Operando ETEM study on solid oxide cells

Zhongtao Ma[1], Mr. Christodoulos Chatzichristodoulou[1], Mr. Kristian Speranza Mølhave[2], Mr. Søren Bredmose Simonsen[1]
[1]DTU Energy, Kongens Lyngby, Denmark, [2]DTU Nanolab, Kongens Lyngby, Denmark

Background incl. aims
Solid oxide electrolyzers and fuel cells (SOEC/SOFC) are crucial for reducing global carbon emissions. Understanding their degradation processes is key to making them more durable and cost-effective for widespread use [1]. Usually, researchers examine these cells after electrochemical tests, which limits insights into the ongoing degradation process. Our study introduces a new method that makes it possible to observe the cells in operation. This method combines the TEM characterization and the electrochemical test and correlates the structural and compositional evolution with electrochemical performance.

Methods
The presented method combines a TEM holder with a heating-biasing MEMS chip and an environmental TEM capable of introducing relevant reactive gases such as O_2, H_2, and H_2O, mirroring the operational conditions of SOEC/SOFC. We developed and utilized a special sample preparation procedure to apply three types of stimuli—heat, reactive gas, and electrical bias—directly to the SOEC/SOFC within TEM [2, 3]. The tested cells have lanthanum-strontium-cobalt oxide (LSC) electrodes and an yttrium-stabilized zirconia (YSZ) electrolyte. To study cell degradation, those cells are tested with polarization in an atmosphere of 2.7 mbar O_2 at 700 °C.

Results
STEM imaging revealed that degradation feature development in the negatively polarized LSC electrodes varies with polarization voltage. Crack formation was also observed at the interface between the positively polarized LSC electrode and the YSZ electrolyte. EELS analysis conducted simultaneously showed a Co valence state change, with lanthanum's state remaining constant. Subsequent analyses, including EDS, SAED, and HRTEM, suggested that the newly observed features in negatively polarized LSC are the result of LSC decomposing into Co_3O_4, La_2O_3, and La_2CoO_4. This decomposition process was particularly pronounced near the YSZ interface, indicating a direct correlation between structural changes and electrochemical performance.

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