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Quasicrystal approximants solved by Rotation Electron Diffraction (RED)

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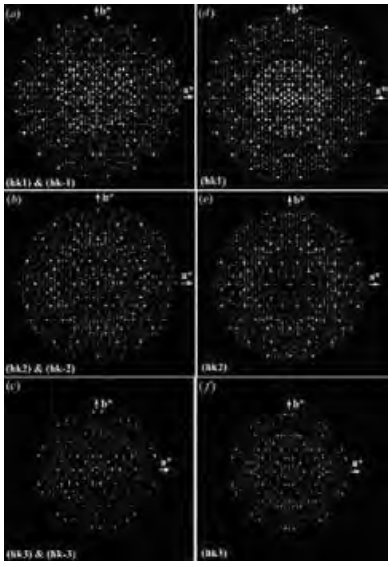
We have developed single crystal electron diffraction for powder-sized samples, i.e. < 0.1µm in all dimensions. Complete 3D electron diffraction is collected by Rotation Electron Diffraction (RED) in about one hour. Data processing takes another hour. The crystal structures are solved by standard crystallographic techniques. X-ray crystallography requires crystals several micrometers big. For nanometer sized crystals, electron diffraction and electron microscopy (EM) are the only possibilities. Modern transmission EMs are equipped with the two things that are necessary for turning them into automatic single crystal diffractometers; they have CCD cameras and all lenses and the sample stage are computer-controlled. Two methods have been developed for collecting complete (except for a missing cone) 3D electron diffraction data; the Rotation Electron Diffraction (RED) [1] and Automated Electron Diffraction Tomography (ADT) by Kolb et al. [2]. Because of the very strong interaction between electrons and matter, an electron diffraction pattern with visible spots is obtained in one second from a submicron sized crystal in the EM. By collecting 1000-2000 electron diffraction patterns, a complete 3D data set is obtained. The geometry in RED is analogous to the rotation method in X-ray crystallography; the sample is rotated continuously along one rotation axis. The data processing results in a list of typically over 1000 reflections with h,k,l and Intensity. The unit cell is typically obtained correctly to within 1%. Space group determination is done as in X-ray crystallography from systematically absent reflections, but special care must be taken because occasionally multiple electron diffraction can give rise to very strong forbidden reflections. At +/-60º tilt with 0.1º steps, a complete data collection will be some 1200 frames. With one second exposures this takes about one hour. There is no need to align the crystal orientation. The reciprocal lattice can be rotated and displayed at any direction of view. Sections such as hk0, hk1, hk2, h0l and so on can easily be cut out and displayed. We have solved over 50 crystal structures by RED in one year. These include the most complex zeolites ever solved and quasicrystal approximants, such as the pseudo-decagonal approximants PD2 [3] and PD1 in AlCoNi. Observed and calculated sections of reciprocal space (cut at 1.0Å) are shown in Fig. 1. Notice the 10-fold symmetry of strong reflections.

[1] *W. Wan, J. Sun, J. Su, S. Hovmöller and X.D. Zou Three-dimensional rotation electron diffraction: software RED for automated data collection and data processing J. Appl. Cryst. (2013). 46, 1863-1873.,* ***[2]*** *Kolb, U. Gorelik, T. Kübel,C., Otten,M.T. and Hubert, D. Ultramicroscopy 107 (2007) 507–513.,* ***[3]*** *Devinder Singh, Yifeng Yun, Wei Wan, Benjamin Grushko,Xiaodong Zou and Sven Hovmöller; A complex pseudo-decagonal quasicrystal approximant, Al37(Co,Ni)15.5, solved by rotation electron diffraction J Appl Cryst . (2014). 47, 215–221.*



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