Oral presentation

Structural analysis of functional materials through Scanning Precession Electron Tomography (SPET)

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Structural analysis of functional materials is the fundamental step towards the ability of tuning their physical properties as desired. For crystalline materials, 3D ED has been proven to be an effective TEM technique to perform ab initio structure solution and refinements of crystals with size well below 1µm [1]. As the crystal or domain size reduces, the capacity to collect 3D ED data with smaller electron beams is the key to access the structure of individual embedded nanodomains. Tracking the area of interest becomes then a major issue. To circumvent this problem, we considered an alternative approach of acquiring 3D ED data by combining PEDT with a scan across the sample for each tilt angle. SPET (Scanning Precession Electron Tomography) has already been used for analysing the domains volume and orientation in known crystalline materials [2-3], relying, as 4D-STEM, on the comparison of the acquired diffraction patterns with theoretical ones taken from a reference database.

In the present work, SPET is used to acquire PEDT data over an area, extract the intensities collected at different positions and used them to obtain structure solution and accurate structure refinements, making it suitable to characterize unknown samples as well. As of proof-of-concept, we performed a SPET experiment on a 35 nm thick perovskite $PrVO_3$ film deposited on $SrTiO_3$ substrate using a probe size of about 10 nm [4]. This way, it was possible to analyse small changes in the PVO structure along the film thickness from the variation of unit cell parameters to accurate atomic positions and tilt amplitude of the VO_6 octahedra (Fig. 1).

This TEM data collection protocol allows to get an overall view of the fine crystal structure of the sample, whether we are interested to characterize a nanodomain embedded in a matrix (ceramics, planetary materials, …) or the fine evolution of the structure inside a single domain (thin films). As it will be shown in this contribution, automatizing the diffraction pattern sorting and data processing can significantly speed up the analysis. We believe that SPET has the potential to become the standard procedure to characterize unknown films or embedded domains as small as 10 nm.

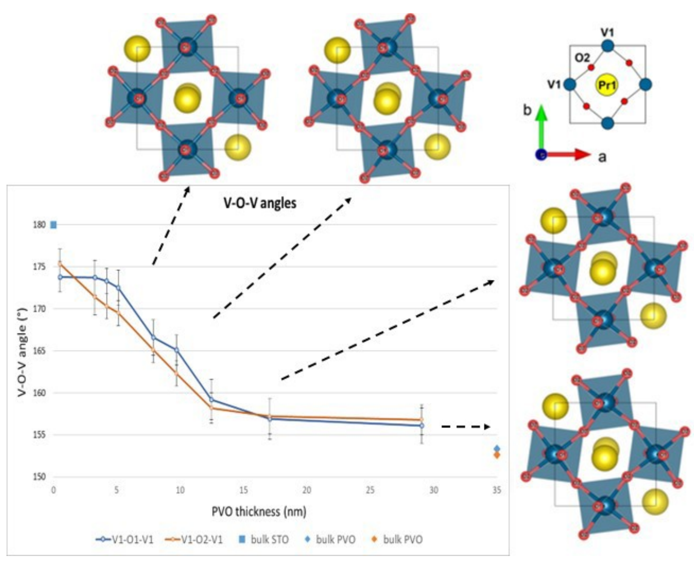

Figure 1. Evolution of the VO_6 octahedra tilting in PVO thin film obtained from dynamical refinements, going progressively further away from the film-substrate interface

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[2] Eggeman, A. S et al. Scanning precession electron tomography for three-dimensional nanoscale orientation imaging and crystallographic analysis. Nature communications 6(1) (2015) 7267.
[3] E. F. Rauch et al., New Features in Crystal Orientation and Phase Mapping for Transmission Electron Microscopy, Symmetry 13 (2021) 1675.
[4] Passuti, Sara, et al. "Scanning Precession Electron Tomography (SPET) for Structural Analysis of Thin Films along Their Thickness." Symmetry 15.7 (2023): 1459.

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