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Keywords: implantation, X-ray diffraction, amorphous scattering

MS82.29.3

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Direct Observation of a H_2 Molecule Swallowed by Open-mouthed C_{60}

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Various types of endohedral fullerene complexes are known to date. However the metallofullerenes are generally produced by arc-discharge method, but the use of such extremely drastic conditions is apparently not suitable for encapsulation of unstable molecules or gases. We recently succeeded in incorporation of a H_2 molecule in 100% into a derivative of an open-cage C60 [1]. In order to observe the endohedral H_2 molecule directly, the X-ray diffraction analysis using synchrotron radiation were carried out. We observed a single H_2 molecule encapsulated in fullerene cage using structure analysis and maximum entropy method [2]. This H_2 molecule is floating inside of the hollow cavity and considered to be completely isolated from the outside (Fig.1).

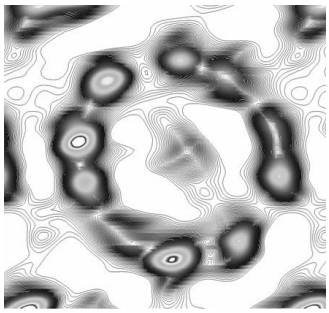

Fig.1. The MEM electronic density distributions of H2 endohedral open-cage C60.

[1] a) Murata Y., Murata M., Komatsu K., *J. Am. Chem. Soc*., 2003, **125**, 7152-7153; b) Murata Y., Murata M., Komatsu K., *Chem. Eur. J*., 2003, **9**, 1600-1609. [2] Sawa H., Wakabayashi Y., Murata Y., Murata M., Komatsu K., *Angew. Chemi.*, 2005, 13.

Keywords: fullerenes, synchrotron X-ray diffraction, single-crystal structure analysis

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Anomalous X-ray Scattering Methods for Structure Investigations of Semiconductor Nanostructures

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X-ray study of the structure of semiconductor nanostructures (quantum dots and wires) is a challenging task. Several methods have been developed for the determination of shape and local chemical composition of quantum dots and wires (see [1] for a review); the methods are based on a surface-sensitive scattering geometry, where the penetration depth of the incoming x-ray beam is limited by a very small incidence angle. Usually, a direct determination of the structure from the measured data is not possible; instead, the experimental data are fitted to a suitable structure model.

An anomalous x-ray scattering experiment uses two different x-ray energies close to and far away from the absorption edge of a selected element [2]. By comparing the data obtained at these energies one can determine the local chemical composition in the nanostructures without using any model *a-priori*. Moreover, measuring the energy dependence of the diffracted intensity close to the absorption edge (diffraction anomalous fine structure – DAFS) one can determine the local atomic ordering in the nanostrucures [3].

Several examples of anomalous scattering and DAFS experiments will be presented and the limits of these methods will be discussed.

[1] Stangl J., et al., *Rev. Mod. Phys.*, 2004, **76**, 725. [2] Schuelli T.U., et al, *Phys. Rev. Lett.*, 2003, **90**, 066105. [3] Letoublon A., et al., *Phys. Rev. Lett.*, 2004, **92**, 186101.

Keywords: nanostructures, surface X-ray scattering, anomalous scattering

MS82.29.5

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Magnetic Imaging of Biquadratic Coupling in Ferromagnetic Bilayers*

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Physical properties of thin magnetic nanostructures are dominated by exchange interactions between the layers. These interactions in most cases induce collinear coupling of spins. The much smaller noncollinear coupling of spins is also allowed through the biquadratic term in the exchange Hamiltonian. Recently, Vlasko-Vlasov et al. [1] observed unusual coupling of two ferromagnetic layers in contact. They studied junctions of SmCo and Fe layers and deduced noncollinear magnetic configurations based on magneto-optical imaging of the top Fe layer. To simultaneously probe the magnetization in the surface Fe layer and in the buried SmCo layer, we used circularly polarized synchrotron radiation. Element-specific hysteresis loops were performed by tuning the energy of the synchrotron radiation to the absorption edges of Fe (7.110 keV) and Sm (6.710 keV). In addition, fluorescence imaging of magnetic domains was performed by using focused circularly polarized x-rays (1 μm by 1 μm). Hysteresis and imaging data unequivocally demonstrated that the Sm and Fe magnetizations were perpendicularly coupled. *This work is supported by the U.S. DOE, Office of Science under Contract No. W-31-109-ENG-38.

[1] Vlasov V.K., Welp U., Jiang J.S., et al., *Phys.Rev.Lett*., 2001,**86**, 4386.

Keywords: magnetic structures, synchrotron X-rays, X-ray imaging

MS83 COMPUTATIONAL PROBLEMS AND SOLUTIONS FOR APERIODIC CRYSTALS

Chairpersons: Christer Svensson, Vincent Favre-Nicolin

MS83.29.1

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Towards the Routine Application of Computing System Jana2000

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The program Jana2000 [1] was originally written as a special refinement tool for modulated structures. During almost 20 years of development it integrated many crystallographic methods that gave the program exceptional flexibility. On the other hand, it has been always difficult keeping all the possibilities accessible not only for specialists, but for everybody interested in solution of a modulated structure. With progress in the experimental methods the number of modulated structures grows amazingly. Many of them can be solved relatively easily using just the basic knowledge about aperiodic crystals.

We anticipate this trend by creating set of wizards for guiding the user to solution of simple structures, keeping - of course - all possibilities opened to complicated cases. This development is far from completion but important steps have been already done. First of all - unified tools are available for standard and modulated structures and also for powder and single crystal data that allow solving the basic and modulated structure using the same program. The phase problem (in three dimensions) can be resolved by rendering the data to SIR or EXPO and reading back the results. Automatic symmetry determination is possible for space as well as super space groups. Adding of hydrogen atoms (and generally defining geometry constraints for standard and modulated structures) has been automated to the extent usual in routine crystallography. In the lecture we shall present the outlined methods and envisaged features of Jana2005.

[1] http://www-xray.fzu.cz/jana/Jana2000/jana.html
Keywords: modulated structures, aperiodic crystals, Jana2000

MS83.29.2
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Modulation Functions of Aperiodic Crystals Determined by the Maximum Entropy Method
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The Maximum Entropy Method (MEM) can be used for the reconstruction of the electron density in the unit cell from phased structure factors [1]. The maximum entropy density map (ρ^{MEM}) may provide information about disorder and anharmonic temperature movements. If an informative prior density is employed, ρ^{MEM} may be used to derive the electron density in the chemical bond.

The MEM can also be applied to the electron density in the *n*-dimensional (*n*D) unit cell of the superspace description of aperiodic crystals [2]. We have thus shown that ρ^{MEM} in superspace may provide a model-independent, quantitatively correct determination of the shapes of the modulation functions of incommensurately modulated crystals and incommensurate composite crystals [3]. All procedures have been implemented into a computer program BayMEM, that can be used for a Maximum Entropy analysis of both periodic and aperiodic crystals. In this contribution the features and problems of the MEM will be discussed, and selected applications of BayMEM will be presented [4].

[1] Gilmore C.J., *Acta Cryst.*,1996, A**52**, 561-589. [2] Weber S., Yamamoto, A., *Phil. Mag.*, 1997, A**76**, 85-106. [3] van Smaalen S., Palatinus L., Schneider M., *Acta Cryst.*, 2003, A**59**, 459-469. [4] Palatinus L., van Smaalen S., *Z. Kristallogr.*, 2004, **219**, 719-729.
Keywords: maximum entropy method, aperiodic materials, aperiodic crystals

MS83.29.3
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***Ab Initio* Solution of Incommensurately Modulated Structures by Charge Flipping**
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"Charge flipping" is an algorithm for *ab initio* structure solution from X-ray diffraction data, which is based on alternating modifications of a trial electron density in direct and reciprocal space [1]. This algorithm makes no use of the atomicity of the chemical structure. It is instead based on the observation that the electron density of a crystal consists of a small number of high-density areas separated by large areas with very small electron density. This property is common to both electron densities of periodic structures and superspace electron densities of modulated structures. Therefore, charge flipping can be generalized towards superspace and used to reconstruct the superspace electron densities [2].

Up to now the structure solution of incommensurately modulated structures has been a two-step procedure involving solution of the basic periodic structure followed by the determination of the modulation. Charge flipping offers for the first time the possibility to solve the modulated structures directly in superspace, avoiding the often tedious task to construct and refine the basic structure.

Charge flipping was successfully applied to solve several modulated crystal structures including several organic and organometallic compounds with complex modulations [2].

[1] Oszlányi G., Sütő A., *Acta Cryst.*, 2004, **A60**, 134. [2] Palatinus L., *Acta Cryst*.,2004, **A60**, 604.
Keywords: ab initio structure determination, incommensurate structures, superspace electron density

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Pyrrhotites Revisited in Superspace with *ab-initio* Calculations Insights
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Superspace formalism has been used to describe families of compounds with flexible composition [1] with a unique structural model. A superspace model for the Pyrrhotite (Fe1-xS) family is proposed in this work.

Pyrrhotites have metal-deficient NiAs structures. S atoms form a hexagonal sublattice with Fe atoms in the octahedral interstices. Fe-deficient layers alternate regularly with full Fe layers to form superstructures with different composition, symmetry and c parameter [2].

In the common superspace construction, the atomic domains representing the Fe and S atoms are crenel functions. The only parameters which change with the composition are the size of the crenels for the Fe atoms and the modulation weave vector. The structure of a particular composition is obtained through a 3D cut of the superspace construction. For different compositions, distinct stacking sequences are obtained, in agreement with the reported 3D structures.

Ab-initio calculations have been used to study the stability of the different stacking sequences.

[1] Pérez-Mato J.M., Zakhour-Nakhl M., Weill and Darriet F., *J. Mat. Chem.* , 1999, **9**, 2795. [2] Yamamoto A., Nakazawa H., *Acta Cryst.*, 1982, **A38**, 79.
Keywords: aperiodic structures, layered systems, ab-initio calculations

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New Tool based on the Superspace Concept to discover Structure Relations
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Relations between crystal structures imply relations between their space groups, which can be expressed by group-subgroup relations. Recently, the superspace formalism has been extensively used for the unified description of sets of commensurate structures [1, 2]. The method consists of finding possible structures by identifying their space group symmetry from rational cuts in superspace. For practical purpose, the inverse problem is more useful but requires a large amount of compilation.

We constructed a complete tree linking (3+1)- and the corresponding 3-dimensional space groups derived by rational cuts. The aim is to discover possible (and impossible) space group chains for structural relations and phase transitions. The corresponding tree for (3+2)-dimensional groups will soon be completed.

A database providing the subgroup-supergroup relations is available at http://lcr.epfl.ch/page55041.html

This tool is particularly helpful in finding common superspace denominators for various series of modular ('composition-flexible') structures. We shall illustrate the use of the database with a few examples drawn from various fields of crystal chemistry.

[1] Perez-Mato J. M., Zakhour-Nakhl M., et al., *Journal of Materials Chemistry*, 1999, **9(11)**, 2795-2808. [2] Zakhour-Nakhl M., Darriet J., et al., *International Journal of Inorganic Materials*, 2000, **2(6)**, 503-512.
Keywords: superspace symmetry, composition-flexible structures, modular structures