

MS28 STRUCTURE/PROPERTIES RELATIONSHIPS OF TECHNOLOGICALLY RELEVANT INORGANIC AND MINERAL COMPOUNDS**Chairpersons:** João Rocha, Alessandro Gualtieri**MS28.25.1***Acta Cryst.* (2005). A61, C40**Chrysotile, a Naturally Occurring Nanotube Material****Bernard Grobety**, *Department of Geosciences, University of Fribourg, Fribourg, Switzerland.* E-mail: bernard.grobety@unifr.ch

The potential of nanosized tube-shaped materials range from the use as wires, wire-templates, microreactors, hydraulic tubes, gas storage devices and optical devices among others. A naturally occurring, nanosized, tube-shaped phase is the silicate mineral chrysotile, $Mg_3Si_2O_5(OH)_4$. The structure of chrysotile consists of enroled sheets composed of a layer of tetrahedrally coordinated silicon cations and a layer of octahedrally coordinated magnesium/aluminum cations. It is one of the rare natural materials with five-fold symmetry.

Chrysotile is common in metamorphosed ultramafic rocks and easy to extract in large quantities. Like for carbon nanotubes, different enrollment schemes (cylindrical, helical etc.) are known. The chrysotile nanotubes differ from the carbon nanotubes in some important physical parameters e.g. they are non-conducting and their length can reach the cm-range.

A drawback for the application of these natural nanotubes is, that chrysotile is carcinogenic. It is one of the minerals that are classified as asbestos material. Research is, therefore, oriented in different directions: 1. the study of the physical and chemical properties of chrysotile in order to tailor replacement materials (membranes used in water electrolysis, fiber enforced concrete etc.), 2. the behavior of chrysotile fibers in the environment and during the handling of asbestos containing materials, 3. the search of new applications for chrysotile nanotubes, requiring only small quantities. The structure of chrysotile and projects in all three research fields will be presented.

Keywords: nanotube, asbestos, five-fold symmetry**MS28.25.2***Acta Cryst.* (2005). A61, C40**Solid State NMR Crystallography of Inorganic Materials****Francis Taulelle**, *Tectospin, Institut Lavoisier, University of Versailles, Versailles, France.* E-mail: taulelle@chimie.uvsq.fr

Solving structures on polycrystalline powders is still today a challenge that has many demands: intensity of the diffraction signal, prior knowledge and a reasonable structural models. It has been shown that in solution, NMR can solve a structure in the sense of a molecular structure. Solving structures of inorganic compounds or organic-inorganic crystals requires profoundly different strategies to what has been done up to now by NMR.

The principles of structure determination has therefore been reinvestigated, with special emphasis to space group determination [1] to unit cell estimation by NMR. Actually the principles of NMR crystallography on powders will be explained, with a special focus on the use of Wyckoff spectra. Once those principles exposed, we will show how we can combine efficiently NMR, diffraction and modeling [2] to propose proper structure elucidation and therefore new strategies for solving the many cases where crystals can not easily be obtained.

Aluminophosphate structure elucidation, solved by combination of modeling diffraction and solid state NMR, on powders will be exemplify this strategy. Furthering the discussion on such strategies will lead us to engage along the polymorphism analysis by solid state NMR, a key area in many pharmaceutical applications.

[1] Taulelle F., *Solid State. Sc.*, 2004, **6**, 1053. [2] Dutour J., Guillou N., Huguenard C., Taulelle F., Mellot-Draznieks C., Férey G., *Solid State. Sc.*, 2004, **6**, 1059.

Keywords: NMR of solids, NMR Crystallography, Polymorphism**MS28.25.3***Acta Cryst.* (2005). A61, C40**Combined XRPD and Spectroscopic Study of Molecules Hosted in Zeolitic Channels****Carlo Lamberti**^a, Carmelo Prestipino^a, Marco Milanese^b, Gianluigi Marra^c, Andy N. Fitch^d, Gilberto Artioli^e, ^a*Dip. Chimica IFM, Turin (I).* ^b*Dip. di Scienze e Tecnologie Avanzate, Alesandria, (I).* ^c*Polimeri Europa S.p.A., Novara (I).* ^d*ESRF, Grenoble (F).* ^e*Dip. di Scienze della Terra, Milan, (I).* E-mail: carlo.lamberti@unito.it

Zeolites are tridimensional organized microporous systems hosting positively charged cations balancing the negative charge of the framework and acting as strong Lewis acid centres. Such materials play a great role in industrial chemistry, as they are very efficient catalysts exhibiting remarkable shape selectivity. The pore dimension and topology select the reactants molecules able to penetrate the framework and to reach the internal active sites. The same holds for the intermediate molecules formed on the sites and for the final products able to leave the zeolite framework that actually acts as a nanoreactor of molecular dimension. The understanding of the site reactivity can be achieved by comparing the cations position in the activated catalyst (zeolites subjected to thermal treatments in vacuo conditions, exhibiting no molecules in the channels) with that occupied upon interaction with selected molecules dosed in situ either from the gas or from the liquid phases. The combination of high resolution XRPD data (collected in situ at the ID31 instrument of the ESRF) with XAFS, IR and Raman spectroscopies is a complete approach to face this complex problem, see e.g. [1-3].

[1] Marra G. L. et al., *J. Phys. Chem. B*, 1997, **101**, 10653. [2] Turnes Palomino G. et al., *J. Phys. Chem. B*, 2000, **104**, 8641. [3] Lamberti C. et al., *Nucl. Instr. Meth. B*, 2003, **200**, 155.

Keywords: Zeolite reactivity, in situ XRPD, in situ spectroscopy**MS28.25.4***Acta Cryst.* (2005). A61, C40**Diffraction Study of the Semiconducting $Cu_2(Se,Te)_3-(Ga,In)_2(Se,Te)_3$ Systems****Jose Miguel Delgado**^a, J. Contreras^a, G. Marín^b, S.M. Wasim^b, C. Rincón^b, ^a*Laboratorio Nacional de Difracción de Rayos-X.* ^b*Centro de Estudios de Semiconductores, Facultad de Ciencias, Universidad de Los Andes, Mérida 5101, Venezuela.* E-mail: miguel@ula.ve

Many contributions have been reported in the literature about the synthesis and characterization of chalcopyrite-related In- and Ga-rich ordered defect compounds of the ternary systems Cu-In-Se, Cu-In-Te, Cu-Ga-Se and Cu-Ga-Te. Particular interest has been paid to the compounds that can be derived from the formula $Cu_{N-3}In_{N+1}Se_{2N}$, where N=4, 5, 6, 7, 8 and 9, because some of these materials have been already used in important solar cell applications. In the present work some of the most important structural aspects of these materials will be discussed. Among others, the different structural models reported for some of the phases (including our own). The difference in the dimensions of the unit cell (unit cell parameters and volume) among these materials can be rationalized based on the fraction of cation vacancies and the amount of interacting donor-acceptor defect pairs [$In_{(Cu)}^{2+}$, $2V_{(Cu)}^{-1}$] per unit formula. The structural studies carried out in several phases prepared by the Bridgman technique in a multi-zone vertical furnace were performed using powder diffraction data collected with a SIEMENS D-5005 diffractometer.

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Keywords: powder X-ray diffraction, semiconductors, solar energy conversion