	Experiment title: Measurement of tortuosity within Solid Oxide Fuel Cells (SOFC) by x-ray nanotomography	**Experiment number**: MA-1155
Beamline: ID22NI	**Date of experiment**: from: 03rd November 2010 to: 06th November 2010	**Date of report**: 21/02/2011
Shifts: 9	**Local contact(s)**: H. Suhonnen	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

This experiment aimed at performing a 3D nanocharacterization of Solid Oxide Fuel Cells (SOFC) samples in order to have an estimate of their porosity and tortuosity. Samples have been prepared in our lab, using polishing and sawing tools routinely used for microelectronics. Samples are 100micrometers cross-section and about 1mm height, and were encapsulated within glass capillaries provided by the ID22NI beamline staff. This carefull sample preparation was essential to limit local tomography artefacts and because samples are highly absorbent (essentially made of Zr, Y, Ni).

Among the nine shifts available, one shift was dedicated to beamline alignement (pink beam setup, KB focusing, spot measurment, CCD and rotation axis alignement), which is a good number. A final spot size of 100nm in both directions has been obtained at the energy of 17keV.

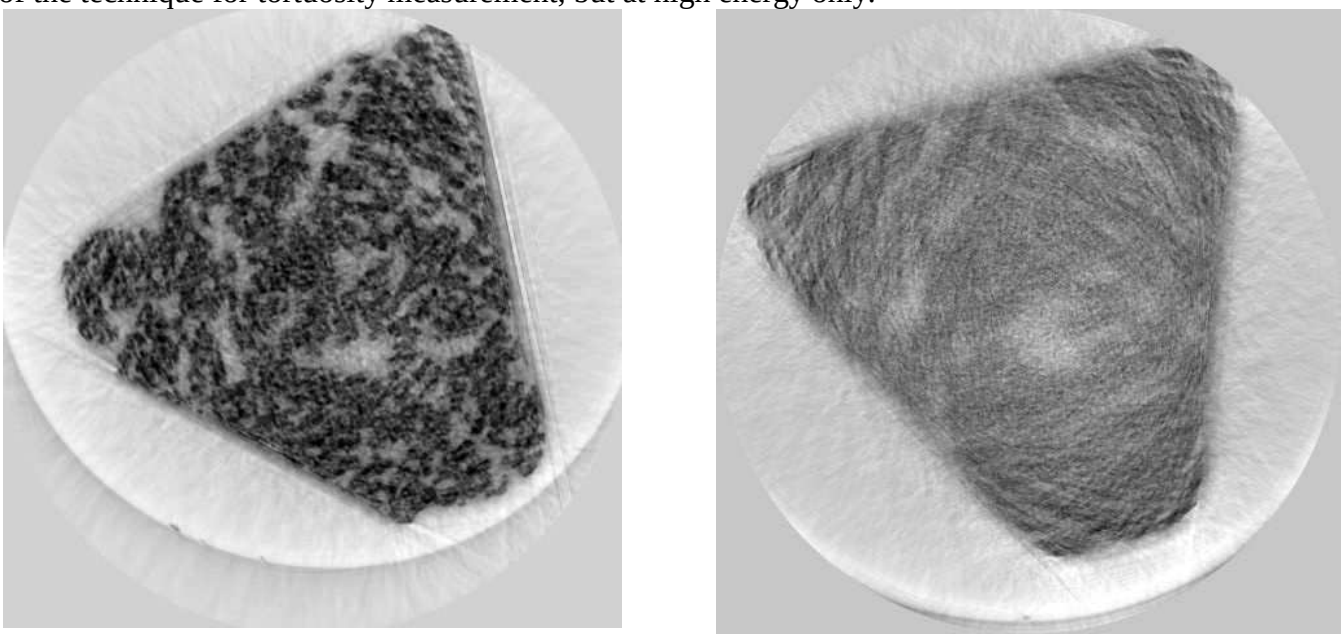
Samples analyses started the first afternoon. Daytime analyses consisted in scanning a series of single reference samples (without redox cycling), which are the most important samples. With respect to previous experiments carried out there, an important improvement is the enlargement of the field of view, that allows to use the full 2048 pixels of the Frelon camera, pushing the field of view to about 100 micrometers. With 3000 projections per turn, 4 distances, and 0.1second exposure time, a whole sample scan took less than 2 hours.

Nighttime was dedicated to automatic scanning of a stack of volumes along the 1mm height of the samples (about 6 volumes).

From the second day, cycled samples have been measured, which were more tricky to prepare and analyze because of a highher brittleness and cross-section. At noon the last day, decision has been taken that a sufficient amount of data was available, and a minor evolution of the setup has been asked to the local contact in order to switch to scanning tomography and perform a single fluorescence tomography slice of a single sample. Unfortunately, there was no way to use the continuous scanning scheme because the piezoelectric motor range was too small given our sample cross-section (one must mention that this measurement were beyond the proposal request). A step-by-step calculation has been run through the night, that could go to the end.

Data are now under processing, thanks to a dedicated post-doctorate. Most efforts are spent on phase-retrieval approach, which is particularly challenging for such highly absorbent samples. Common work and reflexions have been performed with the beamline staff. It rapidly turns out that there was no way to perform the phase

retrieval at the energy of 17keV on such complex, dense and high-Z materials. For this reason, a single scan has been perfomed (courtesy of the local contact, Heikki Suhonen) at a higher energy, showing the potential of the technique for tortuosity measurement, but at high energy only.



Two reconstructed cross-sections of the same sample. Left, acquisition at 17keV. Right, 29keV.

We do work on the 29keV data set, aiming to extract quantitative parameters on this single scan. A publication will be submitted before summer 2011.
A new proposal to continue MA115 will be written and submitted in March 2011.