Quantitative comparison between the diffuse scattering from three-dimensional electron diffraction and single-crystal X-ray diffraction

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Background incl. aims
In contrast to perfectly periodic crystals, materials with short-range order produce diffraction patterns that contain both Bragg reflections and diffuse scattering. In this talk, we will show a quantitative comparison between three-dimensional electron diffraction (3D ED) and synchrotron single-crystal X-ray diffraction, both for the Bragg reflections and the diffuse scattering. The thermoelectric material Nb0.83CoSb was chosen as a reference material.

Methods
3D ED allows the study of nanometre-sized crystals, which are too small to be studied with single-crystal X-ray diffraction. The average structure (occupancies and atomic positions) was refined from the Bragg reflections, whereas the local structure (the vacancy distribution) was refined from the diffuse scattering. A model of the short-range order in Nb0.83CoSb was created by assuming that nearest and next-nearest neighbour vacancies avoid each other. The correlations between the first and next-nearest neighbour vacancies were refined using both a Monte Carlo refinement in DISCUS [1] and a three-dimensional difference pair distribution function (3D-ΔPDF) refinement in Yell [2].

Results
We found that diffuse scattering data used for quantitative analysis are best acquired in selected area electron diffraction (SAED) mode using an energy filter. Both the average and the local structure could successfully be refined from both the 3D ED and synchrotron single-crystal X-ray diffraction data acquired on Nb0.83CoSb. Fig. 1 shows that a good agreement was achieved between the simulated and the experimental intensity distribution of the diffuse scattering. The higher R-value for 3D ED compared to single-crystal X-ray diffraction is likely due to residual multiple scattering. The model of the short-range order in Nb0.83CoSb can easily be applied to determine the short-range order parameters in other materials with similar diffuse

scattering, such as the lithium-ion battery cathode material LiNi0.5Sn0.3Co0.2O2.

Conclusion

The diffuse scattering in 3D ED data can be obtained with a quality comparable to that from single-crystal X-ray diffraction. Short-range order parameters could successfully be refined from the diffuse scattering in 3D ED data. As 3D ED requires much smaller crystal sizes than single-crystal X-ray diffraction, this allows to refine short-range order parameters in many technologically relevant materials for which no crystals large enough for single-crystal X-ray diffraction are available.

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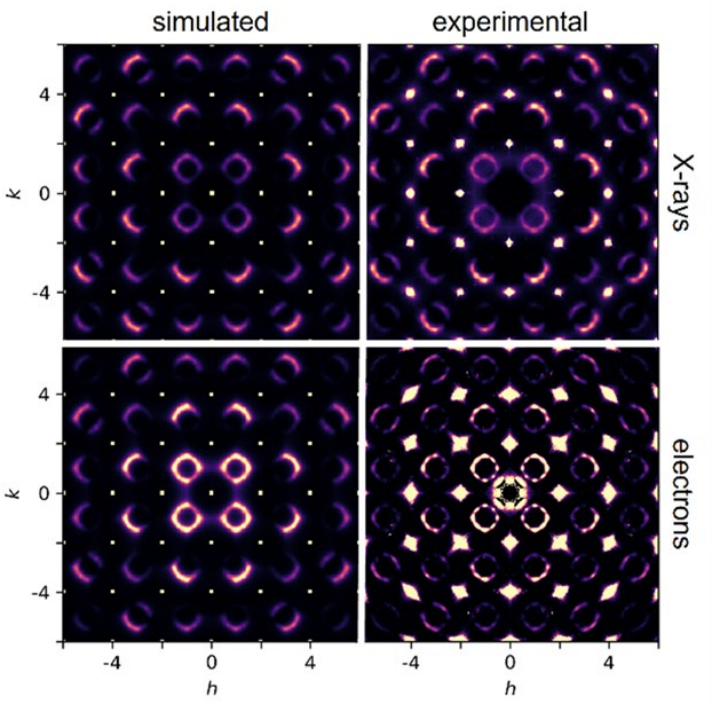
Graphic:



Keywords:

3D ED, diffuse scattering, thermoelectrics

Reference:

[1] Proffen, Th. & Neder, R. B. (1997). J. Appl. Cryst. 30, 171–175.
[2] Simonov, A., Weber, T. & Steurer, W. (2014). J. Appl. Cryst. 47, 1146–1152.