	Experiment title: Development and Application of X-ray Absorption Spectroscopy (XAS) Tomography on SOFC and SexGa1-x Alloys	**Experiment number**: MA-1155
Beamline: ID24	**Date of experiment**: from: 27th November 2014 to: 03rd December 2014	**Date of report**: 02/03/2015
Shifts: 18	**Local contact(s)**: Vera Cuartero	*Received at ESRF:*

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Report:

The aim of this experiment entitled, Development and Application of X-ray Absorption Spectroscopy (XAS) Tomography on SOFC was to study and depth-resolve the 2D and 3D spatial distribution of the chemical states and local atomic structure for a selected atomic species, by reconstructing the local XANES and/or EXAFS spectra. This can be of great interest both from the scientific and technological point view. Indeed, many materials have a 3D heterogeneous distribution of their oxidation state and local atomic structure (geology, engine materials, etc), and no existing technique makes it possible to retrieve the local XAS spectra in 3D at high resolution and with a high sensitivity. In this frame, we intend to identify the spatial chemical state distribution of a typical electrode of SOFC made of porous (La0.6Sr0.4)(Co0.2Fe0.8)O3-x (LSCF). The local structure around Co and Fe elements will be analysed before and after ageing - the deep knowledge of these oxidations states is crucial since conductivities and their evolution in time directly depends on the local elements valence states.

Series of horizontal scans at several ϕ rotational sample orientations (see Fig. 1) were performed to reconstruct the local XANES and/or EXAFS spectra. The spacing between each horizontal position is of about 1 µm to 2 µm.

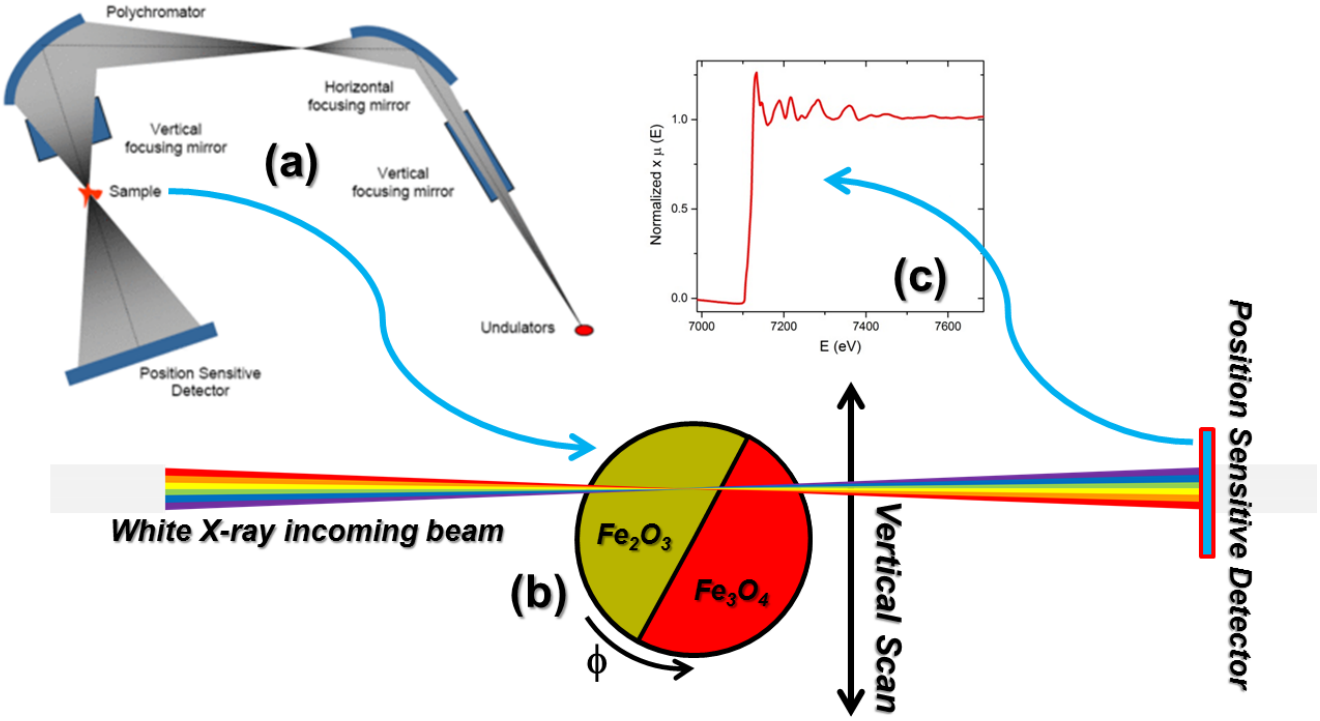

Figure 1: *The experimental setup: (a) the ID 24 beamline scheme; (b) experimental scheme with an illustration of the used iron oxide sample and (c) an example of a measured XANES spectrum.*

We have investigated through a μXAS tomography experiment at ID24 three different samples: (i) a well-known phantom sample with Fe_2O_3 and Fe_3O_4, connected with a glue mixed with metallic Fe (see Fig. 2a); (ii) an as prepared SOFC with LSCF sample, and, (iii) a long term electrochemically tested one (called here as aged "SOFC"), maintained at 0.8 A/cm2 for 1200 hours at 850°C. All the samples were finally prepared by plasma focused ion beam (pFIB). All samples were measured at the K-Fe edge, and samples (ii) and (iii) at K-Co edge.

The initial idea was to analyse samples (ii) and (iii) at the K-Fe edge too, but since after a first attempt to identify the Fe XANES edge the Fe concetrantion was apparalntly too low, we analysed those samples only at the K-Co edge. First day we spent calibrating the beam (energy at the K-Fe edge and focus). The next two days we have performed the experiments at the K-Fe edge. The forth day we spent calibrating the beam (energy at the K-Co edge and focus), and then we have performed the experiments at the K-Co edge.

Through tomography reconstructions, we could retrieve local hematite and magnetite XANES spectra (Figs. 2c to 2d), and, observe an oxidation of Co on the aged SOFC sample with respect to the as prepared one (see Fig. 3). A spatial resolution of about 4 μm was obtained (voxel of 4x4x4 μm3). The obtained spatial resolution was better than expected, smaller than the spot size at the focal point, because of the non-homogeneously energy distribution on a spot cross section.

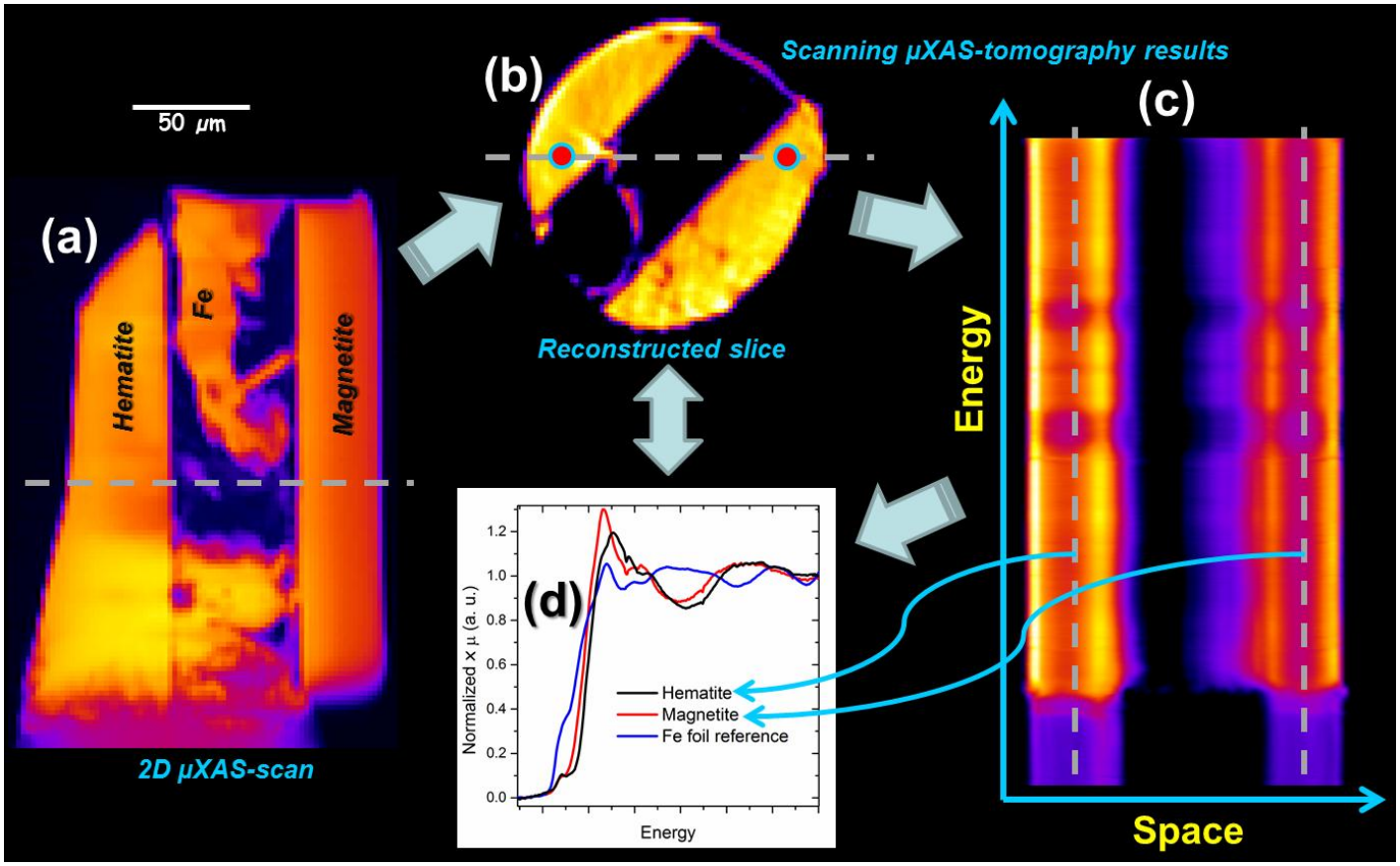

***Figure 2**: (a) 2D μ-XAS map of the iron oxide sample; (b) a tomographic reconstructed slice of this sample; (c) an energy profile along a line of this slice, and (d) the XANES spectra of both iron oxides compared to the Fe referenc*

Despite the very interesting and promising results shown here, the quality and repeatability of the obtained local spectra are still required to be improved in order to achieve more reliable results. We learned that several key issues can be corrected and improved (as already discussed with the ID24 staff in a meeting on Januray of 2015), such as: (a) smaller/less absorbent samples are essential to obtain higher quality tomographic reconstructions; (b) full 360º rotation motor instead of 180º in order to minimize the harmful effect of the intrinsic asymmetric beam shape; (c) a more dedicated macro developed for this kind of experiment and (d) more stable rotational and translational motors.

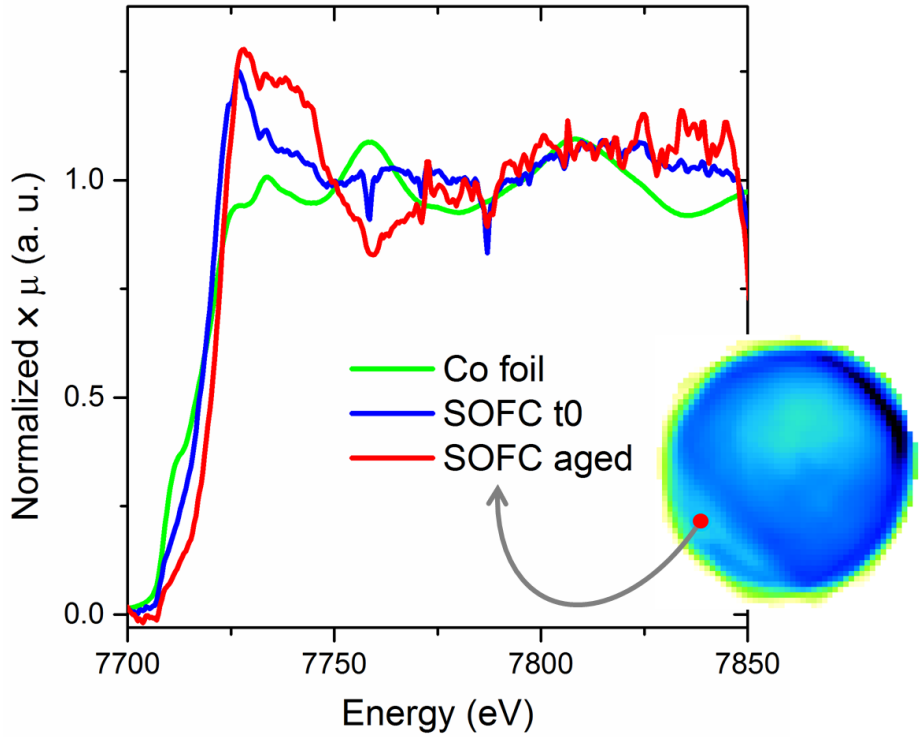

***Figure 3**: Above, local spectra (from tomography) of SOFC samples, as prepared t0 and after aging, compared to the Co reference*

A new proposal to continue MI-1193 was written and submitted in March 2015.