Combined high-resolution FIB-Nanotomography and 3D-EDS of solid-oxide electrolysis cells

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In reverse mode, solid oxide electrolysis cells (SOEC) are being developed intensely for storing growing excess renewable electricity into gas (H2, CH4), reaching close to 100% electrical-to-H2 efficiency. The challenge remains to mitigate the performance degradation with time. The degradation stems from morphological, chemical and crystallographic changes, due to intrinsic material instabilities and (exo- and endogenous) impurities [1]. 3-D imaging largely contributed to the current understanding of the relationships between the microstructure and performance of SOFC/SOEC materials [2-4].

FIB Nanotomography offers a high resolution when the electron beam energy is in the range of 1-2 keV. At this energy the escape depth of electrons is in the range of a few nanometers. Tomography data with isometric voxels of a few nanometers in size can be acquired automatically [5]. EDX analysis however requires beam energies typically in the range of 8-15keV generating a larger interaction volume. Automatic switching between imaging conditions and EDX conditions at regular intervals was used for the acquisition of a combined dataset.

The region comprising the yttria-stabilized zirconia (YSZ) electrolyte, the gadolinia-doped ceria (GDC) compatibility layer, the Ni-YSZ hydrogen electrode and the lanthanum strontium cobalt ferrite-based oxygen electrode has been imaged by FIB-SEM/EDX. The samples were extracted from a cell operated for 11'000 h in SOEC mode. FIB-SEM imaging was performed at an acceleration voltage of 1.8 kV with 10nm imaging pixel size and 10nm slice thickness. Every 10 slices, the acceleration voltage was switched from 1.8 kV to 10 kV for EDX. The reconstructed volume was 35x10x10 m3, which corresponds to the representative volume element for the effective properties of the heterogeneous electrode materials plus the electrolyte and compatibility layer thicknesses.

The combination of the 3-D image data with high spatial resolution and 3-D EDX elemental maps (Fig.1) provides new insights into the microstructural alterations that occur during operation (a). Significant changes were observed SOEC sample, including the formation of a SrZrO3 insulating secondary phase (b). Localized changes include intra-phase micro-cracks in the O2 electrode and inter-phase cracks in the Ni-YSZ electrode (c), predominantly close to the YSZ electrolyte. Closed porosities form along the grain boundaries in the YSZ electrolyte, on the oxygen electrode side (d.). Ni coarsening and depletion close to the electrolyte was also observed.

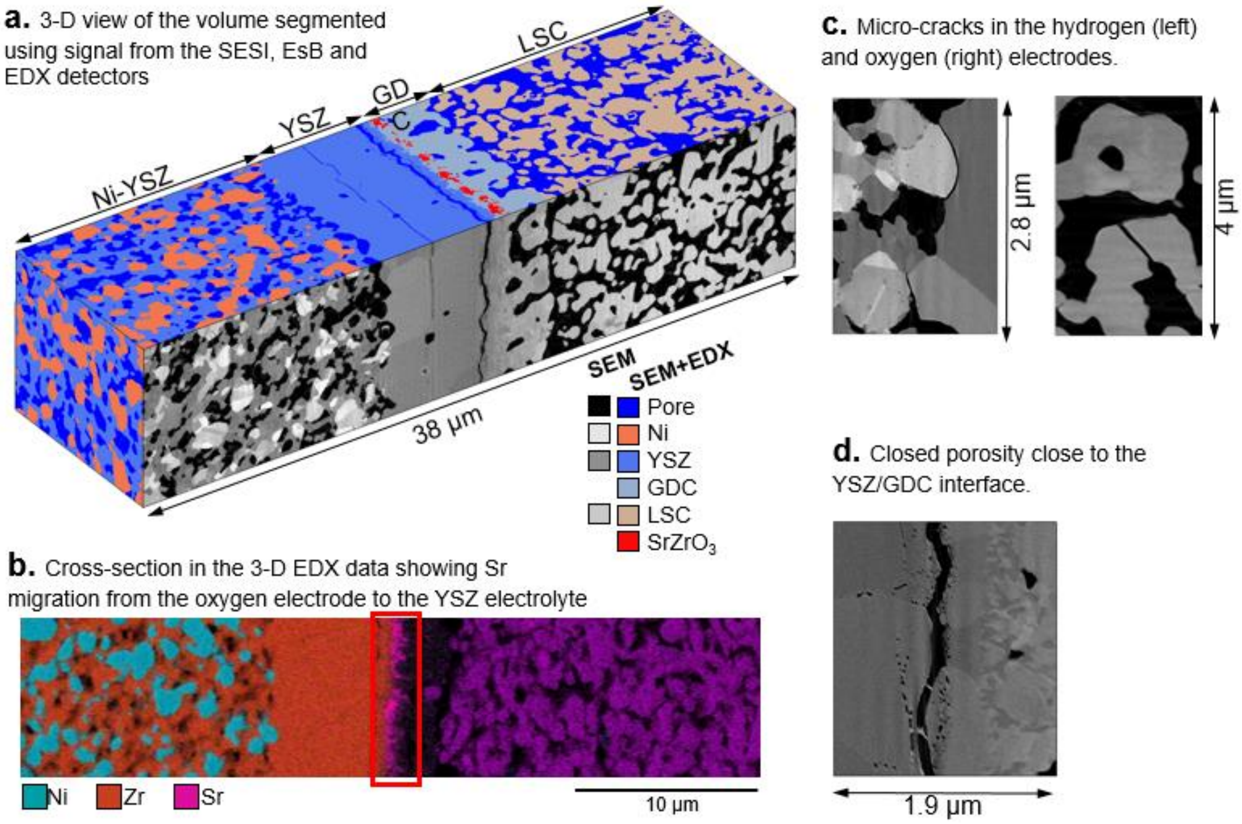

Fig 1: 3-D reconstruction of a sample operated in SOEC mode for 11'000 h obtained by FIB-SEM/EDX.

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