
CNR-Institute of Crystallography (IC), via Amendola 122o, Bari 70126, Italy. *Dept. of Geomineralogy, University of Bari, via Orabona 4, Bari 70125, Italy.

E-mail: angela.altomare@ic.cnr.it

Fourier syntheses providing electron density maps always show series-termination errors due to the limited experimental resolution. The worse the resolution, the worse the quality of the map; peaks are misplaced with respect to the true positions, positive and negative peaks occur, the peak profile is deformed. Two new algorithms have been developed aimed at reducing the resolution bias. The first one works in direct space [1,2], the second one in reciprocal space [3]. They are able to improve both peak positions and peak form in such a way that the map better fits the real atomic electron density. The new procedures have been introduced in EXPO2009, the strengthened and updated version of EXPO [4] and applied to the electron density maps provided by Direct Methods. Organic as well as metallicorganic powder structures with non-atomic resolution experimental data have been tested. In spite of the difficulties in structure solution by powder data, the new approaches succeeded in several cases which could not be solved without the resolution bias correction. The theory of the resolution bias correction and its application by EXPO2009 will be presented.


Keywords: ab-initio powder structure determination; resolution; electronic density


IC, Sede di Bari, via Amendola 122/o, 70126 Bari, Italy. *Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-106 91 Stockholm, Sweden.

E-mail: annagrazia.moliterni@ic.cnr.it

Despite its long history and the recent advances in theoretical methods, computer speed and experimental devices, powder pattern indexing can still be a challenge. More plausible different unit cells are often suggested, among which the identification of the correct cell can be difficult. The cell reliability is generally assessed via historical figures of merit (i.e., $M_{tot}$ by De Wolff [1] and $F_c$ by Smith & Snyder [2]) that sometimes fail. Usually the space group identification follows the indexing. We propose a new approach which simultaneously determines the unit cell and the extinction symbol by N-TREOR09 (the evolved version of the indexing package N-TREOR [3]). A new figure of merit has been also introduced in N-TREOR09. It has been proved to be more powerful than $M_{tot}$ and combines the contribution by $M_{tot}$ with additional parameters like the number of unindexed lines, the agreement between the calculated and observed profiles, the degree of overlap in the pattern, the symmetry suggested by the automatic evaluation of the extinction group. N-TREOR09 has been also enriched by an improved indexing procedure in the triclinic system and integrated into EXPO2009, the updated version of EXPO2004 [4]. The main features of N-TREOR09 and its applications to a large set of test structures will be described.


Keywords: powder indexing; space-group determination from powders; crystallographic computing

Avarage and Local Structure of Laves Phase Deuterides YFe$_{20}$D$_x$. Joanna Ropka, Radovan Černý, Valerie Paul-Boncour, Michel Latroche, Thomas Profen. Laboratory of Crystallography, University of Geneva, quai E.-Ansermet 24, CH-1211 Geneva, Switzerland. *CMTR, ICMPE, CNRS, 2 rue H. Dunant, 94320 Thiais Cedex, France. †Lujan neutron Scattering Center, LANL, Los Alamos, NM 87545, USA.

Email: Joanna.Ropka@unige.ch

Between all cubic (C15) Laves phases, widely investigated for years, the YFe$_{20}$D$_x$ system is especially interesting because of its magnetic properties. The structure of particular YFe$_{20}$D$_x$ deuteride depends on deuterium content, likewise deuterium and magnetic ordering temperatures [1]. The most rich phase of this system under ambient condition - YFe$_{20}$D$_{13}$ – is formed as a cubic (Fd$ar{3}$m) and monoclinic (Pc) above and below deuterium ordering temperature, respectively. The deuteriums in disordered (cubic) structure partially occupies 3 sites, while occupancies in fully ordered monoclinic structure equal to 1 for almost all of 18 deuterium sites. Deuterium atoms make polyhedras (1 tetrahedron, 7 trigonal bipyramids — fig.1) around iron with distances Fe-D between 1.62-1.83 Å. [2]
The Rietveld analysis gives us information about changes of average structures (cubic ↔ monoclinic) but Pair distribution Function (PDF) methods let us describe the local configuration of deuterium atoms which stays almost the same in order and disordered 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].


Figure 2. Observed PDF (points) of YFeO$_3$ in disordered state and modeled (solid line) by same local order of deuterium atoms around iron as in the ordered phase.

Keywords: inorganic solid state chemistry; boron compounds; rietveld structural analysis

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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.

E-mail: Radovan.Cerny@cryst.unige.ch

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 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, 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 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,