MS31 P06

FoxGrid – multi PC execution support for powder diffraction structure solution by Fox program. Jan Rohlíček, Michal Hušák, Bohumil Kratochvíl *Department of Solid State Chemistry, Institute of Chemical Technology Prague, Czech Republic.* E-mail: rohlicej@vscht.cz

Keywords: parallel computing, powder structure determination, software

For most structures (small molecules) the existing global optimisation algorithms and programs are sufficient to find the structure solution from powder data very quickly. But there are still a number of problems for which more computing power is required: this includes complex structures (>50 independent atoms or >30 internal degrees of freedom), and problems for which a wide range of structural models (different unit cells, space groups, building blocks,...) or algorithm parameters has to be tested.

We had try to solve the performance problem by modifying the FOX[1] structure solution code for an automatic multi PC parallel run. The concept uses simple client-sever architecture for distributing the computation. Any PC can be configured either as a server or a client. The data is passed as an XML file between the client and the server. Grid computing can even be used on a single computer with multiple cores or hyper-threading technology, to fully use the available computer power while gathering all the results in a single Fox instance – the one configured as a server.

All the additional code works along the existing Fox code, using cross-platform socket code from the wxWidgets library.

The current work on the code development is targeted on benchmarking, automatic analysis of the obtained results and implementation of an intelligent task generation module. This module can help the user to solve the problem by "brute force", i.e. preparing tasks trying to solve the structure in different space groups and with different lattice parameters (and other input conditions) automatically.

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MS31 P07

Structural Characterization of Nanocrystalline Apatites Obtained by Mechanochemical Synthesis Ivonne Rosales[a], Lauro Bucio[a], Eligio Orozco[a] and Carlos R, Magaña[a]. [a]*Department of Physics, National Autonomous University of Mexico. Mexico City, México.* E-mail: rosales@fisica.unam.mx

Keywords: Nanocrystalline apatites, Mechanochemical synthesis, X-Ray powder diffraction.

Apatite compounds have gained recently considerable attention because their wide range of physicochemical properties such as high ionic conductivity at low temperatures and low activation energies associated with the migration of oxygen ions [1-3]; host material for the nuclear industry [4]; as ceramic pigment [5]; and so on.

The compounds considered here, were those characterized by the general formula $A_{10}(BO_4)_6X_2$ in which A: Na, Ca and Ho; B: P and Si; and X: OH^-, F^-, Cl^- or O^{2-}.

The studied systems for obtaining compounds type apatite from the systems $NaCl-Ho_2O_3-SiO_2$, $NaF-Ho_2O_3-SiO_2$ and $CaCO_3-(NH_4)_2HPO_4$; were treated by mean mechanochemical synthesis method with few hours of milling. This method was used since it allows us to obtain compounds of nanocrystalline size.

The structure and microstructure of apatites were characterized by X-ray powder diffraction. The Rietveld refinement in space group P 6_3/m for all the systems in study leads to lower R values. Scanning electron microscopy (SEM micrographs) of the samples in $NaCl-Ho_2O_3-SiO_2$ and $NaF-Ho_2O_3-SiO_2$ system revealed compact crystallites of ~ 60 nm.

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MS31 P08

Retrieval system for qualitative and standardless quantitative phase analysis Igor Yakimov, Institute of non-ferrous metals and gold, Siberian federal university, Krasnoyarsk, Russia. E-mail: I-S-Yakimov@yandex.ru

Keywords: search/match, quantitative phase analysis, standardless QPA.

A search/match retrieval system (Q&QPA Retrieve) has developed for qualitative and standardless quantitative phase analysis of complex powder patterns and their groups. The system includes itself retrieve based on high selective query language and problem oriented GUI for visualization and operating under powder patterns and reference spectrums from DB. A Boolean logic is complemented in the query language by elements of calculus predicate theory. A qualitative phase analysis is executed by programs made from sequence queries. Hidden program are created automatically during mouse manipulations on analysing pattern, searching inquiry reference spectrums and elemental table over GUI. The GUI includes two main graphical windows for comparison of patterns and reference spectrums and for model spectrum. The model spectrum is optimal line combination of matching reference spectrums in limits of analyzing powder patterns.

Two automatic standardless quantitative phase analysis (QPA) methods have incorporated into search/match system. The first is intended for QPA of individual powder patterns and based on reference intensity ratio (RIR) method modified for using of powder pattern model spectrum. Line combination coefficients are included into known variants of RIR: normalized RIR and RIR with internal standard for without and in presence amorphous phases accordingly. There is possibility to refine a model spectrum by known quantitative elements composition during QPA. The modified RIR more resistance to preferred orientations and other distortions of powder patterns but need to use reference intensity ratio values ("corundum numbers" of phases) adequately to analyzing

powder patterns. The best accuracy is achieved with calculated corundum numbers for the best matching reference spectrums. The second method has developed for simultaneous QPA of group of powder patterns with identical qualitative phase composition of samples. The method is development of J.Rius [1] and K.Zangalis [2] methods but differ by iterative refinement mass absorption coefficients of samples and calibrate constants or corundum numbers of phases which made by least squares or simplex methods.

The QPA accuracy of both methods are discussed on data of Round Robin on Quantitative Phase Analysis CPD IUCr [3]. Identification possibilities of system are discussed on data of Search-Match Round Robin - 2002 CPD IUCr [4] where only the Q&QPA Retrieve identified all phase compositions exactly.

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MS31 P09

Detections of the Pattern Deformations in the Search/Match Procedure Akihiro Himeda, Hiroki Yoshida, *X-Ray Research Laboratory, Rigaku Corporation, Japan.* E-mail: himeda@rigaku.co.jp

Keywords: powder patterns, phase identification, lattice distortion

Phase identification with the powder patterns is important as a starting point for the quantifications or the whole pattern fitting analyses. Although there are a lot of search/match programs, they often fail when the powder patterns are distorted from those in the database. Two main reasons of the deformations are the lattice distortion and the intensity deformation due to the preferred orientations. Especially, when the lattice distortion is anisotropic, which is often the case in organic compounds, it is hard to identify the correct phase.[1]

We have developed a new search/match algorithm which detects and evaluates the anisotropic lattice distortion and intensity deformation due to the preferred orientations. In Figure 1, overlay of the observed powder pattern downloaded from the Search-Match Round Robin-2002 web site [1] and the pattern registered in the ICDD database [2] is shown. The inconsistency between these patterns is due to the lattice distortion of the observed pattern with its lattice constants (a, c)=(9.09, 13.47) from the ICDD pattern (a, c)=(9.19, 13.40). Our search/match procedure automatically detects the anisotropic deformation while searching the card from the ICDD database. After finding the appropriate pattern, the correct lattice constants are readily obtained. In Figure 2, the modified pattern is shown and it agrees well with the experimental pattern.

[1] See the result of Sample 2 in the Search-Match Round Robin-2002, http://sdpd.univ-lemans.fr/smrr/
[2] John Faber and Tim Fawcett. (2002) *Acta Cryst.* B58, 325-332.
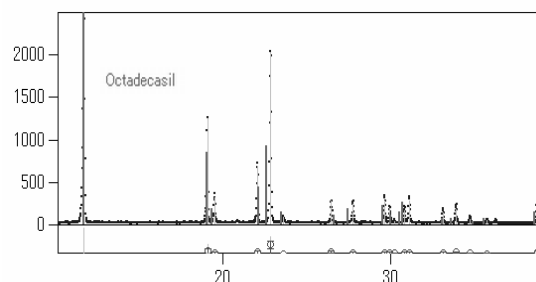

Figure 1 Overlay of the observed powder pattern(dots) and the pattern(spikes) registered in the ICDD database**.**
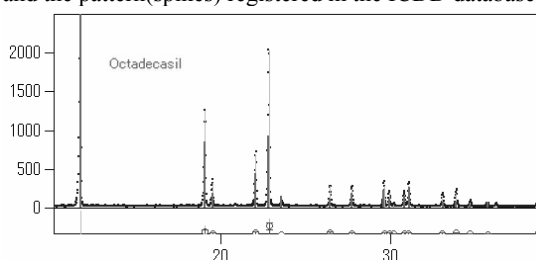

Figure 2 Overlay of the observed powder pattern(dots) and the modified pattern(spikes) based on the detected anisotropic lattice distortion.

MS31 P10

Protein powder diffraction – pH variation studies of insulin. Lisa Knight[a], Irene Margiolaki[a], Andy Fitch[a], Jon Wright[a], Mathias Norrman[b], Gerd Schluckebier[b].
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Keywords: Powder Diffraction Techniques, Protein Crystallography, pH.

Modern developments of the powder diffraction technique have allowed the investigations of systems with large unit cells like proteins [1-3]. One of the main advantages of powder diffraction is the flexibility in crystallisation conditions, as well as being able to observe phase transitions and multiple phases in situ. Variatiation in the structure of microcrystals is important particularly in the treatment of diabetes where medication is often administered as insulin microcrystals. Dependent on the crystal form, the medication can have different time/action profiles. The present study aims to investigate the effect of pH on the lattice parameteres of various forms of insulin microcrystals at room temperature. Variation of the cell axes with increasing pH has been seen in the hexagonal and cubic forms, these variations along with phase transitions observed in orthorhombic forms will be presented. Preliminary data interepretation correlating the variations with the structural and microstructural characteristics of the systems under study will be shown.

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[3] Basso S. et al.Acta Cryst. D61, 1612-1625 (2005).