

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 LiBH_4 [2,3], $\text{Mg}(\text{BH}_4)_2$ [4,5], $\text{Mn}(\text{BH}_4)_2$ [6] and many others will be discussed.

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

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Solving Complex Structures by Combining Precession Electron Diffraction and X-ray Powder Diffraction Techniques. Dan Xie^a, Christian Baerlocher^a, Lynne B. McCusker^a. ^aLaboratory of Crystallography, ETH Zurich, CH-8093 Zurich.

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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 intensity extraction procedure. Generally the intensity ratios of overlapping reflections are set to one (equipartitioning), so it is quite possible that weak reflections in an overlap group are assigned medium or even large structure-factor amplitudes. By eliminating these weak reflections from consideration, a more correct partitioning of the remaining reflections in the group is obtained. In the second case, phase information is extracted from 2-dimensional PED data using the charge-flipping algorithm [1-2]. The number of correct phases retrieved in this way was found to be

comparable to that that can be derived from an HRTEM image. These phases are then used in combination with the 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.

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MAD Techniques Applied to the Structure Solution from Powder Data: A New Probabilistic Approach.

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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 $I_h = |F^+|^2 + |F^-|^2$ are experimentally available; b) dispersive differences between $(|F_2^+|^2 + |F_2^-|^2)$ and $(|F_1^+|^2 + |F_1^-|^2)$ 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_h 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 $|F_o|$ 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.

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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^a.

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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 l of the strongest reflex in the series $000l$ for hexagonal and $00l$ for monoclinic structures and also of the second strong reflex in the series $hh2hl$ ($h=\text{const.}$) for hexagonal and $0kl$ ($k=\text{const.}$) 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*, *TTOTTE*, *TOTE0OE*, *TOTE0OETOTE* 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.

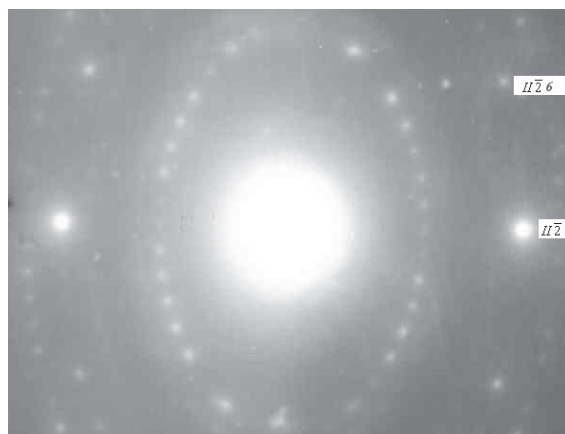
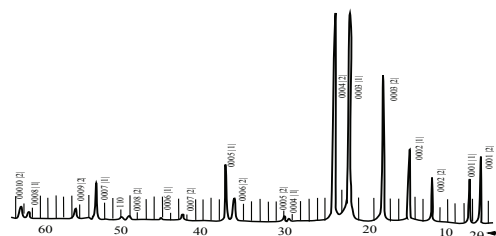


Fig 1. Electron diffraction pattern of 2H polytype of CdInGaS_4 monocrystal with *TOTE* type packet.



The figure 2 demonstrates the powder X-ray diffraction of $\text{GaFeS}_{2.7}$, where layered microcrystals are oriented parallel to substrate. The 0003 [1] peak indicates first *TOTE* phase with the parameters of lattice $a=3,653\text{\AA}$ and $c=12,033n\text{\AA}$, and the 0004[2] peak indicates *TOTTE* phase with the parameters of lattice $a=3,653\text{\AA}$ and $c=14,868n\text{\AA}$, where n is the quantity of layers in polytypes.

Keywords: diagnostics; diffraction data; layered compounds

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Powder Neutron Diffraction of Hydrogenous Materials-What is Possible Today? Marc Schmidtman^a, Chick C. Wilson^a, Valeska P. Ting^b, Mark T. Weller^b, Paul F. Henry^c.

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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