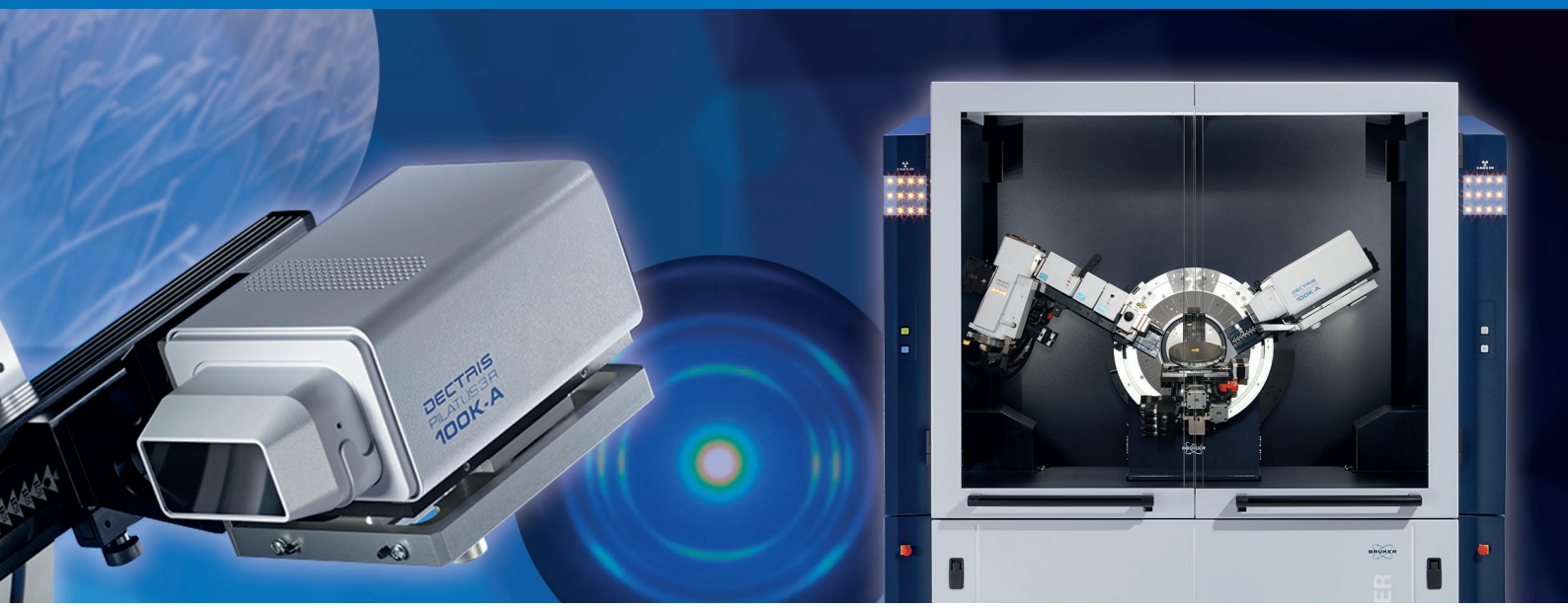

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D8 DISCOVER with PILATUS3 R 100K-A XRD² of Polymers

The D8 family of diffraction solutions combined with the DECTRIS PILATUS3 R 100K-A hybrid photon counting (HPC) pixel detector is an innovative 2D x-ray diffraction (XRD²) solution that is uniquely suited for multipurpose modern materials research characterization. In this report, we present the capabilities of this system in a transmission diffraction configuration for the analysis of polymer samples for applications such as phase identification (Phase ID) and texture analysis.

Introduction

A polymer is a macromolecule that is made up of long chains of repeating subunits. The composition, structure, and form of the polymer determine its properties and therefore proper characterization of these parameters is critical. Polymers are often synthesized into fibers, sheets, and other solid forms. The properties of these types of polymers are strongly influenced by their crystallinity, crystal structure, and texture which can be investigated using X-ray diffraction (XRD). As these types of polymers typically have large d-spacings, low x-ray absorption, and some degree of preferred orientation (texture), transmission scattering utilizing a 2D detector (XRD²) is an ideal way to characterize these samples.

Measurement

Polypropylene samples collected from a sheet and an extruded tube were examined. The samples were measured in transmission with a D8 DISCOVER equipped with the IμS micro-focus source and PILATUS3 2D detector. A 300 micron collimator was used for this analysis. Due to the high count rate capability of this detector, no beamstop was required.

Scattering patterns were collected at a close sample to detector distance to maximize scattering coverage and speed and at an extended distance to achieve best peak resolution. At larger distances the full diffraction pattern was collected by acquiring several images at different diffraction angles. The sample to detector distance was automatically detected and no special detector aberration corrections were required enabling both measurements with very little effort.

Measurement

The images in Figure 1 were collected with a sample to detector distance of 2.7 cm for 5 minutes, with 360˚ gamma coverage to 2θ = 30˚ and limited gamma coverage to 2θ=56˚. This coverage is ideal for mapping and high throughput applications.

In order to maximize resolution, the detector was pushed back to a detector distance of 7 cm and 3 images were collected, 2θ=-20˚, 0˚, +20˚ resulting in 360˚ gamma coverage to 2θ = 30˚. The images resulting from these measurements are shown in Figure 2.

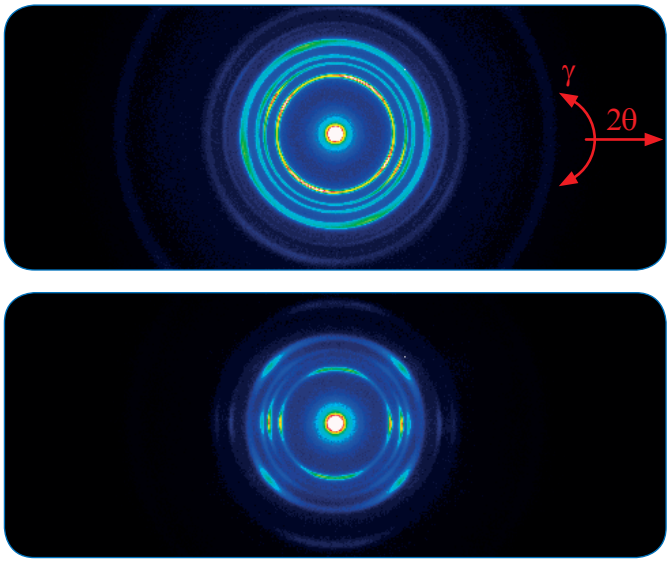

Figure 1: Images of two polypropylene samples collected with a sample to detector distance of 2.7 cm.

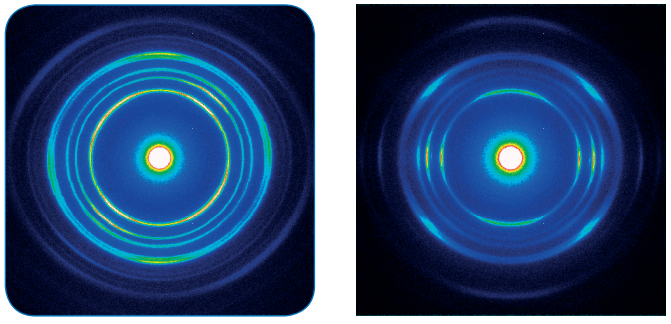

Figure 2: Images of two polypropylene samples collected with a sample to detector distance of 7 cm.

Results

To perform further analysis, the images from Figure 2 were brought into DIFFRAC.EVA, and integrated into a conventional 2θ plot. In addition to phase identification, peak width, correlated to the microstrain and crystallite size of the polymer, can be used to understand the effects of processing on the structure of the material.

Figure 4 shows a radial integration of the (110) polypropylene reflection, located at ~14˚ 2θ. By measuring the FWHM of the reflections in the radial integration, the degree of orientation can easily be quantified.

With conventional 1-dimensional systems, important morphological information, such as texture, is easily missed. With exceptionally large 2 theta and gamma coverage, this morphological information is available in every measurement performed with the D8 DISCOVER with PILATUS3 detector in a 2-dimensional diffraction (XRD²) configuration.

Author

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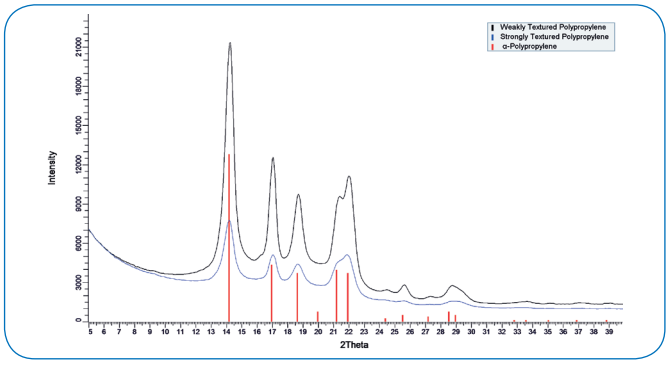

Figure 3: Radial Integration of the images shown in Figure 2.

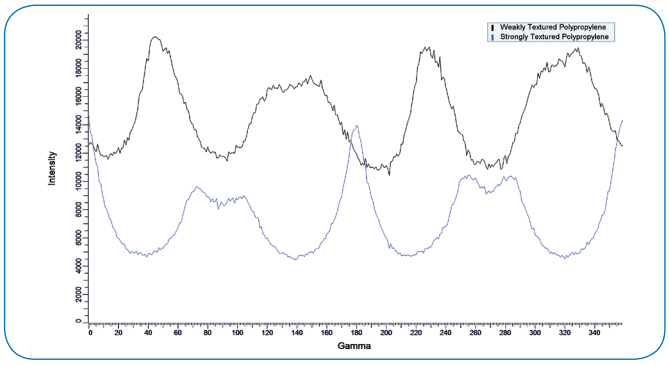

Figure 4: Polar Integration of the images shown in Figure 2.

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