TEM characterization of BN films grown on metallic substrates by CVD

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Boron nitride (BN) is identified as a strategic material for many purposes related to the integration of graphene and 2D materials in devices and the fabrication of van der Waals heterostructures. Thus, it becomes mandatory to have scalable synthesis and characterization procedures for providing suitable and reliable boron nitride material according to these three identified needs [1-3]. In this work, we are interested in the TEM characterization of boron nitride films grown on copper or nickel foil by CVD, using H_2 as a carrier gas and borazine as BN source. The growth on polycrystalline copper foil leads to few nanometer thick films, with a not highly crystallized turbostratic structure. These films exhibit a rough surface at the atomic scale, which is not suitable as a graphene substrate but can be used as capping material of a 2D crystal in a device. Conversely, highly crystalline boron nitride is synthesized on polycrystalline nickel foil. We performed a detailed analysis of the influence of nickel grain orientation on the structure of the BN layers in terms of morphology, thickness, orientation, domain size etc.[4].

To that aim, we have developed a specific wet transfer protocol from the nickel foil onto the TEM grids. We show that this polymer-free technique does not create any contamination and, preserve the integrity of the film. Therefore, we are able to correlate the BN film structure with that of the underlying Ni grains, by proceeding as follows. First mapping of the Ni grain orientations has been achieved using EBSD operated in a SEM prior to the transfer of the BN film. Second, we have combined HRTEM, STEM-HAADF imaging and electron diffraction for the characterization of the transferred BN films. We show that the layers grow differently depending on the crystalline orientation of underlying Ni grains, resulting in different morphologies as illustrated in Figure 1. For (110) and (100) Ni grain orientations, the growth follows a Volmer-Weber growth mode leading to a hill-and-valley morphology with important thickness variations, discontinuity between the islands and multiple orientation domains. For the (111) Ni orientation, BN film consists of epitaxially grown layers according to a van der Merwe layer-by-layer mechanism. We highlighted the single crystalline BN character over the whole underlying Ni grain, in consistency with the almost perfect lattice match between BN lattice and this nickel orientation [5]. On the basis of these findings, we designed synthesis conditions on Ni (111) surfaces prepared to that aim. As shown in Figure 2, HRTEM imaging and electron diffraction of cross-section samples cut by FIB reveal the uniform thickness of the film and its sharp interface with the Ni crystal. The layers are found to be highly crystalline layers and stacked according to the AB sequence. Route is now open for exploiting these films as the desired substrates in the fabrication of heterostructures and devices.

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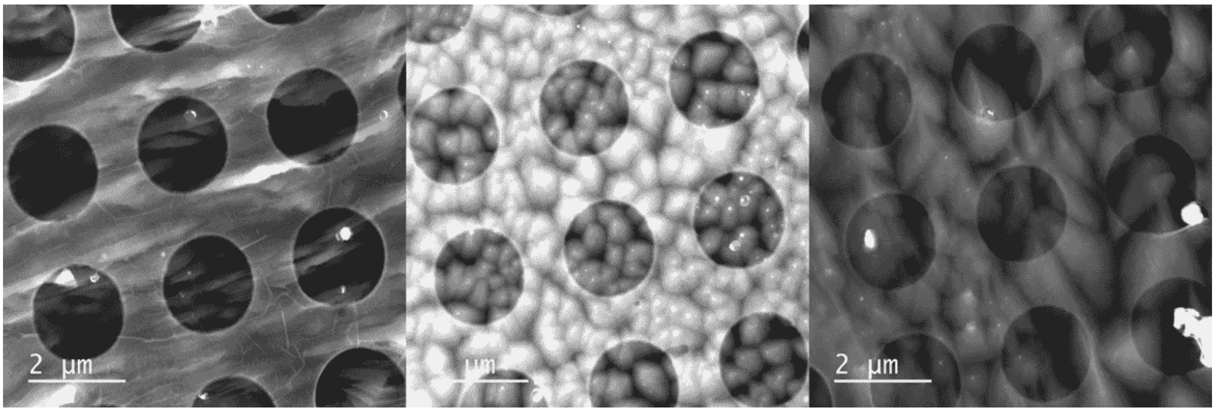

Figure 1 : HAADF image of BN deposit transferred on TEM grids. The 3 different images correspond to different morphologies of the film related to the orientation of the Ni grains.

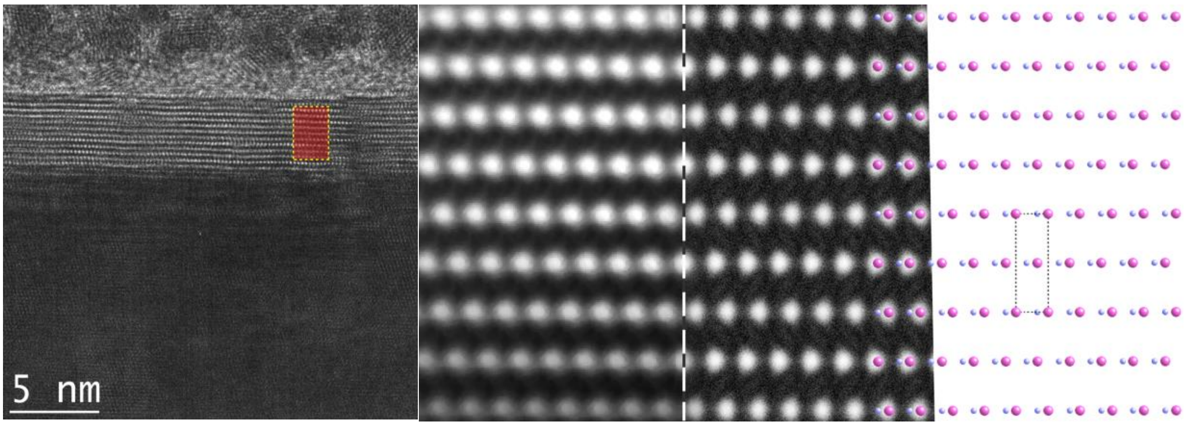

Figure 2 : (left) HRTEM image of the cross section of a BN film grown on a Ni(111) surface ; zoom on stacking sequence :(right) filtered image, simulated TEM image with JEMS and AB stacking representation