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Enabling Science and Technology through European Electron Microscopy

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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

Protecting thin film surfaces during FIB sample preparation by carbon coating

Standard focused ion beam preparation protocols for (oxide) thin films samples usually involve deposition of a Pt or Pt/C deposition (so-called electron-beam or e^--beam deposited material) on the surface of the thin film prior to sample preparation in a focused ion beam (FIB) instrument. This Pt or Pt/C coating is deposited on the thin films in order to protect them during the Ga^+ ion milling and thinning step, which are part of the standard FIB lift-out procedure.

However, deposition of Pt or Pt/C directly on top of a delicate thin film has proven to be detrimental to the thin film properties, as shown in the example of a $LaAlO_3$ (LAO) thin film grown on top of a $SrTiO_3$ (STO) substrate in Figure 1. The Pt/C layer deposited on top of the thin film has clearly damaged the top unit cell of the LAO film (arrows).

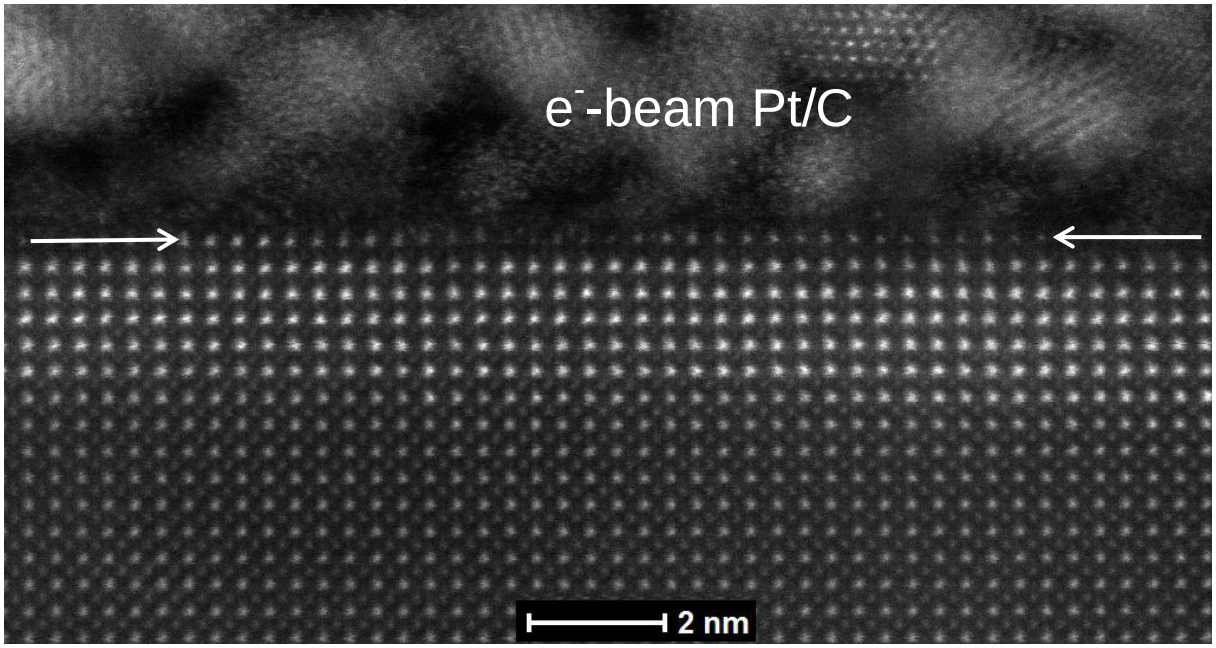


Figure 1: Standard FIB sample of a LAO (6 unit cells) thin film grown on top of a STO substrate

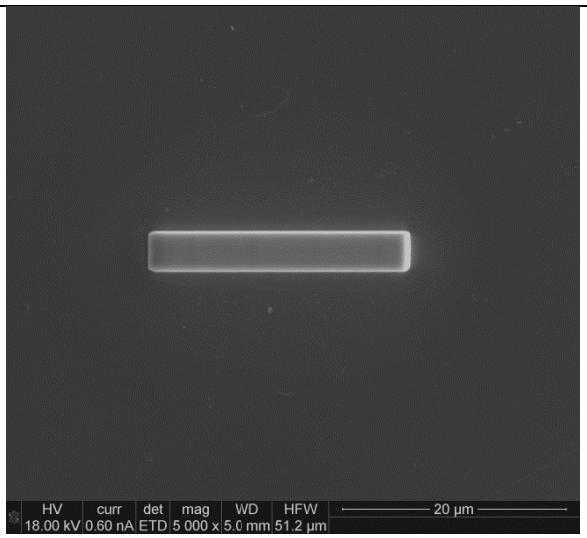
In this protocol, we describe the use of a thin carbon film prior to standard FIB sample preparation. This carbon film is deposited on top of the (oxide) thin film, in order to create a soft buffer in between the area of interest and the e^- beam Pt/C deposited in the FIB.

This thin carbon coating is applied to the surface of the thin film using a LEICA ACE600 high vacuum carbon coater (Figure 2). The evaporation of carbon at a vacuum level of $5x10^{-6}$ mbar is achieved by carbon thread pulsing of a carbon thread (260 W pulse power, 500 ms pulse). This allows a gentle deposition without disturbing the thin film material. In this way, the deposited carbon film is approximately 40 nm in thickness, which is found to be sufficient in order to protect the outer surface of the thin films (see example of a $BiFeO_3$ (BFO) thin film in Figure 3)

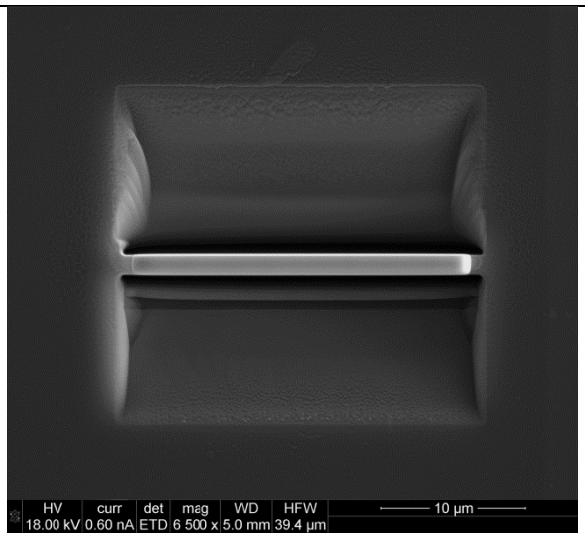
Figure 2: LEICA ACE600 high vacuum carbon coater

The full sample preparation protocol is then as follows:

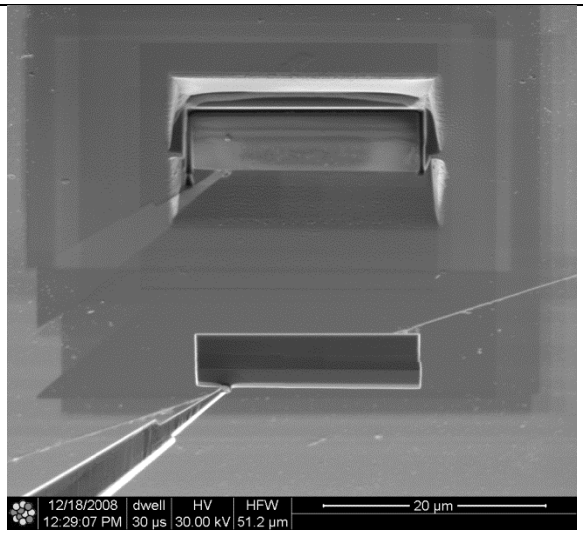
1. Carbon coating in the LEICA ACE600 instrument, using a 500 ms pulse at 260W applied to a thin carbon thread.
2. Transfer of the coated specimen to the FEI Helios FIB-SEM instrument at EMAT, ready for typical FIB sample preparation (Figure 3).
3. Coating of the sample using electron beam deposited Pt (e^--Pt, fine grains, approximately 1 micrometer thick layer, gentle deposition) followed by ion beam deposited Pt (Pt, coarser grains, approximately 2-3 micrometers thick).
4. Milling of two pits at either side of the lamella using Ga^+ ions at 30 kV beam energy at 9nA beam current.
5. Undercutting of the sample on one side, mounting of the omniprobe for sample lift-out, followed by the final cutting and release of the sample.
6. After lift-out, the sample is mounted in a two beam geometry onto a pre-milled Cu support omniprobe grid.
7. Gentle thinning of the sample for electron transparency, using 30kV Ga^+ ions for initial thinning, 8 kV Ga^+ ions for finer thinning and 2 kV Ga^+ ions for final thinning and sample cleaning.



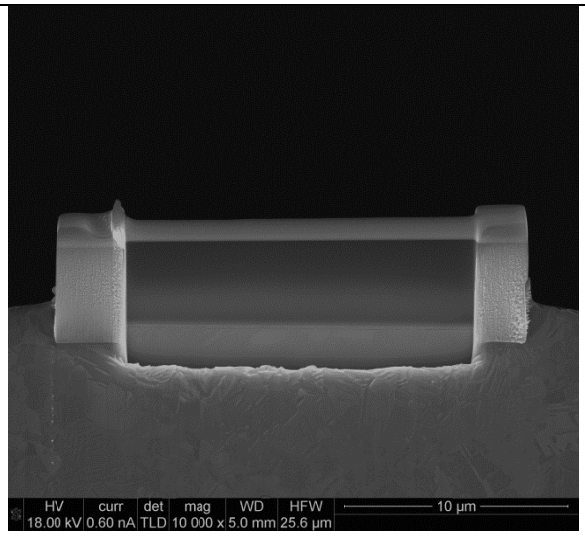
e⁻-beam Pt and Pt/C deposition



Milling of two pits next to the lamella



Undercutting and Lift-out procedure



Sample mounting on Cu grid, ready for final thinning

Figure 3: Typical FIB sample preparation steps

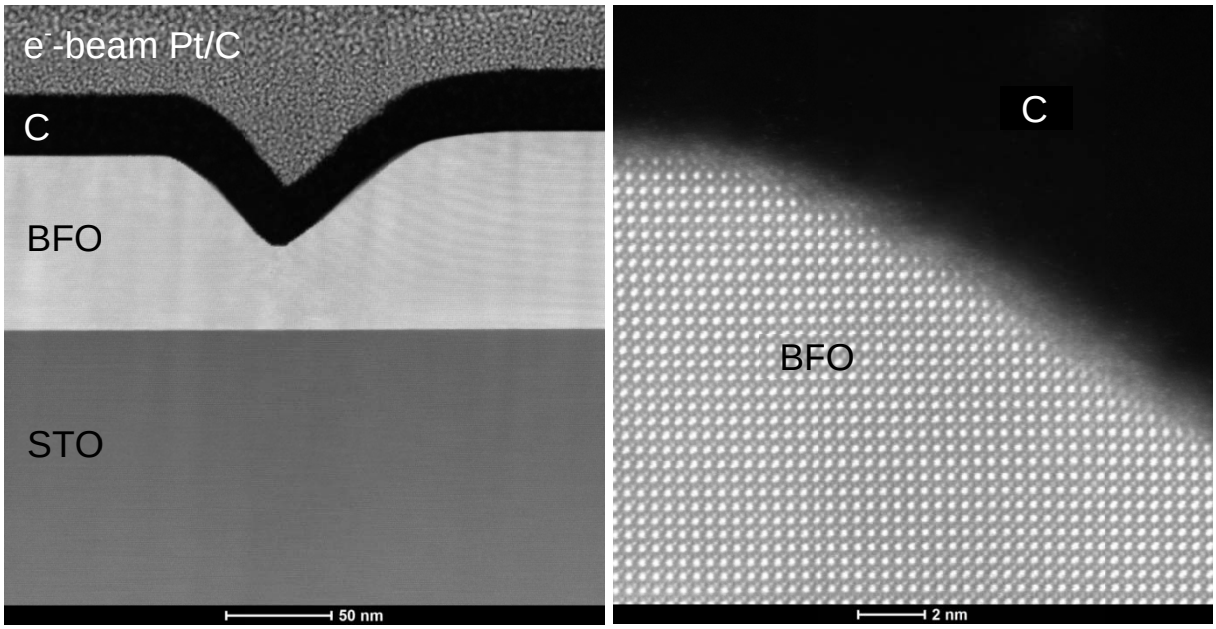


Figure 4: Finished FIB lamella of BFO on STO using a carbon buffer layer of 40 nm thickness