

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

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Direct Localization of Atoms in Nanoporous Powders by Resonant Contrast Diffraction

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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

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

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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. 30 Å 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, TiO₂, 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

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Zeolite and MCM Nano- and Mesoporous Structures

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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 “Zeolite”, 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 Zeolite-1 shows two types of pores: hexagonal and triangular with sides of 2.0 nm and 2.6 nm, respectively. In Zeolite-1, nanoslabs are forced into face sharing, double units, and then linked to form a pattern with hexagonal symmetry. Zeolite-2 is built from very similar (double) units as Zeolite-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.

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Keywords: zeolite, mesoporous materials, electron microscopy

MS19 NEWS FROM INCOMMENSURATE CRYSTALS

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Nowotny Chimney Ladders and Giant Cubic Structures: Electron Driven Interfaces

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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 TiSi₂ structure type. The interfaces between the slabs act as electron sinks to achieve electron counts optimal to the TiSi₂ structure. The same thing happens in giant cubic intermetallic phases based on Friedel polyhedra, such as NaCd₂. This phase the NaCd₂ structure, with over 1000 atoms per unit cell, is also built from blocks cut from a simple structure type, this time the MgCu₂ type. Extra electrons are accommodated by the interfaces between MgCu₂-type blocks. While