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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 UNIVERSITY OF ZARAGOZA

Thinning of organic magnetic tunnel junctions at low temperature: Cryo-dual beam FIB preparation

Transmission electron microscopy (TEM) investigations are highly dependent on the quality of the examined specimens. Preparation of lamellae with Dual Beam systems involves optimization of several ion beam working conditions. Usually, it is necessary to optimize the ion current and voltage values during the experiments in order to minimize the damages on the surface of the material and eventually avoid processes such as amorphization or gallium implantation. In the case of organic materials, which are in general delicate, optimization of other working conditions beyond the ion current and voltage could be helpful. Thus, the use of low temperature techniques opens venues to obtain better experimental conditions and, consequently, better results. Here we present a protocol to thin lamellas of organic magnetic tunnel junctions at low temperature although part of the process is still carried out at room temperature.

First step (room- temperature)

1-With the help of our technician expert in machining at LMA-UNIZAR, we made a copy of sample holder (following the original design from Quorum-technologies) but where it is possible to put the sample together with the grid. See figure 1c and 1d. The sample could be fixed with silver paint, copper tape, etc. And the grid holder is fixed in the groove with a screw.

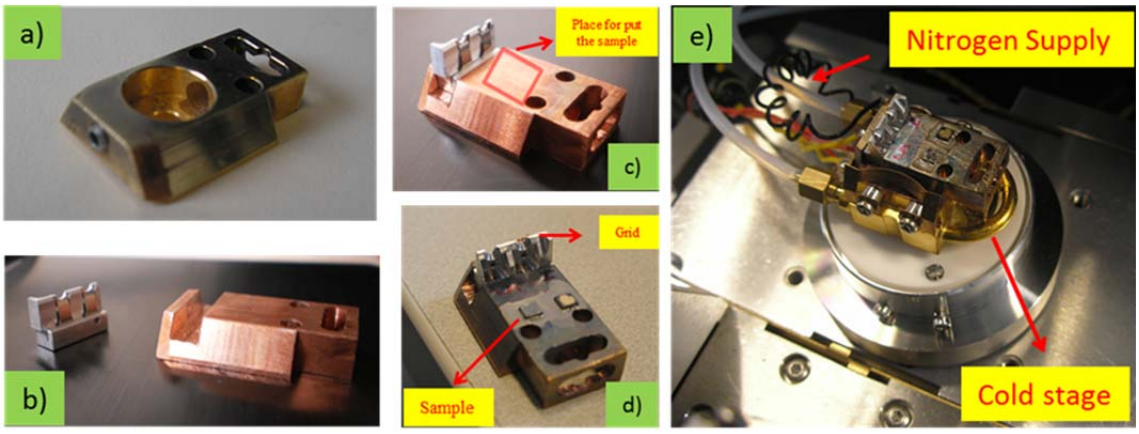

Figure 1a) Original sample holder from Quorum Technologies b) sample holder designed and home-made in INA&LMA c) Sample holder together with grids holder d) whole sample holder, samples and grid e) whole inside to the DB chamber together with the cold stage.

2- After that the sample holder is put on the cold stage see figure 1e, the Dual Beam chamber is closed, when the vacuum reaches a value around $1x10^{-6}$ mbar, we can start with the conventional process (Platinum deposited by e- and i+), the corresponding milling by ion beam and the lift out process. In the figure 2 some parts of the process are shown.

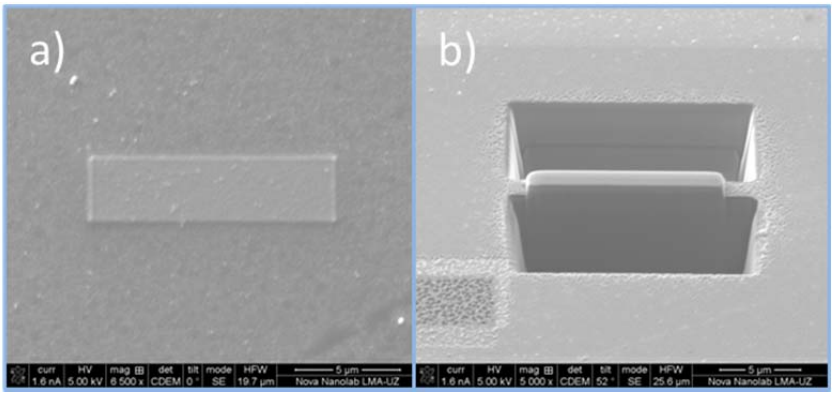

Figure 2 a) Pt deposited by e- over multilayer of Si/NiFe/Al_2O_3/CCO-7(organic)100nm/Al_2O_3/Co, b) View of milling by i+ beam during the process to make lamella previous to the lift out.

3– After the lift-out of the lamella, it is soldered on the grid. See figure 3

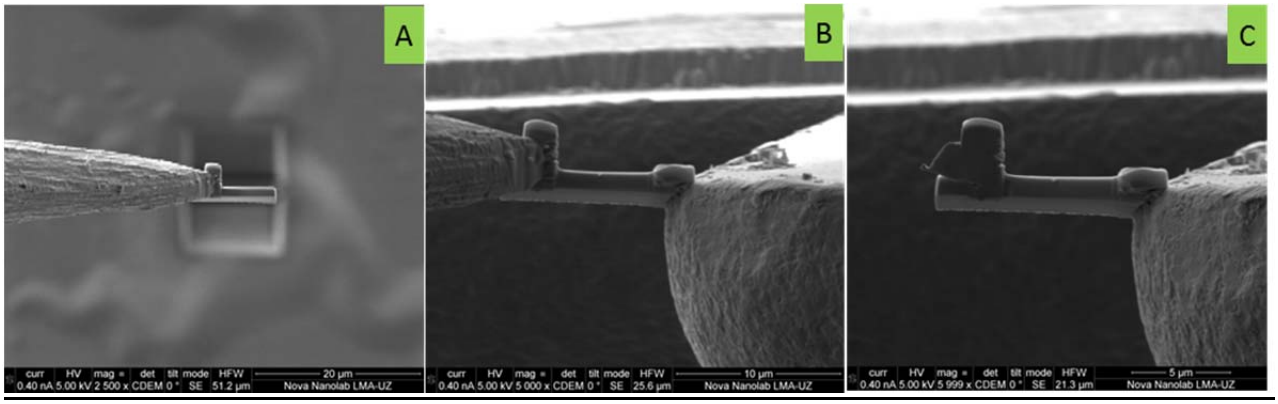

Figure 3 a) The lamella on the nanomanipulator after to lift out process. b) and c) part of process to solder the lamella on the grid. The material in this image correspond to a multilayer of Si/NiFe/BF-T2 (organic)/Co off axis/Al

Second step a Low temperature.

4-Once the lamella is soldered on the grid; the temperature is lowered to 110 K thanks to the gas nitrogen supply, which is cooled after passing by the Dewar which is filled with liquid nitrogen. When the temperature is stable, we can start the final thinning using the ion beam, until reaching a thickness about 60-80 nm. See figure 4

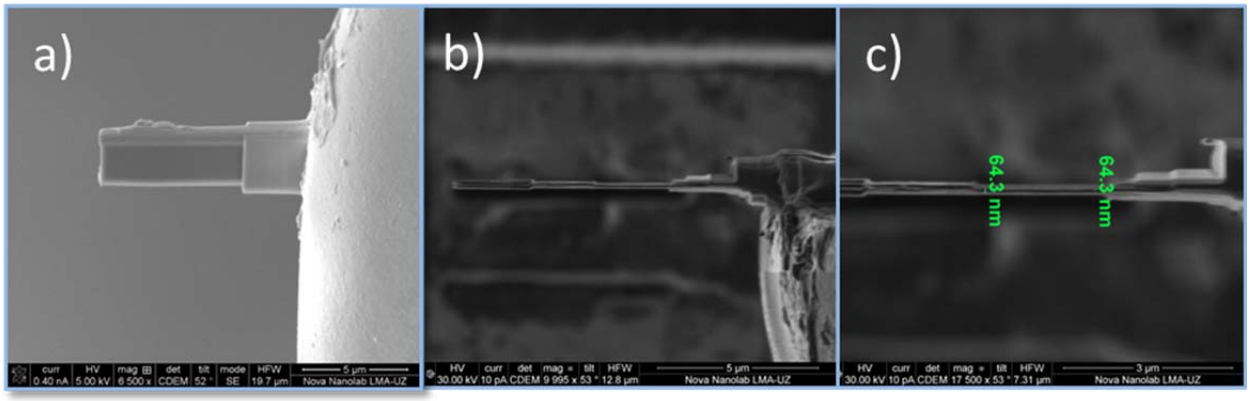

Figure 4. a) Electron image of lamella on the grid. b) Final view of the lamella, ion image c) details and thickness of lamella by ion image.

5- After the previous process is finished, the temperature is raised until room temperature in vacuum conditions. At this moment we can break the vacuum and open the Dual Beam chamber. In the figure 5 some TEM and EFTEM results are showed in the multilayer organic tunnel junctions.

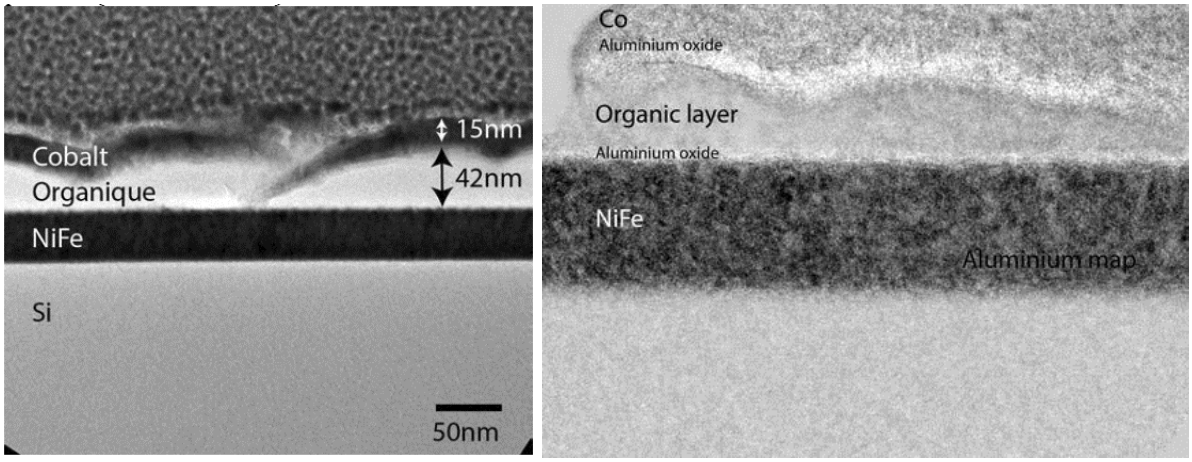

Figure 5. TEM and EFTEM images of the following stack Si/NiFe/Al_2O_3/CCO---4 (organic) 100 nm/Al_2O_3/Co. Electrical measurements on this sample showed no short-cuts but no magnetoresistance. TEM study by B.Warot-Fonrose (CEMES, Toulouse), sample from J.F.Bobo, M.Palosse and I.Séguy (LAAS, Toulouse)

Typically Materials

Until now, the only kind of studied material corresponds to organic magnetic tunnel junctions, provided by the Toulouse group. However, we believe that the protocol could be extended to other kinds of soft material to test the viability.