single-crystal X-ray diffraction (XRD) is not generally used. Here, we wish to show how a combination of powder XRD and advanced solid-state NMR (among other) techniques allow the resolution of the crystal structures of these materials. A brief account of some of the materials properties will also be given.

[1] Rocha J., Anderson M. W., *Eur. J. Inorg. Chem.*, 2000, 801. [2] Rocha J., Carlos L. D., *Curr. Opin. Solid State Mater. Sci.*,2003, **7**, 199.

Keywords: zeolites, structure, lanthanides

MS18.25.3

Acta Cryst. (2005). A**61**, C29

Direct Localization of Atoms in Nanoporous Powders by Resonant Contrast Diffraction

Jean-Louis Hodeau[a], Herve Palancher[c], Christophe Pichon[b], John Lynch[b], Bernadette Rebours[b], Jean-Francois Berar[a], Juan Rodriguez-Carvajal[d]. [a]*Lab. cristallographie, CNRS BP166X 38042Grenoble, France.* [b]*Institut Français du Pétrole, 1-4 av. Bois Préau, 92852Rueil-Malmaison, France.* [c]*C.E.A. Cadarache, DEN/LLCC 13108 Saint Paul-lez-Durance France.* [d]*Lab. Léon Brilloin Saclay, 91191Gif/Yvette, France.* E-mail: hodeau@grenoble.cnrs.fr

Based on the experimental improvement of data collection achieved for DAFS spectroscopy and applied using the MAD principles, a methodology has been developed for powder sample analysis in order to optimise the use and the interpretation of anomalous information. By using a zeolite example[1] our aim was to validate the resonant scattering method for contrast studies in complex powders with mixed occupancy sites. On one hand we show that even in powder samples with considerable reflection overlap and with a basically known framework the use of "***dispersive difference***" electron density maps allows an easy localisation of resonant atoms. On the other hand, the use of "***anomalous difference patterns***" enables good agreement factors to be achieved for accurate localisation. These two specific difference tools can also be used in powders containing several phases. The application of these methods can be extended to materials in geology, industry, environmental studies to localize transition metals or RE atoms. It can also give their valence by the use on powders of Diffraction Anomalous Near Edge Structure spectra. The extension of resonant contrast diffraction to in situ measurements can be foreseen to analyse the evolution of atomic order during chemical reactions.

[1] Palancher H. et al., *Angew. Chem. Int. Ed.*, 2005, **117**, 1753.

Keywords: anomalous diffraction, resonant scattering, zeolites

MS18.25.4

Acta Cryst. (2005). A**61**, C29

Structural Characterisation of Encapsulated Nanoparticles Inside Mesoporous MCM-48 with XRD, TEM and EXAFS

Bernd Marler, H. Gies, M. Bandyophadyay, O. Korsak, A. Birkner[a], K. V. Klementiev[b], M. W. E. van den Berg[b], W. Grünert[b], *Institut für Geologie, Mineralogie und Geophysik,* [a]*Lehrstuhl Physikalische Chemie I ,* [b]*Lehrstuhl Technische Chemie, Ruhr-Universität Bochum, 44780 Bochum, Germany.* E-mail: bernd.marler@rub.de

Mesoporous silica-MCM-48 has been synthesized making use of the liquid crystal templating technique. In the synthesized form the organic amphiphil occupies the pores of the silica framework. Because of the amorphous structure of silica in the framework wall, diffraction experiments only show the periodic order of the wall/pore system, i.e. the diffraction contrast, which has cubic symmetry and has been assigned to space group I a3d. Depending on the wall thickness and the periodicity of the silica framework diffraction signals only up to 8 °2θ for copper radiation can be observed. After calcination the pore space becomes open and accessible for sorbate molecules.

Using dip impregnation methods metal organic salts have been introduced inside the pore system of MCM-48 which is ca. 30Å in diameter. Subsequent calcination has transformed the salt into the oxide and furthermore, the oxides were reduced to the elementary metal. The contribution will discuss the structural characterization of ZnO, CuO, Cu, TiO_2, and Au deposited as nanoparticles inside the pore system using TEM, XAS, and PXRD and discuss their specific nature.

Keywords: mesoporous MCM-48, nanoparticles, EXAFS

MS18.25.5

Acta Cryst. (2005). A**61**, C29

Zeolite and MCM Nano- and Mesoporous Structures

Oleg I. Lebedev, D. Liang, G. Van Tendeloo, *EMAT, University of Antwerp, Groenenborgerlaan 171, B-2020, Antwerpen, Belgium.* E-mail: oleg.lebedev@ua.ac.be

Ordered micro- and mesoporous materials are well known for their catalytic and highly selective adsorbent properties. However they also offer a wealth of possibilities for creating materials with additional functionality.

Detailed X-ray diffraction interpretation of mesoporous materials is hampered by their large unit cell, the variable stacking and the limited crystal size. Electron microscopy combined with electron diffraction is therefore a more powerful technique to determine the structure of mesoporous materials on a local scale.

A crystalline silica material "Zeotile", with two levels of porosity and a clear structural order can be produced by tiling nanoslabs with the Silicalite-1 structure type. They can be tiled in various ways into materials with a well-defined mesoporosity. Microscopy of Zeotile-1 shows two types of pores: hexagonal and triangular with sides of 2.0 nm and 2.6 nm, respectively. In Zeotile-1, nanoslabs are forced into face sharing, double units, and then linked to form a pattern with hexagonal symmetry. Zeotile-2 is built from very similar (double) units as Zeotile-1, but has a body centred cubic symmetry (SG Ia$\bar{3}$d). Hexagonal MCM-41 can be turned into cubic MCM-48 and finally into spherical particles (SSP) by the addition of alcohol to the synthesis of a mesoporous silica material. XRD suggests that the structure of these spherical particles is of the MCM-41 type. However, TEM reveals that the structure of mesoporous SSP consists of a core in the form of a truncated octahedron with the MCM-48 cubic structure and radial pores grown on the surfaces of the truncated octahedron. Spherical MCM particles therefore have a mixture of cubic and hexagonally arranged pores.

We would like to acknowledge S. Kremer, C. Kirschhock, J. Martens (K.U. Leuven) and P. Cool, E. Vansant (U. Antwerp) for their scientific collaboration.

Keywords: zeotile, mesoporous materials, electron microscopy

MS19 NEWS FROM INCOMMENSURATE CRYSTALS

Chairpersons: Sander Van Smaalen, Sven Lidin

MS19.25.1

Acta Cryst. (2005). A**61**, C29-C30

Nowotny Chimney Ladders and Giant Cubic Structures: Electron Driven Interfaces

Daniel Fredrickson[a], Stephen Lee[a], Roald Hoffmann[a], Joshua Teal Schmidt[a], Junliang Sun[a], [a]*Department of Chemistry and Chemical Biology, Cornell University.* E-mail: dcf22@cornell.edu

Sometimes intermetallic phases can adopt bewildering structures with hundreds or thousands of atoms per unit cell, or no detectable 3-dimensional periodicity. Using quantum mechanical calculations as a guide to view these structures, we will see one reason that such structures form: many complex intermetallics are, at heart, simple structures that have made room for extra electrons through the formation of interfaces. The Nowotny chimney ladder phases are an example: these are beautiful examples of two component composite crystals, in which the two incommensurable components form helical motifs. Electronic structure calculations reveal that the complex chimney ladder structures each chimney ladder consists of slabs of $TiSi_2$ structure type. The interfaces between the slabs act as electron sinks to achieve electron counts optimal to the $TiSi_2$ structure. The same thing happens in giant cubic intermetallic phases based on Friauf polyhedra, such as $NaCd_2$. This phase the $NaCd_2$ structure, with over 1000 atoms per unit cell, is also built from blocks cut from a simple structure type, this time the $MgCu_2$ type. Extra electrons are accommodated by the interfaces between MgCu2-type blocks. While in the chimney ladders, the blocks are separated by planar interfaces, the interfaces in $NaCd_2$ form a minimal surface, the P surface.

Keywords: intermetallic structures, electronic structure calculations, aperiodicity

MS19.25.2

Acta Cryst. (2005). A**61**, C30

Ordering as Occupational Modulations, and their Superspace Description

J. Manuel Perez-Mato, L. Elcoro, *Dept. de Fisica de la Materia Condensada, Facultad de Ciencia y Tecnología, Universidad del Pais Vasco, Apdo. 644, 48080 Bilbao, Spain.* E-mail: wmppemam@lg.ehu.es

A new perspective in the field of modulated structures has emerged with the use of so-called *crenel* occupational functions in one-dimensional modulations. These functions limit the atomic occupation to a certain interval along the internal space and, as in quasicrystals, we can talk of atomic domains. The concept of average structure becomes ambiguous and, in general, it is possible to define alternative average periodicities, the division of diffraction peaks between main and satellite reflections being not unique. Also, a clear borderline between composite and single modulated structure disappears. The use of crenel functions allows a very efficient unified description of homologous series of mixed layered compounds. Their composition dependent layer stacking sequences can be understood as *uniform* sequences, which order a minority motif as uniformly as possible. This uniform orderings are reflected in superspace into a so-called *closeness* condition of the corresponding atomic domains, forcing a direct relation between the modulation wave vector and the size of the domains, and therefore, composition. This *closeness* condition, earlier proposed in another context for quasicrystals, is a universal feature of these mixed layered compounds. We will show that it can also be used in (3+2)d superspace to obtain and describe uniform ordering schemes within two-dimensional lattices. Two-dimensional ordered patterns of vacancies observed in various materials can be explained within this framework.

Keywords: superspace, ordering, modulated structures

MS19.25.3

Acta Cryst. (2005). A**61**, C30

Flexible Local Crystal Chemistry and its (modulated) Consequences

Ray Withers[a], Frank Brink[a], Lasse Norén[a], Yun Liu[a], [a]*Research School of Chemistry, Australian National University, Canberra, Australia.* E-mail: withers@rsc.anu.edu.au

There exists an extraordinarily broad range of compositionally and/or displacively flexible "modulated" crystalline materials (displacively flexible framework structures, materials susceptible to Fermi surface driven structural instabilities, substitutionally disordered solid solutions *etc*) whose reciprocal spaces at one temperature or the other exhibit either sharp satellite reflections and/or highly structured diffuse intensity distributions accompanying the strong Bragg reflections of an underlying, well-defined average structure. The existence of such additional scattering reflects strong local crystal chemical "rules" underlying, for example, the correlated rotations of neighbouring polyhedra in flexible framework structures, the local distribution of dopant ions in substitutionally disordered systems or the correlated displacements of neighbouring ions induced by temperature-dependent, charge density wave (CDW) type structural instabilities. The local crystal chemistry as well as the associated physico-chemical properties of such flexible phases can not be understood until such modulations are recognized and properly taken into account. The results of recent structural investigations of some flexible framework structures [1], of O/F ordering in transition metal oxyfluorides [2] and of the low temperature Kondo effect in ThAsSe [3] will be used to highlight these different types of local flexibility and their structural consequences.

[1] Liu Y., Withers R.L., *J. Solid State Chem.*, 2003, **172**, 431. [2] Brink F.J., Norén L., Withers R.L., *J. Solid State Chem.*, 2004, **177**, 2177. [3] Withers R.L., Vincent R., Schoenes J., *J. Solid State Chem.*, 2004, **177**, 701.

Keywords: modulated structures, adaptive systems, order-disorder

MS19.25.4

Acta Cryst. (2005). A**61**, C30

Incommensurate Crystallographic Shear Structure of $Ba_xBi_{2-2x}Ti_{4-x}O_{11-4x}$ (x=0.275)

Yuichi Michiue[a], Akiji Yamamoto[a], Mitsuko Onoda[a], Akira Sato[a], Takaya Akashi[b], Hisanori Yamane[c], Takashi Goto[d], [a]*National Institute for Materials Science, Tsukuba, Japan.* [b]*Graduate School of Engineering, Hokkaido University, Sapporo, Japan.* [c]*Center for Interdisciplinary Research, Tohoku University, Sendai, Japan.* [d]*Institute for Materials Research, Tohoku University, Sendai, Japan.* E-mail: MICHIUE.Yuichi@nims.go.jp

The 4d structure of the title compound consists of atoms with sawtooth-like modulation functions and step-like occupation functions (occupation domains, OD). Most of the structural parameters defining OD are concerned with γ (=0.36693), the **c*** component of the modulation wavevector. The 3d structure is closely related to the β-$Bi_2Ti_4O_{11}$ structure, where the linkage of TiO_6 coordination octahedra constructs the host framework providing the one-dimensional tunnel-like space for the accommodation of Bi ions. Domain boundaries are introduced by a kind of the crystallographic shear (CS) operation in the present structure. Namely, the layer unit consisting of $Bi_2Ti_2O_8^{2-}$ is removed from the β-$Bi_2Ti_4O_{11}$ structure, and remained blocks are displaced to fill the gap. The negative charge of the removed unit is compensated by the substitution of Ba^{2+} ions for Bi^{3+} ions in tunnels.

The unique character of this incommensurate structure is the aperiodic insertion of domain boundaries in contrast to usual (*i.e.* commensurate) CS structures. To the best of our knowledge, the present study is the first example of the quantitative analysis of the incommensurate CS structure.

Keywords: incommensurate structures, higher-dimensional structure analysis, crystallographic shear structures

MS19.25.5

Acta Cryst. (2005). A**61**, C30

Solving Modulated Crystals of Profilin:Actin

Gloria Borgstahl[a], C. Murphy[a], J. Lovelace[a], K. Narayan[b], C. Svensson[c], U. Lindberg[d], C. Schutt, [a]*Eppley Institute.* [b]*Princeton University.* [c]*University of Lund.* [d]*Stockholm University.* E-mail: gborgstahl@unmc.edu

Cellular motility, regulated through cytoplasmic profilin:actin (PA) interactions, is intrinsic to many cellular functions. Profilin both sequesters actin monomers and delivers it to filamentous assemblies. Detailed structural information on monomeric actin in complexes with various actin binding proteins have been provided by X-ray crystallography, but currently no atomic structures for filamentous actin have been determined, although several hypothetical models have been proposed. PA crystals retain the dynamic nature of actin and provide an excellent way to study the protein-protein interactions involved in filament formation. When exposed to conditions known to promote actin filament formation, PA crystals can be transformed into a modulated state characterized by unusual off-lattice satellite reflections. Evidence suggests the presence of a modulated or periodic structure that is occupied by metastable actin filaments. Methods to determine modulated structures are known to "chemical" crystallographers, yet macromolecular crystallographers have, to date, mostly avoided this class of crystal structures. The biological importance of these challenging modulated crystals of PA has prompted the development of methods to solve the underlying atomic structure(s). Progress on this structure determination will be presented.

Keywords: macromolecule, incommensurate, modulation