

MS24 3D electron diffraction

MS24-01

3D ED on epitaxial thin films

P. Boullay¹, **S. Passuti**¹, **A. David**¹, **E. Rauch**²

¹CRISMAT, Normandie Université, ENSICAEN, UNICAEN, CNRS - Caen (France), ²SIMAP, Grenoble INP, Université Grenoble Alpes, CNRS - Grenoble (France)

Abstract

Using strain engineering, metastable phases can be stabilized in the form of epitaxial thin films. Besides their intrinsically small diffracting volume, these films are clamped onto a thick crystalline substrate that significantly complicates their analysis by X-ray diffraction methods and usually prevents any structure determination of unknown complex phases stabilized in the form of thin films. Over the past decade, 3D Electron Diffraction (3D ED) [1] and, in particular, precession-assisted electron diffraction tomography (PEDT) has proven to be a powerful tool to address this challenge. To introduce the topic and the method, some results obtained on a series of Bismuth-based films [2,3] will be briefly outlined.

While challenging, the determination of unknown structures does actually not represent the primary need for epitaxial thin films. In most cases, the deposited materials have a known structure. The question is not to solve the structure but to know how it differs from a reference structure (bulk). In a second part, we will address the accurate structure refinement of our film based on the dynamical diffraction theory. Although this type of refinement is now well established for PEDT [4], we will be confronted here with a complication related to the microstructure of epitaxial thin films which generally resemble more polycrystalline samples with a strong texture than single crystals. The influence of the presence of oriented domains (twinned domains) on our ability to obtain relevant information about the structure of a film will be illustrated with results obtained on tilted perovskite films [5].

In the last part, we will present a 3D ED protocol adapted for a systematic analysis of epitaxial thin films with the use of scanning precession-assisted electron diffraction tomography (see 3D SPED approach already used for nanocrystalline microstructures analysis [6]). In this approach, a parallel electron nanobeam (typically less than 10nm) is scanned along the film growth direction in order to collect series of PEDT data as a function of film thickness for different tilt angles of the sample holder. This can be a line scan or an area scan which will allow us to extract relevant crystallographic information from a specific part of the film. This will be illustrated with our most recent results obtained on 35 nm thick PrVO₃ films deposited on SrTiO₃ [7].

The most recent results are obtained within the framework of the European project NanED (Electron Nanocrystallography – H2020-MSCA-ITN GA956099).

References

- [1] M. Gemmi et al., “3D Electron Diffraction: The Nanocrystallography Revolution”, ACS Central Science 5 (2019) 1315-1329.
- [2] L. Li, P. Boullay et al., “Novel Layered Supercell Structure from Bi₂AIMnO₆ for Multifunctionalities”, Nano Lett. 17 (2017) 6575-6582.
- [3] L. Li, P. Boullay et al., “Self-assembled two-dimensional layered oxide supercells with modulated layer stacking and tunable physical properties”, Materials Today Nano 6 (2019) 100037.
- [4] L. Palatinus et al., “Structure refinement using precession electron diffraction tomography and dynamical diffraction: tests on experimental data”, Acta Cryst. B 71 (2015) 740-751.
- [5] G. Steciuk, A. David et al., “Precession electron diffraction tomography on twinned crystals: application to CaTiO₃ thin films”, J. Appl. Cryst. 52 (2019) 626-636.
- [6] E. F. Rauch et al., “New Features in Crystal Orientation and Phase Mapping for Transmission Electron Microscopy”, Symmetry 13 (2021) 1675.
- [7] D. Kumar, A. David et al., “Strong Magnetic Anisotropy of Epitaxial PrVO₃ Thin Films on SrTiO₃ Substrates with Different Orientations”, ACS Appl. Mater. Interfaces 12 (2020) 356060-35613.