In-situ STEM investigation of thin films in a dual-beam equipment

J. M. De Teresa [a], *R. Córdoba* [b], *P. Strichovanec* [b], *N. Marcano* [b], *M.R. Ibarra* [b]

[a] *Instituto de Ciencia de los Materiales de Aragón, Universidad de Zaragoza-CSIC, Facultad de Ciencias, Zaragoza, 50009, Spain*
[b] *Instituto de Nanociencia de Aragón, Universidad de Zaragoza, Zaragoza 50009, Spain*

deteresa@unizar.es

We use a dual-beam equipment (Nova 200 NanoLab from FEI) that integrates a 30 kV field-emission electron column and a Ga-based 30 kV ion column to prepare electron-transparent lamellae (<100 nm) out of thin films for subsequent cross-section transmission electron microscopy studies. An Omniprobe nanomanipulator is used to place the lamellae on suitable Cu TEM grids for in-situ STEM imaging under 30 keV. The STEM detector is retractable and allows bright-field, dark-field, and high-angle-annular-dark-field STEM imaging. In general, resolution below 5 nm can be obtained in these conditions. If further resolution is required, the same lamellae, already placed on TEM grids, can be straightforwardly used for HRTEM, where we have regularly obtained atomic resolution under 200 keV.

In this contribution we report, as an example, the successful fabrication of lamellae out of three different types of thin films and their in-situ STEM imaging under 30 keV. The first example is a multilayer grown by MOCVD that consists of 50 repetitions of (AsGa/$Al_{0.3}Ga_{0.7}As$) bilayers covered by a GaAs layer. This kind of multilayer, grown in Bratislava (Slovakia), is being investigated for their optical properties arising from the formed quantum-well structure [1]. As can be observed in the STEM image (figure 1), the basic AsGa and $Al_{0.3}Ga_{0.7}As$ layers have typical thicknesses of 5.6 nm and 36 nm respectively and the cover layer is much thicker, 440 nm. The images indicate that all the layers are flat and continuous in lateral distances of 1 μm. The second example is a Bi thin film grown on a Si_3N_4//Si substrate by thermal evaporation in UCM (Madrid), which we are studying for their outstanding magnetransport properties [2]. The STEM images (figure 2) indicate that the average film thickness is about 150 nm and the microstructure of the films consists of grains with lateral size of the order of 100 nm that extend from the substrate up to the film surface. The third example is an ion-beam-induced Pt deposit on a Si_3N_4//Si substrate grown in-situ by injecting the precursor gas $(CH_3)_3Pt(CpCH_3)$ in the proximity of the substrate. The Ga ion column in the voltage/current conditions 30 kV/10 pA is used to break the precursor gas and produce the local deposition of Pt. This kind of deposits is useful in several applications in Nanotechnology such as for sample protection in lamellae preparation, circuit editing, nanocontacting, etc. [3]. The STEM imaging (figure 3) indicates that a 50 nm damage-region occurs at the interface of the sample and substrate, which matches the stopping range of the Ga ions in this substrate. Also, the microstructure of the deposited film consists of small Pt grains of the order of 5 nm inside a carbonaceous matrix. Subsequent HRTEM investigation indicated the crystalline nature of the Pt grains and the amorphous nature of the matrix [3].

References:

[1] J. Novak, P. Strichovanec, I. Vavra, R. Kudela, M. Kucera, Microelectronics Journal, **37** (2006) 1515.
[2] N. Marcano, S. Sangiao, J.M. De Teresa, L. Morellon, M.R. Ibarra, M. Plaza, L. Pérez, to be published.
[3] J.M. De Teresa, R. Córdoba, A. Fernández-Pacheco, O. Montero, P. Strichovanec, M.R. Ibarra, submitted.

Figures:

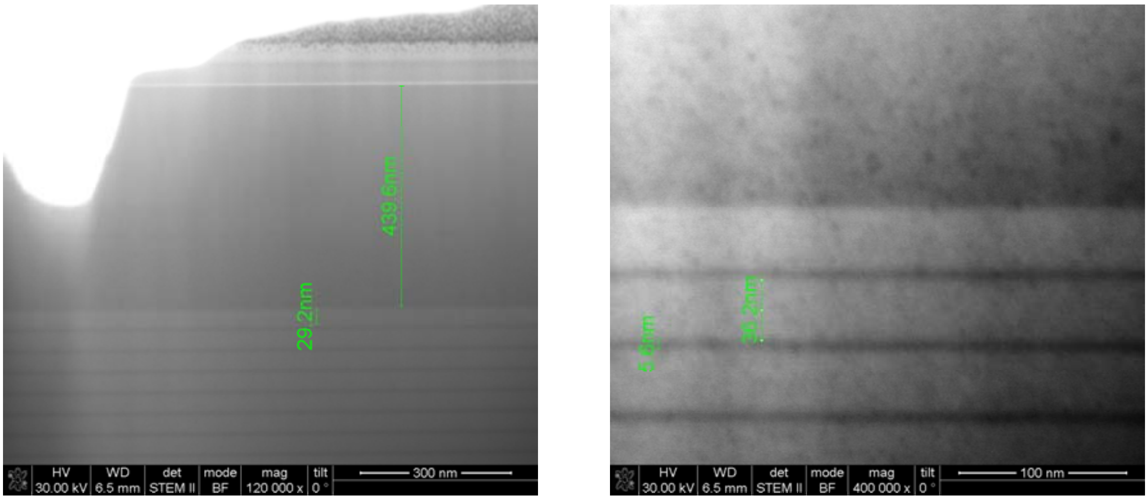

Figure 1. In-situ bright-field STEM images under 30 keV of a lamella prepared inside our dual beam equipment from a GaAs/GaAsAl multilayer grown by MOCVD.

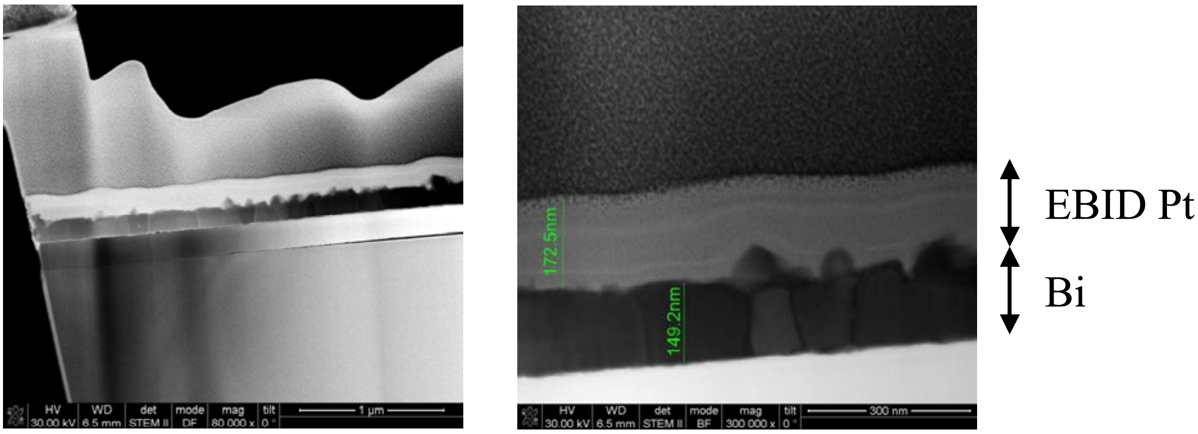

Figure 2. In-situ STEM images under 30 keV of a lamella prepared inside our dual-beam equipment from a Bi thin film grown by thermal evaporation on a Si_3N_4//Si substrate. The left picture is a dark-field STEM whereas the right one is a bright-field image.

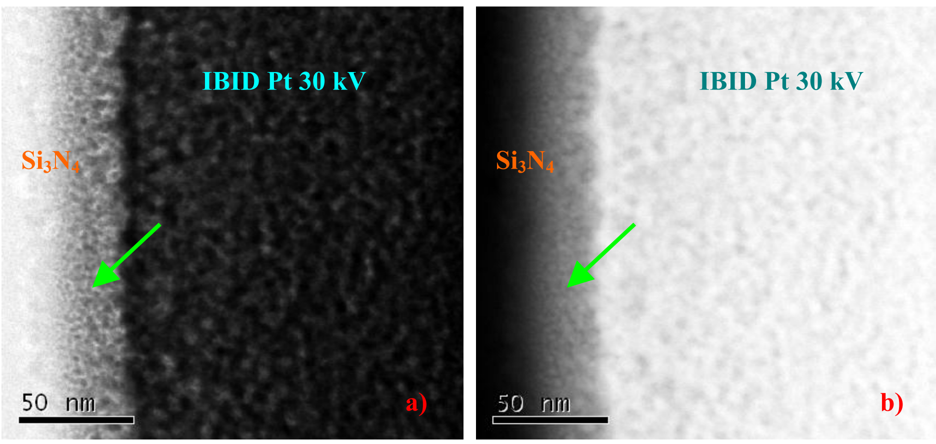

Figure 3. In-situ STEM images under 30 keV of an ion-beam-induced deposit of Pt grown on a Si_3N_4//Si substrate in our dual-beam equipment. The left picture is a bright-field image whereas the right one is a high-angle-annular-dark-field image.