MS27-O5 Improving rotation electron diffraction data quality by data merging

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Automated three-dimensional electron diffraction methods, for example automated diffraction tomography (ADT)[1] and rotation electron diffraction (RED) [2-3], can be used to collect and process completed 3D electron diffraction data from nanometer sized crystals and have been very successful in application of solving complex structures. However, limited crystal tilting range and steps, electron beam damage and dynamical scattering of electrons degrade the RED data quality and result in high R values and inaccurate structural parameters from refinement. Often structure models obtained from RED data are refined against powder X-ray diffraction data for confirmation and better accuracy.

Here we propose an approach for improving RED data quality through merging multiple datasets collected from different crystals with different orientations. Individual datasets are first processed using RED following the standard procedure. The initial model for the refinement is obtained by direct methods using SHELXS. The HKL lists from the individual datasets are merged together for refinement with the initial model using SHELXL. To study the effects of merging datasets on data quality, we performed refinement by sequentially including the individual datasets from a test sample. We show that the number of observed reflections increases and the R values decrease while more datasets are merged. The structural parameters after refinement are improved by merging more datasets. The apparent improved data quality may be attributed partly to the increased data completeness and resolution, and reduced effedcts of dynamic effects through averaging. These factors will be examined carefully to evaluate their potential in improving RED data.

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[2] D.L. Zhang, et al, Z. Kristallogr. 225 (2010) 94
[3] W. Wan et al, J. Appl. Crystallogr. 46 (2013) 1863

Keywords: RED, electron diffraction, data quality

MS28 New approaches in electron crystallography

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MS28-O1 When Precession Electron Diffraction Tomography goes dynamical

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The use of electron diffraction (ED) data for the refinement of crystal structures as routinely as what is done in X-ray diffraction (XRD) has long appeared to be inaccessible. While the strong electron/mater interaction allows studying crystals of nanometer size, the kinematic approximation is not valid, which makes the analysis of the diffracted intensity more complex than for XRD. More complex, but not impossible, as the formalism to describe dynamic conditions in ED is known since long. However, its use by programs aiming structural refinements using ED patterns obtained from parallel beam met no success despite several attempts. [1]

In 2013 [2], L. Palatinus and co-authors have studied the feasibility of refining crystal structures from few zone axis patterns considering the dynamic conditions of diffraction, and shown, the importance of Precession Electron Diffraction (PED) [4] to get better convergence. Early in 2015, the Prague group reached a decisive step by implementing in JANA2006 a module dedicated to ED and such “dynamical refinements” [5]. Integrated in an interface familiar to most structural crystallographers, this module now allows to process PED data collected in a “tomography mode” [6] aiming to reach a high level of data completeness. Several groups across Europe have joined forces to test and validate this new approach and offer the community a reliable and functional tool. [7]

From structure solution to structure refinement (kinematical then dynamical), Precession Electron Diffraction Tomography (PEDT) is a mature technique readily to be used in any materials science laboratory. In the present communication, we will show what astonishing results can be achieved and what are the limitations using examples from incommensurate

modulated structures, thin films and zeo-type materials. No doubt that combining PEDT data and "dynamical refinements" will allow to solve and refine an increasing number of structures on materials inaccessible by other diffraction techniques.

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[5] V. Petricek et al., Z. Kristallgr. **229** (2014) 345.

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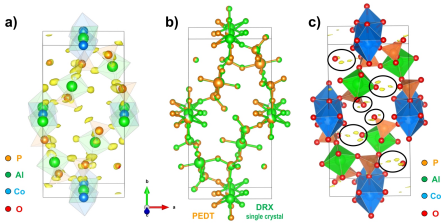

Figure 1. Co-AlPO zeo-type material: a) density map from PEDT data, b) structure from single crystal XRD (green) and PEDT "dynamical" (orange), c) residues in the Fourier difference map after dynamical refinement of PEDT data. Residues inside black circles would correspond to hydrogens.

Keywords: dynamical refinement, incommensurate modulated structures, thin films, zeo-type materials

MS28-O2 MicroED: Three Dimensional Electron Diffraction of Microscopic Crystals

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My laboratory studies the structures of membrane proteins that are important in maintaining homeostasis in the brain. Understanding structure (and hence function) requires scientists to build an atomic resolution map of every atom in the protein of interest, that is, an atomic structural model of the protein of interest captured in various functional states. In 2013 we unveiled the method MicroED, electron diffraction of microscopic crystals, and demonstrated that it is feasible to determine high-resolution protein structures by electron crystallography of three-dimensional crystals in an electron cryo-microscope (CryoEM). The CryoEM is used in diffraction mode for structural analysis of proteins of interest using vanishingly small crystals. The crystals are often a billion times smaller in volume than what is normally used for other structural biology methods like x-ray crystallography. In this seminar I will describe the basics of this method, from concept to data collection, analysis and structure determination, and illustrate how samples that were previously unattainable can now be studied by MicroED. I will conclude by highlighting how this new method is helping us understand major brain diseases like Parkinson's disease.

Keywords: MicroED; CryoEM; Electron Diffraction; Crystallography; nanocrystals