Poster

Applications of precession-assisted scanned nanobeam diffraction tomography to sample metrology over large ROIs

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Electron diffraction experiments, such as 3D electron diffraction (3D ED) [1] and 4D-STEM, provide structural information on materials at the atomic level. The new dedicated scanning transmission electron diffraction microscope TESCAN TENSOR provides rapid 4D-STEM acquisition and processing that can be uniquely combined with precession-assisted 3D ED tomography [2] to obtain and characterize structural information in crystalline and polycrystalline samples. To illustrate the unique capabilities of TENSOR, we show two techniques for combining 3D ED and per-tilt 4D-STEM to provide structural information that is challenging to acquire with standard diffraction techniques and methods.

First, we demonstrate how different phases and orientations of grains may be mapped across a polycrystalline lamella using nanoscale 3DED combined with 4D-STEM data acquisition.

Stannite- and enargite-type phases in a copper-rich sulfide ceramic are investigated as a potential thermoelectric energy harvesting material [3]. The elongated grains are shown as planar defect bands in the STEM image (Fig. 1a). A selected region of interest containing both phases was first scanned per tilt step using a parallel beam over a coarse grid. Individual 3D ED tilt series were then extracted and used for structure refinement of the two phases. Subsequently, by using a fine scanning grid at a single tilt angle, precession-assisted 4D-STEM was used to determine the distribution of phases, including their orientations, in the same ROI. This identification was based on template matching [4] generated directly from the crystal structure identified by 3D ED.*[5]

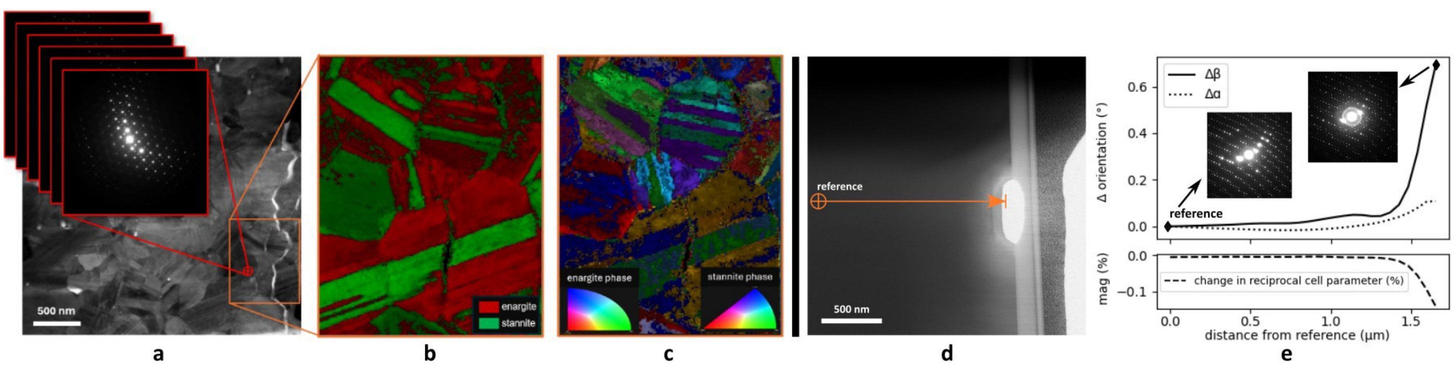

Figure 1. (a) STEM image of the $Cu_{2.3}Mn_{0.7}GeS_4$ nanocomposite. Precession-assisted 4D-STEM tomography was used to obtain the 3D ED data for crystallographic structure analysis used for (b) phase and (c) orientation maps. (d) STEM image of line scan region used for orientation analysis shown in (e) as change in angles Δα (about x-axis) and Δβ (about y-axis) and change in unit cell size.

Second, we show how the combination of scanned diffraction acquisition and 3D ED can be used for determination of lamella bending and strain relaxation. For every 1° tilt step in a series of 60°, a line scan of a known homogenous structure, Lutetium Aluminum Garnet (LuAG), was acquired in a thinned lamella region. The initial crystal orientation was determined from the first pixel in the scan and used to define a reference. The STEM image (Fig. 1d) shows the line acquisition region near the thinned feature of a crystalline lamella sample. Using the tomographic reconstruction of the unit cell for each subsequent pixel in the scan, the lamella bending in this region was measured with greater accuracy than would be possible with qualitative observation of bending contours or template matching.

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*[5] These results were obtained as part of the European project NanED (Electron Nanocrystallography – H2020-MSCA-ITN GA956099).