

Invited Lecture

Better structure models from continuous-rotation 3D electron diffraction data**Paul Benjamin Klar***Faculty of Geoscience, University of Bremen**paul.klar@uni-bremen.de*

3D electron diffraction (ED) is well established as a method for crystal structure analysis of submicron to nanometre crystals, with continuous rotation 3D ED being the predominant experimental protocol [1-2]. This kind of data is mostly used for structure solution and structure model refinement within the kinematical approximation, assuming that electrons are only scattered once. However, ED is governed by multiple electron scattering, which depends on a number of experimental and structural parameters and causes non-linear deviations from the kinematical intensities. These deviations are often considered as unwanted side effects, which make the structural analysis more difficult. On the other hand, these deviations enrich the data with a wealth of information, e.g. on crystal thickness or on the absolute structure in the case of chiral compounds. With the long-term goal of achieving better crystal structure models and better agreement between experimental and calculated intensities, it is essential to consider multiple scattering as described by the dynamical theory of diffraction.

For many decades, ED studies investigated oriented zone-axis patterns by comparing measured with calculated patterns, from which structural parameters were also refined [1]. This approach is, in principle, also transferable to continuous rotation 3D ED. Due to various experimental parameters such as the mosaicity of the diffracting crystal, calculated and measured rocking curves differ significantly and a refinement based on partial reflection intensities is prone to unsatisfactory figures of merit. A successful refinement is possible by grouping the experiment into reflection batches (so-called virtual frames). Using the Bloch wave approach, model-based rocking curve profiles are calculated for each reflection, and during the refinement, the difference between experimental and calculated integrated intensities is minimised [2].

Compared to the commonly used kinematical refinement, the dynamic refinement significantly reduces the noise level in the difference Fourier maps and improves the accuracy and reliability of the resulting structure models. Thus, structural details related to weak scatterers or site disorder are revealed that would otherwise remain hidden in the noise. A particular focus of this contribution is the determination of the absolute structure of several inorganic and organic compounds, which is not possible if the kinematical theory of diffraction is employed [2-3].

Nevertheless, there is still a striking R-factor gap between refinements from X-ray and 3D electron crystallography, indicating that the current approach does not yet adequately describe all relevant experimental parameters. With recent and ongoing developments related to dynamical refinement, e.g., the application of multipolar scattering factors determined with quantum crystallographic methods [4], we are confident that even better crystal structure models will be possible in the near future.

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[2] Klar, P.B., Krysiak, Y., Xu, H., Steciuk, G., Cho, J., Zou, X., Palatinus, L. (2023). *Nat. Chem.* **15**, 848–855.

[3] Brázda, P., Palatinus, L., Babor, M. (2019). *Science* **364**, 667–669.

[4] Olech, B., Brázda, P., Palatinus, L., Dominiak, P.M. (2024). *IUCrJ* **11**, 309–324