**FP7-INFRASTRUCTURES-2012-1**

# Enabling Science and Technology through

# European Electron Microscopy

## Project Acronym: ESTEEM2

**Grant Agreement n°: 312483**

## Deliverable 6.2

## Report on protocols and standards developed in ESTEEM2

**Deliverable leader 6 – Max Planck Institute for Intelligent Systems, Stuttgart**

## REPORT OF ACTIVITIES BY TU GRAZ

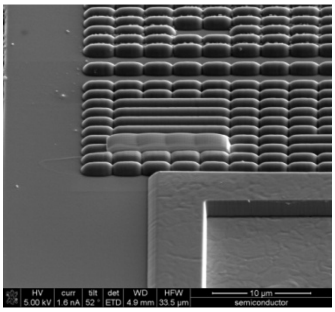
## Input to Deliverable 6.2:
## TEM Sample Preparation of Semiconductors
## with a Focused Ion Beam System (FIB)

**TEM sample preparation of Semiconductors with a Focused Ion Beam System**

The Focused Ion Beam System is a versatile tool to prepare target preparations for transmission electron microscopy. But this technique is not are artifact-free. In case of semiconductor preparation differing materials have different milling rates and complicate the preparation. This results in different milling rates, curtaining effects and various artifacts. To reduce these effects we prepared the samples with the "upside down" method, which shall be described here

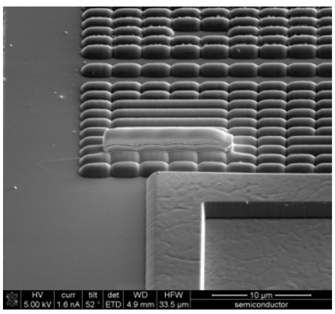
## "UPSIDE DOWN "Sample Preparation

First of all, the sample was sputtered with a conductive carbon layer to avoid charging during the FIB preparation.
After searching the ROI protect the sample with a minimum 250nm thick <u>electron beam deposited platinum layer</u>



| | |
|---|---|
| **rectangle:** | **10µm x 1µm x 5µm** |
| **Pattern:** | **Pt ebeam deposition** |
| **5 kV / 1,6 nA** | **(time ~ 11 min.)** |

<u>Tilt</u> the sample normal to the ion beam. (52°)
<u>Ion beam deposition</u> to protect the ROI during the hole preparation (in our case 2µm is thick enough)
Deposition height depend on the depth of the ROI – ROI near the surface needs a lower Pt layer as an ROI in deeper regions.



| | |
|---|---|
| **rectangle:** | **10µm x 1µm x 2µm** |
| **Pattern:** | **Pt Ibeam deposition** |
| **30 kV / 100pA** | **(time ~ 11 min.)** |

Trench milling step1:

The pattern with and the milling time occurs on the sample material and the depth of the ROI (in our case the ROI is 7µm in the depth, so we have to perform big trenches.)

| |
|---|
| **rectangle pattern - parallel on both sides 12µm x 10µm x 6 µm (time ~ 20min.)** |
| **30 kV 5 nA (reduces drift during the milling - in our FIB system)** |
| **Leave a distance of 1 µm to the ROI to avoid damaging the sample** |

Trench milling step2:

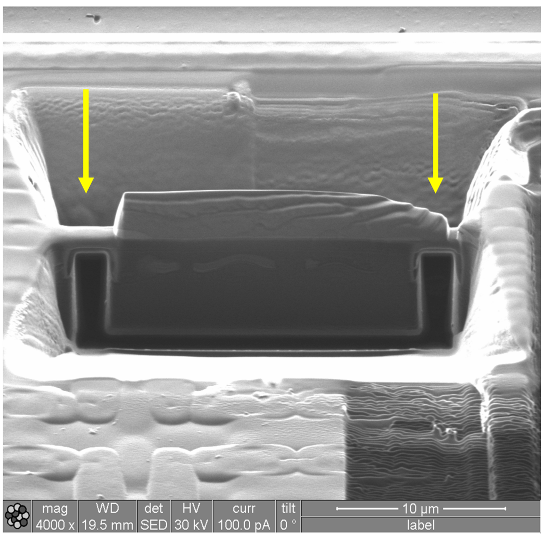
The now free standing lamella became cleaned with an angle of +/- 2° (50° / 54°) on both sides

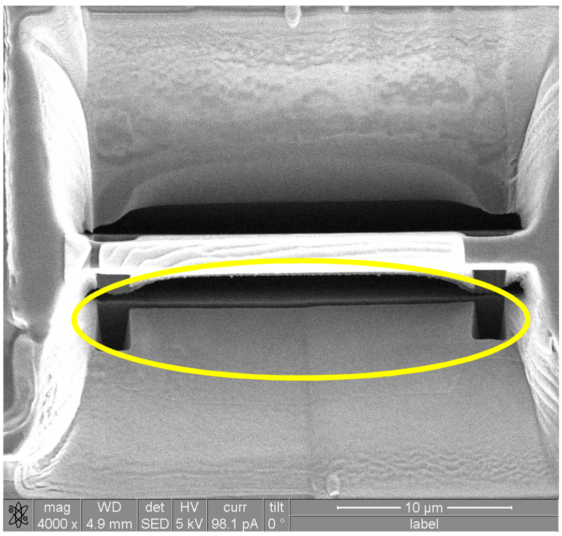
| |
|---|
| **rectangle pattern** |
| **30 kV 1 nA to a lamella thickness up to 1µm** |
| **(time ~ 10min. for both sides)** |

U – cuts to cut the lamella free from the substrates: Tilt the sample normal to the electron beam (0°)

| |
|---|
| **3 rectangle pattern – parallel milling** |
| **30 kV 1 nA / 0,5 nA 800 nm / 500 nm pattern with** |

The milling progress is controlled with the electron beam and stopped when the lamella stands free on the nose-pieces.



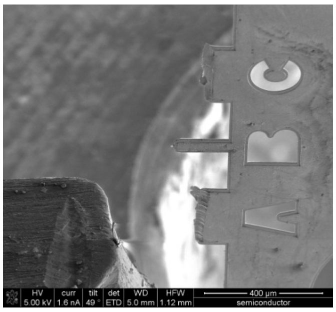

Left picture: ion beam image shows the free standing lamella on the nose-pieces.
Right picture: electron beam image of the lamella – cut marks formed by the ion beam when the U-cut is finished.

Insitu lift out

When the Omniprobe needle is touched and fixed with a Pt deposition on the lamella, the nose-pieces get cut away so the lamella can lift out of the substrate.

The lamella is fixed on a pre-prepared Omniprobe transfer grid that is perpendicular clamped in the TEM grid holder. With a scalpel cut (orange line) all overlay grid structures are cut away so only the middle freestanding finger is left over.

If the transfer is finished the TEM grid must be turned around 180° to perform an "upside down" lamella finishing.



grid pre-preparation for "upside down" performance perpendicular clamped TEM grid

"upside down" lamella finish up to electron transparency

First side finishing: (the milling process should be traceable with the electron beam)

I)

| | |
|---|---|
| **rectangle pattern:** | **Si (prefab by the manufacturer)** |
| **30 kV / 100pA - 50pA** | **milling angle: 2°** |
| **Near to ROI** | |

II)

| | |
|---|---|
| **cleaning cross section pattern:** | **Si (prefab by the manufacturer)** |
| **30 kV / 100pA - 50pA** | **milling angle: 1°** |
| **Up to the ROI** | |

Alternative milling strategy

| | |
|---|---|
| **line pattern:** | **Si (prefab by the manufacturer)** |
| **30 kV / 50pA** | **milling angle: 1°** |
| **Up to the ROI** | |

The milling process is finished when the ROI is reached, all material is milled and no curtaining is visible.

Second side finishing:

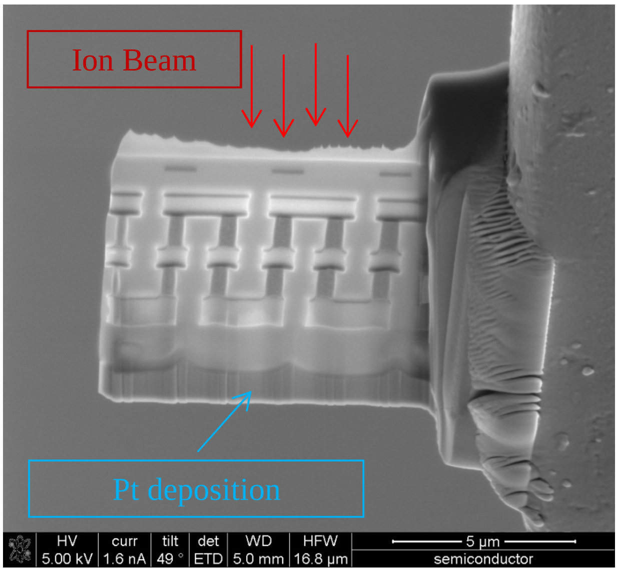
I) Similar to the first side

Cleaning to reduce the amorphous layers:

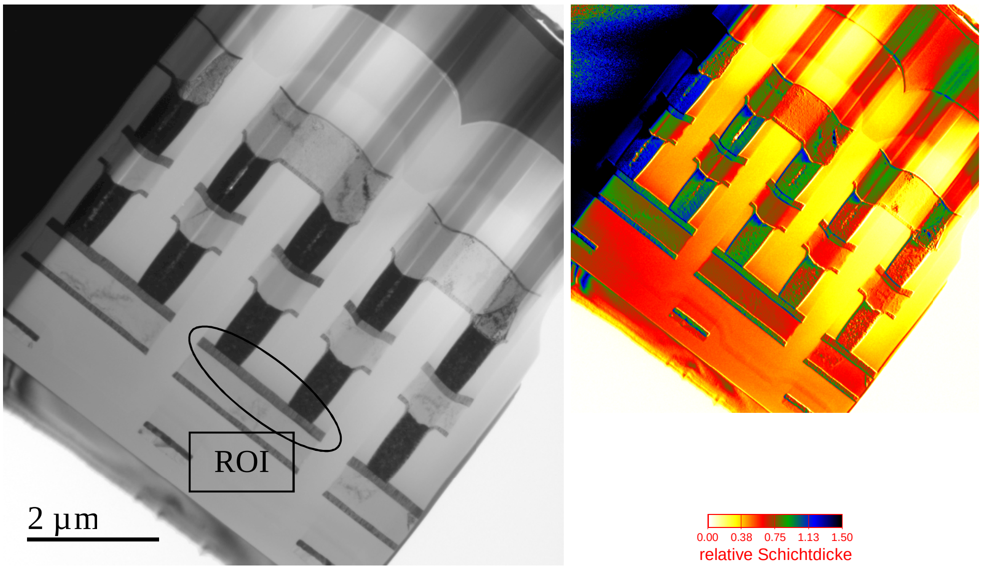
Tilt the sample at +/- 4° (48°/56°)

| | |
|---|---|
| **rectangle pattern:** | **Si (prefab by the manufacturer)** |
| **5 kV / 70pA** | **milling angle: 4°** |

Monitor the process with the electron beam. You should see a face change on the thin lamella edges.




SE FIB image of the finished lamella after 5kV cleaning




TEM BF image and relative thickness map

The preparation of an “upside down” lamella for semiconductor samples allows for a reduction of various preparation artefacts like selective milling and sample induced curtaining effects. Regions of interest in the vicinity or on the substrate can be easily and homogeneously milled, giving better suitable samples for a subsequent analysis.