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USING DARK FIELD X-RAY MICROSCOPY TO STUDY IN-OPERANDO YTTRIA STABILIZED ZIRCONIA ELECTROLYTE SUPPORTED SOLID OXIDE CELL

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Summary: Dark Field X-Ray Microscopy is a promising technique to study the structure of materials in nanometer length scale. In combination with x-ray diffraction technique, the microstructure evolution of Yttria Stabilized Zirconia electrolyte based solid oxide cell was studied running at extreme operating conditions.

1. INTRODUCTION

At ID06 beamline there is currently commissioning a dark field x-ray microscope, enabling to zoom into mm-sized samples and perform 3D mapping of grains and stresses at the 100 nm scale from local regions. This provides unprecedented opportunities for studying microstructural changes in operando materials. Yttria Stabilized Zirconia is a well-known material used as high temperature electrolyte in solid oxide cells for sustainable and renewable power generation. Oxygen bubble formation has been observed at grain boundaries of YSZ near the electrolyte/oxygen electrode interface. This is considered as a degradation process of electrolyser cells running at extreme operating conditions, [1, 2] that decreases the performance and lifetime of the cell. In this work a Dark Field X-ray Microscopy and local X-ray diffraction techniques are used to study microstructural changes of electrolyser YSZ symmetric cell at extreme operating conditions.

2. EXPERIMENTAL METHOD

The experiments were performed at the ID06 beamline of the European Synchrotron Radiation Facility (ESRF), in Grenoble, France. The setup of the local x-ray diffraction is shown in figure a); the incoming beam is approximately 6 microns width, allowing perform several scans across the 250 microns thickness between the electrodes of the sample. The sample was tilted 4 degrees in 0.01 degrees angle step in each scan to improve the statistics of the diffracting grains in that specific layer of the sample. The scans were taken at different times to assess the evolution of the cell for 24 hours, subjected to a temperature of 700 ^{o}C and a potential of 2 volts.
In figure b) is shown the setup of the dark field x-ray microscopy technique, using an objective composed by a set of beryllium lenses, with a magnification of 16X. In both setups Frelon cameras were used to collect the diffraction images.

3. RESULTS

The local XRD scans across the sample, figure a) bottom, show no immediate change in lattice parameter when applying voltage. However, a gradient in lattice parameter is observed in electrolyte after 24 hours (magenta and blue lines), suggesting a compressive stress near the positive electrode/electrolyte interface, possibly induced by the precipitation of high-pressure oxygen bubbles on YSZ grain boundaries [1] observed in the sample by scanning electron microscopy after testing. Dark field x-ray microscopy, figure b) bottom, revealed stresses in a grain from the positive electrode/electrolyte interface and changes in the shape in the boundaries region, after 10 hours under the previously mentioned conditions.

References

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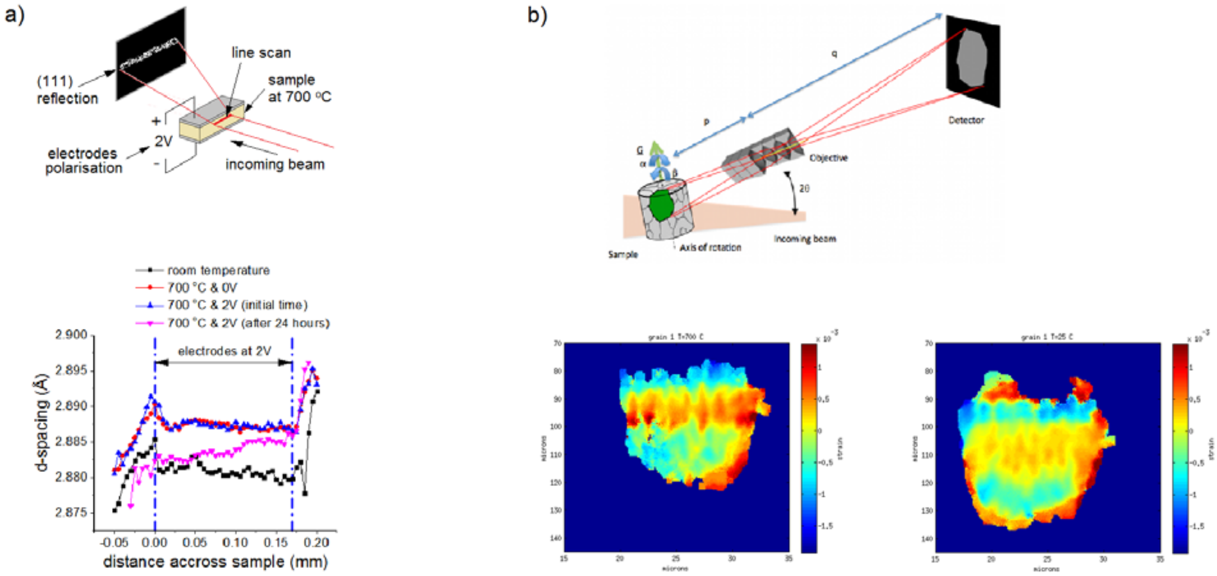

Figure: (a) top: setup of the local x-ray diffraction were the diffraction images are obtained from a line beam of 6 microns width and scans are performed in 50 positions across the sample. Bottom: gradient in lattice parameter observed between the electrodes after 24 hours under extreme operating conditions. In positive electrode (left side) the compressive stress is higher than in the negative electrode (right side), observable in the magenta line.

(b) top: setup of the dark field x-ray diffraction microscope. The objective is positioned between the sample and the detector, allowing zoom into one grain and providing a magnified image of 16X. Bottom: strain comparison in sample at room conditions and after 10 hours running at extreme operating conditions.