Experimental report

13/02/2020

Proposal: 1-04-160 **Council:** 10/2018

Title: SANS investigation of accelerated stress test of fuel cell electrode membrane assembly

Research area: Engineering

This proposal is a new proposal

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Samples: Electrode membrane assembly
Reference catalyst layers (quartz cells)

Instrument	Requested days	Allocated days	From	To
D22	4	4	30/09/2019	04/10/2019

Abstract:

A polymer electrolyte membrane fuel cell for the automotive application must deliver high performance under various operating conditions. However, the operation under unsteady conditions results in degradation of fuel cell materials, particularly the electrode membrane assembly (MEA). Understanding degradation mechanisms of fuel cell components is a key to the commercialization. In the proposed experiment, the MEA of the fuel cell will be investigated with small angle neutron scattering (SANS) during accelerated stress tests (ASTs). We will study how relative humidity and potential cycling impact the water management in the operando fuel cell. The ionomer peak (Q~0.1 and 0.25 A-1) will be tracked for quantifying water uptake in the ionomer, while the total water content can be deduced from incoherent scattering (Q~0.5 to 1 A-1) or transmission. Additionally, reference quartz pocket cells, coated with catalyst inks, will be investigated with and without (heavy) water for calibration. The calibration data will be incorporated to the operando results to improve the accuracy of water measurements.

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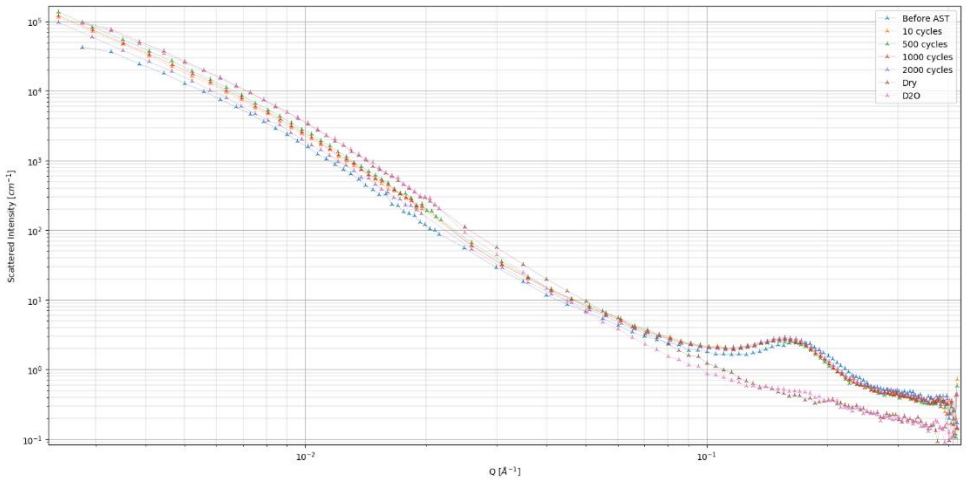
The main objective of this experiment was to determine if there is any evolution of the ionomer nanostructure within the electrode of an operating PEMFC during accelerated stress test. A PEMFC single cell has been specifically designed to be able to record the SANS pattern of the electrode, or catalyst layer, during operation with a very small neutron beam (10 µm x 10 mm). The main difficult is to align the cell and the neutron beam which takes several hours. Compared to our previous studies we aimed at conducting the experiments in operating conditions more representative of real fuel cell, on components closer to the state-of-the-art regarding their thickness. So the thickness of the beam was reduced to 10 µm compared to 30 µm for proposal 1-04-133 or 1-04-141. A new slit has been designed and manufactured at ILL to be less sensitive to temperature variations. Indeed, the slit is very close to the cell so that the footprint of the beam on the electrode is smaller than its thickness. The cell is heated up to 70°C. The thicknesses of the electrode and of the membrane were reduced down to 50µm, compared to 100µm and 125µm respectively in the former experiments. Despite good alignment of the cell with regards of the neutron beam, we were facing some issues because of the waviness of the sample. The thin membrane was too wavy and even after several trials by assembling several cells, it was not possible to find a configuration in which the beam hits solely the catalyst layer. Thus, the signal contains a significant contribution of the membrane and results were not exploitable. Then, we decided to assemble a cell with a thicker and much less wavy membrane. Finally, we have been able to conduct our experiments. We have recorded reference spectra of the cathode, the anode and of the membrane at different relative humidity and saturation, either with H_2O or D_2O, prior to start the cell. Two positions 10µm apart were probed in the cathode in order to make sure that the results are similar in two different areas of the electrode and to have reliable data even if there is a shift in the probed area due to the experimental setup or to the membrane swelling inside the cell. The positions are checked and adjusted for each condition prior to record the SANS spectra by recording a transmission profile across the thickness of the membrane electrode assembly (MEA) from anode to cathode. The SANS spectra were recorded for two sample to detector distances, 2m and 17.6m at 5Å during 10 minutes and 5 minutes. This represents 1 hour of SANS spectra recording on the four positions for each condition. Then, we started the cell and recorded spectra on the three components during operation, with H_2 and H_2O and also with D_2 and D_2O. We have recorded again the reference spectra after operation to check any evolution of the ionomer nanostructure in the catalyst layer after operation. All these steps required several hours, including the time to shift from one condition to the other, to stabilize the conditions, to record the spectra. The longest part is to shit from H_2O to D_2O and reverse, to make sure that the isotopic exchange is complete. Then, we ran 6 hours Accelerated Stress

Test and record time to time the electrochemical performances and properties, as well as SANS spectra. We have been able to extract reliable data during operation as well as the evolution of the ionomer swelling behavior within the electrode due to operation and degradation. So, we finally succeeded in obtaining the data of interest.



SANS spectra obtained on the cathode catalyst layer before, during and after aging.