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APPLICATION NOTE

# FIB TEM Sample Preparation for *in situ* heating in TEM

Around the globe, material scientists and engineers face increased demand for more efficient transportation, enhanced solutions for clean energy, and original synthetically engineered materials. These demands drive research and development to produce better performing materials based on nanostructure phenomena. These enhanced materials are designed to be used in complex systems, special environments, and unique use case scenarios, where performance and reliability is critical.

For maximum insight into the properties of these materials, their structure and performance must be characterized and quantified using methods that go beyond static 2D imaging. By observing these materials under conditions that mimic the actual use case, it is possible to gain deeper understanding of the relationship between structure and composition, unique properties and ultimately, material performance.

Fundamental to *in situ* electron microscopy is the principle of observing the material's behavior in real time, not just under static conditions. Solutions for *in situ* dynamic microscopy from Thermo Fisher Scientific allow materials scientists and engineers to observe morphology and dynamic property changes—in real-time, at the micro- nano- and even atomic scales, in order to gain a complete understanding of how materials will perform in real world applications.

*In situ* experiments that require thermal testing need accurate data collection not only at a specific point but consistently across the complete experiment cycle. The Thermo Scientific™ NanoEx i/v is a MEMS device-based, single tilt TEM holder that enables *in situ* atomic-resolution imaging at elevated temperatures.

Before an *in situ* heating experiment can be performed in a TEM, the nanomaterial of interest needs to be transferred onto the MEMS device. The MEMS device consists of a silicon structure with 4 micron size holes covered with a thin electron transparent silicon nitride film. The subject of study will have to be placed over the holes for TEM observation (c.f. Figure 1). While nanoparticles can be directly dispersed onto the holes of the membrane, bulk material would require FIB TEM lamella preparation. A FIB TEM lamella suitable for MEMS heating can be prepared by ex situ or by *in situ* lift-out. This document describes both methods.

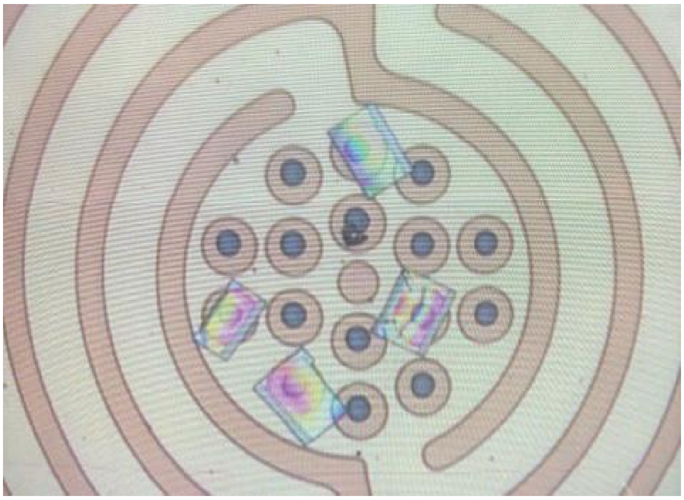

Figure 1. Optical microscope image showing the positioning of FIB lamellae on NanoEx i/v MEMS devices using ex situ lift out.

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### Method 1: Ex situ lift-out

The ex situ lift-out method describes the process of a lamella prepared by FIB and lifted out of bulk onto a suitable TEM grid outside vacuum under an optical microscope. This process was developed initially for high-throughput TEM lamella preparation of semiconductor materials. Automation packages such as Thermo Scientific™ AutoTEM™ 4 software can be used to prepare a batch of electron transparent foils ready for lift-out.

When preparation is finished, the bulk specimen (with the TEM foils inside) is transferred to a lift-out workstation consisting of an optical microscope with long working distance lenses and a sharp glass needle controlled by a micro-manipulator. The glass needle will be maneuvered towards the lamella, where electrostatic forces allow the lamella to stick to the glass needle for lift-out. The lamella can be brought down on a TEM grid or the MEMS heating device by letting needle with the lamella softly touch the surface. The electrostatic forces between the surface of the lamella and the MEMS device will keep the thin lamella in place. The ex situ lift-out process is a very fast and efficient method to prepare FIB TEM foils for the MEMS heater.

The area of interest on the bulk copper alloy is located by SEM, followed by chunk lamella preparation and lift-out at 52 degree tilt. After lift-out we thin the chunk to electron transparency while still attached to the needle, while observing thickness by SEM. We now navigate to the MEMS device. We optimize the orientation of the stage and rotate the EasyLift micromanipulator (G4 systems this would be bulkstage at 38 degree tilt, -26.8 rotation and the Thermo Scientific™ EasyLift micromanipulator at -26.8 rotation). The focused ion beam exposure should now be limited to low currents to avoid any surface damage. After touch down the lamella can be welded using the electron beam and released from the needle by low current FIB. The lamella is now ready, the MEMS chip can be removed from the MEMS chip compatible rowbar holder and placed in the NanoEx i/v TEM holder for TEM investigation. Multiple TEM foils of the copper alloy were placed on a single MEMS chip and imaged in the Thermo Scientific™ Talos F200 S/TEM.

### Method 2: *In situ* lift-out

The *in situ* lift-out method describes the process of a lamella prepared by FIB and lifted out of bulk onto a suitable TEM compatible surface inside vacuum using an *in situ* micromanipulator. The entire lift-out process can be completed inside a DualBeam™ without breaking vacuum.

As an example we take a copper alloy which we would like to study in TEM while performing a heating cycles. First we take a new MEMS chip and place this in a MEMS chip compatible rowbar holder for a DualBeam system. The same MEMS chip will be later transferred to the NanoEx i/v *in situ* TEM holder. The rowbar is placed inside the DualBeam system together with the bulk copper alloy for TEM sample preparation.

The most interesting step in this specific preparation process is to decide when to thin the chunk to electron transparency while preserving the surfaces for TEM investigation. The chunk can be thinned while it is inside the bulk substrate, before attachment to the MEMS device or after attachment to the MEMS device. Thinning after attachment is not recommended due to the navigation restrictions of the holder and charging silicon-nitride membrane. Thinning inside bulk will lead to re-deposition during the process of chunk extraction. Therefore we suggest to thin the chunk to electron transparency when it is already on the lift-out needle. At this point only a very small piece of material is left between the needle and the chunk.

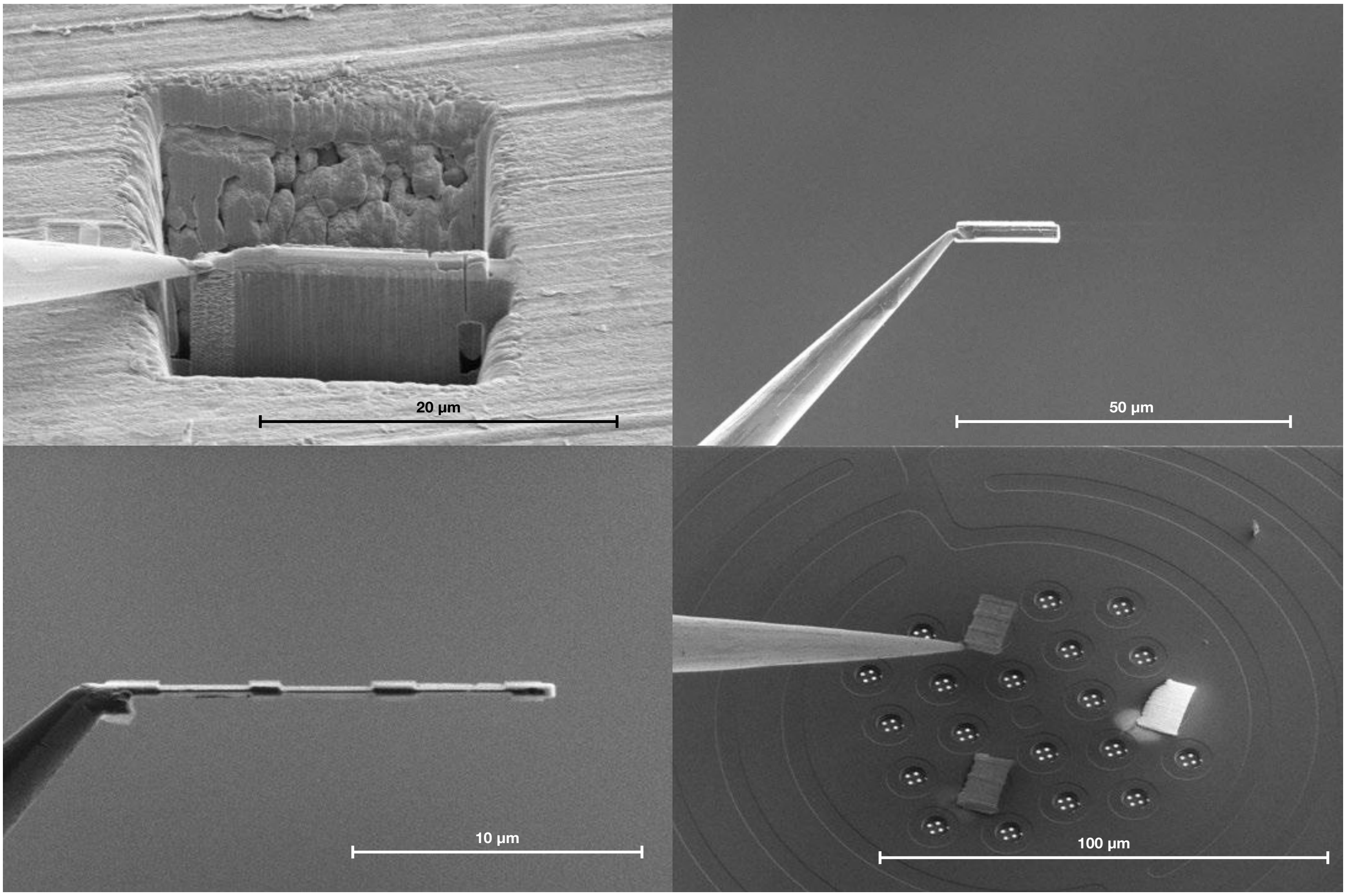


Figure 2. *In situ* lift-out procedure.

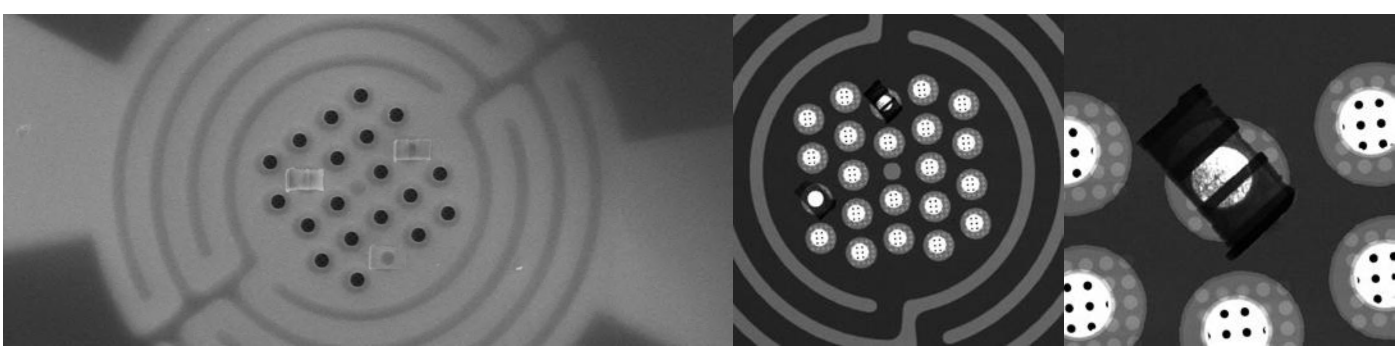

Figure 3. Two copper lamellae lifted out and placed on the MEMS device.

### Lift-out prerequisites:

- A MEMS chip holder for DualBeam (1100668)

#### Ex-situ liftout:

- An external ex-situ lift-out station consisting of a high performance optical microscope with ultra-long working distance lenses and a micromanipulator to control a sharp glass needle.

#### *In situ* liftout:

- A EasyLift micromanipulator for *in situ* liftout.


### References

[1] *A MEMS-Based Heating Holder for the Direct Imaging of Simultaneous In situ Heating and Biasing Experiments in Scanning/Transmission Electron Microscopes, LUIGI MELE et al. Microscopy Research and Technique 2016*

[2] *Convenient Preparation of High-Quality Specimens for Annealing Experiments in the Transmission Electron Microscope, Martial Duchamp et. Al Microscopy and Microanalysis 2014*

| | Top-Down | Top-Down | Planview | Planview |
|---|---|---|---|---|
| | G3 | G4 | G3 | G4 |
| **Bulk Preparation** | | | | |
| • Bulkstage Tilt | 52 | 52 | 52 | 52 |
| **1. Extract from Bulk*** | | | | |
| • Bulkstage Tilt | 52 | 52 | 38 | 38 |
| • Bulkstage Rotation | 0 | 0 | 72.5 | 63.2 |
| 2. On Grid thinning | | | | |
| • EasyLift micromanipulator rotation | 0 | 0 | -101.1 | -95.7 |
| **3. Attach to grid** | | | | |
| • Bulkstage Tilt | 38.0 | 38.0 | 38 | 38 |
| • Bulkstage Rotation | -17.5 | -26.8 | 72.5 | 63.2 |
| • EasyLift micromanipulator rotation | -101.1 | -95.7 | 0 | 0 |

*G3: FEI Quanta 3D, Versa, Scios, Helios G3 series (40° EasyLift micromanipulator port)*
*G4: FEI Helios G4 series (45° EasyLift micromanipulator port)*

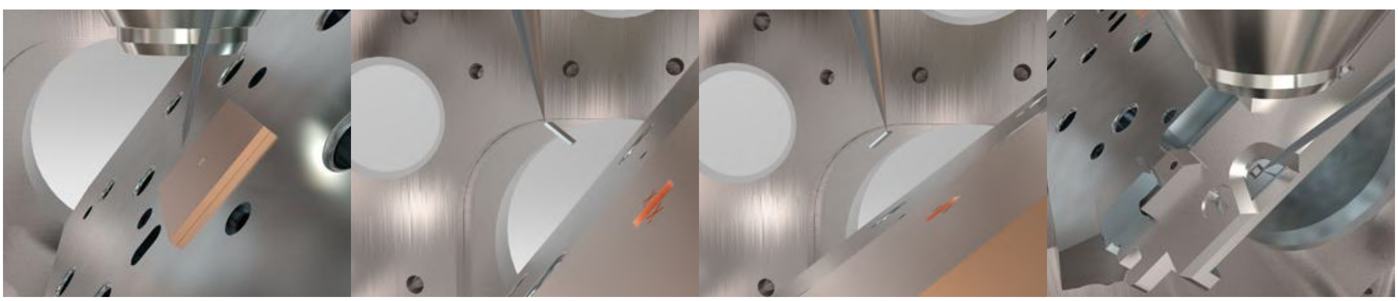

Figure 4.1. *In situ* liftout procedure top-down FIB TEM sample preparation

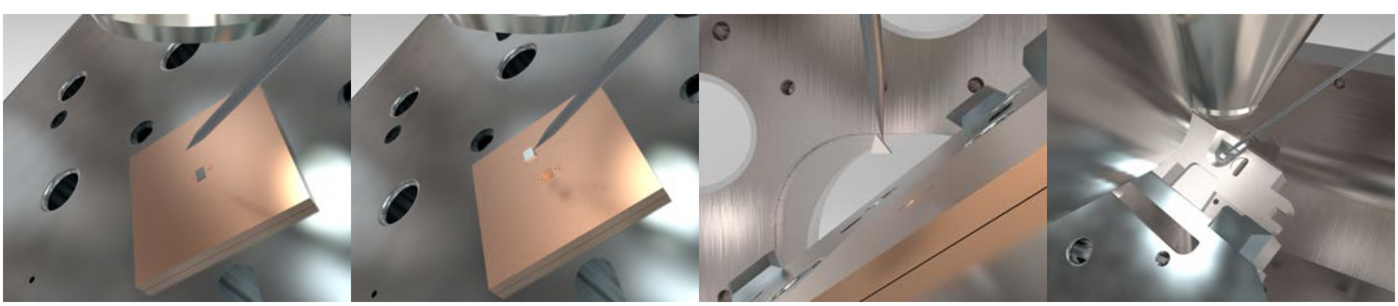

Figure 4.2. *In situ* liftout procedure planview FIB TEM sample preparation

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