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APPLICATION NOTE

# Sample preparation for *in situ* heating experiments in a DualBeam

Direct real-time observation of microstructure evolution at elevated temperatures in electron microscopes has a crucial impact on understanding underlying principles of thermally active processes, such as recrystallization, grain growth and phase transformation.

The MEMS-based Thermo Scientific™ µHeater Holder, in combination with *in situ* sample preparation in SEM/FIB systems, provides new opportunities for convenient, fast and reliable high-temperature *in situ* heating experiments under high-vacuum conditions.

The µHeater holder takes advantage of a Thermo Scientific DualBeam™ instrument by allowing for sample preparation by FIB and transfer to the MEMS chip without breaking vacuum in the DualBeam chamber. First, the area of interest is selected in SEM/FIB imaging mode. Then, a chunk of material is cut with the FIB and attached to the micromanipulator needle using beam-induced deposition. After lift-out, it can then be shaped using the FIB. The chunk is then placed on the MEMS heating holder, attached with beam-induced deposition, and cut free from the needle.

## Method

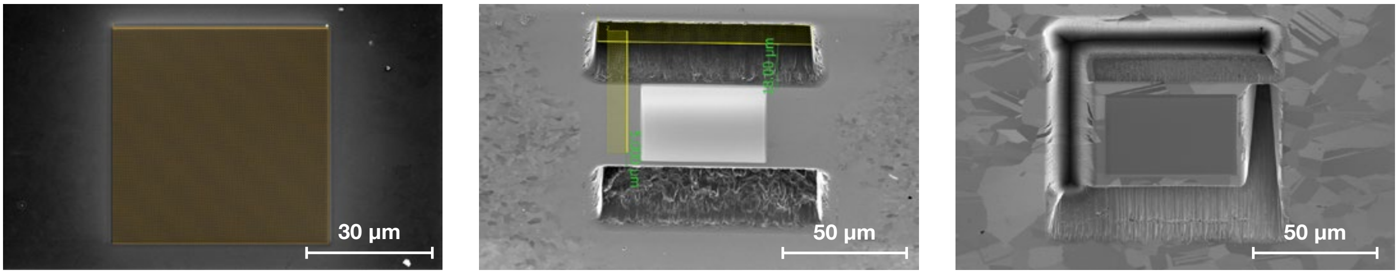
The first step is to determine the region of interest with the electron beam. Often, samples may have a specific orientation requirement for FIB processing to achieve the desired end result.

Thermo Scientific stages are designed for flexibility, allowing you to orientate the region of interest for standard cross-section or planview lift-out.

Prior to material removal by FIB, a protective capping layer is generally placed over the top of the region of interest. This can be achieved by electron beam-induced deposition (EBID), when the surface of the region of interest is of importance, followed by ion beam-induced deposition (IBID).

DualBeams offer a variety of ways to monitor milling processes for cross-section preparation and end-pointing. For the coarse milling, direct observation of the sample is not desirable at such high current, as the protective layer and sample surface are rapidly destroyed by the ion beam. Instead, a snapshot is used to perform a single scan of the viewed area. Patterning progress can be observed with Real Time Monitor if necessary.

At this stage, the first L-shape rectangular box is milled at a shallow glancing angle to create a chunk lift-out. The stage tilt is determined by how deep the region or feature of interest is located below the sample surface. Shallower angles will result in a deeper cut for larger volumes, while shallower cuts will produce smaller volumes. The images below represent the chunk milling steps, deposition and chunk milling. Two intersecting rectangular box patterns are drawn on the chunk back side and then patterned. Next, the second L-shape box is milled by rotating the stage relatively by 180° using compucentric rotation mode. Two intersecting rectangular box patterns are drawn on the chunk front side and then patterned.



An example of a chunk preparation process. Ion SE image of the 50x50µm Carbon protective deposition pattern (left), coarse top and bottom milling (center) and the chunk after the 2nd L-shape box milling. These milling steps are performed using the maximum available FIB current of 65nA for the highest throughput.

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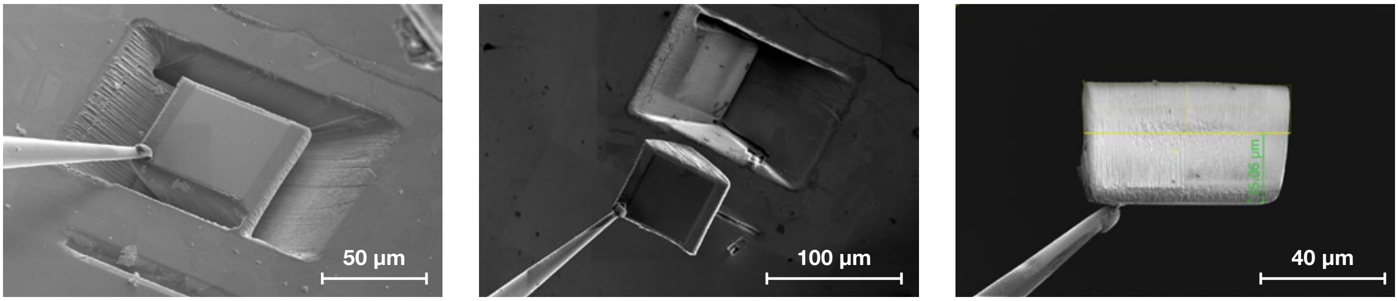
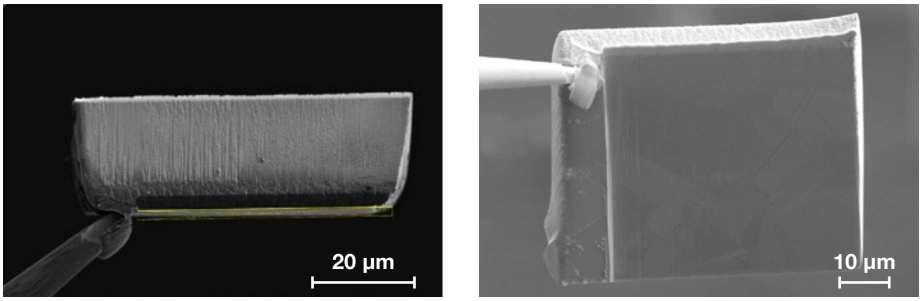


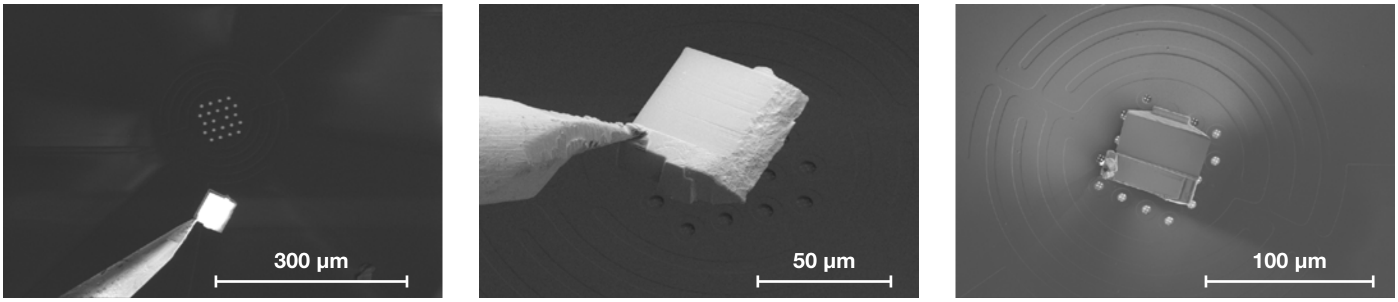
Process of the chunk lift-out using the EasyLift NanoManipulator. SEM image of the chunk and EasyLift needle after C welding (left). The chunk has been cut free from the bulk and lifted out (center). Ion image of the rotated chunk on needle and bottom milling cross section pattern to create a flat surface that will be placed on a MEMS-chip (right).

After milling, it is necessary to clean the cut-faces, making the chunk ready for lift-out by the Thermo Scientific™ EasyLift™ NanoManipulator. With the chunk now lifted out, *in situ* on-needle chunk surface cleaning is performed by first milling off the bottom of the chunk. At this point, the chunk bottom is only roughly cleaned, so the bottom cut-face is now cleaned to ensure a smooth attachment of the chunk to the MEMS chip. Suggested parameters for chunk liftout and on needle polishing are summarized in table 1.



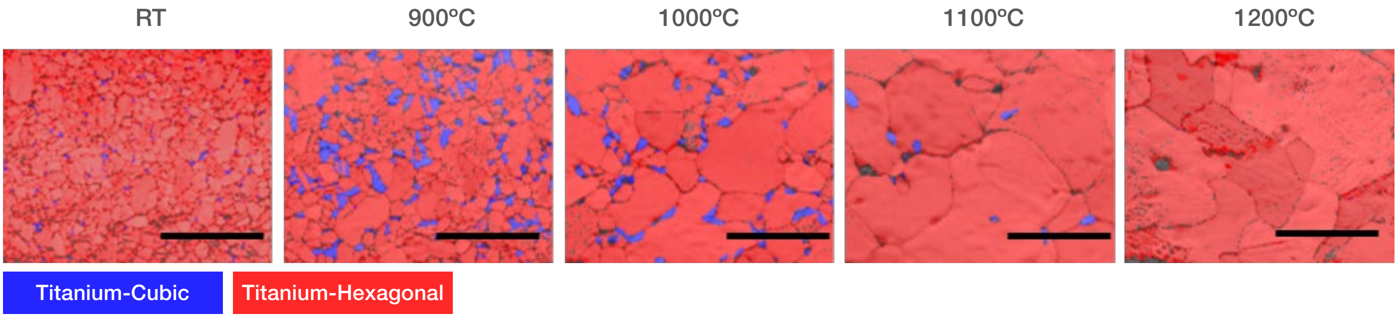
Cleaning pattern placement (left) and the resulting surface (right) of the chunk possess an excellent quality suitable for SEM or EBSD observations at elevated temperatures.

Depending on the material used, low-energy polishing may be desired. If this is the case, the preferred chunk front surface quality can be attained with an initial 30 kV polishing followed by a further cleaning at 5 kV. During the process, cleaning can be directly observed with the SEM. After cleaning, the surface quality should be sufficient for EBSD measurement.

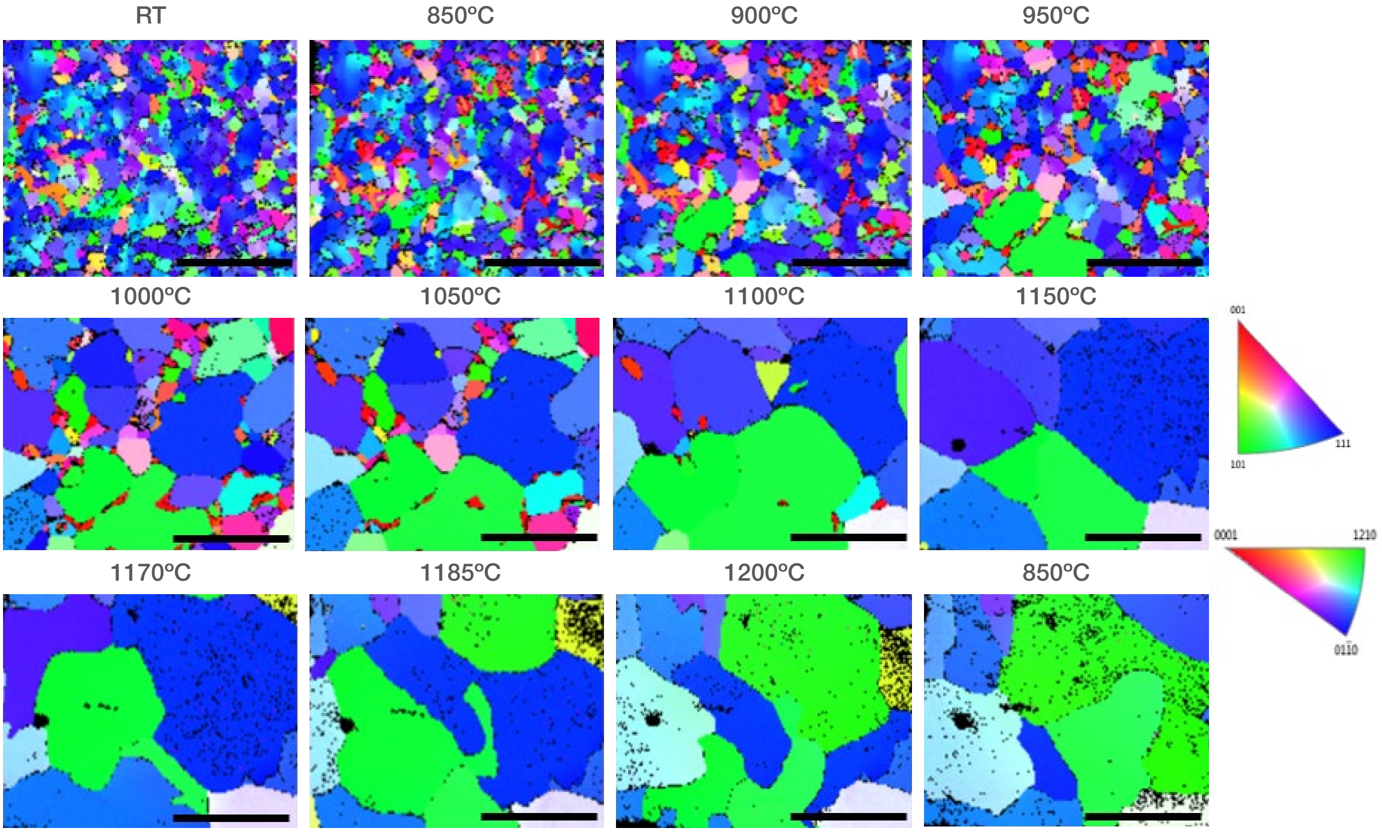


Transfer of the chunk using the EasyLift and its attachment on the MEMS chip. Chunk approaches the MEMS chip viewed by ion (left) and electron (center) beams. SEM image of the chunk on the MEMS chip after Pt welding on one side, EasyLift needle is cut free and retracted (right). Sample is ready for *in situ* heating experimentation.

Finally, the chunk is placed on the MEMS chip with the EasyLift NanoManipulator. Viewing can be done with electron beam and ion beam; however, viewing with ions should be minimized in order to reduce damage to the MEMS chip surface. Suggested parameters for chunk put down to MEMS chip are summarized in table 2.



Ti-6Al-4V alloy sample was heated gradually from room temperature to 1200°C with various isothermal steps at a heating rate of 1000°C/s. The overlayed phase maps with band contrast maps acquired at room temperature, 900°C, 1000°C, 1100°C and 1200°C show a transformation cycle from Ti α(hcp) to Ti β (bcc) to Ti α (hcp) in the *in situ* heating sequence. Scale bar is 20 μm. Each map was collected in approximately 7 mins.



Ti-6Al-4V alloy sample was heated gradually from room temperature to 1200°C then cooled down to 850°C with various isothermal steps at a heating rate of 1000°C/s. *In situ* EBSD IPFZ maps were collected at room temperature and at each isothermal step during the heating and cooling cycle. Sequential original IPFZ maps show a distinct recrystallization and grain growth (grain boundary migration) process at elevated temperatures. Scale bar is 20 μm. Each map was acquired in approximately 7 minutes.

**Table 1: Suggested parameters for chunk liftout and on needle polishing**

| Action | | | Pattern action/ application | Pattern size (suggested) | HV, beam current |
|---|---|---|---|---|---|
| Weld EasyLift to chunk | Stage position | Relative rotate -63.2°, tilt 38° (bridge in left bottom corner) | Pt/C deposition | 5 µm×8 µm×2 µm<br>Over needle and chunk | 30 kV<br>1.2 nA |
| Cut off bridge | | | Box (Si) | 8 µm×5 µm×4 µm<br>Over bridge | 30 kV<br>9.1 nA |
| Cut off chunk bottom | EasyLift needle rotation (actual) | 95.7° | RCS<br>(Si multipass) | 65 µm×15 µm×30 µm<br>Over chunk bottom | 30 kV<br>20 nA |
| Clean chunk bottom | | 95.7° | CCS (Si) | 65 µm×4 µm×30 µm<br>Over chunk bottom | 30 kV<br>9.1 nA |
| Surface cleaning protection | | 95.7° | Pt/C deposition | 46 µm×2 µm×1 µm<br>Over the front edge of the chunk side surface | 30 kV<br>1.2 nA |
| Surface cleaning | | 92.7° | CCS (Si) | 48 µm×2 µm×50 µm<br>Over the chunk front face | 30 kV<br>9.1 nA |
| | | 91.7° | CCS (Si) | 48 µm×1.5 µm×50 µm<br>Over the chunk front face | 30 kV<br>1.2 nA |
| | | 91.7° | CCS (Si) | 48 µm×0.7 µm×50 µm<br>Over the chunk front face | 30 kV<br>440 pA |
| | | 90.7° | Box (Si) | 50 µm×2.7 µm×0.15 µm<br>Over the chunk front face | 5 kv<br>210 pA |

**Table 2: Suggested parameters for chunk put down to MEMS chip**

| | | Pattern action/ application | Pattern size (suggested) | HV, beam current |
|---|---|---|---|---|
| Chunk put down to MEMS chip, weld one side | Stage tilt 38°, EasyLift rotation 0° | Pt/C deposition | 40 µm×4.5 µm×1 µm<br>Overlapping the bottom edge of the chunk and the MEMS chip | 30 kV<br>750 pA |
| | | Box (Si) | Pattern over the joint of the EasyLift and the chunk | 30 kV<br>750 pA |
| Welding the other side of the chunk | Stage tilt 38°, rotate relatively 180° | Pt/C deposition | 40 µm×4.5 µm×1 µm<br>Overlapping the bottom edge of the chunk and the MEMS chip | 30 kV<br>750 pA |

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