Lamella Preparation and Transferring

Either by focused ion beam (FIB) or by electrochemical polishing, the preparation and transferring of the lamella plays an important role in the success of the experiment, as it influences the obtained resolution and the quality of the results.

Contents

I. Focused Ion Beam (FIB) Technique

1.1 Preparation & Transferring Method using a Standard Flat Stub

Source: *M. Duchamp, Q. Xu and R. E. Dunin-Borkowski (2014) "Convenient Preparation of High Quality Specimens for Annealing Experiments in the Transmission Electron Microscope", Microsc. Microanal., pp.1-8*

1. Preparation of the pre-thinned lamella

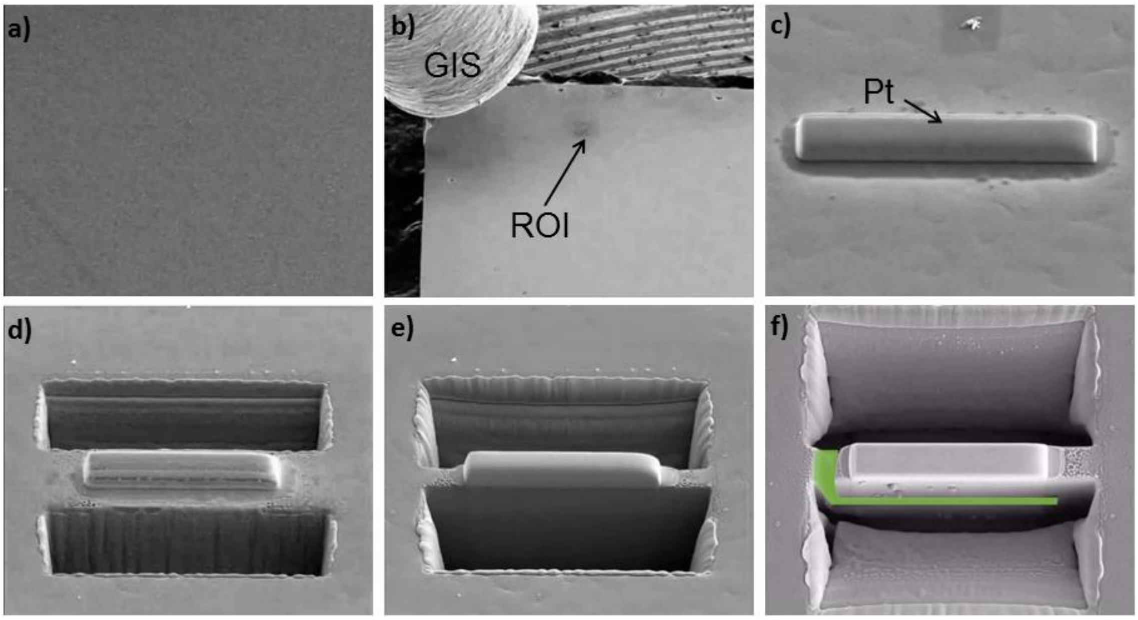


Figure 1. (a) Bring your region of interest (ROI) to eucentric height, (b) Position the GIS needle above the ROI, (c) Deposit a protective layer on top of the ROI; Deposition @30kV and 80pA using regular cross-section; Dimension: L=8-12mm, W=2-3mm, H=2mm, (d) Coarse milling of trenches behind and in front the ROI; Milling @30kV and 9nA and a stage tilt of 52°, (e) Pre-thinning of the lamella on both sides using 30 kV and 770 pA. Try to bring the lamella to a thickness of 100nm, (f) Tilt the stub and perform the undercutting to release the lamella from one side and the bottom (area to be cut is shown in green).

NOTE: The Pt layer is only needed when the material interface is less than 500nm away from the surface.

2. Cut and release of the lamella

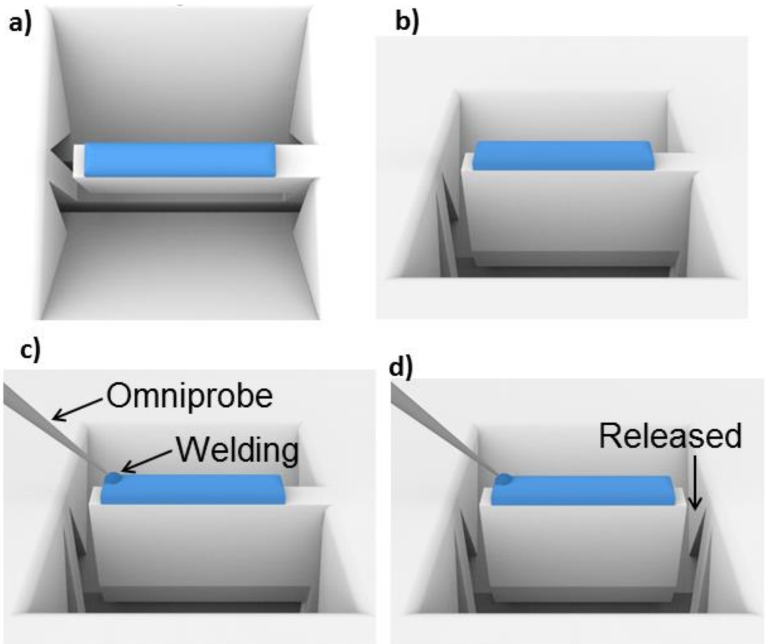


Figure 2. a) Once the lamella has been undercut and released from one side (b) bring the stub back to the original position and (c) insert and weld the Omniprobe for the final release; Welding @ 30 kV and 80 pA. (d) Mill the fixed side of the lamella to complete the full release.

3. Lift-out and rotation of the lamella

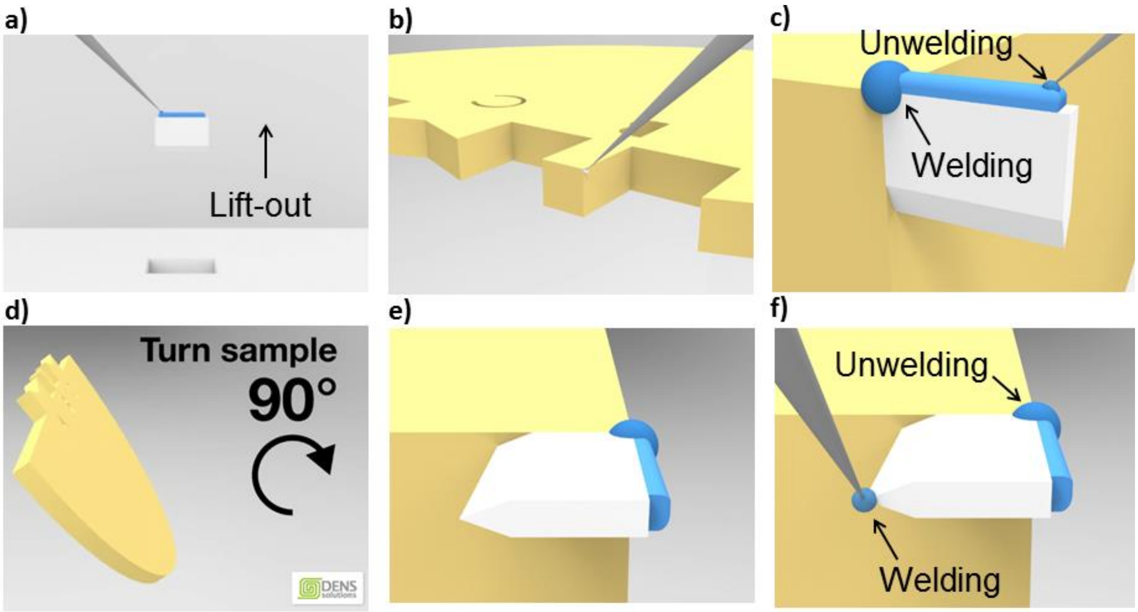


Figure 3. a) Lift-out the lamella, (b) transfer it to the intermediate grid and (c) weld it; Welding @ 30 kV and 80pA, (d) Rotate the grid 90° and (e) approach the lamella by re-inserting the omniprobe. (f) When the probe is the vicinity, weld it to the lamella and unweld the latter from the grid.

4. Transfer the lamella and final thinning

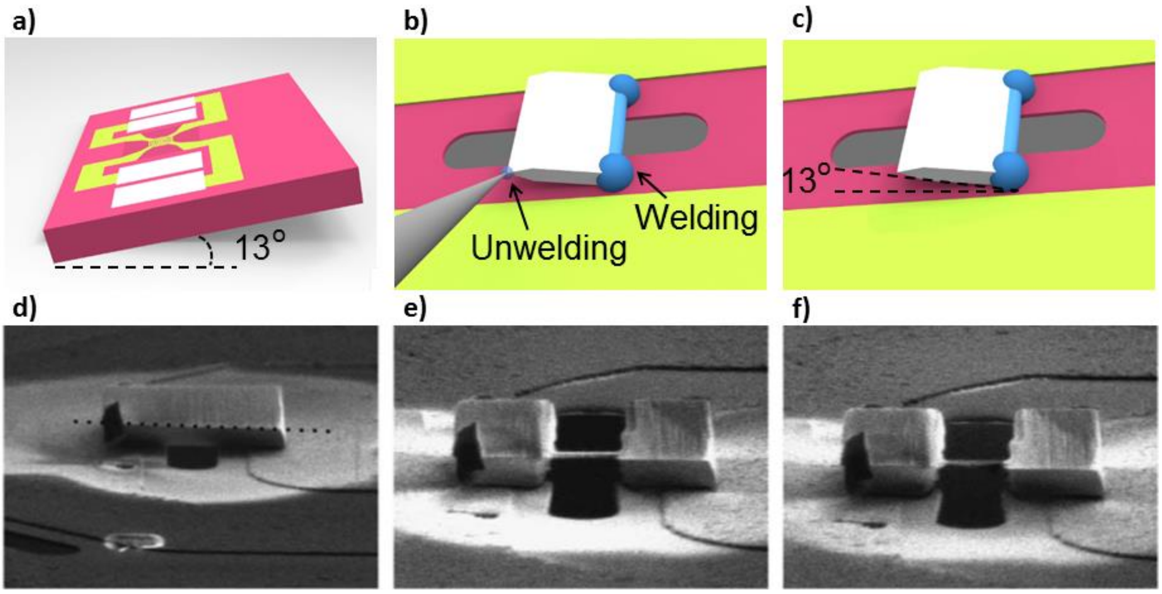


Figure 4. (a) Tilt the flat stub to 13°, (b) Transfer the released lamella onto the window of interest and weld it; Welding @ 30 kV and 80 pA. (c) The lamella should now be at a 13° with respect to the surface. (d) Such opening angle gives access to the top and bottom side of the lamella. (e-f) Do the final thinning by removing from both sides.

II. FIB Preparation & Transferring Method using a 45° Stub

1. Deposition of a protective layer (depo) on top of the specimen surface

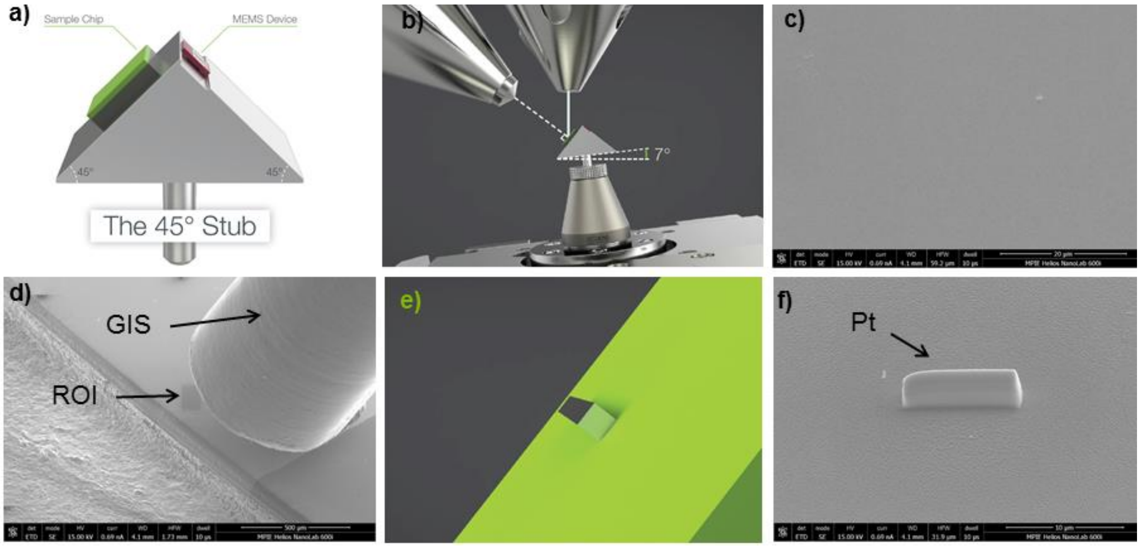


Figure 1. *a)* Coarse milling of trench behind ROI and *b)* in front, and on the side of the ROI; Milling @ 30 kV and 9 nA and a stage tilt of 7°(Dimensions of front/back trenches: x=20µm, y=13µm, z=7µm and Dimensions of side trench: x=5µm, y=10µm, z=7 µm), *c)* Polishing of the lamella walls; Milling @ 30 kV and 2.6 nA and a stage tilt of 7°, *d)* Make sure to have a 2-3 µm long base (no.1) between your ROI and the bulk material and also leave 2-3 µm long base (no.2) for welding the manipulator.

2. Coarse milling of trenches around the region of interest (ROI)

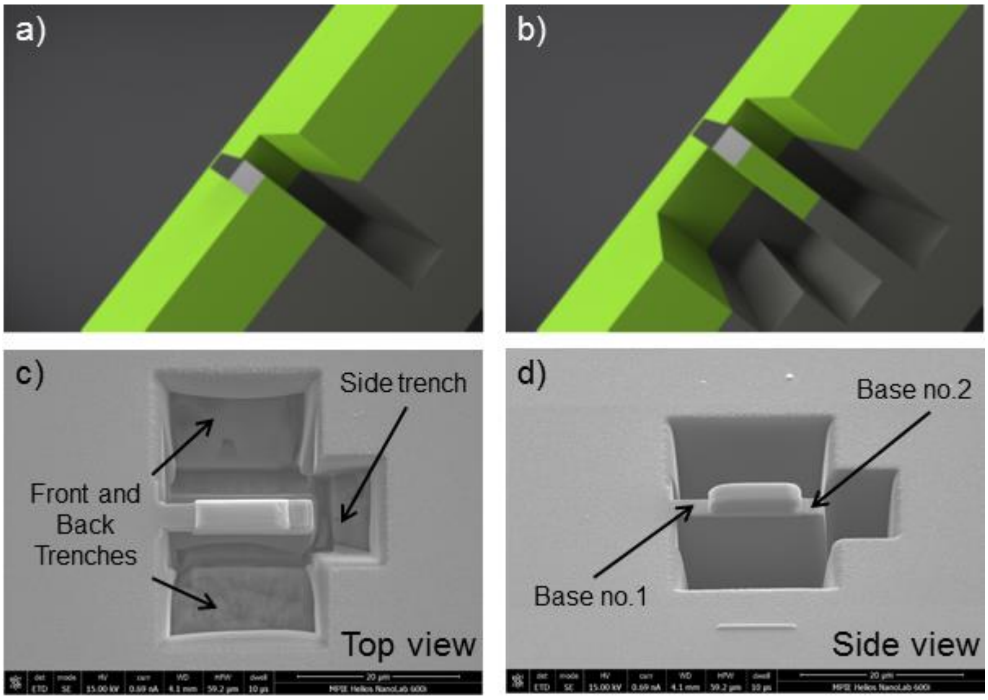
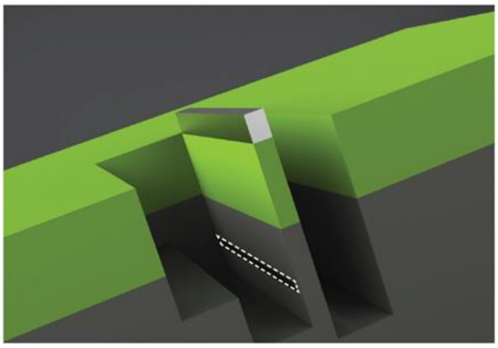


Figure 2. *a)* Coarse milling of a large trench behind ROI and *b)* in front, and on the side of the ROI; Milling @ 30 kV and 9 nA and a stage tilt of 7°(Dimensions of front/back trenches: x=20µm, y=13µm, z=7µm and Dimensions of side trench: x=5µm, y=10µm, z=7 µm), *c)* Polishing of the lamella walls; Milling @ 30 kV and 2.6 nA and a stage tilt of 7°, *d)* Make sure to have a 2-3 µm long base (no.1) between your ROI and the bulk material and also leave 2-3 µm long base (no.2) for welding the manipulator.

3. Perform undercuts to separate the lamella from the specimen



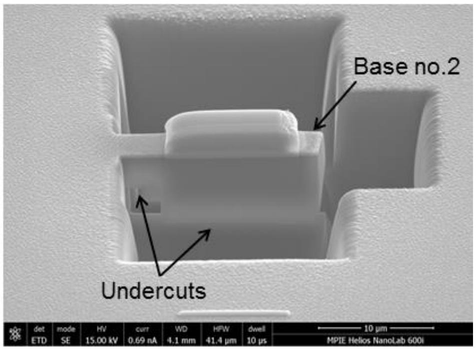
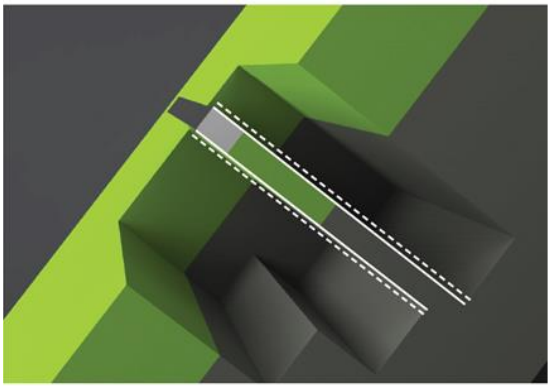


Figure 3. a) Tilt stage to -10°, when cutting free the lamella make sure to leave enough space (2-3 µm) between the bottom part of the lamella and the pit to avoid redeposition during thinning; Milling @ 30 kV and 2.6 nA and reshape base no.2 to have a nice angle for welding the lamella to the chip.

4. Pre-thinning of the lamella



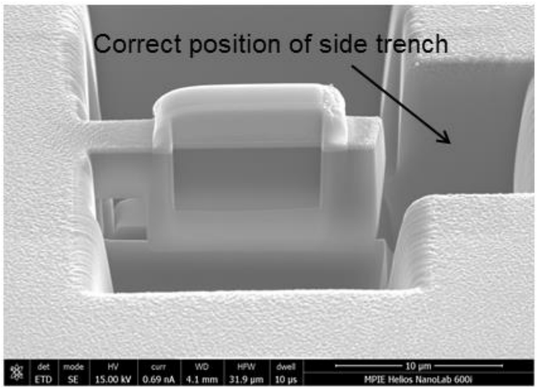


Figure 4. Tilt stage to 7°, start the pre-thinning on both sides using 30 kV and 770 pA. Try to bring the lamella to a thickness of 100nm.

5. Lift-out

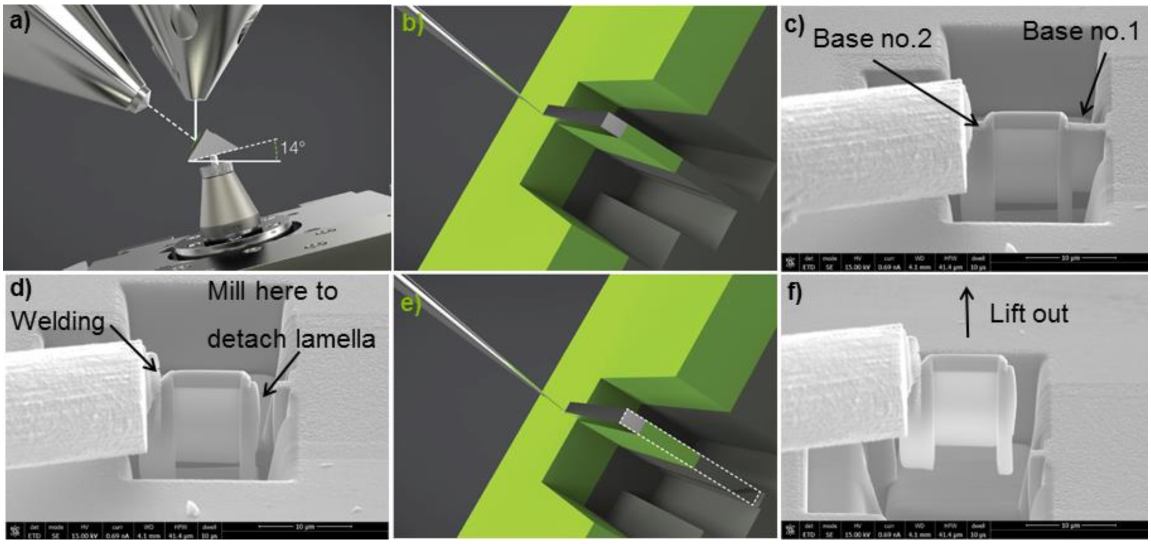


Figure 5. (a) Tilt stage to 14°, (b) bring in manipulator and place it (c) on top of base no.2. (d) Bring in the GIS and perform welding @ 30 kV and 80 pA, (e) mill base no.1 and (f) retract manipulator.

6. Transferring the lamella to the chip

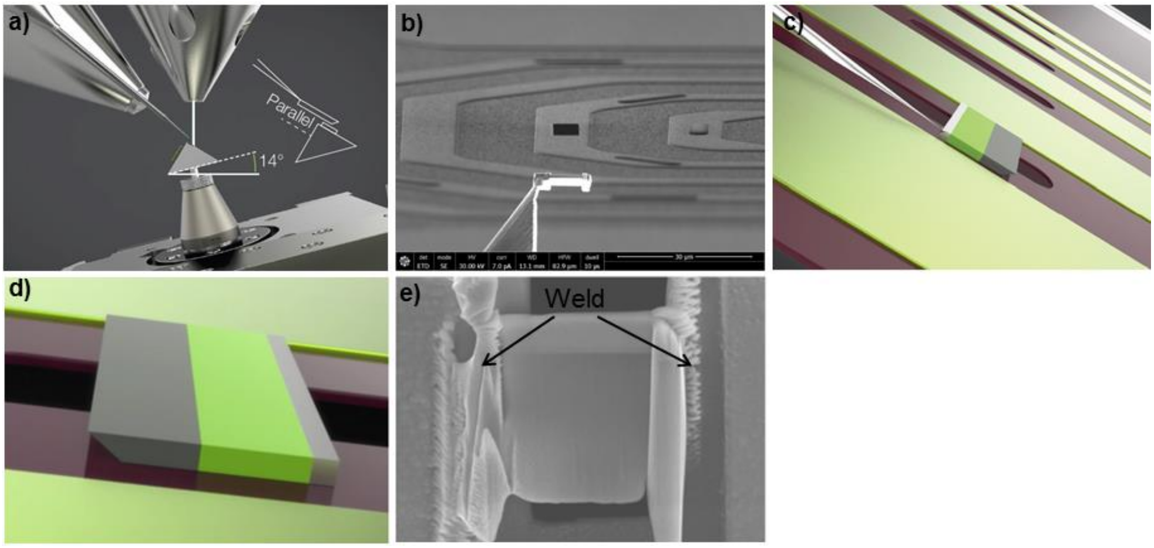


Figure 6. (a) After lifting out the lamella, move the stub to place the chip in the eucentric point. (b) Bring in the manipulator and drive the lamella towards the window of interest. (c) Place the lamella on the window and bring in the GIS; perform welding @ 30 kV and 80 pA. (d) Cut and retract manipulator. (e) Real image of transferred lamella

7. Final thinning of the lamella – Ion beam facing depot

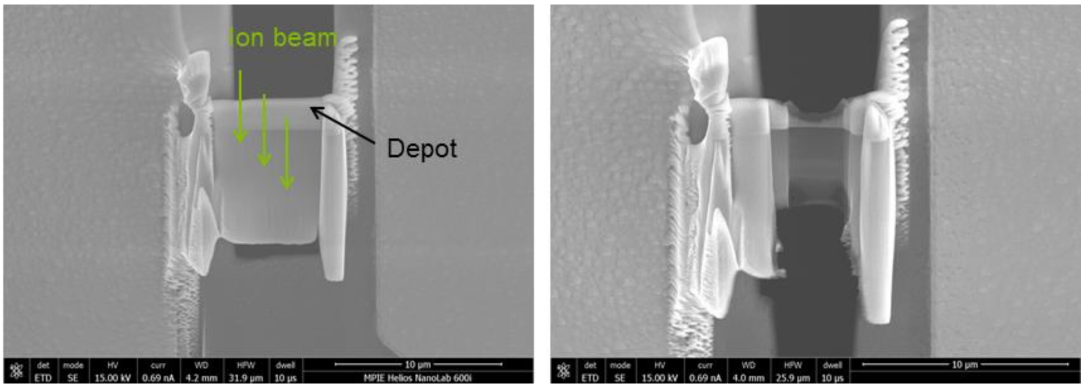


Figure 7. Tilt stage to 12°(to mill the front part) or 9°(to mill the back part) and start thinning down the lamella @ 30 kV and decreasing currents with regular cross sections till it reaches a thickness of roughly 400 nm. Then start using the option *"cleaning cross-sections"* for final milling @ 5 kV and decreasing currents until sample is electron transparent

8. Final thinning of the lamella – Ion beam facing the substrate

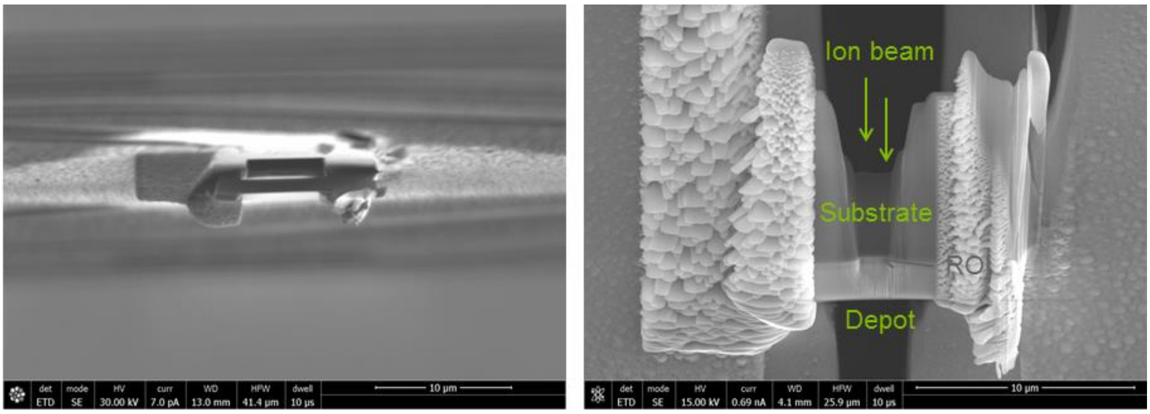


Figure 8. Same thinning procedure like described in step 7. Only difference is that the microscope chamber needs to be opened in order to rotate the chip upside down

III. Using Electrochemical Polishing

1. Electrochemical polishing, cutting of the lamella and transferring onto the MEMS device

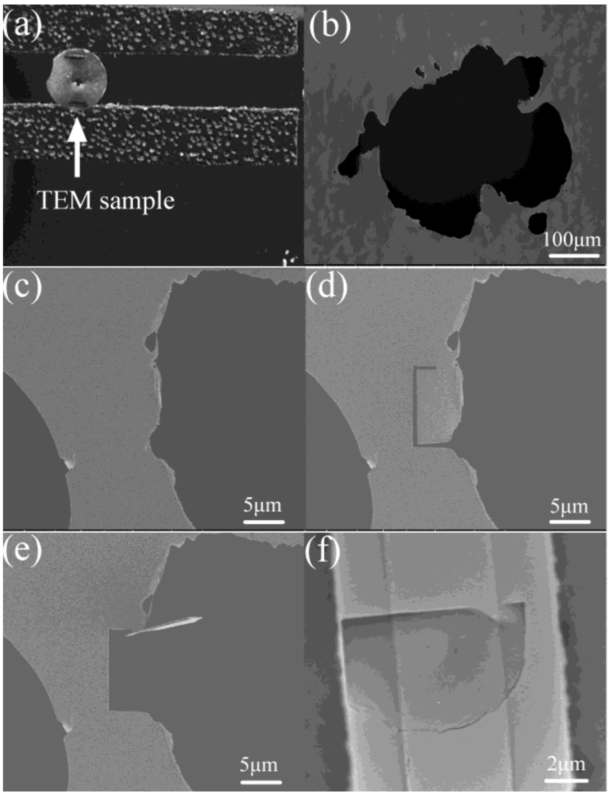


Figure 2. (a) The conventional 3mm disc is sticked onto a clean silicon chip with conductive tapes, while the area close to the hole is suspended. (b) Low magnification SEM image close to the hole. (c) The original thin area selected to cut. (d) Three grooves have to be cut through the sample. (e) The last cutting step leads to the flip up of the lamella. (f) Low magnification TEM image shows the lamella transferred onto the MEMs-based in-situ heating chips. (g) HREM of Ti69Ta30Al1 lamella.

Acknowledgements

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