

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

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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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