s4.m13.o1 **Advances and Accuracy Considerations on Crystal Structure Refinement using highly Dynamical Electron Diffraction Data generated with a Parallel Electron Beam.** Jacob Jansen, *Nationaal Centrum voor HREM, Technische Universiteit Delft, Rotterdamseweg 137, 2628 AL Delft, Nederland. E-mail: joukj@hrem.stm.tudelft.nl*

Keywords: Structure Refinement; Electron Diffraction; Dynamic Diffraction

Crystal structure refinements using electron diffraction data, obtained by using a parallel electron beam, gives accurate results if a full dynamical calculation is performed to calculate the elastic diffracted intensities from the atomic model[1]. Apart from the dynamical diffraction it is of utmost importance to take even a slight tilt from the zone axis and the crystal thickness into account during calculation. Our refinement program MSLS, based on the Multi-Slice algorithm, can cope with all those geometrical constraints. Since only a tiny crystal is used (typically 100x100x100Å), the method is extremely powerful in the area of small precipitates, thin layers and multi-phased samples. MSLS has been applied successfully to any type of material whether it being inorganic (i.e. superconductors), metallic or organic compounds. Comparison between the calculated and observed intensities is usually measured in terms of R-values, whose values appear to be of the same order of magnitude as when single crystal X-ray diffraction would have been used. The accuracy of the method is mainly determined by the accuracy with which the calculations of the intensities of the diffracted beams can be performed. It is known that the calculation method (i.e. Multi-slice as in MSLS, Bloch waves) does not have a significant effect on the resulting intensities. However, all these methods start from the same atomic scattering factors. Currently, for simulations of electron diffraction patterns and HREM images, the Doyle and Turner table[2] is most frequently used. Several other, more recent, tables are available in literature [3-7]. In addition some authors modified the atomic scattering factors in order to include an approximation for absorption of the electrons in the crystal[8,9]. Changing from one to the other table gives some changes to the resulting crystal structure obtained by MSLS Analysis of the refinement results using data from different compounds indicates that the frequently used scattering factors of Doyle and Turner are not the best choice. The absorptive scattering factors are not a good choice either since the background subtraction during the data reduction takes care of the main part of the problems they try to cope with. Further improvement of the calculated intensities can be obtained by including HOLZ reflections by the method developed by Chen [10] . Patterns containing HOLZ circles can also be used to have a rather accurate guess of the crystal tilt, which can be used as a starting point for further refinement.

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[10] J.H.Chen *et al.* (1995) *Phys. Stat.sol (a)* **150**, 13

s4.m13.o2 **The Use of Nanodiffraction for the Structure Analysis of Beam Sensitive Material.** U. Kolb and T. Gorelik, *Institute of Physical Chemistry, Johannes Gutenberg-University, Welderweg 11, 55099 Mainz, Germany, e-mail: kolb@uni-mainz.de URL: http://www.uni-mainz.de/~kolb*

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].

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