Determination of polymer crystallinity

The level of crystallinity of a polyolefin 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

Most polymer transformations from raw material to a finished product necessitate a processing step involving a melt (or molten state). Once cooled down, the final product exhibits a semi-crystalline state that is a result of how it was processed.
The semi-crystalline nanostructure developed during the solidification process primarily controls the mechanical and physical properties of solid polymers. Investigation and definition of the crystallinity is therefore of great interest both for fundamental and industrial approaches. Wide-Angle X-ray scattering (WAXS) measurements of isotactic polypropylene (i-PP) were performed under temperature control.

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 200 s exposure time. Figure 1 shows the 1D scattering curves and corresponding analysis of the crystalline and amorphous fraction of sample 16 minutes after quench. Crystallinity is then calculated from the ratio of crystallized versus amorphous curves integral following the procedure described elsewhere[1].

Since the Xeuss 2.0 SAXS/WAXS system has a very low signal-to-noise ratio, investigation in the lab of low levels of crystallinity is enabled, even during real time crystallization. Hence, determination of levels of crystallinity well below 5% is allowed[2], as demonstrated in Figure 1. Crystalline level is here estimated to 1,1%.

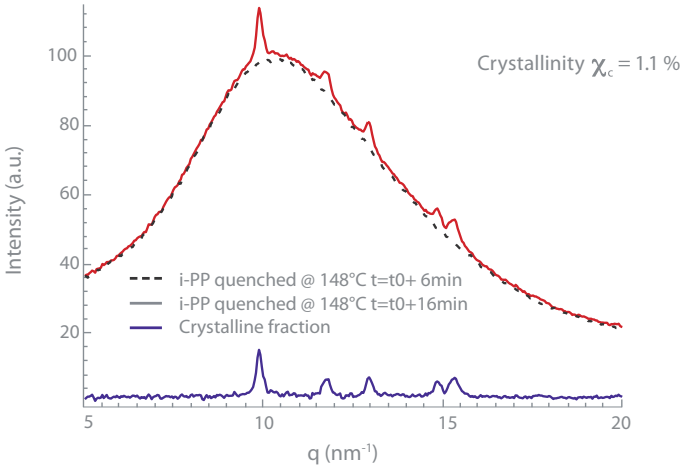

Fig. 1 -1D scattering curves and estimation of crystalline fraction. Time exposure = 200 s

To go further

Mechanical and physical properties of the solid polymer result from its crystallization kinetics that effect both the nanoscale structure and the crystallinity. Morphology of the crystalline phase can be described by characterization at long length scales, which means SAXS investigation, while short length scale measurements (WAXS) enable the description of the crystallographic order. To enhance understanding of the underlying processes, evolution as a function of temperature at both length scales should be investigated simultaneously, as performed at synchrotron facilities[1]. Such simultaneous SAXS/WAXS measurement capability is provided on the Xeuss 2.0 SAXS/WAXS system.

[1]Panine et al, Polymer, 2008, 49, 676-680
[2]Wang et al, Macromolecules, 33, 978-989

Xenocs SA - 19 rue François Blumet - 38360 Sassenage - France
Phone: +33 (0)4 76 26 95 40 - Fax: +33 (0)4 76 26 95 49
E-mail: sales@xenocs.com - www.xenocs.com