

**MS43-P7** Solid-state reactivity explored *in situ* by synchrotron radiation on single crystals of SrFeO<sub>2.5</sub> during electrochemical oxygen intercalation

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Due to the high performance of modern x-ray and neutron diffractometers, the chemical reactivity of solids is studied today routinely under *in situ* conditions on polycrystalline samples in specific reaction chambers. This technique became the standard characterization not only for battery systems but also for catalysis and many other applications. Following up chemical solid-state reactions on single crystals would be even more powerful, as it would allow to scanning the whole reciprocal space. Recent developments concerning fast and low-noise 2D-detectors, together with a high primary beam quality, especially available at large scale facilities but also via  $\mu$ -focus type x-ray sources using standard laboratory equipment, present a huge potential for the study of solid-state reactions on single crystals in real time. It allows to obtain valuable information about diffuse scattering and weak super-structure reflections, as well as microstructural aspects, partially impossible to access by powder diffraction.

We report here on the electrochemical oxygen intercalation reaction on SrFeO<sub>2.5</sub>, followed up *in situ* by x-ray diffraction in a dedicated, miniaturized electrochemical cell, mounted on the BM01A goniometer. We were especially interested to structurally explore the phase diagram of SrFeO<sub>2.5</sub> at ambient temperature, as it is interesting from a crystal chemistry point of view. We report here on the characterization of intermediate phase products in course of the reaction and related changes in the twin structure. In addition, all involved phases show interesting physical properties, beside their outstanding performance as oxygen membranes in SOFCs for energy storage and conversion.

Reference:

[1] Maity A, Dutta R, Penkala B, Ceretti M, Letrouit-Lebranchu A, Chernyshov D, Perichon A, Piovano A, Bossak A, Meven M and Paulus W 2015 Solid-state reactivity explored *in situ* by synchrotron radiation on single crystals: from SrFeO<sub>2.5</sub> to SrFeO<sub>3</sub> via electrochemical oxygen intercalation *J. Phys. D: Appl. Phys.* **48** 504004

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**MS43-P8** Synchrotron Experiments in Large Volume Presses at High Pressure and High Temperature at different Spinels

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For geoscientists, material scientists, physicists and chemists it is very important to study the samples under extreme conditions. It is necessary to use *in-situ* X-ray diffraction experiments at synchrotron beamlines because of the high intensity and the broad energy range to figure out the stability of minerals under high pressure and temperature, the determination of bulk moduli, the thermal expansion, phase diagrams, and the behaviour of kinetic measurements.

The Large Volume Presses were located at the Hamburger Synchrotron Laboratory (HASYLAB) at the DORIS III storage ring. The experiments were carried out using the high pressure multi anvil devices MAX80 (F2.1) and MAX200x (W2). The F2.1 beamline was a bending magnet beamline with a critical energy of 16.6 keV and an energy range up to 75 keV. The W2 beamline was a hard-wiggler beamline with a critical energy of 26.4 keV and an energy range up to 150 keV. Energy-dispersive X-ray diffraction was used to determine the pressure and temperature induced volume change. Isothermal experiments were performed up to 15 GPa at ambient temperature.

Many spinels show phase transformations under pressure (Funamori et al. 1998 [1], Irfune et al. 2002 [2]) and are used as model structures for deep earth mineralogy and for a better understanding of underlying high-pressure phase transition mechanisms. It is widely accepted that the behavior within the transition zone of the Earth's interior (410-670 km depth) is strongly linked to the properties of minerals showing spinel or pseudospinel structure.

The pressure dependence of the thermal expansion coefficient is examined on three different spinels, while systematically varying the iron contents. All three minerals, Magnetite (Fe<sup>2+</sup>Fe<sup>3+</sup>O<sub>4</sub>), Franklinitite (Zn<sup>2+</sup>Fe<sup>3+</sup>O<sub>4</sub>) and Gahnite (Zn<sup>2+</sup>Al<sup>3+</sup>O<sub>4</sub>) adopt the normal spinel structure. Derived from compression experiments using MAX80 apparatus up to 5 GPa at temperatures of 298, 500, 700, 900 and 1100 K.

[1] Funamori, N., Jeanloz, R., Nguyen, H., Kavner, A., Cadwell, W.A., Fujino, K., Miyajima, N., Shimmei, T., and Tomioka, N. (1998) High-pressure transformation in MgAl<sub>2</sub>O<sub>4</sub>, *Journal of Geophysical Research*, **103**, 20813-20818.

[2] Irfune, T., Naka, H., Sanehira, T., Inoue, T., and Funakoshi, K. (2002) *In situ* X-ray observations of phase transitions in MgAl<sub>2</sub>O<sub>4</sub> spinel to 40 GPa using multianvil apparatus with sintered diamond anvils. *Physics and Chemistry of Minerals*, **29**, 645-654.

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