	Experiment title: Water-swollen Ionomer Membranes: -SAXS studies	Experiment number: SC836
Beamline: ID13	Date of experiment: from: 06/10/01 23h00 to 10/10/01 7h00	Date of report:
Shifts: 1 0	Local contact(s): Roth Stephan	*Received at ESRF:*

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Report:

The aim of this experiment was to study, using x-ray diffraction technique, ionic conducting polymer membranes out of the film plane. Since the membrane are usually between 50 and 150 microns thick, the idea was to study them using a very tiny beam (typically 10-15 micron in diameter) through the side of the membrane and to compare the experiment as we shine the membrane perpendicualry to the film surface. We wanted to keep a good angular resolution for SAXS purposes. The samples were placed in a cell in which we controlled the water vapor pressure.

Two types of membranes were studied: Nafion (perfluonated membrane) as reference of a isotropic structure (usually described in the litterature) and PIS (sulfonated polyimide membrane) a system that we have developped at the CEA and for which the experiments of swelling, conductivity and preliminary SANS have demonstrated that the structure is anisotropic.

First of all, the experiments were very successful and the results were ok for some of the membrane (Nafion) and less good for polyimide sample but because of membrane preparation (chemistry not really reproductible).

Nevertheless For nafion (115), we confirm our model of rodlike particle surrounded with the electrolyte. When the film is oriented by stretching we can show that we have typically a scattering spectrum like a nematic mesophase. (see figure 1).

Unfortunetly, the resolution is not sufficient to study the scattering upturn at very low q values (<10-2Å-1) in order to analyse large scale fluctuations parallel and perpendicular to the particles but this is the first time that the structure can be describe as a mesophase.

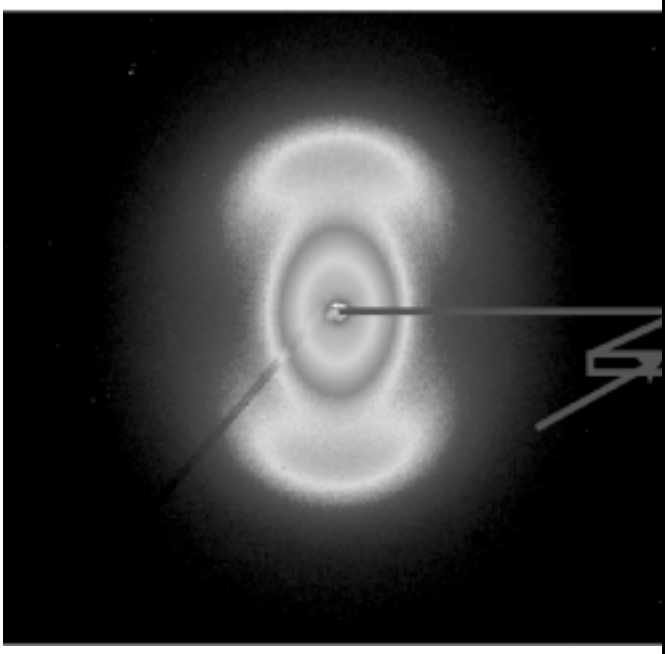

Fig 1

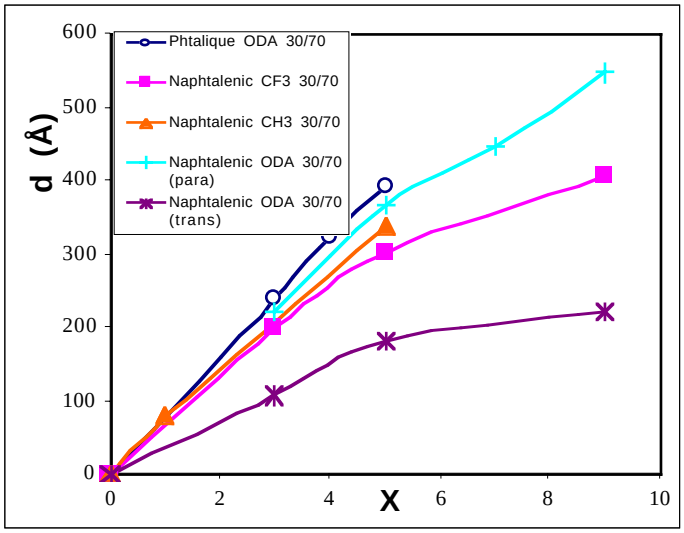

Fig. 2

For PIS, a copolymer with a an ODA3-4';4-4' diamine in the hydrophobic part we have studied different sequences variing the size of the hydrophilic bloc, keeping the total charge constant and reciprocally variing the charge keeping the size ratio betwen both blocks, constant. Since the ionomer peak observed in the plan of the membrane do not move as we swell the membrane even though we observe a thickness variation from 20 to 30%, we expected to observe this ionomer peak shifting in this scattering geometry.

Although the samples were not so good like in the previous experiment (SC701) we confirm what we observed previously but still some difficulties to model the system persist. If we don't take in account two types of population in the bloc copolymer from the chemistry (and the chemist are not able to confirm this) it is impossible to analyse correctly our data.

See in fig. 2 the different characteristic lengths observed in plane and out of plane of the film as a function of the ionic monomer bloc size variation.

First results are submitted to :

J.F. Blachot, O. Diat, J. L. Putaux, A.L. Rollet, L.R. Rubatat, C.Valois, M. Müller and G. Gebel
"**Sulfonated polyimide : structural anisotropy and transport properties.**"
Submitted to J. New Mater. Electrochem. Syst. (2002).

A second article is in preparation.