X-RAY DIFFRACTION

SAXS/WAXS Investigation of Isotactic Polypropylene (iPP) using the D8 Family with EIGER2 R 500K Detector

Lab Report 93

Introduction

This lab report describes the capabilities of the D8 family systems combined with the EIGER2 R 500K detector for complex analyses of semi-crystalline polymers through 2D Small Angle X-ray Scattering (SAXS) and 2D Wide Angle X-ray Scattering (WAXS) measurements. These methods provide insights into different levels of polymer organization.

SAXS examines the morphology related to the size of the crystalline lamellae and their periodic organization, while WAXS investigates the crystalline order and degree of orientation at an atomic length scale.

Semi-crystalline polymers are widely used in various applications due to their stiffness, strength, and toughness. These properties are closely related to their crystallinity and degree of orientation, which can be studied using X-ray scattering methods. Specifically, iPP is often used in blends with other polymers or in composites with fillers or fibers to enhance properties. Applications for these thermoplastic polymers include automobile parts, packaging films, and plastic containers.

Two samples provided by the Department of Mechanics of Solids, Surfaces, and Systems at Twente University were studied. The combined SAXS and WAXS measurements on an iPP film (Sample 1), which was compression molded using a hot press and subsequently cooled to room temperature, enable the study of nanostructural morphology (SAXS) and crystallinity (WAXS).

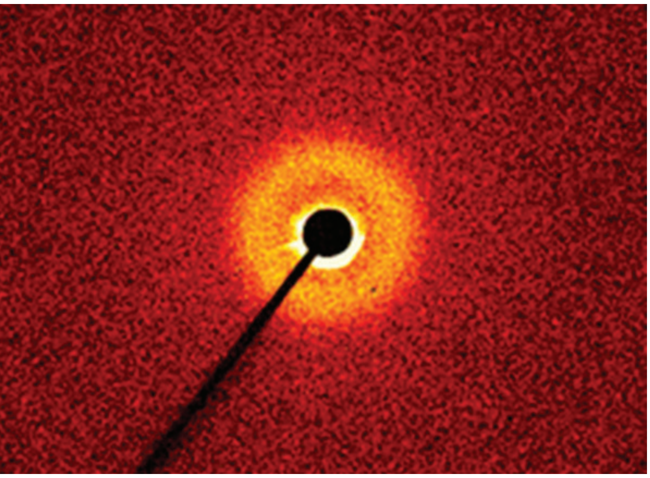

Figure 1a 2D SAXS data of Isotactic Polypropylene (Sample 1).

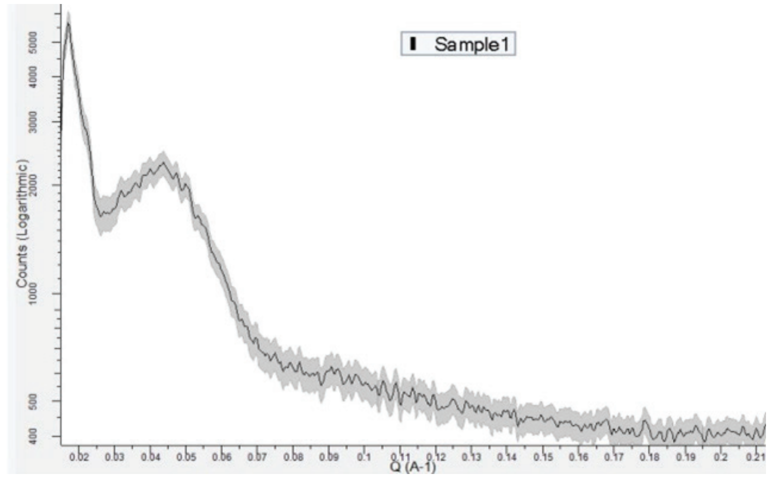

Figure 1b Radial integrated 1D SAXS data of Isotactic Polypropylene (Sample 1), using DIFFRAC.SAXS software. The gray area represents the error band based on the Poisson distribution of counts.

To investigate the anisotropy of crystal orientation induced by a shearing step at high temperature and pressure (1200 bar), another iPP film (Sample 2) was measured in the WAXS region.

The measurements were performed in transmission geometry using the EIGER2 R 500K detector. This detector has the advantage of covering a large part of the scattering signal in a single frame, thanks to its large active area of 2,978 mm² and flexible sample-to-detector distance.

For the SAXS measurements, the sample was positioned at a sample-to-detector distance of 300 mm, optimized for both q-resolution and q-coverage. The primary beam was collimated using a set of three circular pinholes, providing a beam size on the sample of 200 µm. The measurement time per frame was 1000 seconds. To improve the signal-to-background ratio, a panoramic evacuated flight tube (EFT) along with a corresponding beamstop was utilized.

The WAXS measurements were conducted using the same primary collimating set of pinholes at a sample-to-detector distance of 23 mm, allowing for a 2θ coverage of up to 50°. The measurement time per frame was 600 seconds.

The 2D SAXS pattern (Fig. 1a) and the corresponding radial integrated 1D plot (Fig. 1b) show a strong correlation peak, indicating the periodic organization of crystalline lamellae. The DIFFRAC.SAXS software was used for the radial integration of the 2D pattern into the 1D scattering plot.

The WAXS measurements were further analyzed using the DIFFRAC.EVA software: The 2D frame (Fig. 2a) was azimuthally integrated and transformed into a 1D plot. The phase identification is shown alongside the 1D plot. Additionally, the DIFFRAC.EVA software can compute the degree of crystallinity (Fig. 2b). The determination of crystallinity employs a two-phase model, where the sample consists of crystalline and amorphous components. In the 1D plot, the amorphous part is displayed in blue. The crystallinity determined here is 64.3%.

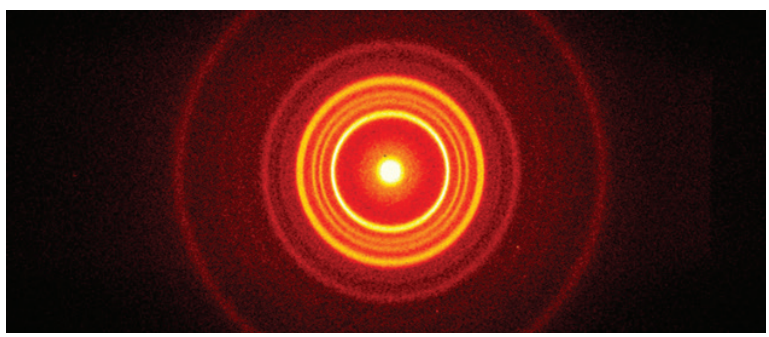

Figure 2a 2D WAXS data of Isotactic Polypropylene (Sample 1).

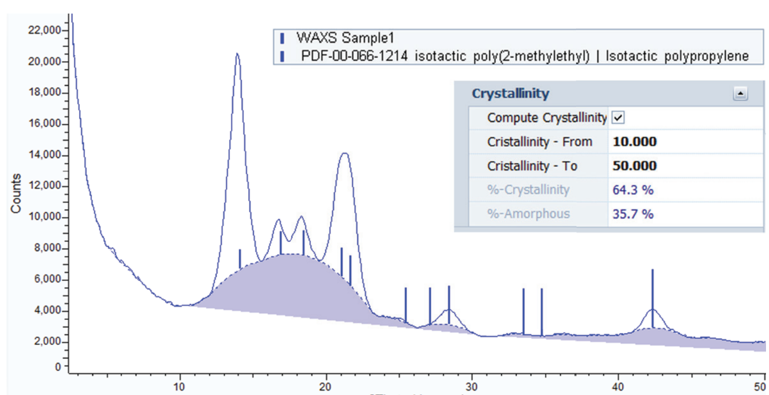

Figure 2b 1D scattering pattern and calculated amounts of crystalline and amorphous content.

A clear preferred orientation is evident in the 2D WAXS pattern of Sample 2. The intensity versus gamma direction is shown in the 1D graph for the (040) reflection, obtained after polar integration of the 2D frame. The black cursor in Fig. 3 indicates the integration area. The degree of orientation can be easily estimated from the value of the Full Width at Half Maximum (FWHM), which is determined using DIFFRAC.EVA. For a measured FWHM value of 36°, a degree of orientation of (180° - 36°)/180° ≈ 80% is obtained.

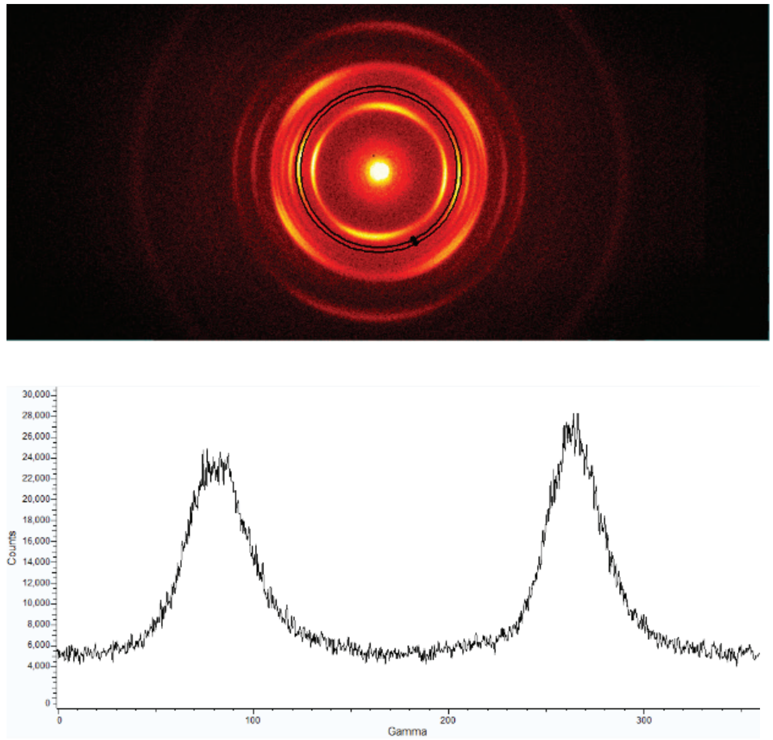

Figure 3 2D pattern and corresponding 1D polar integration (Sample 2).

Summary and Conclusion

This report details the capabilities of the D8 family systems combined with the EIGER2 R 500K detector for analyzing semi-crystalline polymers through 2D Small Angle X-ray Scattering (SAXS) and 2D Wide Angle X-ray Scattering (WAXS). Two isotactic polypropylene (iPP) samples were studied:

- Sample 1: Analyzed for nanostructural morphology and crystallinity after being compression molded and cooled.
- Sample 2: Investigated for crystal orientation induced by high-temperature and pressure shearing.

Measurements were performed using the EIGER2 R 500K detector, optimizing sample-to-detector distances for both SAXS and WAXS.

- SAXS measurements revealed a strong correlation peak, indicating periodic organization of crystalline structures.
- WAXS analysis involved azimuthal integration of the 2D frame, leading to a 1D plot that identified phases and calculated a degree of crystallinity of 64.3%. The WAXS pattern of Sample 2 showed a clear preferred orientation, with the (040) reflection analyzed to determine the degree of orientation to be 80%.

Author: Livia Chitu and Martin Zimmermann

Bruker AXS
info.baxs@bruker.com

bruker.com

Worldwide offices
bruker.com/baxs-offices

Online information
bruker.com/xrd