EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: In situ x-ray diffraction of organic-inorganic hybrid capacitors as a function of electrical poling at different temperatures	**Experiment number**: MA-5418
Beamline: ID11	**Date of experiment**: from: 2nd November 2022 to: 4th November 2022	**Date of report**: 27th February 2023
Shifts: 6	**Local contact(s)**: **LAWRENCE BRIGHT Eleanor**	*Received at ESRF:*

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Report:

We used high spatial resolution X-ray diffraction (HRXRD) to study device structure. An X-ray energy of 43.5 keV was selected and a vertical focussed X-ray beam of 0.8 μm was used. The high X-ray energy and the small beam minimize absorption by the sample and hence degradation by X-rays. The X-ray beam goes through the sample which is mounted on a hot plate positionned on a goniometer. The X-ray diffraction pattern was recorded on a two-dimensional (2D) detector with a 0.63 μm pixel size during the vertical scan of the sample (scan step = 0.5 μm). In order to take advantage of the small beam size, the X-ray pathway inside the sample has to be kept as small as possible. The devices have an elongated shape and the X-ray path across the device is along the smaller sample size, approximately 2 mm. In order to precisely probe the polymer film, the X-ray beam direction was set parallel to the surface. A high-resolution X-ray camera was used for alignment purposes. After the alignment procedure, the polymer film can be considered extended parallel to

the X-ray beam. The requirement of parallelism between the sample surface and the X-ray beam direction is essential to get the optimum spatial resolution.

We have studied thin films of polyvinylidene fluoride trifluoroethylene P(VDF-TrFE) based polymers. A poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) film was deposited by screen printing on a polyimide (PI) substrate. An oven was used for *ex situ* sample annealing. The PVDF-based film is sandwiched between PEDOT:PSS electrodes. The active layer of the devices was either pure P(VDF-TrFE) or P(VDF-TrFE) with oxide particles. The devices were studied *in situ* during hot plate annealing as a function of temperature up to 120 °C and *operando* as a function of electrical polarization.

For each depth position, the HRXRD image is integrated azimuthally in order to get a 1D diffracted intensity as a function of q. As an example, Figure 1 shows the XRD intensity I=f(q) for a 10 µm thick P(VDF-TrFE) film during annealing on a hot plate at 80 °C.

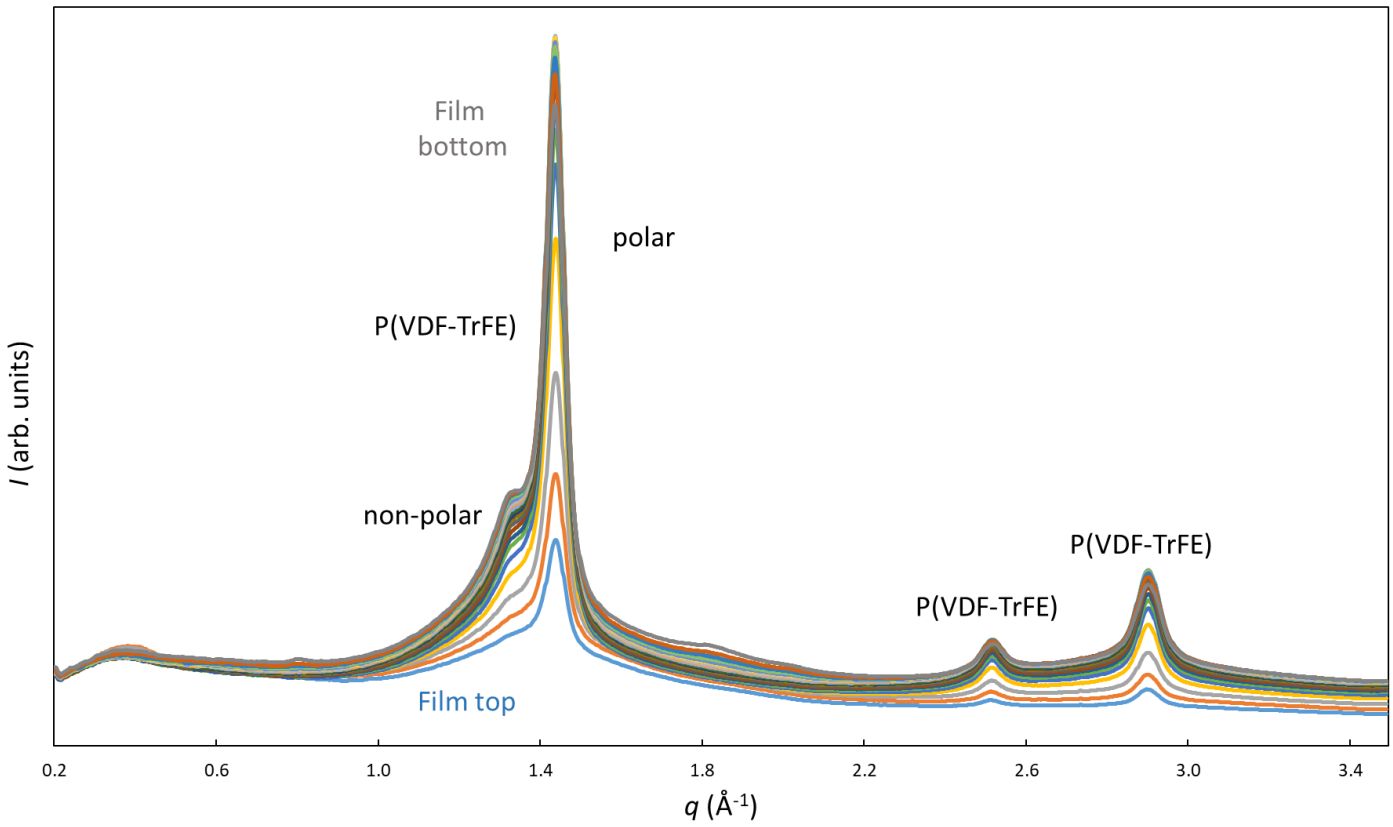

Figure 1: Time-resolved (TR)-HRXRD patterns collected at a hot plate temperature of 80 °C during the vertical scan of the 10 µm thick P(VDF-TrFE) film (scan step = 0.5 µm).

The analysis of the XRD scans as a function of depth is under way for the different devices studied *in situ* as a function of hot plate temperature up to 120 °C and studied *operando* as a function of electrical polarization.