

Examples will be presented of the application of Rietveld analysis of VCT powder X-ray data to investigate chemical and structure variations in synthetic mineral systems. These include studies on cation substitutions in synthetic lovingite, (Ca, Ti³⁺, Ti⁴⁺, Mn)₂₂O₃₈ prepared under reducing conditions, studies on anion vacancy and Fe³⁺/Fe⁴⁺ variations in the solid solution of hexagonal BaTiO₃ with Ba₂Fe₂O₅, and crystal chemistry systematics of sulphate and oxysulphate phases formed during sulphuric acid digestion of ilmenite.

ref. I.C. Madsen and R.J.Hill, *J. Appl. Cryst.* (1994). 27, 385-392

MS02.05.07 INFORMATION ON SYMMETRY IN POWDER DIFFRACTION DATA. M. Ohmasa¹, K. Ohsumi² and H. Toraya³. ¹Department of Life Science, Himeji Institute of Technology, Japan; ²Photon Factory, National Laboratory for High Energy Physics, Japan; ³Ceramics Research Laboratory, Nagoya Institute of Technology, Japan.

Since each crystal family except triclinic, monoclinic and orthorhombic ones doesn't correspond to a Laue class uniquely (holohedral and hemihedral Laue classes) and no method to identify Laue classes of those families had been presented for powder specimens, their space groups had been assigned only with crystal family and reflection conditions. Because summed intensities are measured by powder diffraction methods and they are not separable to individual intensities. Recently Ohmasa & Ohsumi (1995) indicated that weighted reciprocal lattices named composite reciprocal lattices are constructed from powder diffraction data in such a way that summed intensities are distributed to reciprocal lattice points to hold holohedral symmetry and indicated that information on Laue classes can be obtained from concentrations of interatomic vectors in Patterson functions evaluated with intensities at the composite reciprocal lattice points (composite Patterson functions). For hemihedral Laue classes, a composite reciprocal lattices is regarded as a superposed record of weighted reciprocal-lattice points of a single crystal. In this case the superposition yields new symmetry generators (extra generators) which are not intrinsic to the Laue class of the single crystal. The same generators as the extra ones are included in holohedral symmetry and the distribution of the points and their relative weights in the composite Patterson function coincide with those in the Patterson function of the real structure. The same generators as the extra ones are not included in the symmetry generators of the real structure with hemihedral Laue symmetry but the apparent symmetry of the composite Patterson functions is enhanced to holohedral one by the extra generators. However the distribution of the peaks and their relative weights in the composite Patterson functions are not perturbed by the extra generators and the feature of the concentration of the peaks related to interatomic vectors in the real structure is retained. Consequently, distinction of space groups should in principle be possible by interpretation of composite Patterson functions. Composite Patterson functions of some materials will be indicated as examples.

Ohmasa, M. and Ohsumi, K. (1995). *Acta Cryst.* A51, 87-91.

MS02.05.08 THE TANGENT FORMULA DERIVED FROM PATTERSON FUNCTION ARGUMENTS: A USEFUL TOOL FOR SOLVING ZEOLITE STRUCTURES FROM X-RAY POWDER DATA. J. Rius & C. Miravittles, Institut de Ciència de Materials de Barcelona (CSIC), Campus de la UAB, 08193-Cerdanyola, Catalunya (Spain).

The viability of solving the structure type of zeolitic and layered materials applying multisolution direct methods to low resolution (≈ 2.2 Angstroms) powder diffraction data is shown. The phases are refined with the tangent formula derived from Patterson function arguments [Rius (1993). *Acta Cryst.* A49, 406-409] implemented in the XLENS program and the correct phase sets are discriminated with the conventional figures of merit.

The two test examples presented are (a) the already known tetragonal zeolite ZSM-11 (space group I-4m2) at 2.3 Angstroms resolution and (b) the hitherto unknown layer silicate RUB-15 (Ibam) at 2.2 Angstroms resolution. In both cases, the tetrahedral Si units appear as resolved peaks in the Fourier maps computed with the phases of the highest-ranked direct methods solutions.

MS02.05.09 DIRECT PHASING FROM POWDER DATA: THE EXTRA OPTIMIZED PROCEDURE C. Giacobozzo(+), A. Altomare(+), G. Cascarano(+), A. Guagliardi(+), A.G.G. Moliterni(+), M.C. Burla(*) & G. Polidori(*) (+) Istituto di Ricerca per lo Sviluppo di Metodologie Cristallografiche - CNR c/o Dipartimento Geomineralogico - Campus Universitario Via E. Orabona, 4 - 70125 Bari - Italy. (*) Dipartimento di Scienze della Terra, Università, 06100 Perugia, Italy.

Present difficulties in solving crystal structures from powder data are not to be found in the lack of efficiency of direct methods, but in the inaccuracy of the process which extracts structure factor amplitudes from a powder pattern. EXTRA [A. Altomare, M. C. Burla, G. Cascarano, C. Giacobozzo, A. Guagliardi, A. G. G. Moliterni & G. Polidori (1995), *J. Appl. Cryst.* 28, 842-846] has been an efficient answer to this problem: friendly to use, the program is able to automatically decompose, via the Le Bail algorithm, quite complicated experimental patterns.

EXTRA is now incorporating new important features. It is able to take advantage from: a) the Patterson map; b) the pseudo-translational symmetry; c) the preferred orientation; d) the statistical estimates of the diffraction amplitudes via direct methods; e) a molecular fragment.

All the above sources of information are able to dramatically improve the accuracy of the pattern decomposition process so enlarging the size of crystal structures solvable by powder data. We are merging EXTRA with SIRPOW.92 [A. Altomare, G. Cascarano, C. Giacobozzo, A. Guagliardi, M.C. Burla, G. Polidori, & M. Camalli, (1994), *J. Appl. Cryst.* 27, 435-436], our direct methods program for powder data, to provide a unique program, transparent to the user, which should be able to solve a structure directly the diffraction pattern.