In situ TEM implementation of impedance spectroscopy on a model solid oxide electrolysis cell combining reactive gasses, high temperatures and electrical potentials

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Sustainable energy sources are now economically competitive with that from coal and gas, but a main challenge is efficient energy storage solutions. The solid oxide electrolysis cell (SOEC) is a promising technology for efficient conversion of electrical energy to chemical energy (hydrogen or hydrocarbons) for storage [1]. SOECs are operated at high temperatures (≥ 800°C) in reactive gasses (O_2, H_2O, CO_2) and these conditions induce nanoscale changes in the active metals and ceramics in the cells thereby changing the overall cell performance. Conventional post mortem electron microscopy analysis is insufficient to understand the time, temperature and electrical potential dependencies of these changes. It is, however, a great challenge to conduct in situ electron microscopy on SOECs because this requires that the hard and brittle ceramic cells are thinned to electron transparency, that the cells are carefully designed to allow for characterisation of the layer interfaces, and that the cells are characterised during exposure of reactive gasses, electrical potentials and high temperatures.

Here we present a TEM/STEM study where such in situ experiments were performed on symmetric model SOECs composed of materials commonly used in state-of-the-art SOECs, i.e. $La_{0.6}Sr_{0.4}Co_{O3-\delta}$ (LSC) electrodes and a $Zr_{0.8}Y_{0.2}O_{2-\delta}$ (YSZ) electrolyte (fig. 1a). This was achieved in an environmental TEM in combination with both custom-made and commercial heating/biasing TEM holders. STEM-EDS revealed segregation and grain formation for the LCS electrodes, which increased as a function of temperature, and that this degradation was enhanced by the presence of O_2 compared to vacuum [2]. Spectral imaging showed that the oxidation state of LSC near the LSZ-YSZ interface increased at temperatures above ca. 400°C both in O_2 and even in vacuum. It was furthermore possible to induce structural degradation, similar to that from heating, by applying an electrical potential across the cells.

One of the most important characterization methods for determining electrochemical resistances for different processes (ion transport, gas diffusion, catalytic reactions etc.) in an SOEC is electrochemical impedance spectroscopy (EIS) [3]. In this work we extended the TEM in situ tool box by implementing EIS on the model SOECs in the TEM. Preliminary EIS-TEM experiments show a decrease in resistance over the model cells (observed as decreasing semicircular impedance arcs vs. temperature in fig. 1b). This trend is expected since the oxygen ion conductivity increases with temperature and similar trends are observed for full scale SOECs. We are currently working on a more detailed understanding the EIS-TEM results.

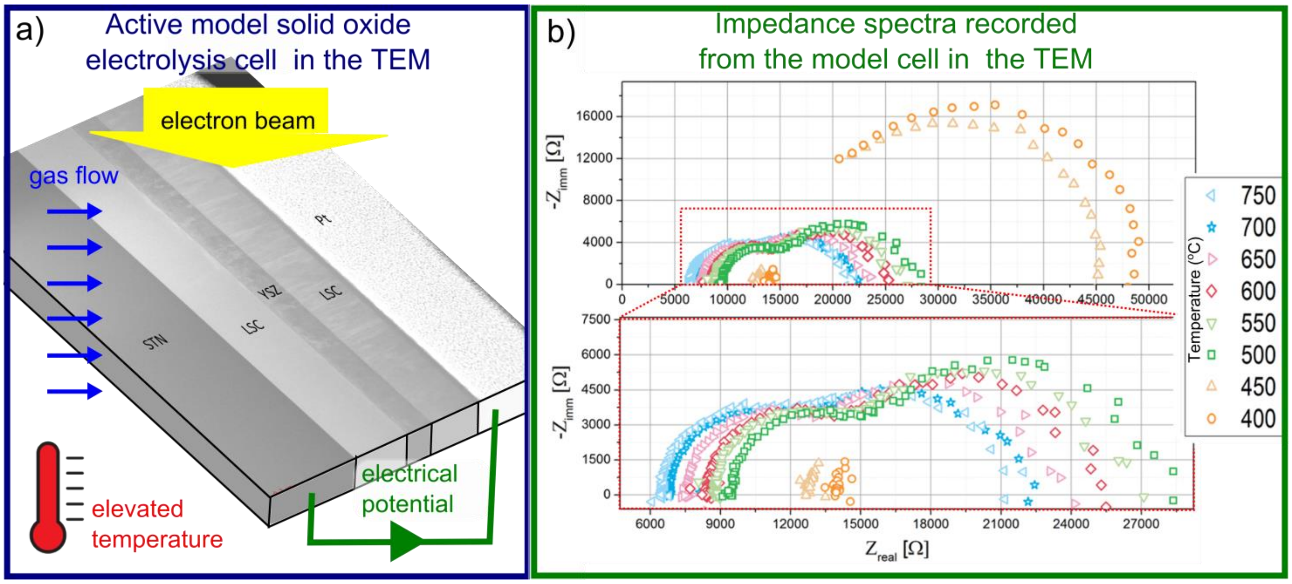

Fig. 1: (a) TEM image of the model SOEC with drawings added to illustrate the cell geometry and the in situ conditions: gas flow, elevated temperature and electrical potential. (b) Impedance spectra recorded from the model cell in (a) during exposure of O_2 at 400-750°C.

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References

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