



	Experiment title: In-operando XRD/SAXS of polymer electrolyte fuel cells during conditioning and degradation	Experiment number: CH-5165
Beamline:	Date of experiment: from: 20 Sept 2017 to: 27 Sept 2017	Date of report: 24/03/2020
Shifts:	Local contact(s): Jakub Drnec	<i>Received at ESRF:</i>
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Report:

This experiment was extremely successful. Several datasets from this beamtime, and further experiments have been assembled into a more comprehensive article currently under review in a high impact journal.

All of the major objectives described in the proposal towards in operando PEM fuel cell XRD and SAXS were met. The electrochemistry and aging of catalyst layers under several Department of Energy stress testing cycles was performed and observed. The dynamic hydration of the membrane was observed with SAXS and XRD. The catalyst Pt nanoparticle size distribution could be observed with SAXS and through Rietveld analysis of the XRD. The initial conditioning of the fuel cell was observed, and the in situ cell was able to reach extremely high performance and current densities, in excess of 1.5 A/cm².

Further work is necessary to improve and optimize the data analysis, and the speed of data acquisition. Presently, the ability to perform complex in-situ spectroelectrochemical measurements is limited by on-line data analysis at ID31. The actual data acquisition was fast enough to resolve the desired physical phenomena (1s), but not fast enough that diffractive imaging was trivial.

The single largest innovation of this beamtime was the implementation of multiple simultaneous in situ sample environments. One sample inside an electrochemical cell was mounted on each end of a translation stage. The durability testing required periodic measurements over several days, while the catalyst surface structure diffraction required shorter periods of more detailed experiments. Shuffling back and forth between the cells doubled the scientific output of the beamtime, and is being used as a standard approach in future experiments.