Poster

# Crystal structures at the nanoscale with Scanning Electron Diffraction Tomography

**P. Boullay[1], S. Passuti[1], A. David[1], E. Guilmeau[1]**

[1]*Normandie Université, ENSICAEN, UNICAEN, CNRS, CRISMAT, Caen, France*

*philippe.boullay@ensicaen.fr*

Significant advancements in precise structural analysis using electron diffraction have been made in the past decade, largely attributed to the adoption of 3D electron diffraction (3D ED) methods [1]. In this study, we investigate the constraints linked to the size of crystalline domains and the degree to which we can monitor structural alterations at the nanoscale. For this, we propose an approach that uses an appropriately sized parallel beam to collect simultaneously 3D ED patterns for different region of interest (ROI). One way to perform such an analysis is to combine a scanning procedure with precession-assisted 3D ED where the electron beam is scanned across an area with the desired step size while collecting the precession-assisted diffraction patterns. Afterwards, the sample is tilted and the scanning procedure is performed again, and so on, obtaining this way 3D ED data from a volume (Fig. 1).

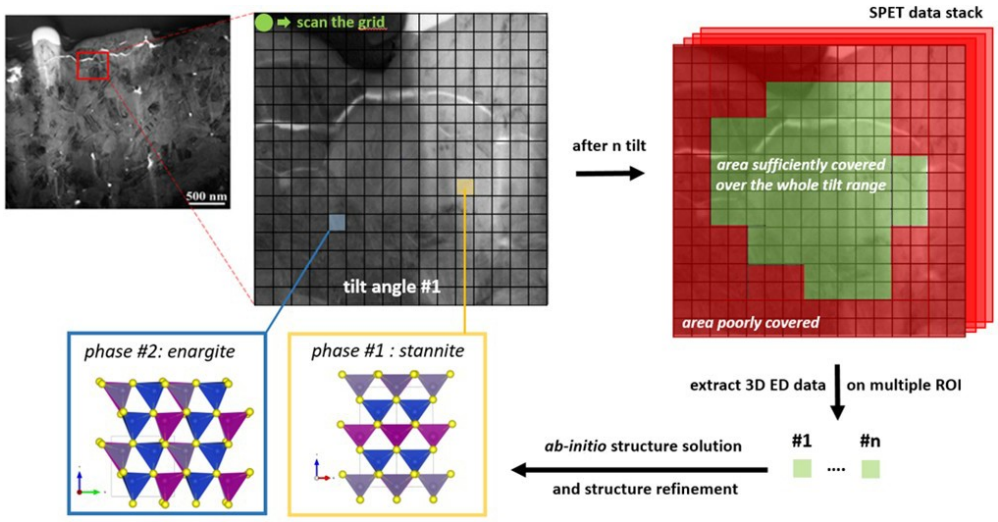


**Figure 1**. Illustration with the case of a $Cu_{2.3}Mn_{0.7}GeS_4$ nanocomposite [2], a thermoelectric material characterized by two different phases (enargite and stannite), which form interconnected domains at the nanoscale. Upper part: a scanning area is defined (here 16x16 grid), and a PED pattern is acquired at each position of the grid. The sample is rotated (tilt angle) and the scanning is done again. Even if the grid is not placed exactly at the same position, some parts of the initial area shall be scanned. Lower part: after the tilting series is obtained, multiple ROI can be extracted. Each ROI consist of a 3D ED dataset that can be used to obtain accurate structure analysis.

Eggeman et al. [3] actually exploited a similar approach for analysing the volume and orientation of domains in crystalline Ni-based superalloys relying on the comparison of the acquired diffraction patterns with templates simulated from a reference structure. We extend here the use of so-called Scanning Precession Electron Tomography (SPET) for the ab-initio structure solution and accurate structure refinement of nanodomains. This implies that after the acquisition, we are able to distinguish, and sort accordingly, the diffraction patterns generated by different ROI in the sample. We demonstrated the usefulness of SPET for accurate structural characterizations of subtle structural changes in a 35 nm thick $PrVO_3$ thin film using a line scan in a direction perpendicular to the film/substrate interface [4]. In this contribution, we take a step forward employing it for the analysis of nanodomains embedded in ceramics (Fig. 1) using an area scan and show how it is possible to extract 3D ED data corresponding to single domains/ROI from the SPET data stack.

[1] Gemmi, M. et al. ACS Central Science 5 (2019) 1315.

[2] Pavan Kumar, V. et al. Angewandte Chemie International Edition 61 (2022) e202210600.

[3] Eggeman, A. S., Krakow, R., & Midgley, P. A. Nature communications 6 (2015) 7267.

[4] Passuti, S. et al. Symmetry 15 (2023) 1459.

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