chemical and positional disorder, structural solution from low quality data (powder patterns), joint use of several data sets, resonant scattering and fast in-situ data collection. The direct space approach is currently the powder diffraction method mostly used in hydride research for its simplicity of use, ability to work with powder patterns of low quality (broad peaks), easy way to treat the occupation disorder on hydrogen sites and active use of simple geometrical constraints. Crystal structures containing as many as 55 independent atoms (including hydrogen) have been fully characterized using powder diffraction. This is of great importance, because rapid collection of powder data thanks to modern synchrotron and neutron time-of-flight sources opens the possibility for fast in-situ studies, mapping of phase transitions induced by the temperature, pressure, hydrogen content, and chemical reactions. The crystallography of important materials for hydrogen storage like LiBH4 [2,3], Mg(BH4)2 [4,5], Mn(BH4)2 [6] and many others will be discussed.

Keywords: powder diffraction; ab-initio structure solution; hydrogen storage

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X-ray powder diffraction (XPD) and electron microscopy are remarkably complementary techniques for analyzing the structures of polycrystalline materials. Several ways of combining them have been developed recently to address structures that cannot be solved by either technique alone. Here we present two different ways of using precession electron diffraction (PED) data in combination with XPD data for structure solution. In one case, PED data are simply used to identify the weak reflections in a projection, and then these reflections are eliminated from the XPD intensities extracted from the XPD data to initiate structure solution using the powder version of the charge-flipping algorithm [3] in the program Superflip [4]. The approaches were first developed using data for the moderately complex zeolite ZSM-5, and then tested on TNU-9 [5], one of the two most complex zeolites known. In both cases, including PED data from just a few projections facilitated structure solution significantly. It should be noted that the methods are generally applicable (i.e. not zeolite specific), so it should be possible to apply them to any polycrystalline material. Furthermore, introducing information from PED data to the structure solution procedure is not restricted to charge flipping. It could also be used to advantage in other programs (e.g. in the form of better intensities in a direct-space global optimization program or as starting phases for direct methods trials). Thus, the PED technique offers a relatively simple route to valuable information that can be used to complements that in a XPD pattern.


Keywords: precession electron diffraction; X-ray powder diffraction; structure determination

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MAD Techniques Applied to the Structure Solution from Powder Data: A New Probabilistic Approach. Maria Cristina Burla, Angela Altomare, Corrado Cuocci, Carmelo Giacovazzo, Fabio Gozzo, Anna Moliterni, Giampiero Polidori, Rosanna Rizzi. *Dip. Scienze della Terra Università di Perugia, P.zza Universita’ 1, Perugia, 06100, Italy; **IC, CNR, Via Amendola 122/a, Bari, 70126, Italy; **Dipartimento Geomineralogico Università di Bari, Campus Universitario, Via Orabona 4, 70125, Bari, Italy. *Paul Scherrer Institute, Swiss Light Source, Villingen PSI, 5232, Switzerland.
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Phase determination via multiple-wavelength anomalous dispersion (MAD) techniques, owing to the tunability of the wavelength of the synchrotron radiation, is one of the most popular approaches for the solution of the phase problem in protein crystallography. The classical MAD technique is essentially a three-step procedure: a) the estimation of the structure factor moduli of the anomalous scatterer substructure; b) the location of the anomalous scatterers via Patterson or Direct Methods; c) the protein phase estimation. MAD techniques were not very beneficial for powder crystallography owing to the unavoidable peak overlapping in powder patterns. Indeed: a) the reflections $F^+$ and $F^-$ systematically overlap: consequently, anomalous
differences $|F|^2 - |F'|^2$ cannot be measured, and only the intensities $I = |F|^2 + |F'|^2$ are experimentally available; b) dispersive differences between $(|F|^2)^2 + (|F'|^2)^2$ and $(|F|^2)^2$ may be estimated from the experiment, however the estimates may be heavily affected by the casual and/or by the systematic overlapping present in the diffraction patterns. The evident minor experimental information provided by a powder diffraction experiment discouraged the use of MAD and its applications up to now. This new approach combines the joint probability distribution function method with MAD techniques to solve the phase problem from powder data. The probabilistic bases of the method were established and the distributions are calculated by assuming the prior knowledge of the scattering intensities $I$ collected at two wavelengths, the first close to the absorption edge of the anomalous scatterer and the second far away from it. The method is able to derive from these quantities the formulas providing estimates of the substructure structure factor moduli $|F_{aa}|$ which allow to retrieve the anomalous scatterer positions by means of Patterson deconvolution or Direct Methods [1]. Given the anomalous scatterer substructure the method leads to formulas estimating the full structure phases and their reliability [2]. The related procedure was implemented into a modified version of EXPO2004 [3]. Applications to synchrotron data will be shown.


Keywords: anomalous dispersion; powder data; structure solution

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The Indicator - Reflexes and Influences of Various Factors on Their Intensity, Mahmud G. Kyazumov. Institute of Physics of the National Academy of Sciences of Azerbaijan, H.Javid av.33, Baku Az-143. E-mail: elmar@physics.abs.az

On diffraction patterns the indicator-reflexes (IR) pointing to the types of packages which form crystal structures of many layered semiconductors have been revealed. It has been established that the magnitude $f$ of the strongest reflex in the series 0001 for hexagonal and 001 for monoclinc strutures and also of the second strong reflex in the series hh2hl (h=const.) for hexagonal and 0kf (k=const.) for monoclinic structures defines quantities of filled by cations the polyhedral (T and O) layers in the cell and points to the TOTE, TOTTE, TOOTE, TOTTEOE, TOTEOETOTE types of packages. Where $T$ is the tetrahedron, $O$ is the octahedron, $E$ is the empty layer. The research of influence of various factors on the intensity of IR results in the use of these reflexes as the indicator of quality.

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Powder Neutron Diffraction of Hydrogenous Materials—What is Possible Today? Marc Schmidtmann, Chick C. Wilson, Valeska P. Ting, Mark T. Weller, Paul F. Henry. WestCHEM, Department of Chemistry, University of Glasgow, UK. School of Chemistry, University of Southampton, UK. BESC, Helmholtz Zentrum Berlin, Germany. E-mail: marcsc@chem.gla.ac.uk

Understanding structure–property relationships plays a key role in current material science research and hydrogen is undoubtedly a key element to a wide range of materials. The accurate structural determination of hydrogen, however, is at least difficult and often impossible with current experimental methods. One experimental method, powder neutron diffraction (PND) was long perceived as inapplicable for hydrogenous materials due to the large incoherent scattering contribution of the $^1H$ nucleus – thus the determination of hydrogen has been a particular problem when the naturally abundant $^1H$