

MS17 Total scattering studies and disorder

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Recent advances in data quality and versatility of laboratory PDF experiments

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Abstract

Although the very first laboratory-based X-ray pair distribution function (PDF) analysis had been carried out in the 1930s [1], laboratory PDF studies have been pretty uncommon till a few years back. [2] Limited momentum transfer vector Q_{\max} , insufficient instrumental resolution and very long measurement times impeded a routine use for structural refinements. A few years ago, we have significantly pushed instrumental resolution of PDF data in the laboratory by reducing air scattering and optimizing beamstop positioning for a diffractometer in transmission geometry, using a MYTHEN2 4K module to cover a large angular range. [3] The use of monochromatic Ag $K\alpha_1$ radiation permitted laboratory PDF data to be fitted over interatomic distance ranges of several dozens of Angstroms, e.g. to refine PDF data of 7 nm TiO₂ nanoparticles with a goodness-of-fit R_w as low as 0.222 over 70 Å. This paved the way to study local disorder in superionic conductors [4] and the structure of 1D lanthanide polymer powders in the laboratory [5]. Since then and as very specialized PDF setups are not available in many laboratories, more diverse attempts to optimize laboratory PDF analysis have been carried out. Diffractometers using Goebel mirrors provide $K\alpha_{1,2}$ mixtures, which results in overlap of PDF peaks beyond 20 Å, preventing a continuous and simultaneous fit of the short-range order < 20 Å and the medium- to long-range order beyond. However, recent advanced data processing evolved and TOPAS can nowadays deconvolute the instrumental contributions such as wavelength mixtures, emission profiles or axial divergence, yielding less instrumentally dampened PDF data [6] – in particular for crystalline materials. This approach is showcased and compared to monochromatically measured laboratory PDF data. To conclude, and in light of recent pushes in high-energy X-ray detectors such as the HyPix-Arc series (Rigaku) or Photon III (Bruker) combined with high-flux microfocus sources, single crystal diffractometers can accomplish very decent PDF data in only about two hours. This approach is based again on $K\alpha_2$ stripping, necessary for data fitting beyond 30 Å, and highlighted for poorly crystalline powders and solutions of polynuclear lanthanide complexes. [7]

References

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