Fig.1. Coordinates polyhedra around Fe atoms:
a) tetrahedron, b) trigonal bipyramid

The Rietveld analysis gives us information about changes of avarage structures (cubic ↔ monoclinic) but Pair distribution Function (PDF) methods let us describe the local configuration of deuterium atoms wich stays almost the same in order and disordere phases (fig.2). Neutron Time-of-Flight (ToF) data were collected at IPNS, Argonne, and Lujan Center, Los Alamos on series of samples with different deuterium content. Each sample was measured below and above the temperature of deuterium ordering. The local deuterium configuration in disordered phase have been modeled by DISCUS using Revers Monte Carlo technique [3].

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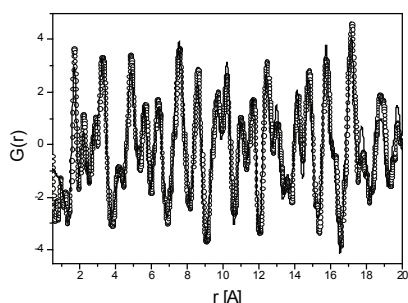

Figure 2. Observed PDF (points) of $YFe_2D_{4.2}$ in disordered state and modeled (solid line) by same local order of deuterium atoms around iron as in the ordered phase.

Structural Investigations of $LnBO_3$ (Ln= Y, La, Nd, Sm, Eu, Gd, Dy, Ho, Er,Yb, Lu) by Rietveld Method. Semih Seyyidoglu[a], Katrin Hoffmann[b], Barbara Albert[b], Aysen Yılmaz[a]. *[a]Department of Chemistry, Middle East Technical University, 06531, Ankara, Turkey. [b]Eduard-Zintl-Institut für Anorganische und Physikalische Chemie, Petersenstr. 18, 64287, Darmstadt, Germany.*
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Numerous efforts have been devoted to the structure determination of these compounds in literature. While $LaBO_3$ and $NdBO_3$ exhibit the aragonite type (orthorhombic Pnma) structures [1-2], the structure vaterite type rare earth borates (Y, Gd-Yb) are still under investigation since the structure of these compounds are directly related with their synthesis method. The structure of $GdBO_3$ were defined in rhombohedral space group R32 by Lin and coworkers [3]. While in Sheptyakov's work[4], Eu doped yttrium orthoborate structure is monoclinic space gropu C2/c, Chadeyron produced YBO_3 single crystals with P63/m space group [5]. Since there is no systematic structural analysis work of whole rare earth borate series, in this work we synthesized and solved their stucture by using Rietveld Method [6] with GSAS program[7]. We prepared $LnBO_3$ (Ln=Y, La, Nd, Sm, Eu, Gd, Dy, Ho, Er, Tm, Yb, Lu) powder samples by using Ln_2O_3 and H_3BO_3 (ratio=1:2) heated at 900 °C for 10 hour and 1000 °C for 5. Then, their XRD patterns were collected on a PANalytical X'PERT PRO diffractometer equiped with PIXCEL detector. Among these rare earths, $LaBO_3$ and $NdBO_3$ were solved based on Pnma orthorhombic structure. The crystal structure of YBO_3, $DyBO_3$ and $HoBO_3$ were C2/c monoclinic and $SmBO_3$ showed P-1 triclinic structure. The structure of $TbBO_3$, $TmBO_3$ and $YbBO_3$ were solved based on P21/m.

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Metal and Chemical Hydrides: Highlights and Pitfalls of Powder Diffraction. Radovan Černý. *Laboratory of Crystallography, University of Geneva, 24, quai Ernest-Ansermet, CH-1211 Geneva 4, Switzerland.*
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The methods of structural characterization of metal and chemical hydrides are reviewed (see also [1]). It is shown that powder diffraction is essential component of hydrides research where the structural characterization is currently undertaken by X-ray and neutron diffraction. In the case of chemical hydrides like borohydrides of light alkaline metals/earths X-ray diffraction alone can provide the structural parameters with sufficient accuracy. A crystallographer analyzing hydrides has to face numerous crystallographic challenges which include complex structures, superstructures, pseudo-symmetries, twinning,

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], $Mg(BH_4)_2$ [4,5], $Mn(BH_4)_2$ [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. <u>Dan Xie</u>[a], Christian Baerlocher[a], Lynne B. McCusker[a]. *[a]Laboratory 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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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.
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Phase determination *via* multiple-wavelength anomalous dispersion (MAD) techniques, owing to the tunability of the wavelength of the synchroton 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