MS13-O3 Accurate structure refinement from electron diffraction tomography data

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Structure analysis by electron diffraction has made an enormous progress in the last decade. From a status of a method deemed by many unsuitable for ab initio structure solution and very complicated for refinement it turned into an accepted tool of structure analysis, which can be used almost routinely to solve unknown structures. This is especially true for inorganic materials, which, in general, suffer less radiation damage under the electron beam than organic or organometallic compounds.

This progress was caused by the introduction of the electron diffraction tomography method (EDT) [1]. This method allows collecting complete diffraction information from a large part of reciprocal space. Such data can be processed in a way similar to single crystal x-ray data collection, and then used for structure solution and refinement. Unfortunately, the strong interaction between electrons and matter cause that the kinematical theory of diffraction is not valid for electrons to sufficient accuracy, the consequence being that the structures refined against EDT data lack accuracy and statistical reliability. The problem can be partially remedied by the combination of the EDT method with precession electron diffraction [2], but even then the dynamical character of diffraction has negative impact on the accuracy of the refinement results.

A solution to this problem is the method, which uses for structure refinement the full dynamical diffraction theory. Such method was developed recently [1] and implemented in the crystallographic computing system Jana2006 [2]. The method was tested on a range of inorganic materials. The comparison of the refined structures with the reference single crystal x-ray structures shows that the average distance between corresponding atoms in the refined and reference structure is typically less than 0.02 Å. With this accuracy, the method can be claimed to be comparable, if not superior to the Rietveld refinement from powder diffraction data, and allows an accurate single-crystal analysis from a single nano-sized crystal.

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MS13-O4 Serial snapshot crystallography for materials science with SwissFEL

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With the development of X-ray free-electron laser (X-FEL) sources that create ultra-fast X-ray pulses of unprecedented brilliance, a new option for the structural characterization of micro-crystalline inorganic materials is arising [1]. The SwissFEL facility (PSI, Switzerland), a new X-FEL source, is scheduled to come online in 2017 [2]. Any crystal placed in the extremely intense SwissFEL beam will be destroyed, but not before a diffraction pattern is generated. To get a full data set, therefore, many randomly oriented stationary crystals will have to be measured. One of the unique features of the SwissFEL facility will be that the bandwidth of the X-ray beam can be adjusted to give as much as a 4% wavelength spread. To evaluate the possibility of using the full energy range of the SwissFEL beam, we first simulated data for inorganic crystals. With the 4%-energy-bandpass mode, not only can more reflections be recorded per shot, but the intensities will also be measured more reliably [3]. To test the viability of this approach experimentally, the single-crystal diffractometer on the Swiss-Norwegian Beamlines at the ESRF was used to mimic the SwissFEL setup. The broad bandpass mode was achieved by collecting a diffraction pattern while the monochromator was scanned over a 4% energy range. Three test samples, with unit cells typical of small-molecule and inorganic structures were used: the zeolite ZSM-5, a hydrated cesium cyanoplatinate, and the mineral sanidine. In order to index the single-shot patterns of randomly oriented crystals of such materials, we have developed two indexing algorithms, one using Laue diffraction concepts and the other starting with a monochromatic approximation [4]. Both algorithms were optimized to index several orientations from a single pattern. We show that the individual patterns of up to 10 crystals measured simultaneously can be indexed, and the intensities extracted reliably for structure investigation. Even with a single shot, at least a partial analysis of the crystal structure will be possible, and this offers tantalizing possibilites for time-resolved studies. [1] H. N. Chapman, et al., *Nature*, 2011, **470,** 73–77. [2] B. D. Patterson, et al., *Chimia*, 2014, **68,** 73-78. [3] C. Dejoie, et al., *J. Appl. Cryst., 2013,* **46**, 791-794. [4] C. Dejoie, et al., *IUCrJ*, 2015, accepted.

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