

## FP7-INFRASTRUCTURES-2012-1

## **Enabling Science and Technology through**

**European Electron Microscopy** 

Project Acronym: ESTEEM2

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## **CNRS-CEMES: Input to deliverable 6.2**

**Report on protocols and standards developed in ESTEEM2** 

## **FIB preparation of insulating samples**







Preparing a FIB lamella for TEM is usually started with the deposition of a protective layer in two steps. A first layer is made with electron induced deposition (EBID) over a few hundred nanometers thickness. Such a thin layer is mandatory to protect the sample from any damages that could occurs with the second thick layer (several  $\mu$ m) made by ion induced deposition (IBID). This second layer is used for protection of the sample from any damage in the whole FIB preparation procedure.

A problem arises when an insulating sample has to be prepared (glass, MgO, etc...). With such sample type it is impossible to properly make that initial layer due to charging effect:

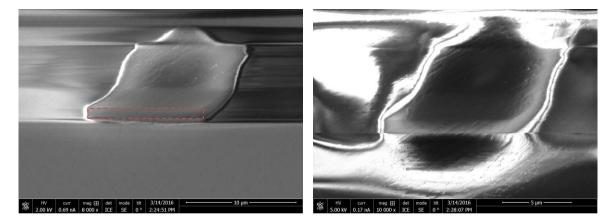


Figure 1 : Platinum EBID on an insulating MgO sample. Charging effect prevent from making a welldefined protection area (initial wanted shape was a rectangle depicted by dashed area in left image). The two images are displaying two various accelerating voltage (2 kV – left – and 5 kV – right) confirming the huge charging effect on such sample.

In such cases EBID has to be replace by a former ink deposition that will be used for a good protection layer for second step IBID deposition. The aim is thus to deposit a thin ink layer onto the sample prior to entering the FIB.



Figure 2 : Marker used for ink deposition.



http://esteem2.eu/





The full initial sample preparation can be sketched as follows:

- A line is drawn on a selected area of the insulating sample. One can adjust the final ink thickness by superposing several lines.
- All remaining insulated surfaces have to be cover with silver paste (or copper tape) to avoid any charging problem.

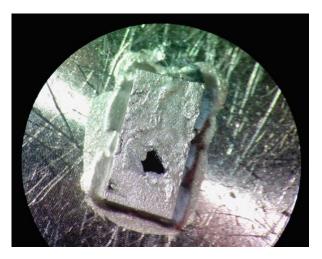


Figure 3 : Insulating sample drown into silver paste letting just see an ink covered area of the sample.

- Several hours have to be spent for a complete drying process of the ink (we recommend to wait for a full night time).
- One have then to find a good ink thickness area by simply milling some holes making various ink profile analysis (a value of few 100's of nm is acceptable)

Once the area is found one can proceed to the IBID layer before finishing the whole lamella preparation as recommended in other protocols.

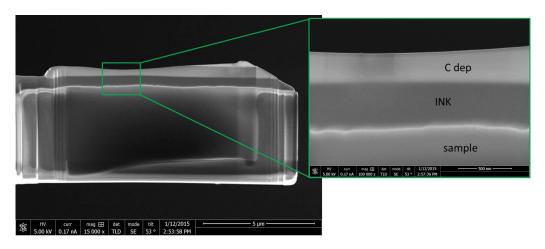


Figure 4 : Final sample with the ink and Carbon layer deposition using IBID.

