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In operando Raman spectroscopy for investigation of solid oxide electrolysis cells

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Summary. How can in operando Raman spectroscopy increase our understanding of degradation and activation processes in solid oxide electrolysis cells? Recent experimental results are reported, including experiments showing remarkable polarisation induced compositional changes in infiltrated perovskite electrodes.

Abstract. Traditional evaluation of solid oxide electrolysis cell (SOEC) durability and performance relies on electrochemical methods including voltammetry and impedance spectroscopy. In operando electrochemical studies are then followed by post mortem investigations of changes in microstructure and elemental composition. As a supplement to these well-established characterization methods various in situ and in operando spectroscopies have received increased attention in the solid oxide cell community during the last decades[1-2] and may offer a great potential for improving the understanding of degradation processes in solid oxide electrolysis cells. The advantage of these in situ and operando techniques is they may *reveal changes in elemental electrode compositions in real time as the changes occur.*

For solid oxide cells in operando spectroscopy is defined as spectroscopy with the three operating parameters: 1) Temperature, 2) Atmosphere and 3) Electrical polarization, held at values similar to those experienced during real operation of the cells. Previous work using operando Raman spectroscopy to study solid oxide cells and materials has examined coking by carbon containing fuels[3] as well as changes in a material's oxidation state[4] under atmospheric pressures and at temperatures as high as 800 °C.

In the present poster, a number of recent cases are presented, where Raman spectroscopy has been applied in situ and in operando to study solid oxide cells and solid oxide materials. Special emphasis is put on an experiment applying in operando Raman spectroscopy on an air electrode prepared by BaO infiltration of LSM ($(La_{0.85}Sr_{0.15})_{0.9}Mn_{3\pm\partial})$, as this experiment led to several interesting observations[5]. The first surprising discovery was that the BaO had formed a secondary $Ba_3Mn_2O_8$ phase due to reaction with the LSM. This phase was likely the cause of a decreased polarization resistance observed at OCV conditions. Secondly, this $Ba_3Mn_2O_8$ phase reversibly decomposed with electrical polarization with Mn changing its oxidation state sequentially from 5+ to 2+ (MnO). When the electrical polarization was removed, the MnO was transformed back into $Ba_3Mn_2O_8$. These findings emphasize the need to directly observe material changes in high temperature solid oxide electrodes under realistic working condition, especially electrodes prepared by infiltration.

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