of this metal. There is increasing interest in pressure acid leaching (PAL) as a means of extracting nickel from laterites. PAL involves leaching of laterites in sulphuric acid under hydrothermal conditions, typically 250°C and 45 atmospheres pressure.

The saprolitic component of the ore is known to undergo rapid changes upon cooling following PAL thus making it difficult to examine using traditional post-mortem techniques. Time resolved, *in-situ*, X-ray diffraction (XRD) studies have been carried out into the reaction mechanisms of this process. The sample environment during this study aimed to closely emulate the conditions used in industrial processing plants. The novel experimental set-up used a capillary reaction vessel, short wavelength radiation and a position sensitive detector to enable rapid, simultaneous collection of a wide range of diffraction data. Quantification of the data via the Rietveld method has allowed the derivation of reaction mechanisms and kinetics.

This paper will present the results of both laboratory and synchrotron experiments within this system and will discuss the practice and perils of *in-situ* experimentation in general.

Keywords: in-situ time-resolved powder diffraction, process kinetics, quantitative phase analysis

MS81.29.4

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***In-situ* Measurement of Cation Ordering in Electroceramics**

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Electroceramics are critical elements in microwave devices widely used in communications equipment and a full understanding of their crystal chemistry is fundamental to future development. High temperature processing is crucial to the performance of these oxide materials in applications, due to the resulting control over both atomic scale structure and domain size over which the ordered structures persist.

We have developed high resolution neutron and synchrotron powder diffraction methods to study the structural development of commercially-used ceramics such as $Ba_3ZnTa_2O_9$ (BZT) and $Ba_3CoNb_2O_9$ (BCN) under industrial processing conditions[1] at temperatures of up to 1500 ºC on timescales of minutes and in some cases seconds. The studies provide insight to domain growth during processing and quantitative information on order-disorder temperatures and thermodynamic processes affecting B-site cation ordering in the perovskite structures.

[1] Moussa S.M., Ibberson R.M., Bieringer, M., Fitch, A.N., Rosseinsky, M.J. *Chem. Mater.*, 2003, **15**, 2527.

Keywords: in-situ powder diffraction, order-disorder structure, dielectric ceramics

MS81.29.5

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Phase Transitions in Metal Hydrides by *in-situ* Synchrotron Powder Diffraction with High Time-resolution

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In-situ powder diffraction studies of hydrogen absorption/desorption in intermetallic compounds can offer valuable information about their real behaviour. The availability of the microstrip detector at the Materials Science Beamline at the Swiss Light Source, able to collect one full high resolution powder pattern in a very short time (~ 5 seconds), has allowed to follow hydrogen absorption/desorption in a sample in one shot.

A reaction tight cell rated up to 25 bar hydrogen pressure has been especially designed and built. Examples of *in-situ* studies on hydrogen absorbing intermetallic compounds like $LaNi_5$ will be shown. Analysis of the collected diffraction patterns has allowed obtaining the nature and amount of the phases involved during the hydrogen absorption/desorption, the evolution of the lattice parameters, and the anisotropic character of the diffraction line broadening. From these data, the out-of-equilibrium phases have been observed.

Temperature and/or hydrogen desorption induced phase transitions in selected light metal hydrides like $NaAlH_4$, $LiBH_4$ were studied in high-temperature chamber Stoe. High angular resolution of the experimental set-up has allowed characterization of lattice defects involved in the phase transitions.

Examples of hydride structure solution by direct space method (program FOX) will be shown too.

Keywords: metal hydride, time resolved study, powder diffraction

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MS82 X-RAY CHARACTERIZATION OF NANOSTRUCTURES

Chairpersons: Ugo Valbusa, Tilo Baumbach

MS82.29.1

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Quantitive Morphological Characterization of Nanostructure Arrays by scanning Probe Microscopy

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Due to the imaging principle in Scanning Probe Microscopies as Scanning Tunneling Microscopy and Atomic-Force Microscopy (AFM), the recorded image represents a topographical information z(x,y) of the surface under investigation. Provided a well calibrated scanner and a sufficiently sharp probe, the three-dimensional shape and size of nanostructures can be determined with high precision. Besides analysis of individual structures, ensembles of nanostructures can be analyzed with respect to size and separation distributions applying power spectral density analysis of the recorded images. For epitaxial nanostructures with well defined facets, integral information on preferential facet orientations can be obtained by calculating histograms of local surface normals from z(x,y). These procedures will be demonstrated for quantitative analysis of self-organized nanostructure arrays in semiconductor homo- and heteroepitaxy [1,2] as well as for ion-bombardment induced pattern formation [2,3].

[1] Teichert C., *Phys. Rep.*, 2002, **365**, 335. [2] Teichert C., *Appl. Phys.*, 2003, **A 76**, 653. [3] Bobek T., et al., *Phys. Rev.*, 2003, **B 68**, 085324.

Keywords: nanostructures, atomic-force microscopy, self-organization

MS82.29.2

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Ripple Structure of Ion Beam Induced Si Wafers

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Ion beam induced ripple formation in Si wafers was studied by atomic force microscopy (AFM) and non-destructive depth-resolved x-ray grazing incidence diffraction (GID). The formation of a ripple structure at high doses ($7x10^{17}$ ions/cm^2), starting from initiation at low ion beam doses ($1x10^{17}$ ions/cm^2) is evident from AFM, while that in the buried crystalline region below a partially crystalline top layer is evident from GID. GID reveals that these periodically modulated wave-like buried crystalline features become highly regular and strongly correlated as one increases the Ar ion beam energy from 60 keV to 100 keV. The vertical density profile obtained from the analysis of Vineyard profiles shows that the density in the upper top part of the ripples is decreased to about 25% to 35% of the crystalline density. The partially crystalline top layer found at low doses transforms to a completely amorphous layer for high doses. The top morphology was found to be conformal with the underlying crystalline ripple structure. The inspection of the amorphous scattering shows that the amorphous form factor of the damaged top layer is strongly textured in azimuth and scales with the ion dose.

[1] Hazra S., Chini T.K., Sanyal M.K., Grenzer J., Pietsch U., *Phys.Rev.*, 2004, B**70**, 121307(R).

Keywords: implantation, X-ray diffraction, amorphous scattering

MS82.29.3

Acta Cryst. (2005). A**61**, C104

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).

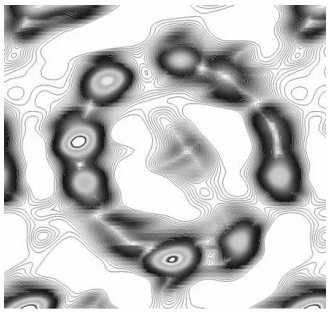

Fig.1. The MEM electronic density distributions of H2 endohedral open-cage C60.

[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

MS82.29.4

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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.