

F.I.B. Operation

Do and Do not

Do check the specimen height to make sure it is low enough

Do not use UHR mode when OMNI Probe in

Do not use UHR mode when gas injector is in

To start software

XP start service

XP

user (user name)+ user (password)

You must not change any settings on the computer system!

The only changes you can make are within the XP2.29 (FIB) program.

On the upper right portion of the screen, there are three buttons that determine what set of microscope controls are displayed. The icons are as follows (Left to right): a KEY, a PENKNIFE, and a HISTOGRAM. The HISTOGRAM will rarely if ever be used during F.I.B. operation. The KEY menu deals with on and off beam controls and the PENKNIFE menu controls most of the imaging / patterning and stage functions.

Install specimen

Press Key icon button.

Turn off the ion beam and isolate the electron beam

Press vent button on vacuum (top of key menu)

It will ask if you really want to vent. YES

Open valve of N2 line on wall

WAIT about 4 minutes. Pull door open gently.

Insert specimen stub into hole on stage, make sure that your sample surface is lower than the plat form of the surrounding subjects, tighten small Allen screw until you can feel resistance on the Allen wrench.

Press pump button on vacuum control.

Microscope Set-Up

1. Press the KEY icon button to display the beam control menu.
2. Turn on source button and check Ion emission reaches 2.2uA (Lower part of the Penknife menu)
3. Turn on the E-beam and I-beam HV buttons once vacuum level in chamber has dropped below the 5×10^{-5} Pa level.
 - a. Each gun has its own control panel.
 - b. I-beam has automatic HV set at 30kV
 - c. E-beam can e adjusted to whatever accelerating voltage is required (5kV is a good default).
 - d. On the left screen, use E-beam controls and on the right screen, use I-beam controls.

- i. Control of E-beam can be switched by selecting the E-beam primary beam icon (labeled with a **BLUE E**) and the E-beam Control button located 2nd to the right.
 - ii. Identical controls exist for I-beam as well (labeled with a **RED I**)
 - iii. **Knob set controls both beams**, (for Magnification/Focus/Contrast & brightness beam shift and astigmatism), but only one at a time and is dependent on the one selected on the menu icons.
- e. Live image can be obtained by clicking on the left-most icon (looks like a pyramid).
 - i. Go to low magnification to ease navigation.
 - 1. If you experience difficulty in adjusting brightness and contrast, press the auto brightness/contrast button on the toolbar. The computer will establish a good starting point so that you can get your bearings and make appropriate manual adjustments to the brightness and contrast.
 - ii. Establish the working distance by setting the focus and using the z-control to bring the specimen into focus. A working distance of 5mm seems to work well.
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 - iv. Establish the working distance by setting the focus and using the z-control to bring the specimen into focus. A working distance of 5mm seems to work well.
- 4. Focus the image of E-beam at a magnification at 2k or above, and link working distance with Z. Then, bring Z to 5 mm gradually.
- 5. Start imaging by finding an area of interest by using E-beam controls at 0° tilt (can check the tilt on the PENKNIFE menu).
 - a. Focus an image using the E-beam conditions.
 - b. Stigmatize the beam at high magnification (>10,000X)
- 6. Eucentric Height Adjustment**
 - a. Center focused image of area of interest at 3500X using X-Y stage controls.
 - b. Tilt the sample 5° by using the PENKNIFE menu and return area of interest to center of screen using z-control **only**.
 - c. Go back to 0° tilt and re-center.
 - d. Gradually work toward tilting the sample 52° such that the area of interest is at the center of the screen at both 0° and 52° tilt*. The sample is now on the eucentric height! Go to the STAGE command at the top of the screen and select “stage is at the eucentric height”.
 - e. Every time the sample is moved in the X and Y directions, the eucentric height should be reexamined.

Ion Beam Set-up

1. Use E-beam and move stage such that area is at center of screen at 3500x as described above.

2. Select the Ion Beam Primary and Ion Beam Control buttons
3. Select 10pA setting under I-beam menu.
4. Obtain a focused I-beam image
5. Go to stage menu and select “zero beam shift”.
6. Use the X-Y beam shifts to center image at 3500x (do not touch stage control !!)
7. Press the **II** icon to capture an image

Usage of OMNI Probe

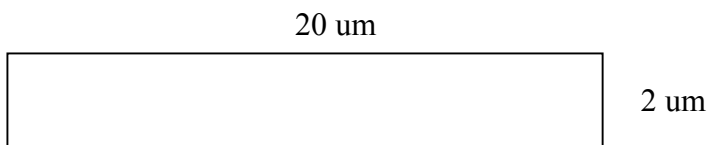
1. Start the software, password=omni
2. Adjustment of needle position when it is changed: lower the stage all the way to its lowest position; adjust the needle position so that the tip of the needle is in the center of both I- and E-beam images. Click eucentric, then edit/store
3. Move the sample left and right, up and down. Two mode: speed and step.

Platinum Deposition

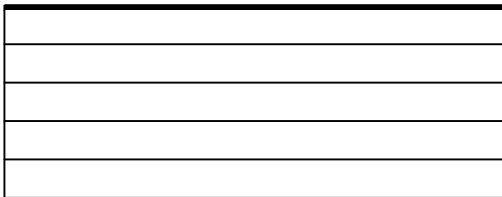
4. Be sure that sample is tilted 52°.
5. Sample should already be at eucentric height.
6. Change I-beam setting to 100pA.
7. Adjust brightness and contrast and refocus image.
8. Gas Injection Needle
 - a. Turn on Gas heater (using GIS control button, key menu) 20 min. before usage.
 - b. Once light is red, go to PENKNIFE, insert has injector needle.
9. Re-center I-beam image
10. Be sure to stop scanning imaging processes.
11. Click on full box tool (2nd button from left).
 - a. Draw a rectangle around the area of interest.
 - b. Dimensions are x=20μm, y=2μm, z=1μm for TEM specimen preparation.
12. Change materials file to “pt.mtr”.
13. Be sure I-beam and I-control buttons are pressed
14. Begin Pt-deposit process by pressing the “Start/Stop pattering” icon. BE SURE TO REMOVE THE NEEDLE WHEN COMPLETE!!!

Nanolift liftout technique

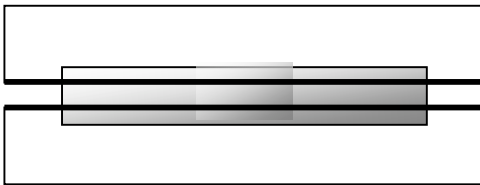
1. Deposit Pt using 100pa, pt_tem.mtr centered on your area of interest. Use ebeam pt first if necessary.



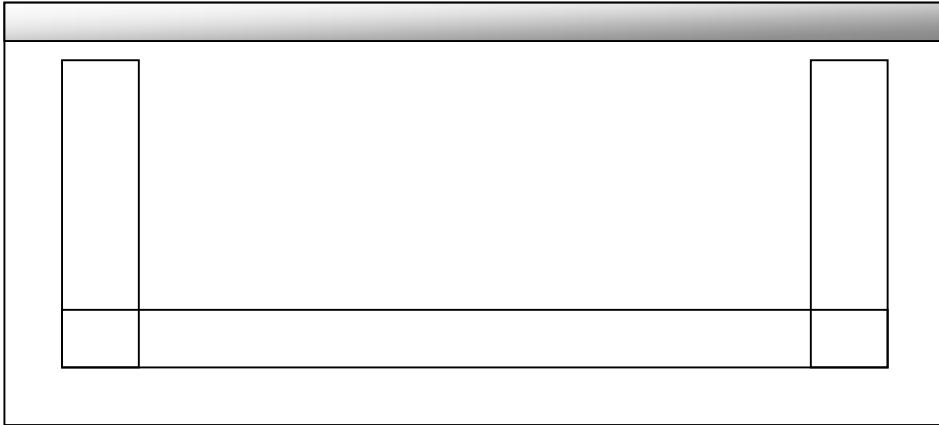
2. Mill a regular cross section on the front about 17 x 7 x 4 using 7000pa, si.mtr, rotate the pattern and mill on the backside.



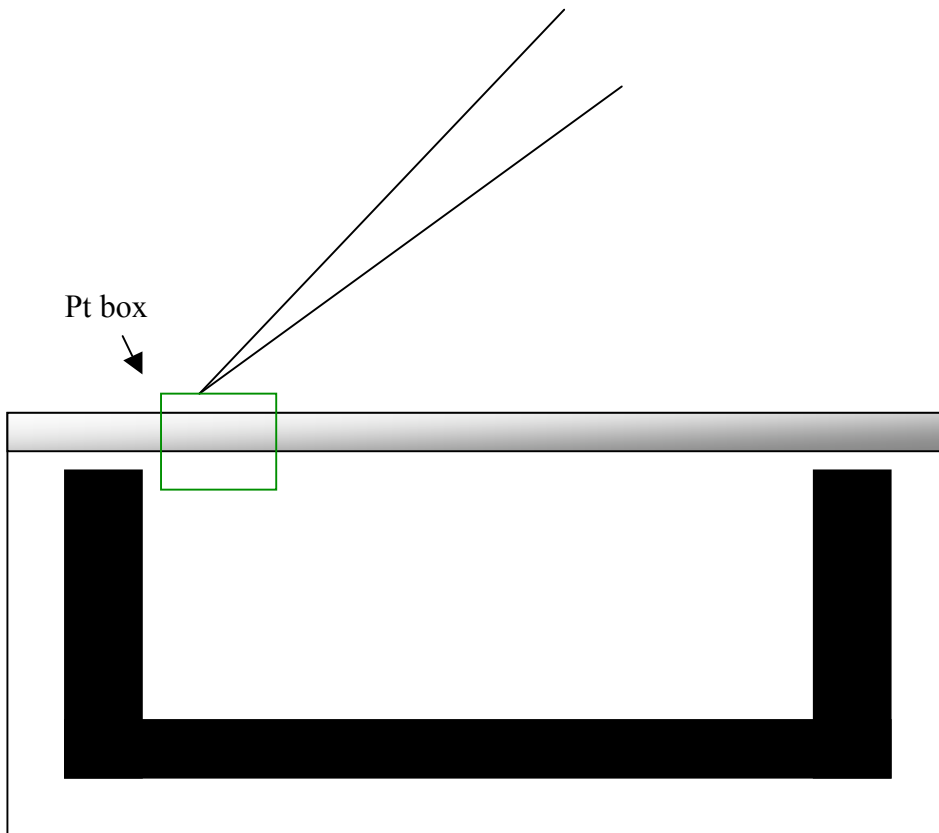
3. Mill cleaning cross sections near the pt using 5000pa, si.mtr until the sample is 1um thick (reduce the pattern z by at least 1/2). On the front side, tilt the stage to 53 degrees. Mill the pattern. On the backside, tilt the stage to 51 degrees. Mill. This will make the sample vertical (less wedge shaped).



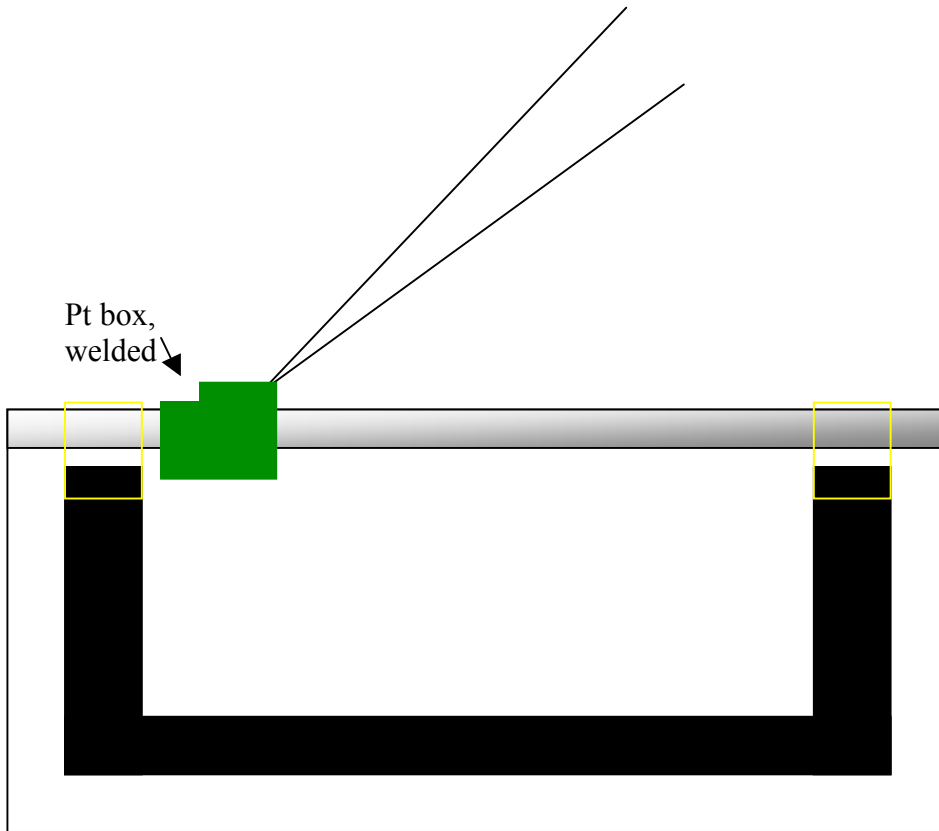
4. Tilt the stage to 0 degrees. Change the beam current to 1000pa. Draw filled box patterns in the shape of a "U" (make the vertical boxes 1.5um wide). Mill until you've broken through the sample using si.mtr (parallel milling).



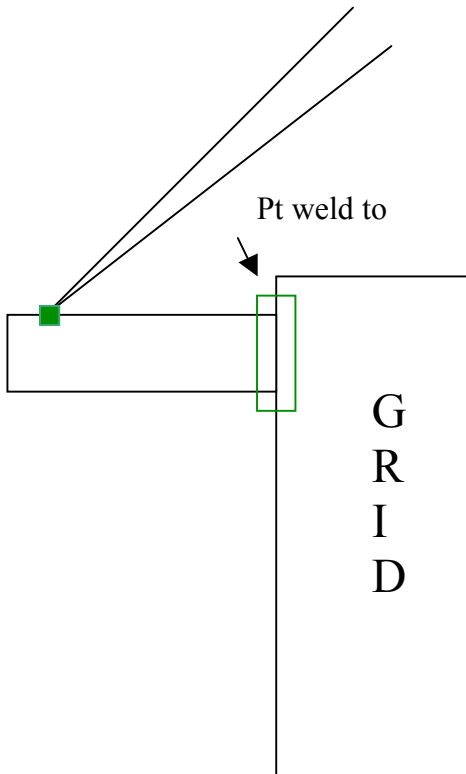
5. Bring in the omniprobe needle. Leave a very small gap between the needle and the sample, then place your pt box just underneath the needle, weld the needle to the sample with 10-30pa pt. Do not retract the pt needle when finished.



6. Cut the remaining “pt bridges” free with 100 – 500 pa, si.mtr, serial milling. LOWER the stage until the sample is out of the trench. Put the omniprobe in the parked location and retract the omniprobe and the pt gis.



7. Drive to the grid location (make sure the stage is 0 degrees). Make sure that the grid is parallel to the E-beam column at zero degree stage tilt. Bring the stage to eucentric height. Maneuver the sample down to the grid and weld it to the grid using pt_tem.mtr and 100pa. Do not retract the pt needle when finished. Cut the needle – membrane weld free with 300-500pa using a box pattern. LOWER the stage until the needle is above the grid. Put the needle in the parked location and retract the omniprobe and the pt gis.



NOTES:

- Before you begin welding with the Pt, take note of the chamber vacuum. During the welding the chamber pressure will increase slightly. After the Pt is done, the pressure will remain at this heightened pressure for a minute or two. You must wait for the pressure to come back to the initial pressure before you mill. Otherwise you will not get a clean cut because of the residual pt in the chamber.
- Good rule of thumb to remember: Whenever you are using the omniprobe, make sure the stage is 0 degrees. Liftout from the bulk and welding to the grid are always done with 0 degree stage tilt.

- Auto TEM: TEM specimen sample preparation
- 1.RunScript TEM
- 2.Button 6 is used for the AutoTEM Wizard.Select 6 and press PLAY to begin TEM preparation process.
- 3.A dialog box prompts you to turn on GIS heating and insert Pt gun.
- 4.New AutoFIB Script Files? Click NEW to create a new file.
- 5.Sample Type? Click Liftout.
- 6.Specifying the Sample Width? 15um is OK. Click OK.
- 7.Membrane position. Adjust patterns to desired membrane position? Using stage movement, beam shift or movement of the patterns, Then click OK.
- 8.Membrane position. Use recognition to recenter crosses? Click No.
- 9.Sepcifying Sample Parameters?
 - target thickness: 0.84
 - finaldepth:4
 - platinum:1
 - Click OK.
- 10.Dimension setup. Click FINISH.
- 11.Deposit grounding layer now? Click Yes.
- 12.Sample definition. Click END.
- 13.Select 1 and press PLAY.
- 14.Move stage to the stored location for this membrane? Click NO.
- 15.Click All.

- Script
- 1.RunScript
2.File->Script1->Open exist file.
3.Click PLAY—Click yes to the question “Do you want to move the sample to the preset position?”

Procedure to scan in bitmaps and convert to milling files for xP

Any bitmap can be converted into a milling file (stream file, also known as a STR file). You can scan in items and save them as bitmaps, or create bitmaps in a program such as *Paint*.

- Create the bitmap via scanning:
Each scanner will be different. In general, it is best to use monochromatic coloration and keep the physical size of the bitmap small. Monochrome bitmaps or 8-bit bitmaps work the best. If an item is colorized, before saving it, I would recommend converting it to monochrome or 8-bit. The attached bitmap will be used as an example in this exercise. The size of this bitmap on disk is 60kB. The size of the resultant STR file is 530kB.



- Once the bitmap has been created via scanning or through creation in a program such as *Paint*, the bitmap needs to be converted to a stream file. A stream file is simply an ascii file that contains dwell time, x, and y locations for each pixel that is in the bitmap. Use the program attached in this email to create stream files.

Using the *Patterns* program to create stream files:

Load *Patterns.exe*. Type in the approximate magnification that you will want to mill the stream file at. Note that stream files do not scale with magnification within xP, as normal milling patterns do. A typical magnification may be 5000x. Press the *save data* check box. Press the *calculate* button and let the software know where you saved the bitmap file. Next, let the software know the name and location of where you want to save the stream file. Be sure to type the name, including the “.str” ending. Location of save is not important, only so that you can relocate it.

Here is the stream file that was created from the bitmap above. If you would like,



it can be opened in Notepad to examine the dwell, x, and y data.

- In xP, go under *File, Open, Pattern Data* and load in the STR file that you just saved. Select a reasonable beam current, typically 300-1000pA. Adjust the magnification and move the stage to where you would like the pattern milled on the sample. Press the mill button as you normally would.

- EDX Operation
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- 1. Preparation
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- (1) E-Beam: 20KV, Spot 3, EDX Mode
- (2) Scan: External Scan
- (3) Computer Switch to 2
- (4) Click on "EDAX Genesis" ShortCut on EDAX computer Desktop
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- 2. Point Analysis
- (1) Select "Maps/Line"
- (2) "Collect e" to get an image
- (2) Select Cross Button; Click a point of interest on Image
- (3) Click Collect Spectrum Button
- (4) Click "Clock Button" to Stop collection
- (5) Click "Peak ID". Click "up and down arrow" button to modify peak ID manually
- (6) To save Date
- (a)File -> Save as ->Save as Type ->Spectrum & Image(BMP,SPC).
- This way both spectrum and Image are saved.
- Saved in this Folder C:\EDax32\User Data
- (b)Click "W"(Send results to Word). This way results will be in a Word File.
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- 3. Line Analysis
- (1)Collect Spectrum
- (2)Peak ID -> Delete some element don't interest
- (3)Press line button
- (2)Click tools "Select Line on Image"
- (3)Draw a Line on image
- (4)Point 100, Dwell 2000, Click ENTER
- (5)Collect Line X-> Save as a file on disk -> Click OK -> Waite Line Scan Finish
- (6)Click "Display Line scan data"
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- 4. Maps Analysis
- (1)Maps/Line -> Collect e to get an image
- (2)Click tools "scan select area" -> Draw a box of interest on Image
- (3)Collect Spectrum
- (4)Peak ID -> Delete some element you are not interested in
- (5)Click Map
- (6)Right Click Reso to Choose the Resolution you want
- (7)Dwell(ms):500
- (8)Click "Collect Maps X"->Save as a file on disk -> Click OK-> Waite Map Scan Finish
- (9)Click "Build maps from spectra" -> OK