The use of nanodiffraction for the structure analysis of beam sensitive material

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Keywords: electron diffraction, computer simulation, structure analysis

Electron crystallography is sometimes the only path to analyse the crystal structure of nanocrystalline small organic molecules or even polymeric compounds where x-ray powder diffractograms deliver only poor data. Unfortunately, these materials are often sensitive to irradiation by electrons. Data for structural analysis is normally taken via several tilts of an appropriate crystal around selected axes [1]. The more effort is used to reduce the beam damage on the sample the higher is the chance to collect a big and reliable 3D data set. Dependent on the habit of the crystals sample holders with different flexibility (rotation-tilt or double-tilt rotation) can be used. In the case of nanocrystalline organic samples nanodiffraction turned out to be a gentle way to sample the reciprocal space. Data for small organic molecules, such as pigments, have been collected by transmission electron microscopy at 300 kV with a FEI Tecnai F30 ST. The use of a field emission gun allowed a nearly parallel illumination with a beam diameter of approx. 10-20 nm. Unit cell parameters have been obtained through tilt series and were subsequently refined by x-ray powder data if available [2]. In a second step data for quantitative structure analysis has been collected. After the cell parameters are determined it is possible to index the zones on-line and to use a cryo holder in order to reduce the beam damage further. Different from x-ray powder diffraction there is no automated routine developed to collect electron diffraction data and to process respectively analyse the collected data in advance. To build up the models for a start conformation of the molecules single crystal x-ray data were taken from CSD and partial charges have been calculated by ESP from semi-empirical approach using MOPAC 6.0 [3]. Structure analysis has been performed by stepwise simulation of the available diffraction data taking only kinematic diffraction into account. For kinematical approach R-values of approx. 20-30% can be achieved, whereas the application of dynamical effects including the refinement of the centre of Laue circle and the crystal thickness leads to values about 5% [4].

[1] U. Kolb and G. Matveeva, Electron crystallography on polymorphic systems, *Z. Krist*., **218**, 259-268, 2003.
[2] Cerius 2 version 4.2 MS, Molecular modeling environment from Accelrys Inc., 9685 Scranton Road, San Diego, CA 92121 – 3752, USA.
[3] CSD – Cambridge structural database, CCDC Software Ltd., Cambridge, GB; J.J.P. Stewart, MOPAC6.0 A General Purpose Molecular Orbital Package, QCPE.
[4] Jansen, J.; Tang, D.; Zandbergen, H.W. and Schenk, H.: MSLS, a least-squares procedure for accurate crystal structure refinement from dynamical electron diffraction patterns. *Acta Cryst.* 1998, **A54**, 91-101.