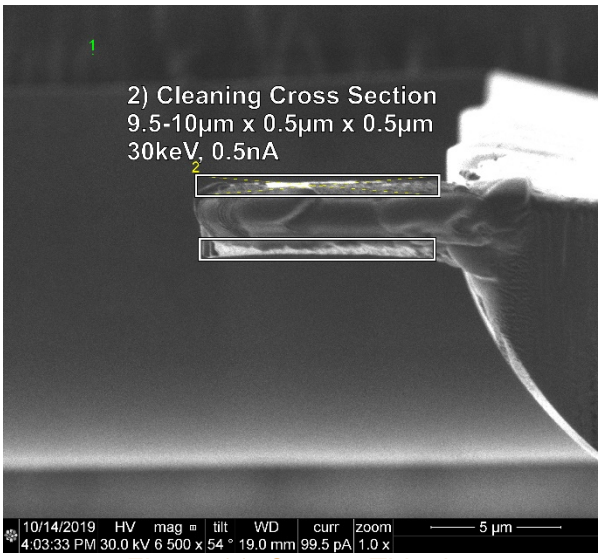

Figure 11. Coarse Thinning

- Rotate sample so that the front face (ridged side of the grid) is facing down in the E-Beam image.
- Stage tilt = 52°.
- I-Beam = 30keV @ 30pA. Align shifts.
- Stage tilt = 53.5°.
- Pattern = Cleaning cross-section 9.5-10µm x 0.5µm x 0.5µm.
- I-Beam = 30keV @ 0.5nA
- Using the cleaning cross-section, remove the platinum redeposition and outer layers of the sample. Repeat until the face of the sample is even with the final Pt cap.
- Stage tilt = 0°.
- Rotate the sample 180° and repeat cleaning.
  Target thickness is 350nm of material remaining.

2.10 Fine Thinning of the Lamella

- Once the sample is approximately 350nm thick, we can start the final thinning process.
- Stage tilt = 55°.
- I-Beam = 5keV @ 0.77nA
- Pattern = Rectangle 6µm x 0.25µm x 0.25µm. Advanced → Overlap = 85%.
- Center the rectangle horizontally on the sample and remove material to create a thinned window on the face.
- Stage tilt = 0°. Rotate 180°.
- Stage tilt = 55°.
- Repeat milling. Target thickness is 275 – 300nm.
- Stage tilt = 56.5°-57°.
- I-Beam = 5-8keV @ 100pA. Align I-Beam.

- Mill rectangle one or two times.
- Repeat for the other side.
- Final target is 250nm.

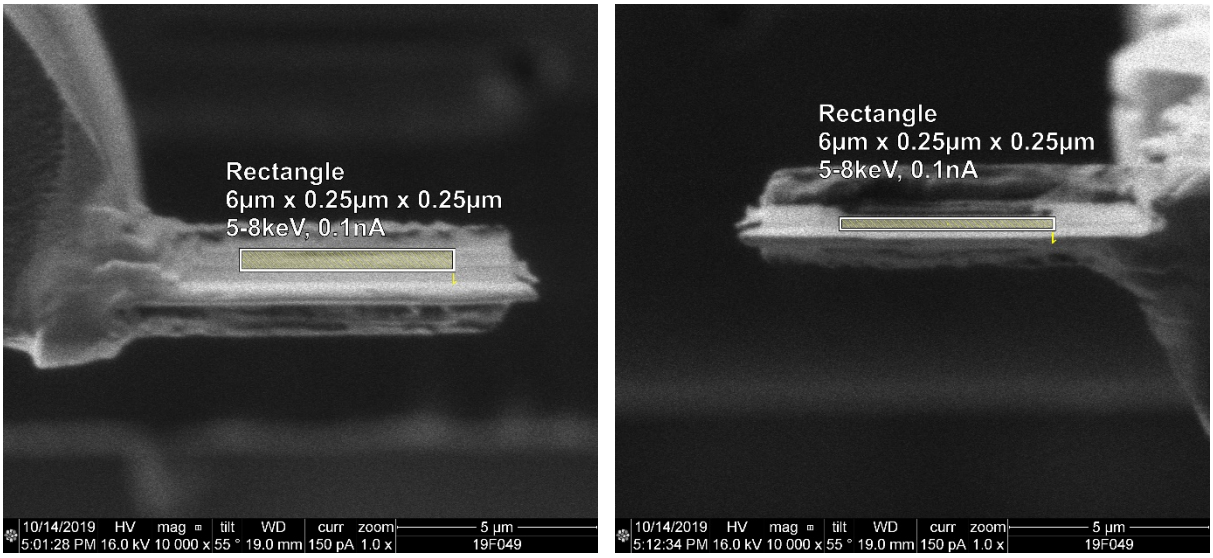

Figure 12. Fine Thinning

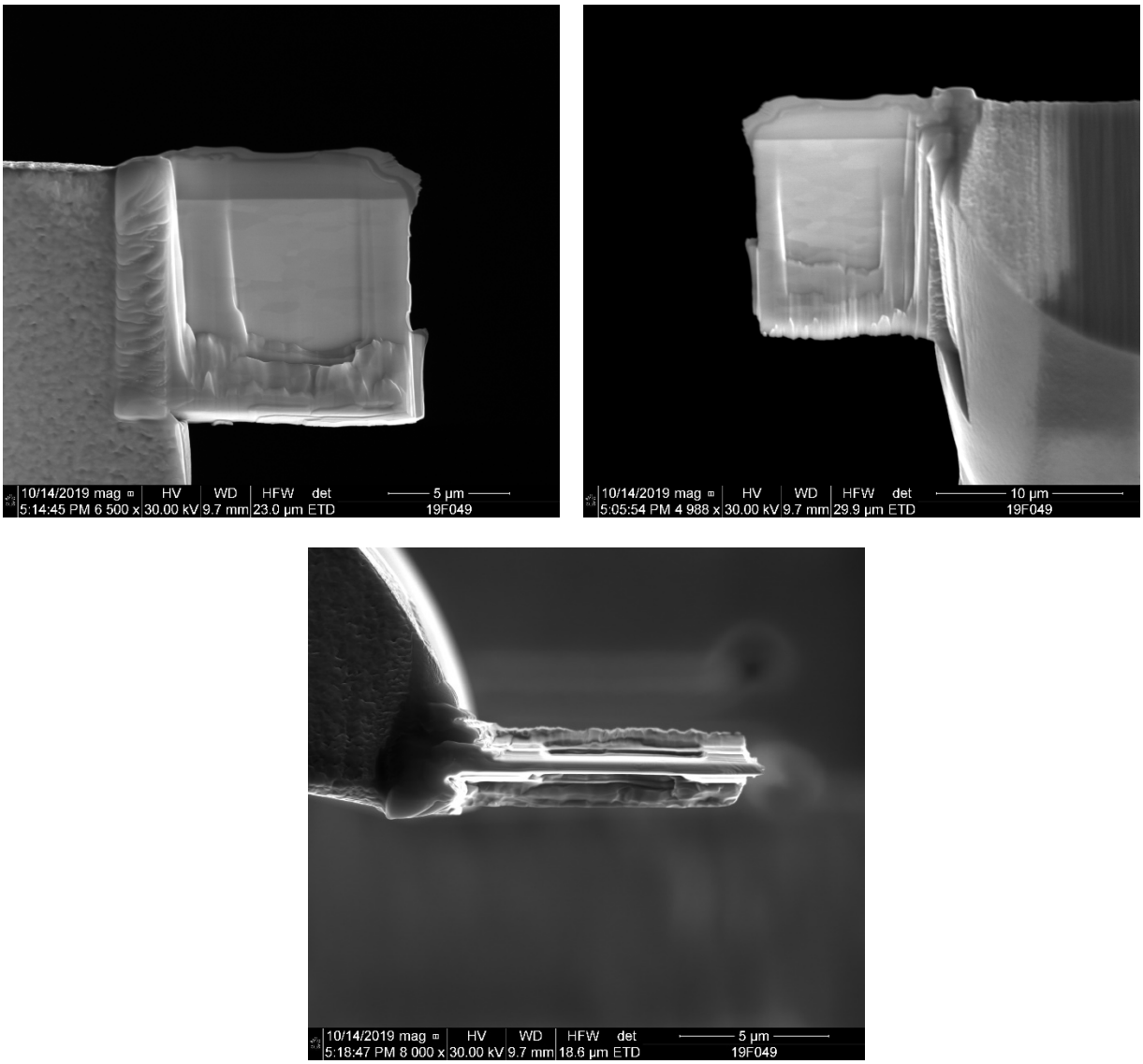

Figure 13. Finished Lamella

Appendix A – Milling Current Reference Chart

Table 1. Milling Current Quick Reference Chart

Step	Voltage	Current
Surface Protective E-Beam Pt Cap	5keV E-Beam	4-16nA
Surface Protective C Cap		
Surface Protective Pt Cap	30keV I-Beam	.45nA
Trench Cuts / Cleaning Cut	30keV I-Beam	15nA / 7nA

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