Microscopy and Microanalysis, **29** (Suppl 1), 2023, 715–716
https://doi.org/10.1093/micmic/ozad067.352

Microscopy AND Microanalysis

Meeting-report

Imaging Structural Phase Transitions with Higher Order Laue Zones Using 4D-STEM

Magnus Nord[1,2,3,*], Giulio Guzzinati[1,2], Gertjan Koster[4], Nicolas Gauquelin[1,2], and Johan Verbeeck[1,2]

[1]*EMAT, University of Antwerp, Antwerp, Belgium*
[2]*NANOlab Center of Excellence, University of Antwerp, Antwerp, Belgium*
[3]*Department of Physics, Norwegian University of Science and Technology, Trondheim, Norway*
[4]*MESA+ Institute of Nanotechnology, University of Twente, Enschede, The Netherlands*

*Corresponding author: magnus.nord@ntnu.no

Structural phase transitions are an important aspect of functional materials due to the strong coupling between the functional properties and crystal structure. Transmission electron microscopy (TEM) is a powerful technique used to examine materials all the way down to single atomic columns. Studying the structure of materials at temperatures from room temperature to above 1000 degrees Celsius with TEM is today routine, thanks to improvements in chip-based in-situ TEM-holders. Studying the structural changes can be done in several ways, from conventional TEM (cTEM), electron diffraction (ED) or atomic resolution STEM. Due to advances in fast pixelated electron detectors it is now possible to acquire a STEM diffraction pattern for each probe position in the scan, yielding a 4-dimensional dataset (4D-STEM). This gives highly detailed information about the crystal structure at the nanoscale, but also gives very large and complex data.

In this work, we utilize higher order Laue zones (HOLZ) [1] to study nanoscale structural phase transitions using 4D-STEM. The studied material is VO_2 films deposited on in-situ heating chips [2]. At room temperature, the diffraction pattern has an "extra" HOLZ ring (Fig. 1a) compared to the high temperature diffraction pattern (Fig. 1b). The appearance and disappearance of the HOLZ ring is a signature of a transition from one crystal structure to another, as the HOLZ is sensitive to the periodicity parallel to the electron beam.

The presentation will focus on the data processing steps for ascertaining if the material is in the low or high temperature structural phase. This is made difficult by the textured nature of the film, meaning the film is arranged in grains with different crystal orientations, resulting in the HOLZ "ring" becoming non-circular. This necessitates a combination of template matching, peak finding and clustering as shown in Fig. 2. This is implemented using the open source Python libraries HyperSpy and pyXem to handle the very large data sizes [3].

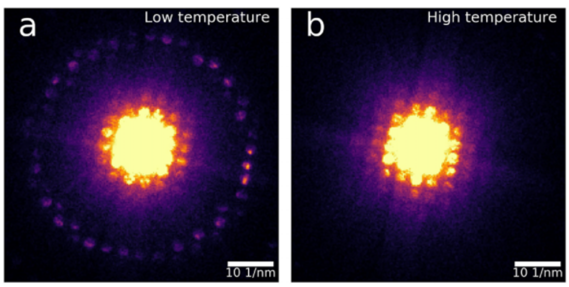

Fig. 1. 4D-STEM diffraction image from the a) low temperature and b) high temperature phase, showing the "extra" higher order Laue zone (HOLZ) ring in the low temperature phase.

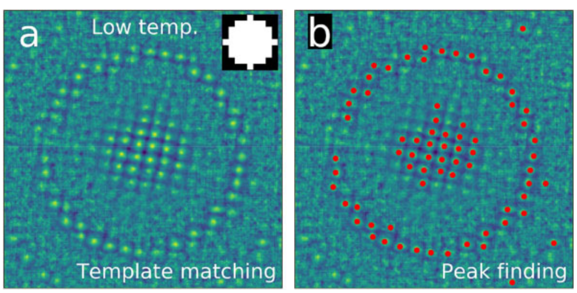

Fig. 2. Data processing steps for finding the presence of the "extra" HOLZ ring, which is made difficult by large changes in intensity and movement of the HOLZ ring: a) template matching with a binary disk, b) peak finding of the "template matched" image.

References

1. PTP Le *et al.*, *Adv. Funct. Mater.* **30** (2020). doi:10.1002/adfm.201900028
2. JCH Spence *et al.*, *Ultramicroscopy* **31** (1989). doi:10.1016/0304-3991(89)90218-0
3. We acknowledge funding for the direct electron detector used in the 4D-STEM studies from the Hercules fund 'Direct electron detector for soft matter TEM' from the Flemish Government. MN was partly funded in this work via the Research Council of Norway grant number (315475). MN was also partly funded via the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 838001. Dr. Y.A. Birkhölzer, Dr. T.P.P. Le, Dr. K. Sotthewes and Prof. J.E. Elshof is acknowledged for providing the sample material used in this study.