

Getting a grip on *in situ* biasing A clean method for sample preparation for *in situ* biasing TEM experiments



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Introduction & Motivation

For state-of-the-art *in situ* TEM characterization of electrical material properties thin sample lamellas are cut from the bulk material and transferred to Micro Electro Mechanical Systems (MEMS) devices, employing a Ga-focused ion beam (FIB) instrument [1,2]. However, FIB based preparation procedures currently used for *in situ* biasing lamella preparation often lead to poor sample quality. The aim of this master thesis is to present an optimized dual beam SEM/FIB based preparation method for high quality *in situ* biasing TEM lamellas.

Bulk milling & Lift-out

Frontside & Backside milling on the gripper

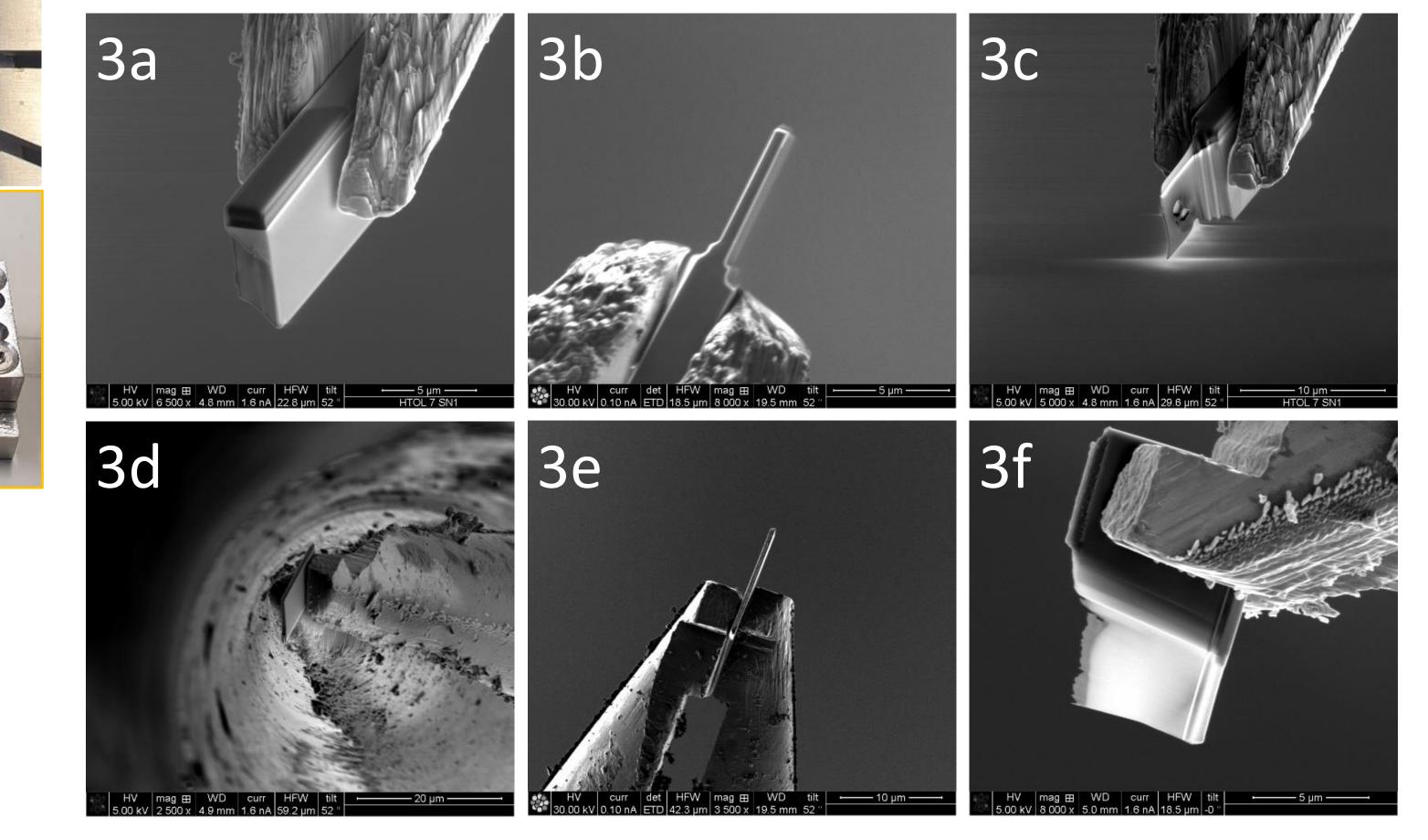
The preparation procedure is performed in a FEI nova 200 nanolab dual beam SEM/(Ga)-FIB system with the usage of the following tools (by Kleindiek and DENSsolutions):

- MM3A-EM Micromanipulator
- ROTIP-EM Rotational Tip
- MGS2-EM Microgripper
- Lightning dedicated biasing chip with custom holder
- FIB stub with AI cylinder

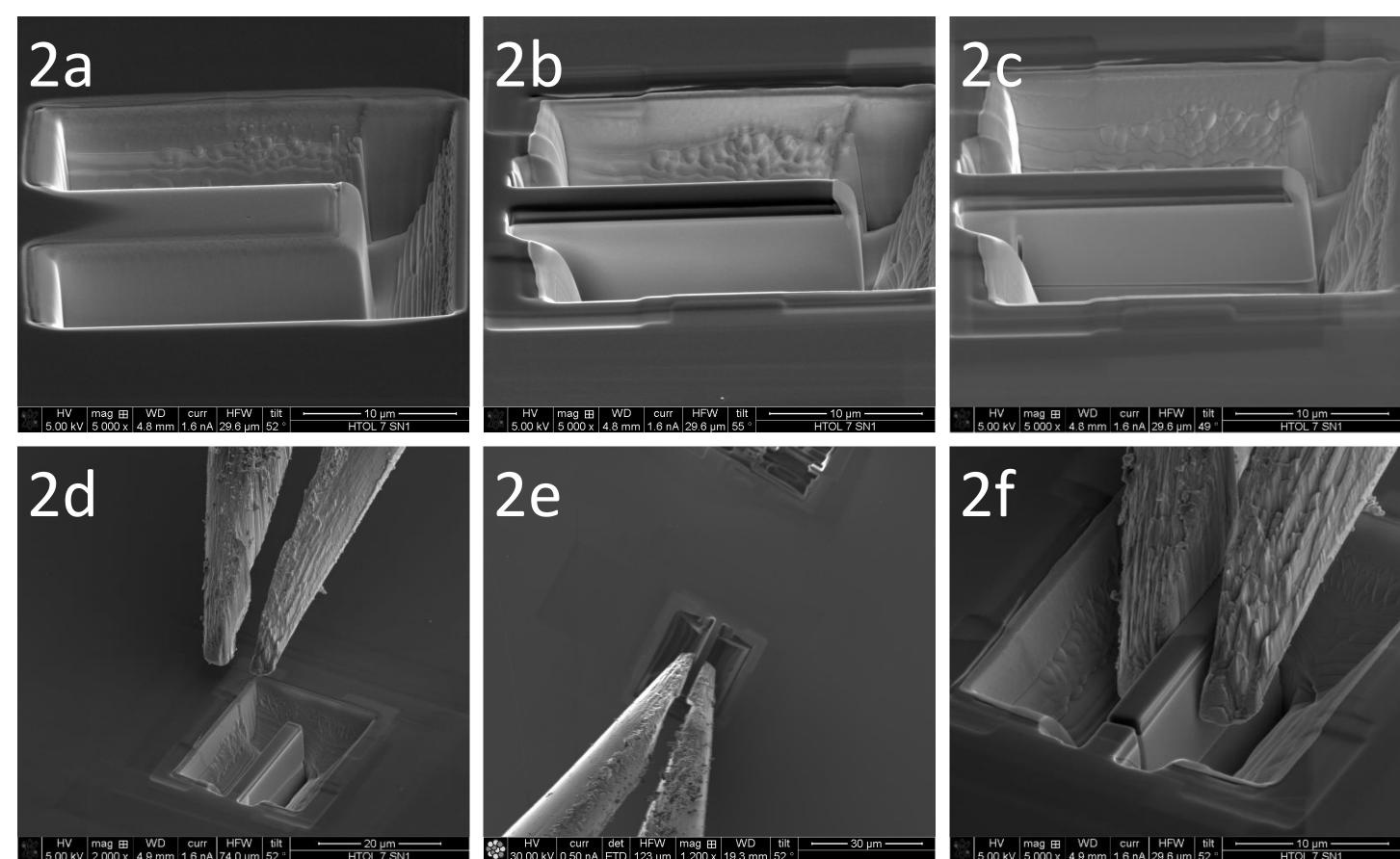
The following steps describe the bulk milling & lift-out procedure:

- 1) In advance: spin-coating of polymer protective layer or FEBID/FIBID deposition of Pt protective layer
- 2) Ion milling of three stepped trenches (fig. 2a)
- 3) Pre-thinning (fig. 2b)
- 4) U-cut & side cleaning (fig. 2c)

Milling is performed on the microgripper (at 52° stage tilt). The incidence angle of the ion beam is changed by the rotation of the ROTIP-EM Rotational Tip. Frontside milling is possible (fig. 3a-c) as well as backside milling (fig. 3d-f).



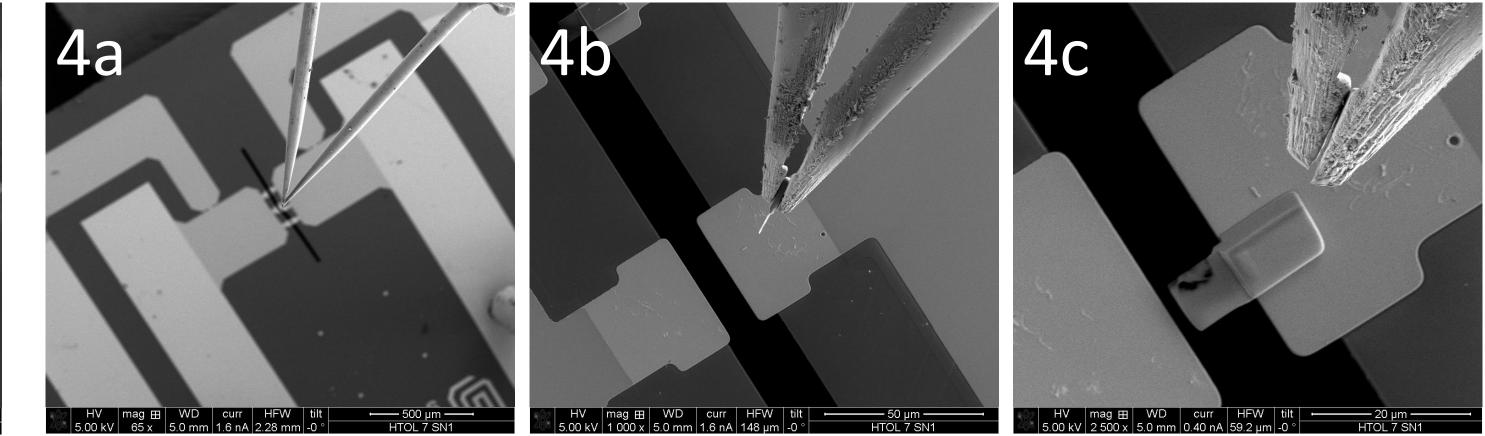
- 5) Approaching the gripper (fig. 2d)
- 6) Lift-out at 52° stage tilt (fig. 2e,f)



Transfer & Fixation on the chip

The lamella can be fixed on one electrode (for biasing) or on both electrodes (for measurements with current flowing):

- 1) Transfer of the lamella to the chip (fig. 4a)
- 2) Drop off (fig. 4b)
- 3) Positioning of the lamella (fig. 4c)
- 4) Fixation of the lamella with our specifically developed FEBID protocol (on one or both electrodes)
- 5) Optional: Electron Beam Curing (EBC) of deposits



TEM-results & Conclusion

2 µm

TEM investigations show that this method allows for a high sample quality and little to no Pt contamination (fig. 5a,b). Thus, imaging with atomic resolution is achievable with ease (fig. 5c). However, especially for poor electrical conductors it is difficult to get the lamella off the gripper in a controlled manner due to charging and polarization effects. The present work has established a framework for artefact free sample preparation for future research in the field of biasing TEM material characterization.

Acknowledgements & References

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