MS09 Combined XAS and XRD Techniques in Physics Chemistry and Materials Science
Chairpersons: Settimio Mobilio, Joaquin Garcia-Ruiz

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XAS and XRD: Complementary Tools to Explore Matter under Extreme Pressure
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The study of matter under extreme conditions introduces strong experimental constraints that, in many cases, ask for the use of complementary techniques for structural characterization. These constraints can include the presence of a complex sample environment with different absorbing and/or scattering elements, a reduced k-space domain or the presence of non isotropic constraints.

I will present here a condensed review of examples where the successful combination of X-ray absorption spectroscopy and X-ray diffraction has contributed to the understanding of the structure and physical properties of matter under extreme conditions of pressure and/or temperature.

Emphasis will be given to those cases where the actual local structure is different from that obtained from the long range order structural analysis and/or when crystallography does not succeed in getting the full structure of a system.

Examples will range from simple molecular crystals with only 4 atoms in the unit cell to the silicon clathrate case with more than fifty atoms in the Bravais lattice. The study of the local compressibility combining both techniques constitutes a further example of the complementarity of both techniques that will be illustrated in the case of pseudobinary alloys and lamellar systems.

Keywords: high pressure research, absorption spectroscopy experimental, high-pressure X-ray diffraction

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Element- and Site-specific Study of the Atomic Origin of Magnetic Hardness in Modern Magnets*
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We combined resonant diffraction and absorption of circularly polarized x-rays to probe the atomic origins of magnetic hardness, or coercivity, in permanent magnetic materials. Modern permanent magnets gain both intrinsic stability against demagnetizing fields and large magnetization through alloying of rare-earth and transition metal ions. The resultant complex crystal structures not only feature more than one magnetic element type but also elements of the same species in inequivalent crystal sites, making it difficult for even state-of-the-art probes of magnetism to pinpoint the atomic origins of the desirable magnetic properties of these materials. The element specificity of x-ray magnetic circular dichroism, combined with the site selectivity of resonant magnetic diffraction allow for a more thorough understanding of the rare-earth role. We show that the magnetic hardness of currently the best permanent magnet has its atomic origin predominately at one of the two inequivalent Nd crystal sites.

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Keywords: resonant diffraction, xmcd, permanent magnets

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X-rays to Elucidate the Structure of Catalysts: Probing the Local and the Crystalline Structure by XAS and XRD
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Both X-ray diffraction and X-ray absorption spectroscopy are widely used in heterogeneous catalysis, since the synthesis of heterogeneous catalysts often aims at high surface area materials. Hence, the materials are often at least partly X-ray amorphous and X-ray absorption spectroscopy is an ideal complementary tool to XRD to monitor the local structure. Recent examples in the field of heterogeneous catalysis and the field of nanomaterials are discussed, where novel synthesis methods, such as flame spray pyrolysis, were used to produce high surface area materials.

An important advantage of the use of X-rays is that the solid catalysts can be studied in situ. Recently, we studied the formation of MoO_3 nanorods from $MoO_3{\cdot}2H_2O$ by XAS, monitoring both the liquid phase and the solid phase. XAS uncovered the formation of soluble species as soon as the transformation to MoO_3 started. No evidence for an intermediate product was found, which is supported by in situ XRD results. Another key field is the identification of the active species under reaction conditions [2-4]. Also here X-ray based techniques are powerful, as will be illustrated using recent examples.

[1] Michailovski A., Grunwaldt J.-D., Baiker A., Kiebach R., Bensch W., Patzke G.R., *submitted.* [2] Clausen B. S., Topsøe H., Frahm R., *Adv. in Catal.*, 1998, **42**, 315. [3] Sankar G., Thomas J. M., Catlow C. R. A., *Topics Catal.*, 2000, **10**, 255. [4] Grunwaldt J.-D., Clausen B. S., *Topics Catal.*, 2002, **18**, 37.
Keywords: EXAFS, XRD, catalysis

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Structural Properties of Semiconductor Nanostructures Determined via X-ray Anomalous Diffraction (DAFS) and Absorption (EXAFS)
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We will report novel results on the structural properties of encapsulated semiconductor nanostructures obtained by an ultimate application of anomalous diffraction combined with ray absorption spectroscopy. We have been studying InAs/InP Quantum Sticks (QSs) and GaN/AlN Quantum Dots (QDs). Reciprocal space mapping and fixed-Q anomalous diffraction, mesured as a function of energy, in grazing incidence, gives access to the the partial structure factor of the embedded nanostructures (QSs or QDs), allowing to determine their size, strain and atomic composition. Quantitative analysis of the Grazing Incidence Diffraction Anomalous Fine Structure (GIDAFS) oscillations above the resonant edges, gives strain accommodation and coordination at a local atomic scale for the diffraction-selected iso-strain region inside the nanostructures. On the other hand EXAFS measurements provide a comparison with the average atomic environment in the whole island. These methods have been applied succesfully both to InAs sticks and to the GaN dots.

[1] Létoublon A., et al., *Phys. Rev. Lett.*,2004, **92**, 186101. [2] Létoublon A., et al., *Physica B*, 2005, **357/1-2**, 11-15.
Keywords: nanostructures, EXAFS, GIDAFS

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The Power of the Multi-disciplinary Approaches to the Study of Minor and Trace Element Incorporation in Geo- and Technological Materials
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Much of our knowledge of the origin and differentiation of the terrestrial planets arises from the study of trace elements behaviour.