	Experiment title: Investigation of the oxygen electrode degradation of solid oxide cells by X-ray nano-fluorescence and diffraction measurements	**Experiment number**: MA-5526
Beamline: ID16B	**Date of experiment**: from: 16/11/2022 to: 21/11/2022	**Date of report**: 02/08/2023
Shifts: 15	**Local contact(s)**: Julie Villanova	*Received at ESRF:*

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Report:

Solid Oxide Cells (SOCs) are high temperature electrochemical energy-conversion devices. They can be operated in fuel cell mode (SOFC) to produce electricity or in electrolysis mode (SOEC) to produce hydrogen. Thanks to their reversibility and high efficiency, this technology is considered as the most promising solution to match the fluctuation in energy production inherent to the development of the renewable energy sources. Nevertheless, the durability of SOCs is still insufficient to envisage a large-scale deployment of this technology. The degradations are mainly related to morphological evolutions in the complex electrode microstructure associated with material instability and inter-diffusion between the cell components. Among all these phenomena, the destabilization of the oxygen electrode material contributes significantly to the cell degradation. However, despite many studies, the driving force of this degradation phenomenon remains unclear especially in electrolysis mode. Therefore, this experiment performed analyses down to the nanoscale coupling X-ray Fluorescence (XRF) and X-ray Diffraction (XRD) to bring new insights on the degradation mechanism of the oxygen electrode. The beamline ID16B for X-ray Nano-Analysis available at the ESRF is especially relevant to perform these coupled characterizations.

Experimental method

The lamellae for the synchrotron nano-XRD and nano-XRF were prepared using a Xe^+ plasma-Focused Ion Beam (pFIB) Vion (FEI™), following a protocol already validated at the laboratory. The lamellae of approximately 60 × 60 × 2 μm^3 were fixed on an aluminum pin. Synchrotron XRF and XRD were performed at the ID16B beamline in ESRF. The beamline provides a high flux (10^{12} ph/s) focused X-ray beam, which has a spot size of 60×60 nm^2. An energy of 29.6 keV was selected for this study in order to analyze all the cell elements. A ROI of 7×8 μm^2 was defined focusing on the oxygen electrode. Both XRF and XRD 2D maps were acquired with a step of 50 nm and an exposure time of 1000 ms per point. PyMca software (27) was used to fit the XRF data while the diffraction data were first treated with XRDUA software (28)

Results

A first paper has been published using the results obtained during this beamtime. The XRF and XRD analyses have been combined with other techniques in order to unravel some degradatino mechanism in the oxygen electrode of SOCs. Reference of the apper is given hereafter:
"Advanced Nanoscale Characterizations of Solid Oxide Cell Electrodes", G. Sassone, O. Celikbilek, M. Hubert, K. Develos Bagarinao, T. David, L. Guetaz, I. Martin, J. Villanova, L. Rorato, B. Morel, A. Léon, J. Laurencin, ECS Transactions 111(6) (2023) 885.

Two other papers are under preparation using these results. They should be submitted by the end of the year.