Experimental report

07/08/2019

Proposal:	**TEST-2991**	**Council:**	4/2019
Title:	Operando fuel cell structural studies		
Research area:			

This proposal is a new proposal

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Samples: C + Pt + PTFE (CF2)n

Instrument	Requested days	Allocated days	From	To
D22	2	2	05/07/2019	07/07/2019

Abstract:

TEST-2991 : Operando fuel cell structural studies

Experimental report

The initial goal of this experiment was to study the structure of the electrode of a proton exchange membrane fuel cell (PEMFC) during Operation using Small Angle Neutron Scattering (SANS). The experiment has been simplified and the electrodes have been studied ex-situ as a function of the relative humidity (RH). The porous electrode (40-60% porosity) is made of a catalyst, consisting in Pt nanoparticles (3-5 nm) supported onto carbon nanoparticles (30-50 nm), blended with a proton conducted polymer. This polymer is a perfluorosulfonic acid ionomer, which presents a phase separation between the highly hydrophobic perfluorinated backbone and the acidic aqueous phase with the sulfonic acid groups. This nanophase separation gives rise to the presence of a so called ionomer peak around 0.1 -0.3 A^{-1} easily evidenced by SANS thanks to the contrast between the perfluorinated (CF_2) polymer backbone (SLD ~$4.2x10^{-6}$ A^2) and water ($-0.561x10^{-6}$ A^2) . In the electrode, the ionomer coats the catalyst in a few nm thick ionomer film and also forms aggregates within the pores. This proton conducting ionomer plays a crucial role in the operation and degradation of the PEMFC. The structure, composition and distribution of this ionomer has an impact on the effectiveness of Pt utilization by modifying the wetting properties of the electrode, namely liquid water removal ability, as well as the oxygen and proton access toward catalytic sites. The nanostructure and the transport properties of the ionomer, and at the end of the day, of the electrode, depends on the RH and temperature. SANS with contrast variation is a very suitable and efficient tool to study the structure of the ionomer and of the catalyst layer which can hardly be directly investigated by other techniques.

In this experiment, we have studied the structure of catalyst layers differing in ionomer content and type of ionomer before and after different operation as a function of RH and temperature (30 and 80°C) using H_2O and D_2O. For this purpose, a dedicated cell has been designed to study five samples up to a Q of 0.5 A^{-1}. We demonstrate the possibility of studying ex-situ using SANS the structure of the catalyst layer and ionomer in samples recovered after operation. We show difference in behavior depending on the type of ionomer and history of the sample. Especially, the ionomer in the aggregates within the electrodes swell more water after operation than before (Figure 1 and Figure 2). In the low-Q region, where information arises from the catalyst/ionomer interface, the scattered intensity is higher when D_2O is used as solvent. This result still has to be understood considering that the SLD of D_2O is very close to the SLD of catalyst, $6.335x10^{-6}$ $Å^2$ and $6.6x10^{-6}$ $Å^2$, respectively.

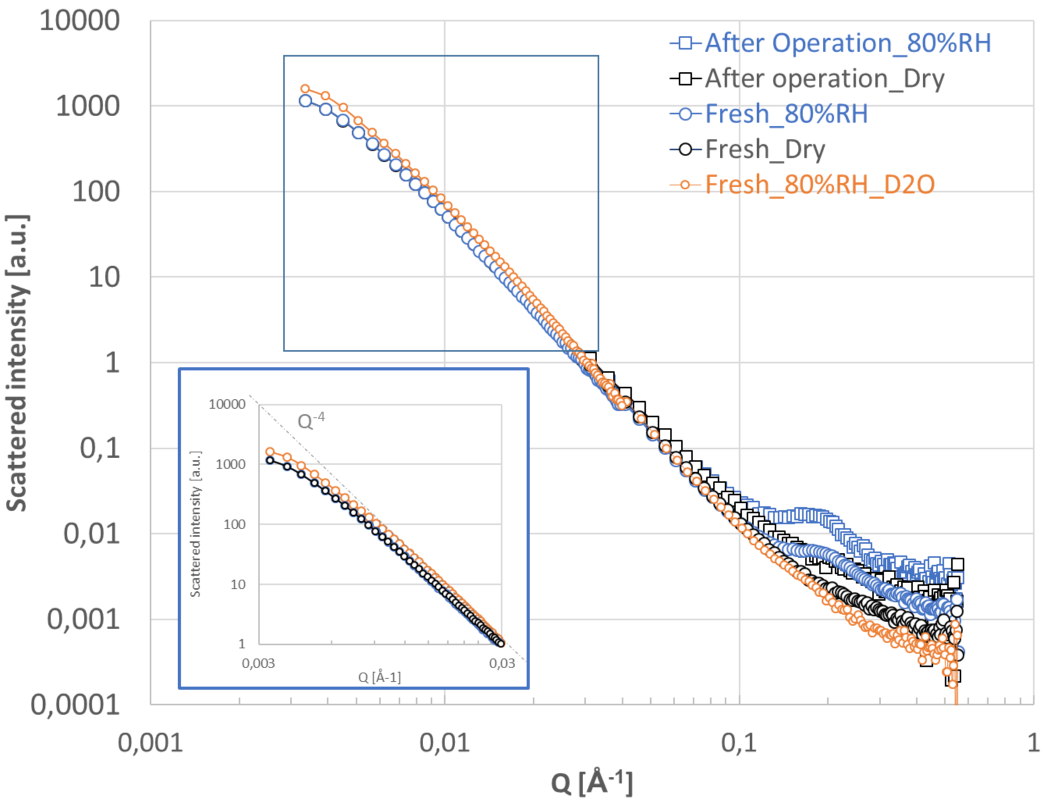

Figure 1: SANS spectra of dry and wet (80%RH) electrode before and after operation. Inserted a zoom-in of the low-Q region which gives information on the ionomer/catalyst interface.

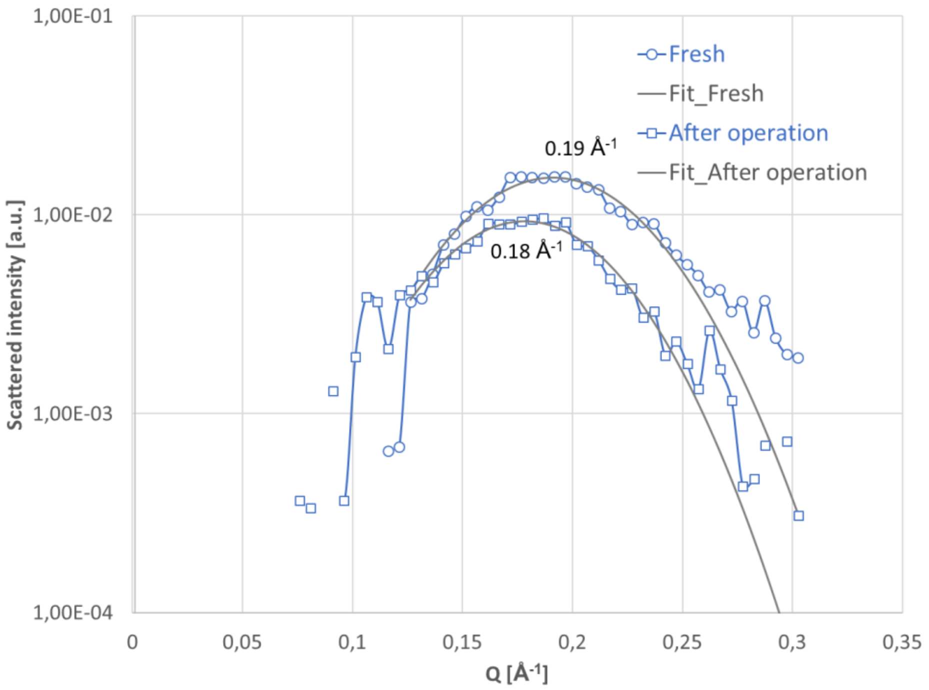

Figure 2: Extracted ionomer peak at 80%RH with its position obtained by fitting. Higher value of Q for the position of the ionomer peak means less water content in the ionomer