



Low detection limit of crystallinity

Low polyolefin crystallinity fraction down to 0.3 % was determined during a kinetic experiment using the Xeuss 2.0 SAXS/WAXS system in combination with an integrated temperature control stage, the Linkam HFSX350.

Introduction

Mechanical and physical properties of solid polymers are resulting from its crystallization kinetic which affects both the nanoscale structure and the level of crystallinity. The semi-crystalline state can be finely characterized at short length scale, identifying the crystallographic order, while the morphology of the crystalline phase can be described by investigation at long length scales.

A Wide Angle X-ray Scattering (WAXS) investigation of the crystalline fraction of an isotactic polypropylene (i-PP) corresponding to a typical processing route has been carried out as a function of temperature.

Measurements & results

A pellet of i-PP (Innovia Films, U.K.) was encapsulated in thin aluminum foil (to avoid direct contact with air and ensure good thermal contact). This polymer sample was then fixed and mounted into the temperature control stage (Linkam HFSX350). Real time crystallization of i-PP was measured in the WAXS configuration of the Xeuss 2.0 SAXS/WAXS system. A quenching experiment at 148°C from full molten state was performed, with repeated WAXS measurements of 200 s exposure time, collected after the quench.

Figure 1 shows the crystalline fraction as a function of the time after quenching, as measured by WAXS, thus proving the ability to characterize the crystallization kinetic. Please see AN-XE01 for crystallinity determination¹. It can be inferred from Figure 1 that the low detection limit of the crystallinity level is 0.3 % for this polymer system.

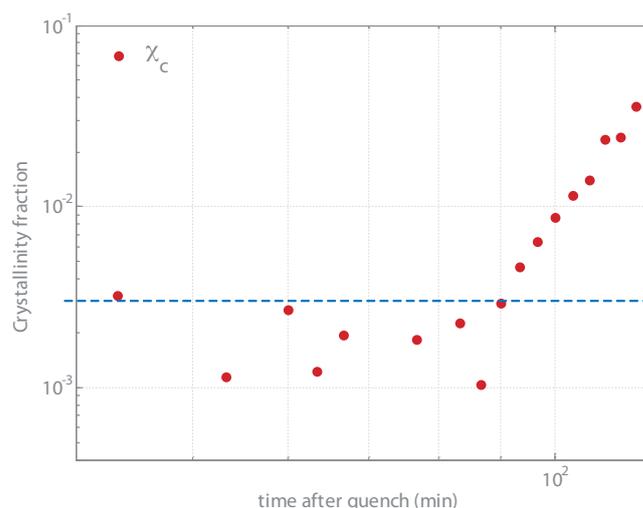


Fig. 1 - Evolution of the crystalline fraction as a function of time. Exposure time = 200 s per point.

To go further

To enhance the understanding of the underlying mechanisms involved during the crystallisation process, a study of the material at a larger scale should be carried out simultaneously with a WAXS measurement. Such a simultaneous SAXS/WAXS experiment as performed at synchrotron facilities² is now available on the Xeuss 2.0 SAXS/WAXS system.

¹AN-XE01 "Determination of polymer crystallinity".

²Panine et al, Polymer, 2008, 49, 676-680.