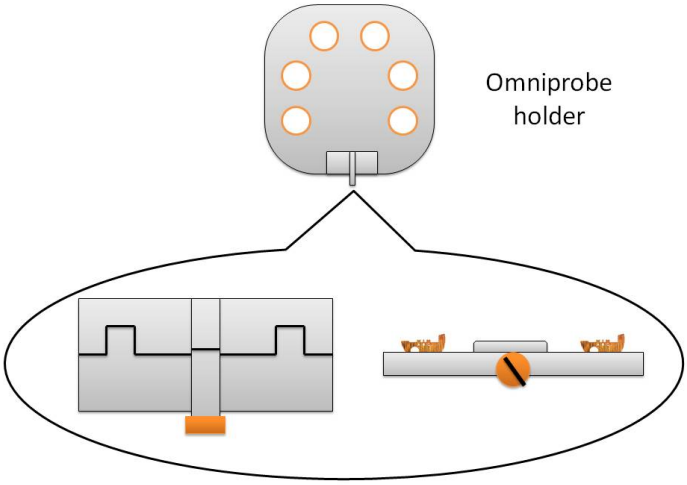
JEOL 4500 Operating Instructions

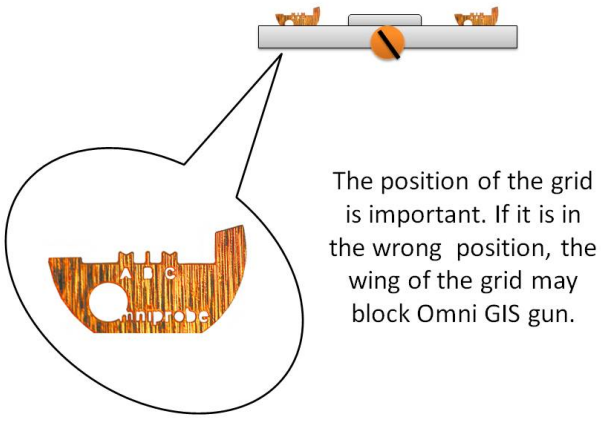
Omniprobe Lift-out

TEM sample preparation (Lift-out).

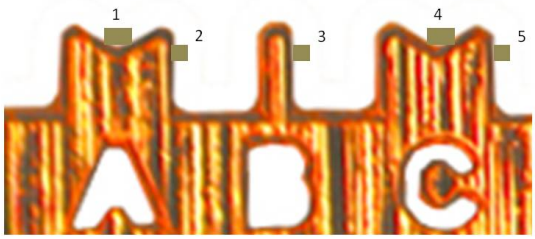
Note: the following steps are based on Si wafer specimen. Settings might change for other specimens.

I. Omniprobe holder:





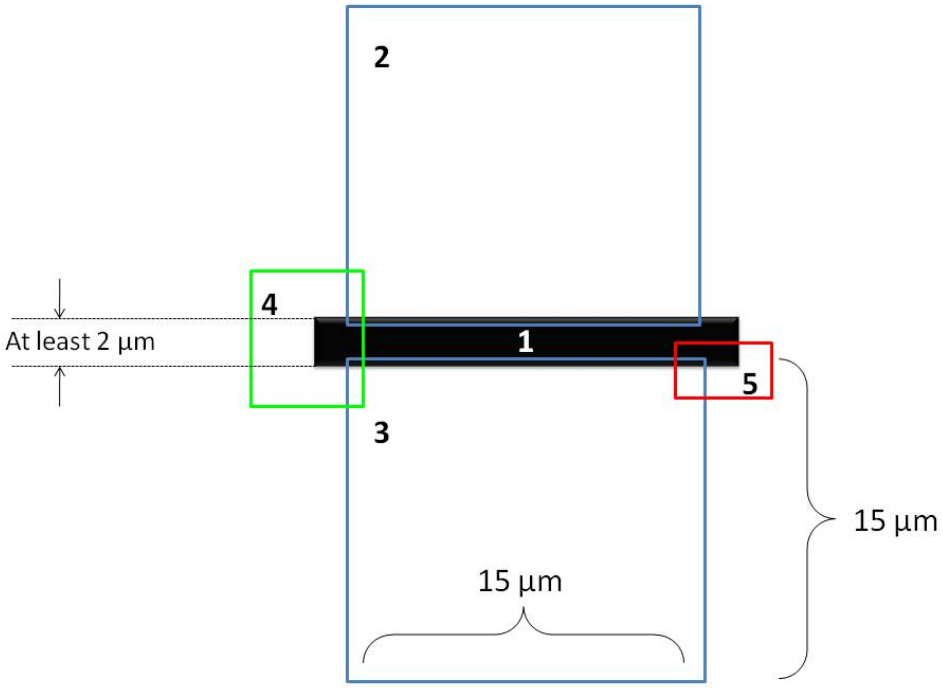
Only five specimen positions for one TEM grid



1) Insert the sample holder.
2) Select “Omniprobe holder”.

II. Making lift-out trenches:

1) Perform all the alignments (fine-z, beam and coincident alignments).
2) Go to “FIB” preset. (T= 52^{o}). Use beam#11 for observation. Find a suitable place to make trenches.
3) Click on “Process Setting” tab on the bottom left side of the screen.
4) Draw boxes for deposition and milling processes as shown in the following figure.



Box #	Process	Beam	Dose	Finish side	GIS
1	DEPO	8	1	N/A	Gun1/Auto
2	RAPID	3	Width (15)	bottom	N/A
3	RAPID	3	Width (15)	top	N/A
4	RAPID	3	Width	right	N/A
5	RAPID	3	Width	left	N/A

Note: Dose number may vary depending on different specimen.

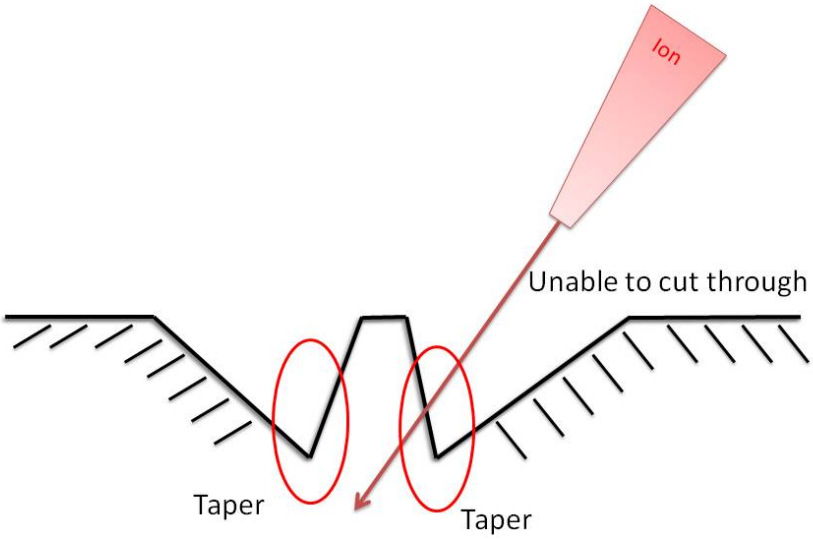
5) After finishing all boxes and settings for each process, go to "Group" tab and click "Apply".
Tip: You can click on "Entry" to make another group so that multiple TEM lift-out trenches can be made automatically once all the processes and groups have been defined. Operator may leave the FIB while the machine is operating by itself.

6) Under "Tuning" tab (the right side control panel), click on "ENTRIES" to finish the beam alignment. (i.e. beam #3 and #8 in this case)
7) Go to "group" tab and click "Review" to confirm all the processes.
8) Hit "start".

III. Bottom side cutting:

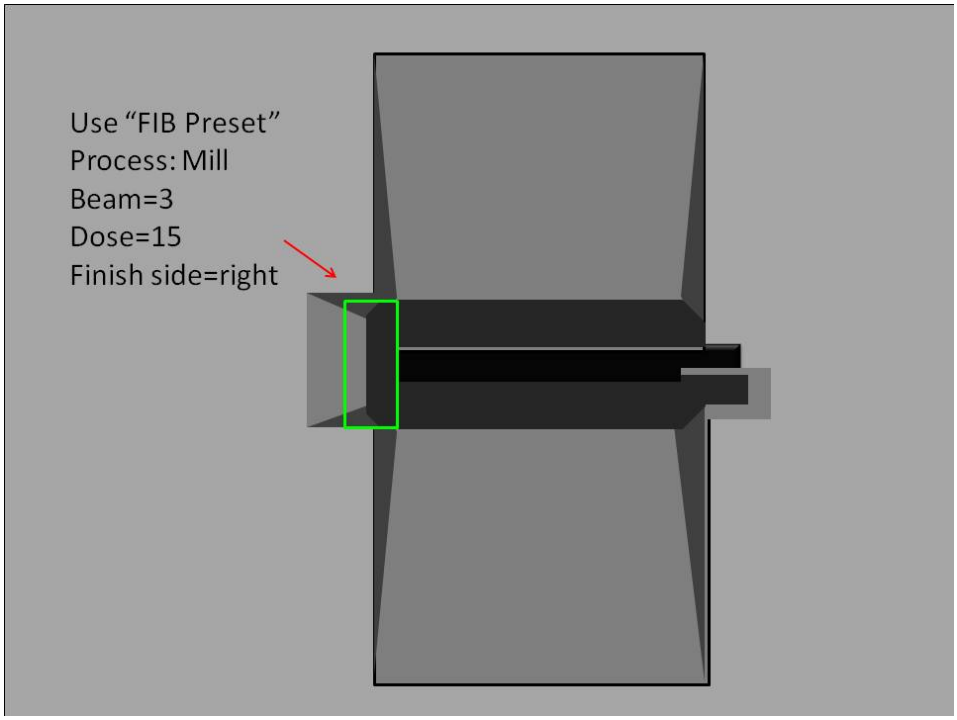
1) Before cutting the bottom side, further milling is required to remove tapers.

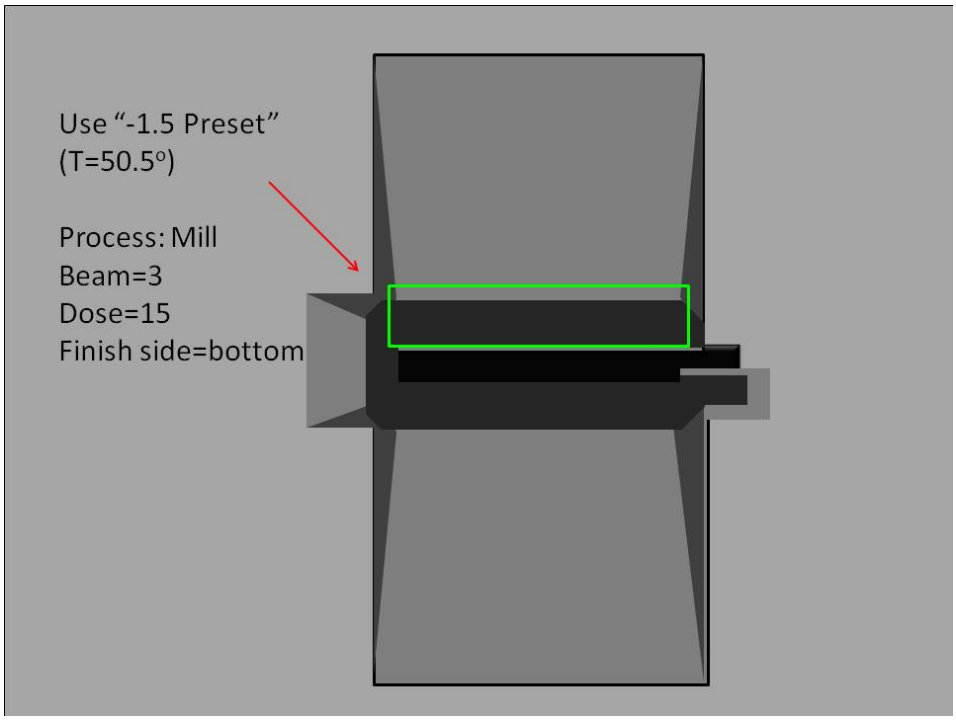
2) Go to “Stage” tab, select “FIB” preset (T=52^{o}).

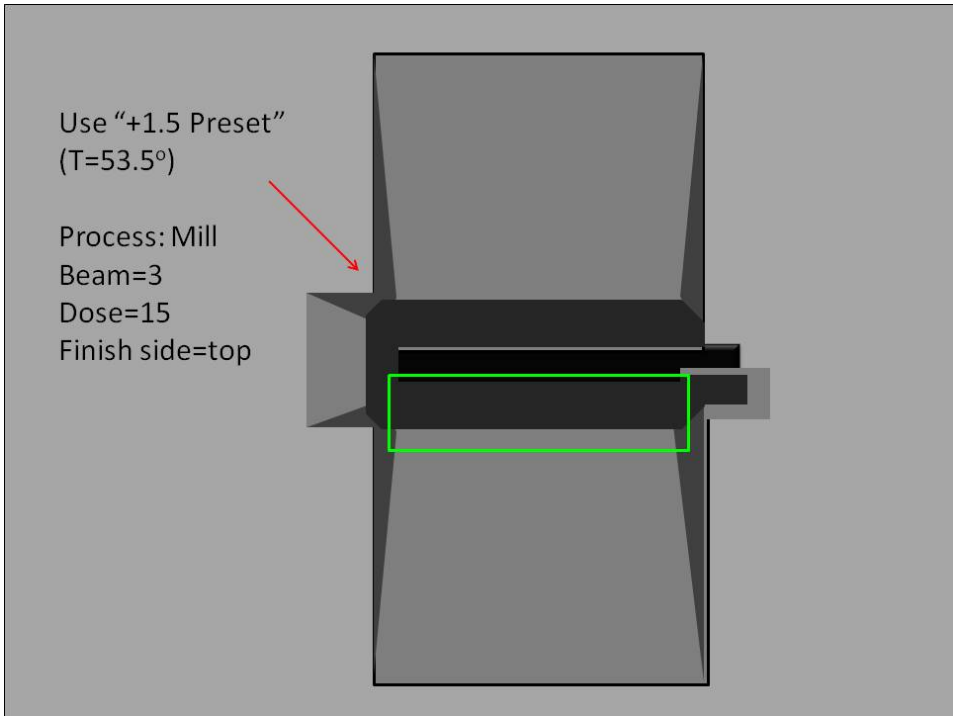


Side view of the trench

3) Remove the tapers according to the following drawing.





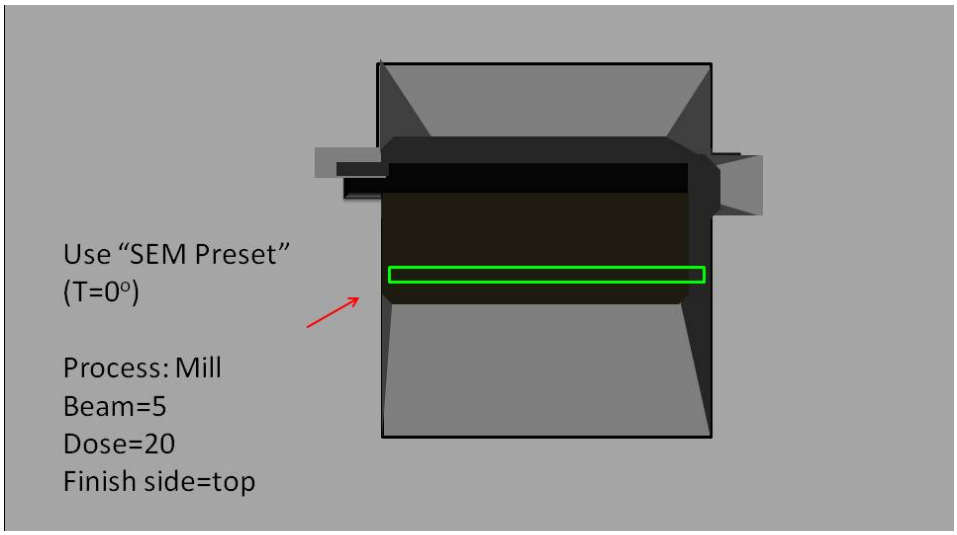


Note: Max T = 54°.

4) After moving the tapers, ready to cut through.
   Use "SEM" Preset to bring T=0°.
5) Go to "Tools" menu → "FIB tools" → "Scan Rotation" → Rotate the image by 180°. (no

actual movement of the stage)

6) Click "Monit" button (activate "FIB_SEI" window first) for the real time image of the milling area.
7) Cut the bottom side in the "FIB_SEI" window according to the following drawing.



8) When it is cut through, stop the milling process manually.
9) To check the cutting, right click on the "FIB_SEI" window and then check "Computer eucentric R".
10) Set R = 180°. (Make sure the stage will not hit anything in the way.)
11) Set T = 10° to see the cutting. (Always use beam 11 for observation.)
12) If the cutting is well done, set R = 0° and T= 0°.

Caution: Do not tilt the stage when tip and nozzle are inserted.

IV. Welding tip on TEM slab: (T=0°)

1) Set T = 0°.
2) Lower the magnification of the "SEM_SEI" image.
3) Turn on the Omniprobe control PC. Open "Autoprobe" and "OmniGIS". (Log on as normal user; no password.)
4) In "Autoprobe" window, press "Insert" button to the tip to the park position.
5) In "OmniGIs" window, right click on "Insert" and then "Goto" to insert Omni GIS nozzle.
6) In "Autoprobe" window, Select "Eucentric high" and click "Goto" button to bring the tip to the eucentric high position.

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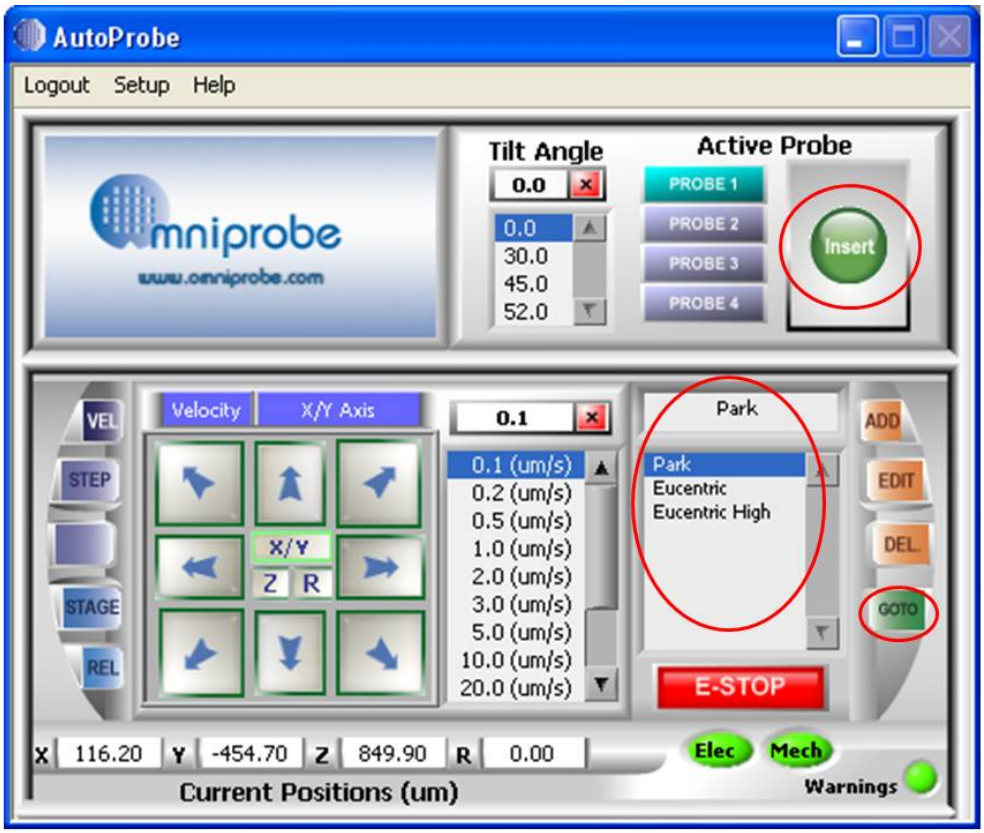
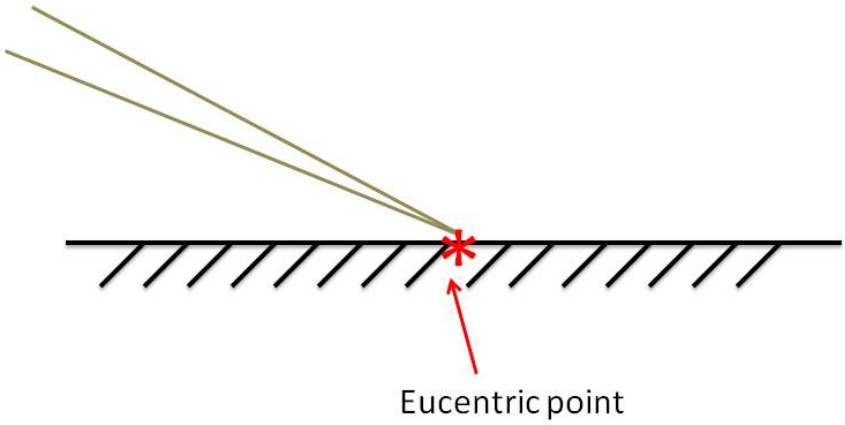
Caution: Do not click on "DEL" button.

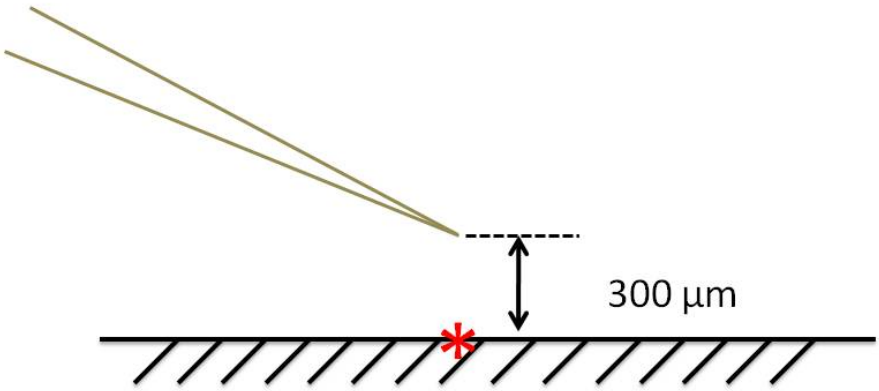


Position of the tip:

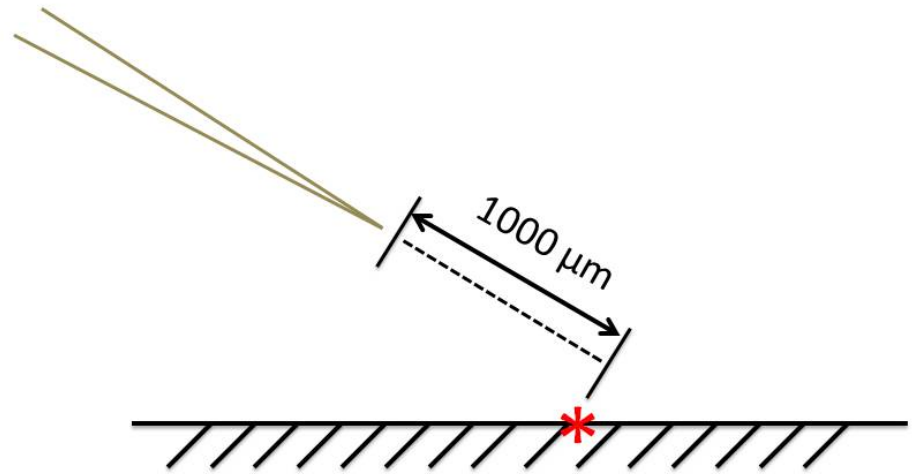
1) Eucentric position (rarely used)



2) Eucentric high position



3) Park position



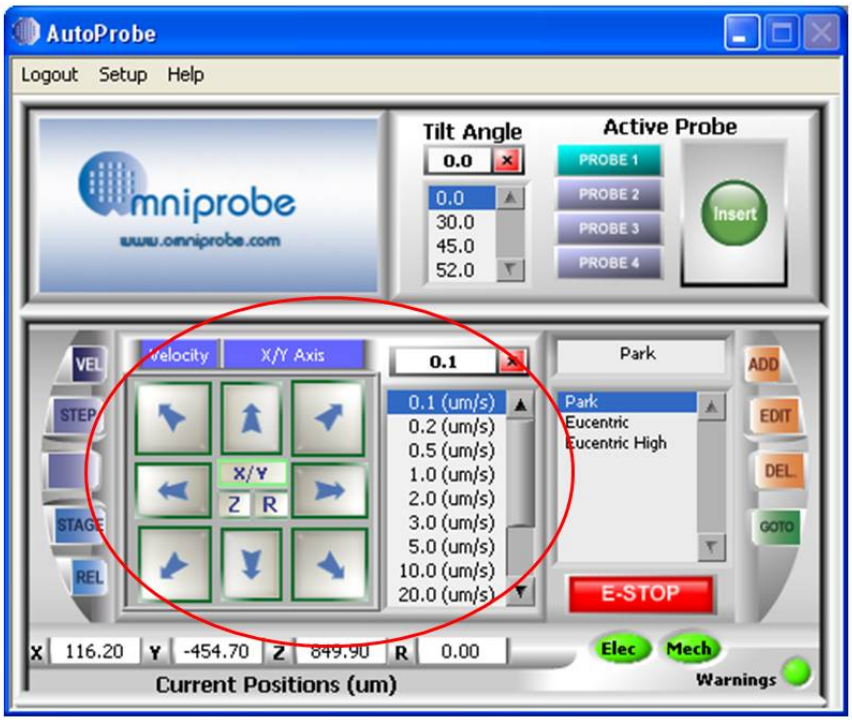
4) Retract position

Note: park the tip before retracting it.

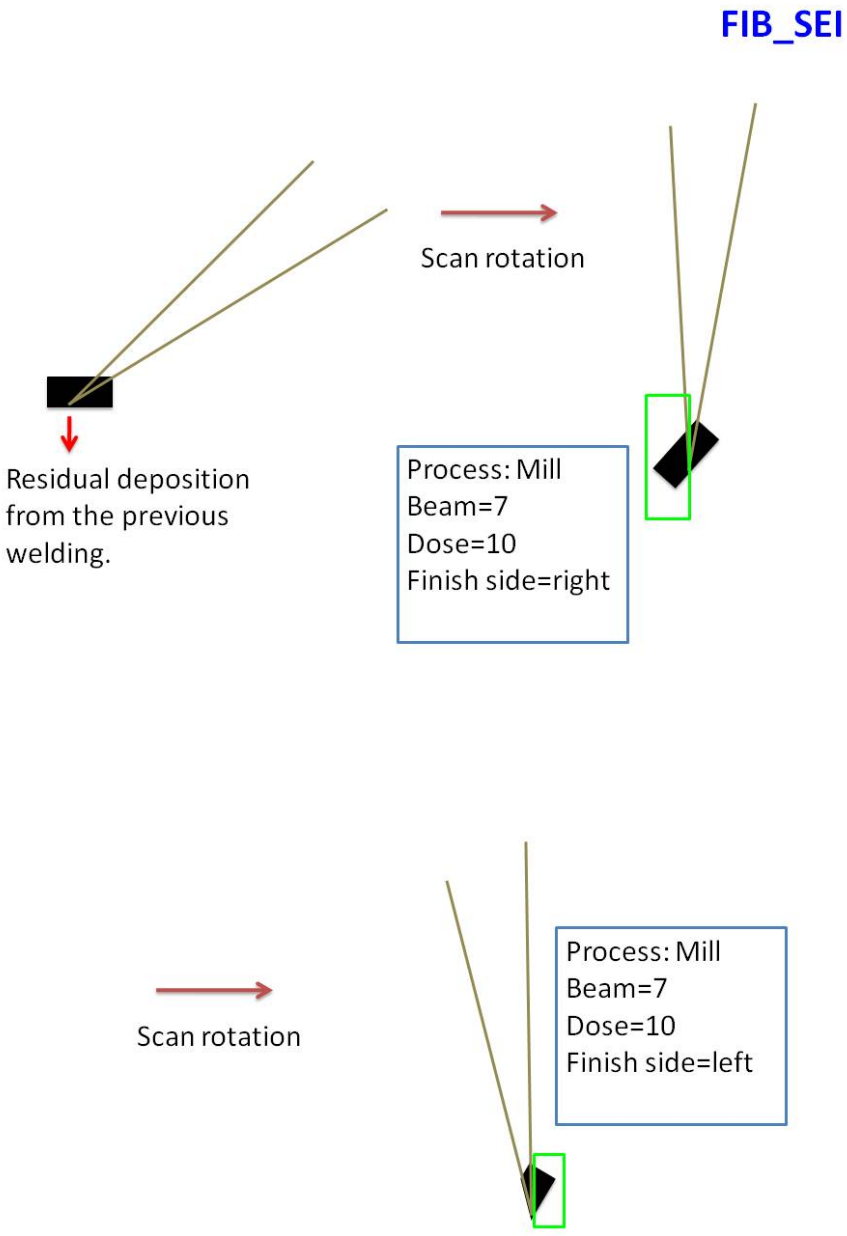
7) Manually move the tip to approach the sample surface using the control.
Click "Z" and press arrow down. At first, select "30um/s". As the tip is approaching to the surface, increase the magnification and speed (10um/s).

JEOL 4500 Operating Instructions

Omniprobe Lift-out



8) Select “X/Y” and move the tip above the welding site with the speed of 1 um/s. Monitor the tip movement in “SEM_SEI” window.
9) Further lower the tip with low speed.
10) Before welding, cleaning of the tip is required. Bring the magnification over 5,000x, therefore there will be no physical movement of the stage. **(Important)**
11) Double click the probe tip in “FIB_SEI” window to center it.
12) Use “Scan rotation” (Tools—FIB Tools) to rotate the image to make cleaning easier.



13) After cleaning the tip, right click on the "FIB_SEI" window and click "Image reset".
14) Use "OmniGIS" for the welding. Beam 9, 10 and 11 are suitable to the welding.
And Pt is selected for the welding element.

Beam #	Minimum Size of the deposition area
9	5 X 5 um
10	2 X 2 um
11	1 X 1 um

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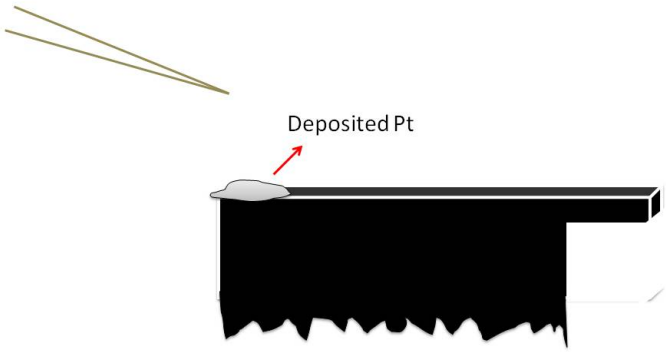
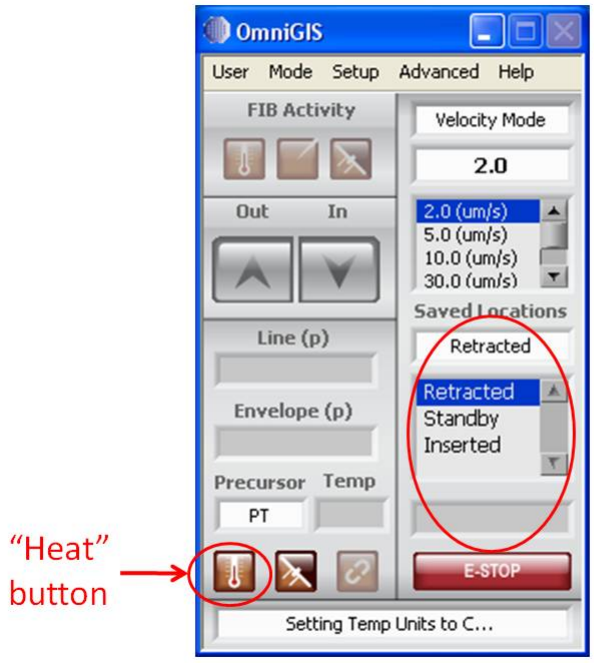
Omniprobe Lift-out

Since Pt on Pt welding is stronger than Pt on carbon welding, a pre-deposition of Pt is necessary. (Optional step)



15) In "OmniGIS" window, press the "Heat" button to increase the temperature of the Pt source. (It takes about 10 seconds.)



16) Back in JIB-4500 control software, set

Mode: **Manual**

Process: DEPO

Condition: Omni-P-DEPO 10 (if beam 10 will be used). Beam 9 and 11 can be selected depending on the size of the deposition area. (Check the table above.)

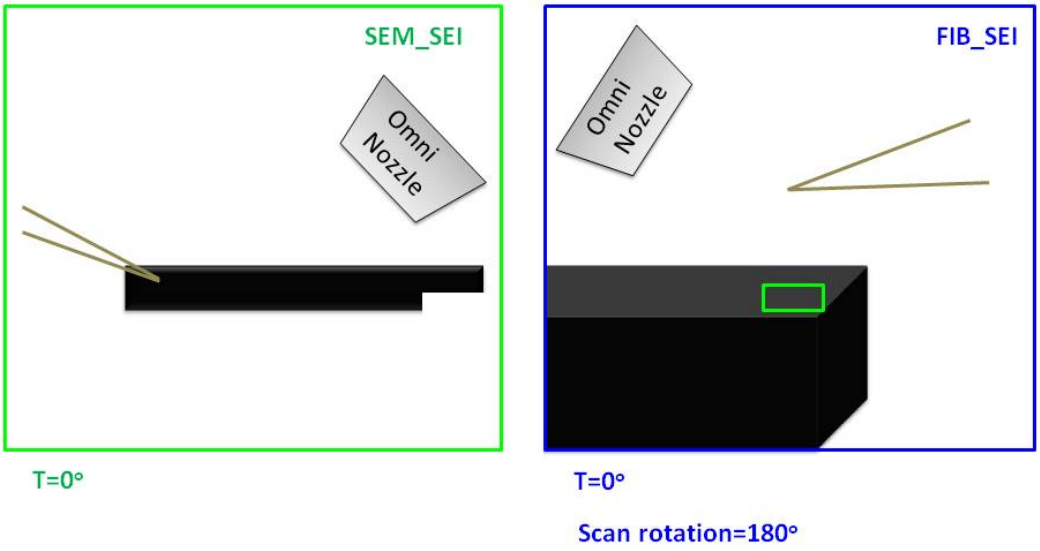
Dose = 1 or 0.8

GIS: Carbon

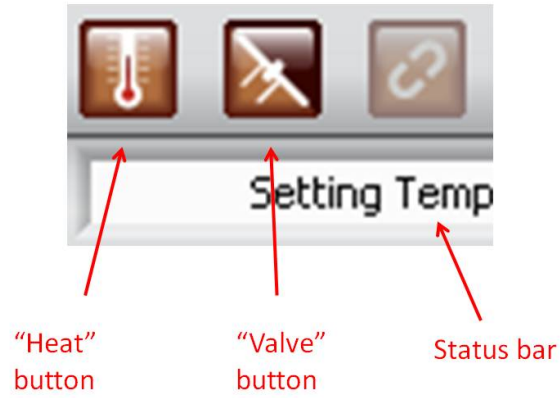
Manual

Caution: If "Auto" is selected, then JEOL nozzle will come in and hit the Omni nozzle!

17) Freeze the image and draw a box as in the following figure.



18) When the heating of the Omni nozzle is done (check the status bar), Click on "Valve" button. It takes about 20 sec for Pt to reach the tip of the nozzle. Then Pt will be released from the nozzle, react with ion beam and finally be deposited on the specimen.

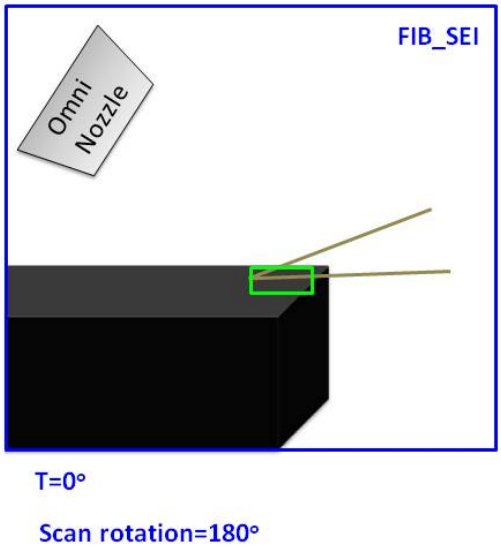


19) Hit "Start". Leave "heat" and "valve" on during the deposition.
20) After Pt deposition, weld the tip onto the specimen.
    Mode: **Manual**
    Process: DEPO
    Condition: Omni-P-DEPO 10

Dose = 1
GIS: **Manual**

21) Slowly lower the tip until it touches the specimen. The contrast will change suddenly when the tip contacts the surface.
22) Freeze the image and draw the box according to the following figure.
23) Hit "Start".
24) When the deposition is done, turn off the "heat" and close the "valve". Press "**E-STOP**" immediately after the valve is closed.

Note: Because purging process can cause vibration, it must be avoided when you close the valve.



Tip: Once the tip is attached on the sample, any vibration or movement could detach it.

Caution: After welding, do not double click the "FIB_SEI" window with magnification less than 5,000x. Otherwise the welding joint will break.

V. Final side cut:

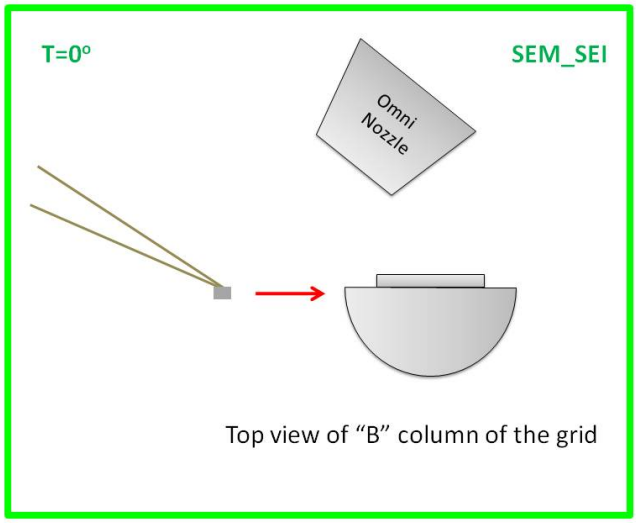
1) **Make sure** "valve" of the Omni nozzle is closed. Otherwise, deposition will occur instead of milling.
2) Cut the last side of the specimen.
   Mode: Auto
   Process: Mill
   Beam = 5
   Dose = 20
   Finishing side = right
3) Click "Monit" to check the real time cutting. Stop the milling when the side is cut through.

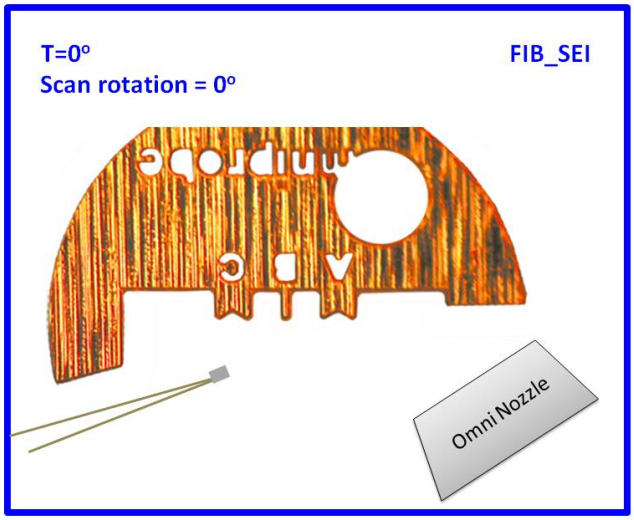
Vibration of the sample piece is a good indication of a cut-through.

4) Switch to Beam 11.
5) Slowly lift the tip to a safe distance above the sample surface. (Z up with 0.5 um/s)
6) Bring the tip to “Eucentric high” position. And then go to “Park” position and “retract” the tip.
7) Retract Omni GIS nozzle and turn off the “heat”. With the tip and nozzle retracted, now it is safe to move the stage.

VI. Attaching to TEM grid: (T=0^{o})

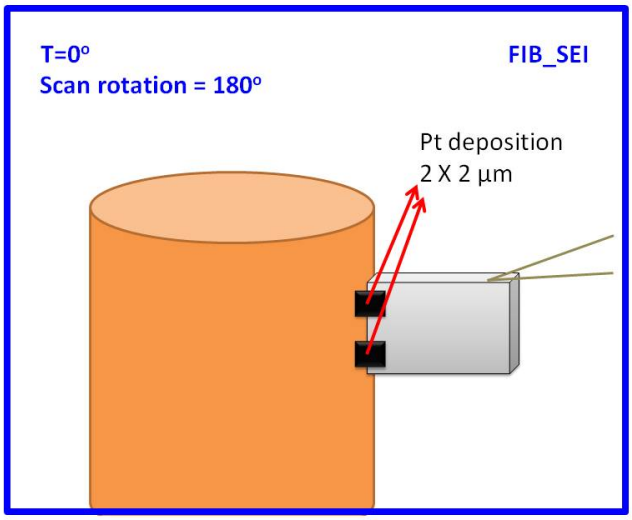
1) Set T = 0^{o}.
2) Locate TEM grid position. (Use the holder navigator)
3) Rotate the stage until the grid looks horizontal in the “SEM_SEI” window.
4) Do the fine Z adjustment again on the spot of the grid where you wish to attach the specimen.
5) The relative position of the grid, Omni GIS nozzle and the Omni probe tip:





6) In “AutoProbe” window, Insert the tip to the “Park” position.
7) Insert the OmniGIS nozzle.
8) Bring the tip to the “Eucentric high” position.
9) Set up “Process Data”:
   Mode: **Manual**
   Process: DEPO
   Beam = 10
   Dose = 0.8
   Condition: Omni P-DEPO 10
   GIS: **Manual**

10) Slowly move the tip to the copper grid until they contact. (0.5 um/s)
11) In “Omni GIS” window, press “Heat” button.
12) When heating is done, turn on the “Valve”.
13) Weld the TEM sample on the grid.



Tips: 1) Finish the deposition process in a short time to prevent any damage or break of the sample.

2) Do not move the stage after the welding. It is recommended to bring the magnification over 5,000x to prevent any mis-movement.

VII. Detaching tip from TEM sample:

1) Make sure the "heat" and "valve" of the Omni GIS nozzle is turned off. (**E-STOP** right after the valve is closed.)
2) Set up the process data:
   Mode: Auto
   Process: Mill
   Beam = 7-9
   Dose = 10-20
   Finish side = top & right
3) Use real time monitor to check the milling.
4) Once the milling is done. Stop the process, switch to beam 11.
5) Slowly lift the tip until it reaches a safe distance away from the TEM sample.
6) Bring the tip to the "Park" position and retract it.
7) Retract the OmniGIS nozzle.
8) (Optional) If you feel the bonding is not strong enough, you could also use JEOL GIS to deposit a carbon layer around the welding joint.
   Mode: Auto
   Process: DEPO

Beam = 8

Dose = 1

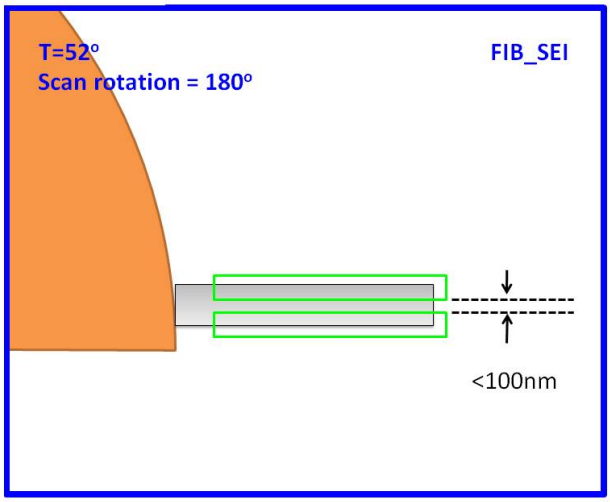
GIS: Gun1

Carbon

Auto

VIII. Final thinning of the TEM sample:

1) Check the "Image shift reset".
2) Set T = 52°. (FIB preset) (**Make sure** the tip and nozzle have been retracted.)
3) Process data:
   Mode: Auto
   Process: Mill
   Beam = 5-7
   Dose = 20
4) Freeze the image and draw the boxes according to the following figure.



5) Switch to finer beams for further polishing until the thickness is around 100nm.
6) Tilt 0.5° to remove the taper if necessary.

Ready to move the grid