

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

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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<sub>3</sub>ZnTa<sub>2</sub>O<sub>9</sub> (BZT) and Ba<sub>3</sub>CoNb<sub>2</sub>O<sub>9</sub> (BCN) under industrial processing conditions[1] at temperatures of up to 1500 °C 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

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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<sub>5</sub> 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<sub>4</sub>, LiBH<sub>4</sub> 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

#### MS82 X-RAY CHARACTERIZATION OF NANOSTRUCTURES

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#### MS82.29.1

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##### **Quantitative 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

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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 ( $7 \times 10^{17}$  ions/cm<sup>2</sup>), starting from initiation at low ion beam doses ( $1 \times 10^{17}$  ions/cm<sup>2</sup>) 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.