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Water dynamics during gas fed CO2-electrolysis revealed by 4D X-ray imaging

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CO2-electrolysis (CO2ELY) is a promising technology to harvest the temporal surplus of renewable energy and convert CO2 into valuable non-fossil feedstock for green chemistry. CO2ELY is similar to water electrolysis. CO2-gas is fed to the cathode to be electrochemically reduced, e.g. to CO, with an adapted catalyst. We employ time-resolved synchrotron X-ray tomographic microscopy (XTM) to study operando the complex water dynamics during CO2ELY and its relation to electrochemical performance. Water plays an essential role as a reactant for the CO2 reduction reaction (CO2RR) in alkaline conditions, while condensed water can block gas exchange in the gas diffusion layer (GDL). Water is further involved in the process through vapor adsorption/desorption, capillary fingering in the GDL and hygroscopic swelling of the polymer electrolyte membrane.

A zero-gap electrolyzer with a bipolar membrane (BPM) in forward bias [1] - alkaline at the cathode, acidic at the anode - is studied. The membrane is sandwiched between two porous electrodes based on carbon paper GDLs. The cathode GDL additionally carries a microporous layer. This membrane electrode assembly is clamped between two polar plates containing flow channels for gas and water at the cathode and anode, respectively. A custom-made miniature electrolyzer with an active area of 10 mm2 is used for XTM [2]. This miniature cell is mounted on the rotating sample stage of the TOMCAT beamline at the SLS, Paul Scherrer Institut, Villigen PSI, Switzerland. During electrochemical operation, fast tomographic scans (1s/scan) of the active region are recorded every minute for 45 min with a voxel size of 2.75 um. The concurrent electrochemical performance is measured and related to the liquid water evolution and gas bubble formation within the cell. Beyond qualitatively correlating the imaging results to the electrochemical data, the different phases in the image data are segmented to study the evolution of the liquid water configuration in detail. Due to substantial deformation of the sample during acquisition, i.e. strong membrane swelling with GDL compression, differential segmentation methods are not applicable. We extend machine learning segmentation (e.g. Weka [3], usually 2D) to exploit information in all four dimensions at once.

Imaging reveals the multilayer system of 0.8 mm-wide flow channels, GDLs, microporous layer, catalyst layers and the BPM, particularly resolving the inter-fiber GDL-pores in the range of 20 um. The temporal resolution allows tracking the evolution of liquid water. It is a delicate balance between feeding the CO2RR with reactant water and not blocking the gas transport. Water condensation is observed in the GDL and, more severely, creates blocking slugs in the flow channels at the cathode. Additionally, hygroscopic swelling of the membrane together with catalyst delamination and BPM-interface fracturing is observed.

The presented experimental results help to better understand water dynamics during CO2-electrolysis to ultimately yield higher power density and stability. The results further reveal important degradation processes, particularly in the BPM. The combined electrochemical and imaging data can further inform modeling to accelerate the search for optimal operation parameters.

Participation

In-Person

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