pl10 State of the art of structure analysis using powder diffraction data. L.B. McCusker, *Laboratorium für Kristallographie, ETH, CH-8092 Zürich, Switzerland*
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Thirty years ago, if a single crystal of a new phase could not be prepared, its structure was likely to remain a mystery. However, even then, if a structural model could be deduced from non-diffraction data (e.g. chemical analysis, IR/UV-Vis/NMR spectroscopy, electron microscopy, etc.), the correctness of the model could be checked by comparing its calculated powder diffraction pattern with the measured one. Twenty years ago, not only could a postulated model be verified with the powder pattern, it had also become possible to refine the atomic coordinates of the model using the Rietveld (whole-profile) method[1] in combination with X-ray data. Ten years ago, whole-profile structure refinement was already considered to be routine, and a few simpler structures were being determined directly from powder data. Today, a moderately complex structural model for a novel polycrystalline material (*ca* 20 atoms in the asymmetric unit) can usually be derived using standard approaches. Even more complex structures can be deduced if the powder diffraction data are combined with crystal chemical information (e.g. known bond distances, bond angles and torsion angles) and/or if special data collection strategies are used.

The reasons for these advances can be traced back to the the remarkable developments that have taken place in instrumentation and computing power, the increasing ease of access to synchrotron radiation facilities, and the growing number of crystallographers who have taken up the challenge posed by powder diffraction data.

The information content of a powder diffraction pattern collected on a (carefully adjusted) state-of-the-art laboratory instrument is impressive. For structure analysis, the relevant features include high angular resolution, strictly monochromatic radiation (CuKα_1), and the option for capillary sample geometry. Of course, with a synchrotron X-ray source, not only can the resolution be optimized even further, but the wavelength can also be selected according to sample requirements (e.g. to avoid high absorption, to exploit anomalous scattering, or to vary the $\sin\theta/\lambda$ limit). The high intensity and parallel nature of a synchrotron beam are also essential to the new texture approach to structure determination from powder diffraction data[2].

Without question, the spectacular developments in the realm of computing power are responsible for the fact that computational approaches that would have been unthinkable only 10 years ago have already been put into practice. This is particularly apparent in the direct-space methodologies that have been developed for structure solution in the last five years. These include the use of a variety of global minimization techniques (Monte Carlo[3], simulated annealing[4], genetic algorithm[5]) for the determination of the structures of molecular materials, the further development of a simulated annealing algorithm[6] and the implementation of an exhaustive topology search combined with Fourier recycling[7] for the determination of zeolite framework structures, and the inclusion of potential energy calculations in the determination of ionic and intermetallic structures[8].

More conventional crystallographic approaches to structure determination, such as direct[9] and Patterson[10] methods, have also been adapted to address the problem of powder diffraction data.

Armed with all of these tools, powder diffractionists are now in a position to perform reliable structure analyses on relatively complex polycrystalline materials, where complexity can be understood to describe the number of atoms in an asymmetric unit, the number of torsion angles in a molecule, the number of T-atoms in a framework structure, the number of phases in a material, or the "crystallinity" of a material. The limitations are dictated by the degree of reflection overlap in the powder diffraction pattern and the general quality of the data, but they are being challenged constantly.

An attempt will be made to give an overview of these developments and of the current possibilities and limitations of structure analysis using powder diffraction data.

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