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"Operando structural studies in Materials Science"

In operando studies of an yttria stabilized zirconia electrolyte supported symmetric solid oxide cell by Dark field X-ray Microscope at ID06

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At ID06 beamline we are currently commissioning a dark field 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 at grain boundaries of YSZ near the electrolyte/oxygen electrode interface has been observed as a degradation process of electrolyser cells running at extreme operating conditions, [1, 2]. In this work a Dark Field X-ray Microscopy and local X-ray diffraction techniques at beamline ID06 are used to study microstructural changes of electrolyser YSZ symmetric cell at 700°C and an applied voltage of 2V during 24 hours. Results from local XRD scans across the sample (figure 1. a) 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 in figure 1. b), 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 (figure 1. c). Dark field x-ray microscopy revealed stresses in a grain from the positive electrode/electrolyte interface and changes in the shape in the boundaries region. Next experiments should include information from more grains using this technique to have a wider overview of the inside microstructure and its changes.



<u>Figure 1</u>: a) Scan position in the sample, parallel to the electrodes. b) Average position of peaks in each scan layer. c) Oxygen bubble formation in YSZ grain boundaries near oxygen electrode

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