Electron crystallography of nanodomains in functional materials

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Abstract

The investigation of functional materials has increasingly focused on samples characterized by nanodomains (ranging from submicron sizes to tens of nanometers) due to their interesting physical properties, such as those observed in thin films and ceramic materials. When unknown phases need to be determined or detailed information on the crystalline structure of these materials is required, this presents challenges for both X-ray diffraction and transmission electron microscopy (TEM). To address this, a novel electron diffraction (ED) technique, Scanning Precession Electron Tomography (SPET), has been employed. SPET combines the established precession-assisted 3D ED data acquisition method (a.k.a. Precession Electron Diffraction Tomography – PEDT) with a scan of the electron beam on a region of interest (ROI) of the specimen at each tilt step. This procedure allows to collect 3D ED data from multiple ROIs with a single acquisition, facilitating structure solution and accurate structure refinements of multiple nanodomains or distinct areas within a single domain, at once. In this thesis, the potentialities of SPET are explored on both oxide thin films and ceramic thermoelectric materials prepared as TEM lamellae. Additionally, a novel methodology was developed to efficiently analyze the large amount of data collected. This method involves sorting the diffraction patterns according to their region of origin, reconstructing the diffraction tilt series of the ROI, and automatically processing the obtained tilt series for structure solution and accurate refinements. This work demonstrates the potential of SPET for the fine crystallographic characterization of complex nanostructured materials. This approach appears to be complementary to what can be done in imaging or spectroscopy by (S)TEM or, in diffraction, by the so-called 4D-STEM and ACOM approaches.