MS20-O2 **Structure refinement against precession electron diffraction data.** Lukas Palatinus,[a] Mariana Klementova,[a] Damien Jacob,[b] Priscille Cuvillier,[b] Wharton Sinkler [c] and Laurence Marks,[d] [a]*Institute of Physics of the AS CR, Prague, Czechia,* [b]*Université Lille, CNRS UMR 8207, Lille, France,* [c]*UOP LLC, Des Plaines IL, USA,* [c]*Northwestern University, Evanston IL, USA*
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Diffraction remains the most frequently used tool for probing atomic structure of crystalline materials. Electron diffraction, in particular, has the advantage that, using transmission electron microscope, crystals as small as a few nanometres can be investigated. There have been many attempts to use electron diffraction as a quantitative tool for crystal structure analysis, dating back to the earliest days [1,2,3]. However, mathematical treatment of electron diffraction is much more complicated than for x-ray or neutron diffraction. The treatment requires the use of dynamical diffraction theory, making a full quantitative analysis difficult. There has recently been a resurgence of interest in quantitative analysis of high-energy electron diffraction data due to the introduction of the precession electron diffraction (PED) technique [4]. The method is now commonly used for *ab initio* crystal structure solution. We demonstrate that precession electron diffraction data can be also successfully used for accurate structure refinement, provided that full dynamical treatment is applied. We have tested the method on three samples: a simple structure of silicon, mineral orthopyroxene (10 independent atoms, V_{UC} = 843 Å^3) and gallium-indium tin oxide (17 independent atoms, V_{UC} = 392 Å^3). The thickness of the samples varies from 30 nm to 110 nm. The structures are refined using full dynamical refinement, and, for comparison, using a simplified two-beam dynamical refinement [5] and the refinement in kinematical approximation.

For all three samples the method yielded stable refinements with acceptable refined parameters and reasonable figures of merit, which were in all cases much lower than the corresponding figures of merit obtained with two-beam or kinematical refinement. In particular, the full dynamical treatment allowed the refinement of atom occupancies at mixed crystallographic sites – an impossible achievement with kinematical approximation. The refined parameters are in a good agreement with the reference structures refined against x-ray diffraction data.

[1] Cowley, J. M. (1953). *Acta Cryst.* **6**, 516-521.
[2] Vainshtein, B. K. (1964). Structure analysis by electron diffraction, *Pergamon Press*.
[3] Spence, J. C. H. & Zuo, J. M. (1992). Electron microdiffraction, *Springer*.
[4] Vincent, R. & Midgley, P. A. (1993). *Ultramicroscopy* **53**, 271-282.
[5] Sinkler, W., Own, C. S. & Marks, L. D. (2007). *Ultramicroscopy* **107**, 543-550.

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MS20-O3 **Solving structures of sub-micron crystals by rotation electron diffraction**, Sven Hovmöller, Xiaodong Zoi, Yifeng Yun, Wan Wei, Faiz Rabbani, *MMK, Stockholm University, Sweden*
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Many compounds have interesting and important structures, yet they cannot be solved because crystals large enough for X-ray diffraction cannot be grown. Recent developments [1,2] within electron crystallography provides a solution: Complete 3D single crystal diffraction data can be collected in a transmission electron microscope, even from crystals as small as 10-100 nm, i.e. a million times smaller than what is needed on a microbeam synchrotron. Several complicated structures including zeolites and metal oxides have been solved by the Rotation Electron Diffraction (RED) method. Diffraction patterns of a tiny crystal of random orientation are collected at 0.1° intervals in a range of about 140°. It takes just over one hour to collect 1400 electron diffraction patterns. After a further hour of data processing, all the diffraction points to 1.0Å or higher are localized and indexed. The unit cell is found and the space group determined from extinction conditions. The data is of sufficient quality to allow solving by standard techniques, such as direct methods. In one case a sample of metal oxide(s) with a very complicated X-ray powder diffraction pattern was studied by RED. The sub-micrometer sized crystals showed different shapes in the EM. Three crystals with different shapes were studied by RED. The crystals had quite different unit cell dimensions and turned out to be of three different compounds. All three structures were solved from the RED data. The X-ray powder pattern contained peaks from all these three compounds, and could now be indexed. RED is a very powerful new tool for structure determination, especially for very small crystals.

[1] Zhang, D.L., Hovmöller, S., Oleynikov, P. and Zou, X.D. Collecting 3D electron diffraction data by the rotation method. Z. Kristallogr. **225** (2010) 94–102
[2] Jiang, J., Jorda, J.L., Yu, J., Baumes, L.A., Mugnaioli, E, Diaz-Cabanas, M, Kolb, U. and Corma,A. (2011) Science **333**, 1131-1134.

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