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Development of an Operando XRD PEMWE Cell for Correlating Catalyst Structure and Performance

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ABSTRACT (oral presentation)

In PEMWE, iridium- and ruthenium-based anode catalysts enable hydrogen production but undergo significant structural changes during operation. In addition to reported crystallite growth and particle dissolution [1], also other structure-related processes occur. For instance, the rapid decrease in performance of initially highly active amorphous IrO_x is associated with partial crystallization to the rutile IrO_2 phase [2]. Such degradation processes are not necessarily detectable by cell tests alone, as they may be masked by effects such as membrane thinning and the resulting increase in proton conductivity [3].

To enable an analysis of catalyst particles during the PEMWE operation, an *operando* X-ray diffraction (XRD) cell is constructed. The concept is based on reflection-mode XRD measurements using a Mo-X-ray source, allowing high penetration depths. Due to the low incidence angle of the X-ray beam, there is a significant offset between the beam entrance point and the probed region on the catalyst-coated membrane (CCM). This is taken into advantage with an input and an output Kapton® window, separated by a supporting structure ensuring the contact pressure to the CCM region below. The X-ray beam enters the cell through the cathode side and must pass through the carbon paper (Toray®) gas diffusion layer to reach the CCM. Due to the low X-ray absorption of carbon, the horizontal offset can be adjusted *via* the thickness of the carbon paper with minimal intensity loss. The geometry is designed such that a line-focused X-ray beam is diffracted at the catalyst layers directly beneath the supporting structure. The cell body is made of glass-fiber-reinforced high-performance polymer (Rigid 10k) to ensure the necessary contact pressure between the cell components. Inside the XRD housing a water supply is implemented with a preheating cell to ensure *operando* PEMWE with temperatures up to 80° Celsius. Automated water and power supply allow long-term operando XRD measurements exceeding 500 h.

Initial tests of the *operando* PEMWE cell demonstrate the appearance of characteristic diffraction peaks of catalyst materials. These signals are sufficient to determine crystallite size, particle dissolution, and potential phase transformations *via* Rietveld refinement. The versatility of the cell is explored in several applications. The cell is used to investigate the reduction of IrO_2 to metallic Iridium at low cell voltages [2]. In addition, the cell is applied to study amorphous IrO_x as anode catalyst. The unfavorable crystallization to the rutile IrO_2 structure is investigated under different operating protocols and *operando* XRD can reveal which conditions particularly promote this crystallization.

References

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