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Pre-ordering and the early stage crystallization of PET (Poly(ethylene terephthalate)

We performed grazing incidence x-ray scattering studies on ultra thin (50 nm -150 nm) films of initially amorphous PET (Polyethylene terephthalate) to study the early stage ordering prior to crystallization. We think that a thorough understanding of the early stage evolution of this industrially important material will help us understand the dynamics of how PET crystallizes and also what impact a free surface has and if there is enhancement in the rate of crystallization at the free interface.

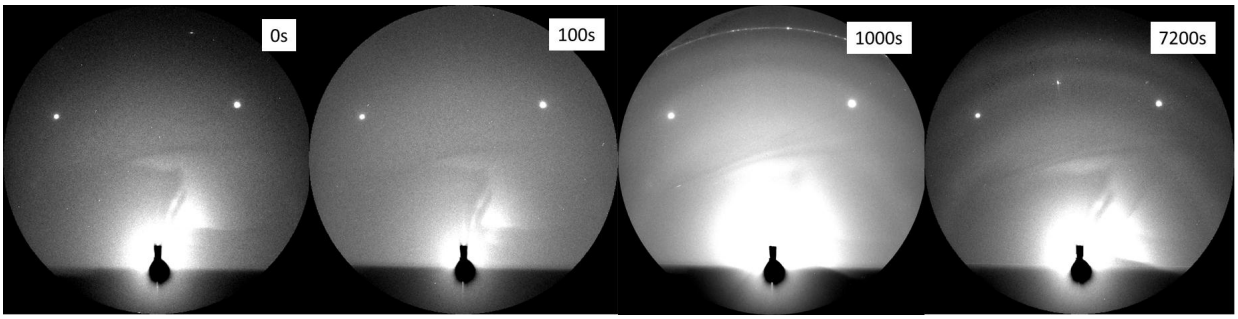

Figure 1. The GIWAXS images were captured at the XMaS CRG beamline at the ESRF. The grazing incidence angle was from 0.12 to 0.6 $^{\circ}$, camera length was approximately 1.0 m, and the x-ray energy was 15KeV. The sample film thickness was 50 nm and the samples were isothermally annealed at 120 $^{\circ}$C for 0, 100, 1000, and 7200 seconds. We can see the diffraction ring patterns appeared after 1000 seconds, and also clearly at 7200 seconds.

From the two dimensional detector data above, we can see the following features.

i) Our sample preparation route was able to produce entirely amorphous PET thin films.
ii) GIWAXS images showed clearly that thin PET films were crystallized by annealing.
iii) The kinetics, that is the evolution of the PET crystallization are coincident with the results of the Ellipsometry and atomic force microscopy.

Optical microscope images the samples show spherulite which indicates the scheme of the crystallization. However, thanks to GIWAXS measurement, we could further confirm the detail of crystals at various time intervals and compare them to bulk crystals, for example, plane spaces. This kind of quantitative analysis is in progress now. In future experiments we will look for the grazing incidence small angle scattering (GISAXS) signal as well as the grazing incidence wide angle signal (GIWAXS).