

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.

[1] Altomare et al., **2009**, *J.Appl.Cryst.* 42, 30-35. [2] Altomare et al., **2009**, *Acta Cryst.* Submitted. [3] Altomare et al., **2004**, *J.Appl. Cryst.* 37, 1025-1028.

Keywords: anomalous dispersion; powder data; structure solution

FA5-MS01-O8

The Indicator - Reflexes and Influences of Various Factors on Their Intensity. Mahmud G. Kyazumov^a.

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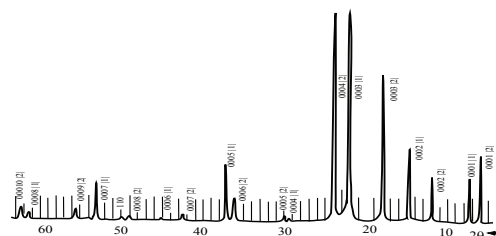
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On diffraction patterns the indicator-reflexes (IR) pointing to the types of packages which form crystal structures of many layered semiconductors have been revealed. It has been established that the magnitude l of the strongest reflex in the series $000l$ 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*, *TOITE*, *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

FA5-MS01-O9

Powder Neutron Diffraction of Hydrogenous Materials-What is Possible Today? Marc Schmidtman^a, Chick C. Wilson^a, Valeska P. Ting^b,

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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

isotope is concerned and deuteration is unfeasible (true for many materials, e.g. hydrogen storage materials, proton conductors, ferroelectrics, etc.). Improvements in neutron and detector technology have changed this long-standing view and a range of hydrogenous materials have now been characterised successfully with PND. [1-6] There are limitations though, naturally dependent on the ^1H content, complexity and thermal motion of the material under study. The general reduction of information inherent in PND data (from both the nature of the powder data and the often high incoherent background contribution from hydrogen-containing materials) can be partially overcome by the inclusion of single crystal X-ray diffraction (SXD) data in joint PND and SXD refinements. Also, imaging proton densities from difference Fourier maps is an old but equally powerful tool to obtain qualitative information about proton behaviour. We intend to show what appears possible today, but also point out the limitations we have found on the basis of recent datasets collected at the high intensity powder diffractometer D20 at ILL, Grenoble, and the recently upgraded HRPD at ISIS, UK. The materials studied include both inorganic and organic, and vary in ^1H content, complexity and data collection temperature.

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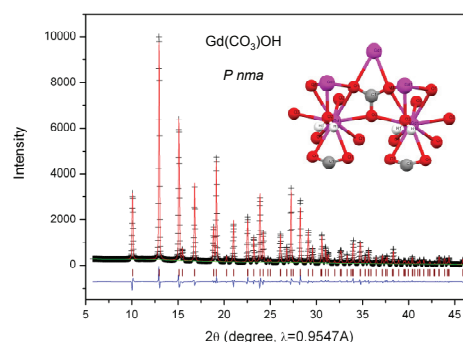
Keywords: powder neutron diffraction; hydrogen compounds; incoherent scattering

FA5-MS01-O10

Crystal Structure and Phase Transitions of $\text{Gd}(\text{CO}_3)\text{OH}$ Studied by Synchrotron PXRD. Hwo-Shuenn Sheu^a, Wei-Ju Shih^a, Wei-Tsung Chuang^a, I-Fang Li^b, Chen-Sheng Yeh^b. ^aNational Synchrotron Radiation Research Center, Hsinchu 300, Taiwan. ^bDepartment of Chemistry, National Cheng Kung University, Tainan 701, Taiwan.
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The crystal structure of $\text{Gd}(\text{CO}_3)\text{OH}$ was solved using synchrotron powder X-ray diffraction (PXRD) (see attached Figure). $\text{Gd}(\text{CO}_3)\text{OH}$ has been known to exist in the form $\text{Gd}_2\text{O}(\text{CO}_3)_2 \cdot \text{H}_2\text{O}$ and its powder pattern is listed in JCPDF (#430604) for decades but the crystal structure has not yet been elucidated. The crystal structure is solved by simulated annealing method using the DASH program. The final Rietveld refinement converged to $R_{\text{wp}} = 6.28\%$, $R_p = 4.47\%$ and $\chi^2 = 1.348$, using the GSAS program. $\text{Gd}(\text{CO}_3)\text{OH}$ crystallized in an orthorhombic crystal system with lattice constants $a = 7.08109(9)$, $b = 4.88436(7)$, $c = 8.45010(13)$ Å, and space group $Pnma$. $\text{Gd}(\text{CO}_3)\text{OH}$ forms a three-dimensional framework structure with an eight member-ring, one-dimensional channel and an OH anion in the cavity. XANES of Gd L_{III} -edge suggests that the oxidation

state of Gd is 3+. Two phase transitions of $\text{Gd}(\text{CO}_3)\text{OH}$ were found at 500 and 650 °C to yield $\text{Gd}_2\text{O}_2\text{CO}_3$ and Gd_2O_3 respectively.



Keywords: structure determination; phase transition; rare-earth compounds