Application note

Determination of the degree of crystallinity in PE using ARL X'TRA Companion X-ray Diffractometer

Introduction

The degree of crystallinity (DoC) in polymers is determined by analyzing their molecular structure and arrangement. This measurement is crucial as it provides valuable insights into the mechanical, thermal, and optical properties of the polymer. X-ray diffraction (XRD) is a widely used technique to determine the degree of crystallinity in polymers.

XRD is the reference method to determine DoC because X-rays interact with crystalline and amorphous parts of the polymer sample which yields a unique diffraction pattern. Refinements of that pattern allow a direct determination of DoC.

XRD is advantageous due to its non-destructive nature, high sensitivity, and ability to provide quantitative data. It can detect even low levels of crystallinity and is applicable to a wide range of polymers. Additionally, XRD can provide information about crystal size and orientation, which are crucial factors affecting the properties of polymers

Figure 1: ARL X'TRA Companion X-ray diffraction system.

Instrument and software

The Thermo Scientific™ ARL™ X'TRA Companion Diffractometer (c.f. Figure 1) is a simple, easy-to-use benchtop XRD instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion instrument uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available as an option. Thanks to the state-of-the art solid state pixel detector (55 x 55 µm pitch), the ARL X'TRA Companion Diffractometer provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS (Laboratory Information Management System).

Experimental

A polyethylene (PE) sample was measured in reflection mode using Cu Kα (1.541874 Å) radiation for 3 minutes using a fixed sample holder (c.f. Figure 2). An individual peak fit was performed using Profex software (BGMN) [1] and from the derived intensities the DoC was calculated. Additionally, average crystallite sizes were calculated for crystalline part

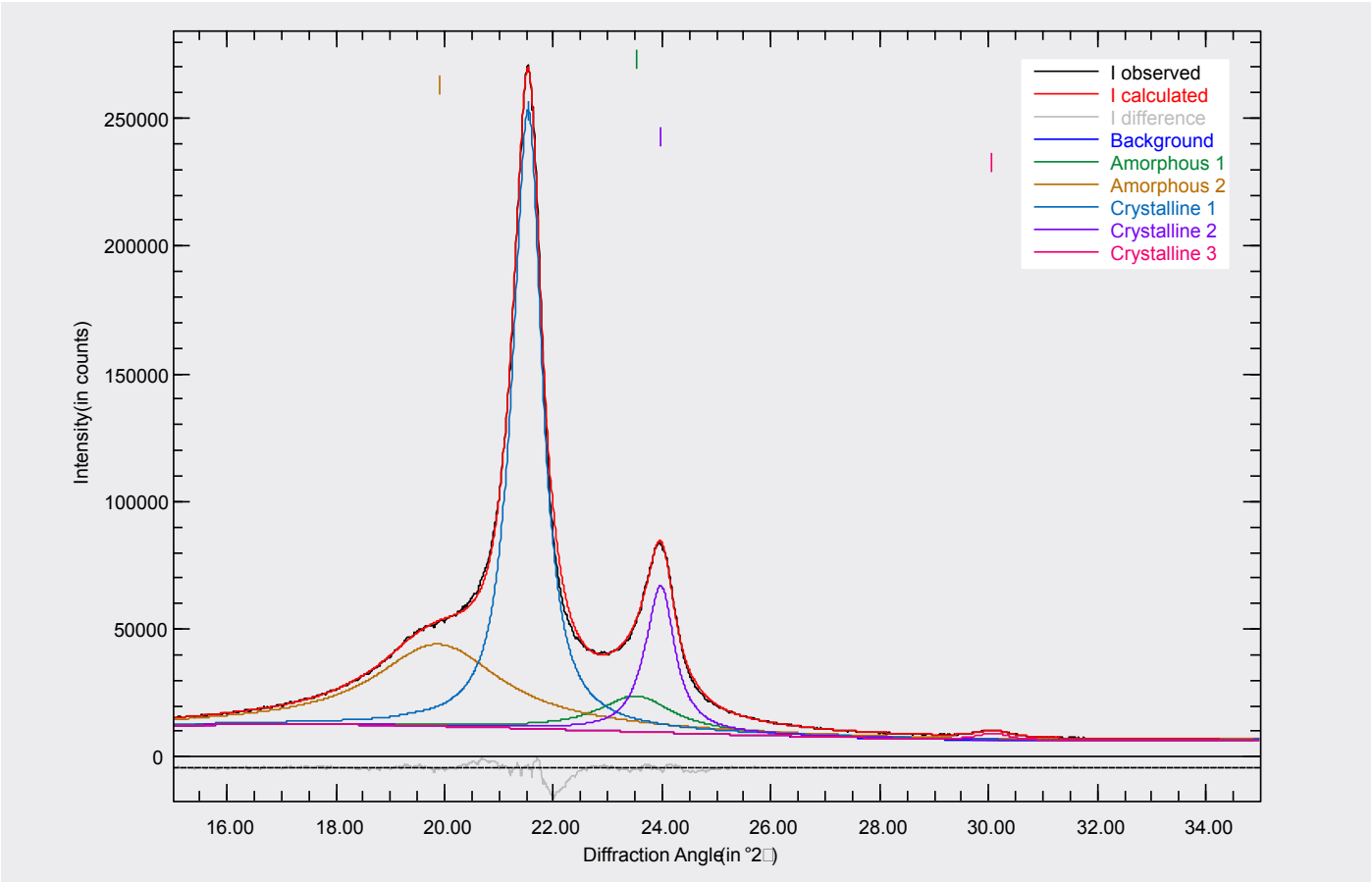

Figure 2: Measurement (3 minutes) of PE; Individual peak fits of amorphous and crystalline parts.

Results

The DoC is calculated as 62.3 % which is in the range of MDPE (medium density PE) with an average crystallite size (CS) of 13.3 nm for the crystalline part.

Conclusion

The ARL X'TRA Companion Diffractometer is perfectly suited to determine the degree of crystallinity and average crystallite size in a PE sample. The instrument is configured with one-click analysis functionality to enable flawless operation in an industrial quality control environment but also keeps the flexibility required for development laboratories.

1. **N. Doebelin, R. Kleeberg, J. Appl. Crystallogr.2017, 48, 1573-1580.**

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