	Experiment title: Relaxation of the ionomer structure after stretching: time-resolved SAXS-WAXS study	**Experiment number:** SC1150
Beamline: ID02	**Date of experiment:** from: 20/02/03 7h00 to 23/02/03 7h00	**Date of report:**
Shifts: 9	**Local contact(s):** P. Panine	*Received at ESRF:*

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Report:

The relaxation studies were not so successful due to our stretching system for we cannot really controlled the applied force. Nevertheless we observed interesting relaxation through the shift o the ionomer peak position and intensity as a function of time. We planned to mount a dynamical stretching machine directly on the beamline to solve our problem.

Anyway we could analyse the interesting WAXS and SAXS data by correlating polymer chain orientation at different length scale and birefringence studies.

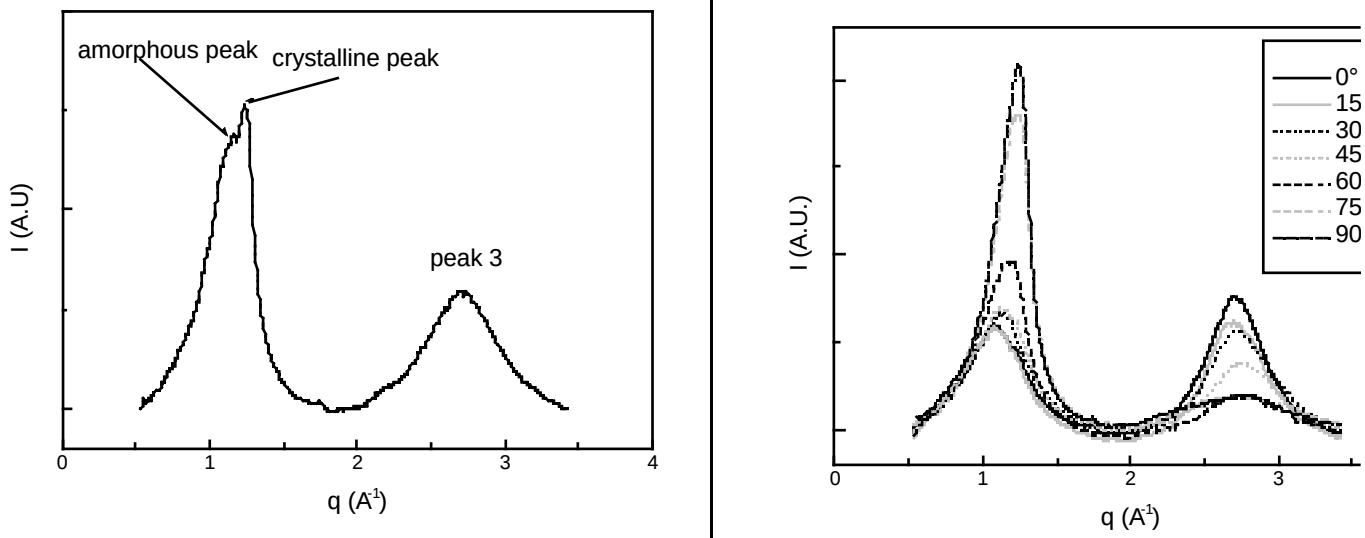

Figure 1: WAXS result Nafion 125: at meridian (left picture) for at DR=100%, and DR=229% for different azimuthal angles (right picture).

We analysed the maximum intensity of the amorphous and crystalline paek as function of the azimuthal angle following a standard procedure. Similar approach was followed for the ionomer peak and the corresponding distribution were analysed using the Hermann factor f which quantify the degree of orientation (for two type of Nafion, more or less crystalline):

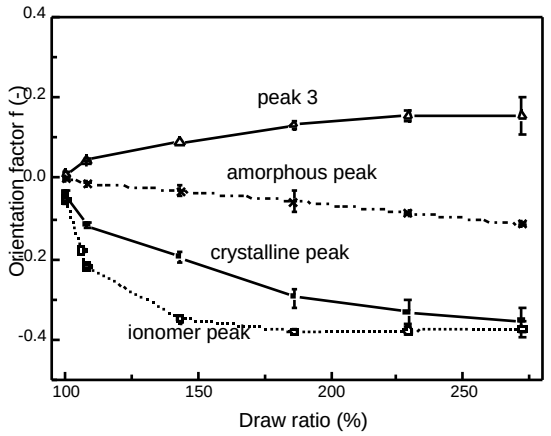

Figure 2 : Orientation factors of the different peaks of Nafion 117 (left) and Nafion 125 (right) as function of draw ratio.

The scattering intensity distribution as a function of the azymuthal angle in the detector plan parallel to the surface of the membrane has been analysed at different q-vector using SAXS and WAXS techniques. These different q-vectors correspond to the position of four characteristic peaks of the Nafion x-ray spectra, which were studied as function of draw ratio. We have determined the orientation parameters at different length scale and pointed out some correlation which can be analysed in two steps as a function of the drawing ratio. In the frame of fibrillar structure of Nafion made of elongated polymeric aggregates, we first observe a pre-alignment of the crystallites through an orientation of bundles of aggregates and second an almost perfect orientation of these crystallites through a creep of the structure at large draw ratio [ref à venir, A. de la Rosa, F. Volino, J-F. Blachot and O. Diat]. From the comparison between the birefringence and the X-ray scattering data, it appears that the obtained values for the intrinsic amorphous and crystalline birefringence are reasonable in comparison with published data for aliphatic polymers. Only the molecular structure and its orientation is of importance for the birefringence.

Results presented at the fuel cell and ion-containing polymer Gordon Conference (July 2003) and submitted to Macromolecules.