MS.03.4

Closing the gap between single crystal and powder diffraction

Henning O Sorensen[1], Soren Schmidt[1], Jonathan Wright[2], Per Christian Hansen[3], Henning F Poulsen[1]

[1]Riso National Laboratory for Sustainable Energy, Technical University of Denmark, Materials Research Department, Frederiksborgvej 399, P.O. 49, Roskilde, Roskilde, 4000, Denmark, [2]ESRF, 6, Rue Jules Horowitz, B.P. 220, 38043 Grenoble CEDEX, France, [3]Department of Informatics and Mathematical Modelling, Technical University of Denmark, Building 321, DK-2800 Lyngby, Denmark, E-mail：henning.sorensen@risoe.dk

One of the major challenges in single crystal structure determination is to obtain crystals of suitable quality and size. If no suitable crystallization conditions one often turns to powder diffraction. Powder diffraction is unfortunately limited in the molecular complexity (unit cell size), which can be solved ab initio and the accuracy of the refined molecular parameters. To fill the gap between single-crystal and powder diffraction, we have being developing an alternative method. The method is based on employing hard and highly focussed X-rays illuminating a polycrystalline sample with grain sizes down to 1-10 microns. From this polycrystal X-ray data set reflections are sorted into sets each associated with a certain grain in the sample [1,2]. Hence, one obtains a series of single crystal data sets. One of the difficulties apart from determining the unit cell parameters and the grain orientations is to extract integrated intensities as the spot overlap grows with the number of grains in the X-ray beam. We have adapted a new approach for extraction of the intensity of the overlapped reflections as well as integration of weak reflection by fitting the intensity using an intensity profile. The approach is to reconstruct the orientation distribution function (ODF) on a grid using the strong non-overlapped reflections and from this the profile any reflection can be calculated. The algorithms for reconstructing the ODF and calculation of the reflection profiles are presented together with results obtained.

[1] S. Schmidt, GrainSpotter, http://fable.wiki.sourceforge.net/GrainSpotter.
[2] J. Wright, ImageD11, http://fable.wiki.sourceforge.net/imaged11.

MS.03.5

Advances in methods and algorithms in EXPO2008

Carmelo Giacovazzo, Angela Altomare, Gaetano Campi, Corrado Cuocci, Anna Grazia Moliterni, Rosanna Rizzi
CNR - Istituto di Cristallografia, Via Amendola 122/o, Bari, Bari, 70126, Italy, E-mail：carmelo.giacovazzo@ic.cnr.it

The program EXPO2008 is able to face all the steps necessary for crystal structure solution from powder data: a) diffraction pattern indexing; b) space group determinations; c) phasing process; d) crystal structure refinement by least squares- Rietveld refinement. Recent advances in the program concern:
a) the indexing. The program N-Treor (1) has been updated to perform a more exhaustive search of the correct unit cell;
b) the space group determination. This step has been improved by integrating probabilistic techniques (2) with a more (automatic) careful analysis of the diffraction pattern;
c) the phasing process. For organic structures Direct Methods usually leads to electron density maps which do not provide useful molecular fragments. That is partly due to the inaccuracy of the diffraction magnitudes caused by the peak overlapping, and partly to the limited data resolution. A recent mathematical approach (3) showed how to minimize the resolution effects in any electron density map. The approach has been implemented into an EXPO2008 routine which combines it with an automatic least squares procedure and proved to be able to solve organic structures ab initio with data resolution up to 1.53Angstrom. A further tool available for the EXPO2008 users is a simulated annealing procedure which allows the crystal structure solution when the geometry of the fragments which constitute the molecule is a priori known.

(1) A.Altomare, C. Giacovazzo, A. Guagliardi, A.G.G. Moliterni, R. Rizzi, P.-E. Werner (2000). J. Appl. Cryst. 33, 1180-1186.
(2) A.Altomare, M. Camalli, C. Cuocci, C. Giacovazzo, A.G.G. Moliterni, R. Rizzi (2007). J. Appl. Cryst. 40,743-748.
(3) A.Altomare, C. Cuocci, C. Giacovazzo, G.S. Kamel, A. Moliterni, R. Rizzi (2008). Acta Cryst. A64, 326-336.

MS.04.1

Supercritical hydrothermal synthesis of organic inorganic hybrid nanoparticles

Tadafumi Adschiri
Tohoku University, Advanced Institute for Materials Research(WPI-AIMR), 2-1-1, Katahira, Aoba-ku, Sendai, Miyagi, 980-8577, Japan, E-mail：ajiri@tagen.tohoku.ac.jp

We propose a new method to synthesize organic-inorganic hybrid nanoparticles at supercritical hydrothermal conditions. By introducing organic legands (aminoacids, carboxylic acids, amines, alcohols, aldehydes etc.) into supercritical hydrothermal synthesis atmosphere, organic -inorganic hybrid nanocrystals are synthesized. This is due to the homogeneous phase formation for the organic substance and metal salt aqueous solutions at supercritical conditions. Particle size is in the range from 2.5 nm to 10 nm, and particle size dispersion is extremely narrow. Crystal shape can be controlled to be sphere, nano-cube, nano-ribbon etc. By selecting a proper modifier, particles can be dispersed perfectly in organic solvents or in aqueous solutions. By drying the colloidal solution, self-assembly structure of nanocrystals can be obtained. This implies a variety of applications of the nanoparticels including nanohybrid polymers, nano-ink, nano-paints, and bio-imaging probe.

MS.04.2

Acidic ammonothermal growth of bulk GaN crystals

Dirk Ehrentraut, Tsuguo Fukuda
Tohoku University, Institute of Multidisciplinary Research for Advanced Materials, Katahira 2-1-1, Aoba-ku, Sendai, Miyagi, 980-8577, Japan, E-mail：dirk@tagen.tohoku.ac.jp

The crystal growth of hexagonal GaN (h-GaN) is currently a hot topic in spite of high demand for free-standing, lattice-matched wafers for the GaN device technology [1]. Among the other GaN growth techniques, the ammonothermal growth of GaN is very promising for future mass-production and basically is derived from the successful