ESRF	**Experiment title:** In-situ SXRD study of fuel cell model cathodes	**Experiment number**: SI2340
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Shifts: 18	**Local contact(s)**: Olivier Balmes	*Received at ESRF:*

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Report:

Solid oxide fuel cells (SOFCs) are regarded as a very promising technology to generate clean electrical energy by direct conversion from chemical energy [1]. Yttria stabilized zirconia (YSZ) is a key material used in SOFCs where it acts as solid electrolyte for oxygen ions. The YSZ surface in addition plays a central role in the relevant reactions on SOFCs electrodes: it is involved in the adsorption and oxidation of hydrocarbons, the formation of H_2O and the oxidation of carbon monoxide at the anode side [1, 2]. At present the bottleneck for the efficiency of solid oxide fuel cells is the dissociation and incorporation of oxygen at the fuel cell cathode side, which requires high temperatures (above 1000 K). The oxygen incorporation is enhanced by the presence of perovskite type oxides such as (La,Sr)MnO_3 and (La,Sr)(Co,Fe)$O_{3-\delta}$ on the cathode. At present, it is not clear if the triple phase boundary between oxygen, perovskite and YSZ plays the dominant role for the incorporation of oxygen or the surface of the pervoskite electrodes. To shed light on these questions the experiment was performed at ID03 with UHV High-Pressure chamber installed on the six circle diffractometer. The sample was YSZ (100) with a microelectrode made of $La_{0.8}Sr_{0.2}MnO_3$. The electrode size of 400x400x0.2μm was chosen such that one can also perform impedance spectroscopy experiments with the sample. Anomalous SXRD data sets were collected at the Y and Zr K edges for 5 different oxygen chemical potentials using a provisional setup (see Fig.2). To be sure about position of the beam footprint with respect to the electrode a fluorescence detector was utilized placed at 90° towards the beam position (see Fig.1). So data from 3 areas on the sample surface were recorded with few μm spatial resolution in the direction perpendicular to the beam: below the electrode (interface), close to the electrode (3 phase boundary) and far from the electrode (free surface as a reference). The oxygen chemical potential has been tunable locally by applying a voltage to a micrometer sized tip, which had been brought into contact with the electrode from the top. The backside of the sample was contacted by a Pt counter electrode. The KB focused beam with a vertical size of 3 μm was matched to the electrode size of 400 μm by setting the incident angle to 0.9°, about three times the critical angle, which resulted in a significantly increased background. Apart from that we have used a possibility of tuning the photon energy to the Y or Zr K edges for an enhanced scattering contrast. In front of the 2D detector a graphite analyzer was mounted to suppress the fluorescence background. The quantitative analysis, which is on its way, will elucidate the chemical potential dependent

interfacial structure and structural differences at the perovskite – electrolyte triple phase boundary during oxygen incorporation.

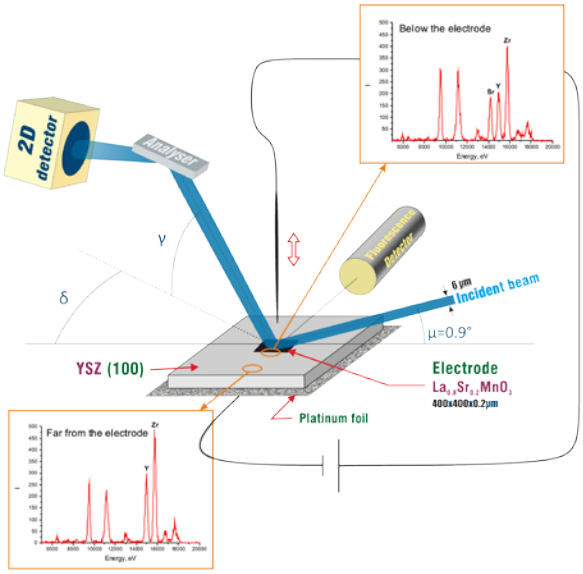

Fig. 1.Scheme of the experiment.

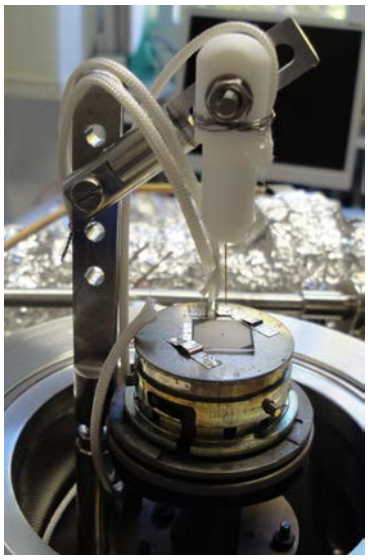

Fig. 2. Experimental setup.

During the experiment we have faced several problems. First of all we were not able to acquire data in the whole region of interest for some CTRs (Fig.2, fig.3). As one can see on Fig.3 in the region between L=1.5 and L=2.5 signal was cut by metal rod of the tip holder (Fig.2).

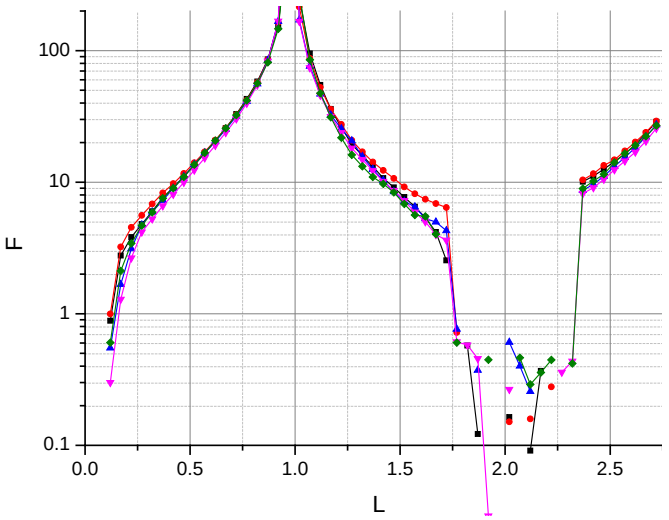

Fig.3. Rod (1, -1), below the electrode, Y edge. All conditions.

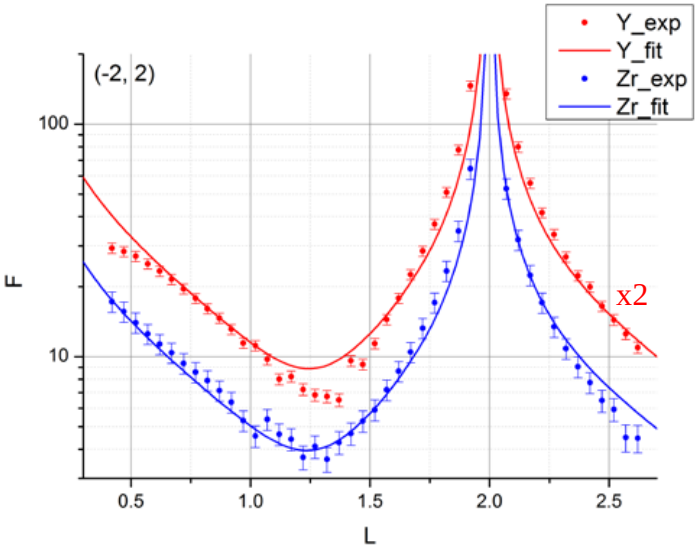

Fig.4. Experimental data (dots) fitted (solid lines) with ANA-ROD program. Conditions: room temperature, 10^{-7} mbar, far from the electrode.

	Y	Zr	O
Occupancy	0.0794	0.2216	0.0705
Displacement	-	-0.0319	-

Table 1. Fit parameters for data set recorded with conditions: room temperature, 10^{-7}mbar, far from the electrode

Second, some of the shifts were lost because of severe misalignment of the diffractometer.
On the other hand we got rather big data set which is possible to analyze. First attempts are presented on Fig.4, for fit parameters see Table 1. Nevertheless, it is desirable to have data with better statistics.