APPLICATION NOTE

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Determination of the crystallinity in polymer samples using ARL EQUINOX 100 benchtop X-ray diffractometer

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Introduction

Polymer materials are used for a vast amount of applications in modern society, ranging from packaging to aerospace engineering. Of course, different type of applications demand different properties, which are determined by the structure of the polymer product. Not only the type of polymer (e.g. Polyethylene PE or Polypropylene PP) but also the crystallinity D are important observables for polymer materials. In a highly industrialized environment, it is equally important for users as well as producers to quickly screen products for the latter properties. A convenient and fast way is the usage of X-ray diffraction (XRD) combined with whole pattern Rietveld refinements to distinguish the crystallinity and type of the compound.

Instrument

The Thermo Scientific™ ARL™ EQUINOX 100 X-ray diffractometer employs a custom-designed Cu (50 W) or Co (15 W) micro-focus tube with mirror optics. Such a low wattage system does not require external water chiller or other peripheral infrastructure, allowing the instrument to be easily transported from the laboratory to the field or between laboratories.

Figure 1: ARL EQUINOX 100 X-ray diffractometer.

The ARL EQUINOX 100 (c.f. Figure 1) provides very fast data collection times compared to other conventional diffractometers thanks to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time. It is therefore well suited for both reflection and transmission measurements.

Experimental

For XRD measurements, sheet samples of PE (Polyethylene) and PP (Polypropylene) were measured in transmission geometry for five minutes under Cu-Kα radiation. The quality of the data obtained with the ARL EQUINOX 100 benchtop instrument is comparable to data from a typical high-power floor standing instrument. Qualitative and quantitative analysis was carried out using MDI JADE 2010 with the ICDD PDF4+ Organic database.

Table 1: Results of crystallinity determination using Rietveld's method in PE and PP materials

Sample	Reference D (in %)	D Rietveld (in %)	Amorphous Wt (in %)
PE 1	59.4	54.1	31.0
PE 2	29.3	23.1	61.9
PP 1	59.9	57.5	47.2
PP 2	45.3	39.7	73.6

Results

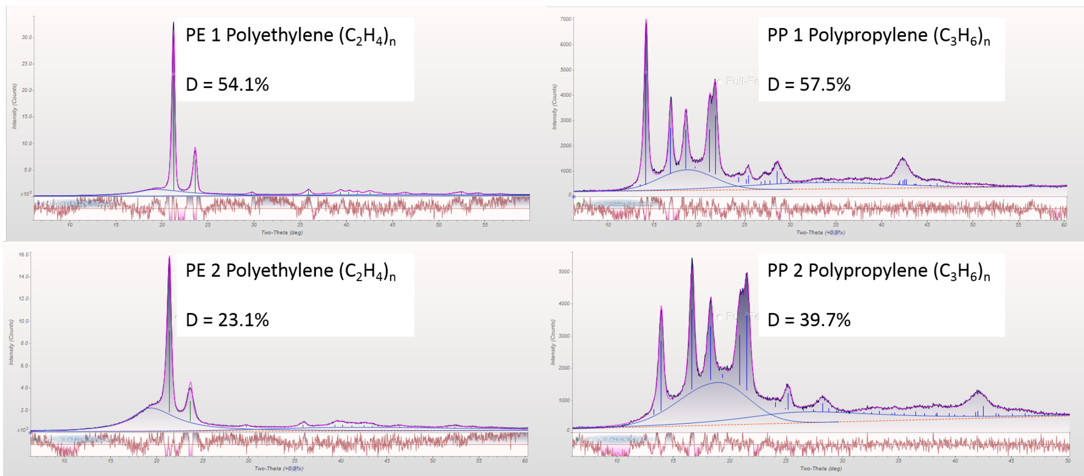
Qualitative phase analyses clearly reveal Polyethylene (PE) and Polypropylene (PP) type materials. (c.f. Figure 2). MDI JADE 2010 offers the possibility to directly determine D by using a standard-less refinement method where the intensity of the amorphous contribution is directly determined by deconvolution of the diffraction pattern using Rietveld's method (c.f. Table 1).

For the calculation of the amorphous content (in wt%) a density of 0.85 g/cm^3 was used. This method yields unambiguous results for PE and PP samples with high and low D values (c.f. Figure 2).

Conclusion

The ARL EQUINOX 100 benchtop XRD instrument in combination with the MDI JADE 2010 software suite and ICDD pdf4+ Organic database is a suitable solution to conveniently determine both, the type and the crystallinity of polymer materials, even for inexperienced users. The values are in the range of high (PE 1) and low (PE 2) density PE and isotactic (α-) PP, whereby the differences in crystallinity are influencing the mechanical properties of the polymers. With the latter method, in less than 10 minutes it is possible to determine the quality of polymer products in research labs as well as during the production process in order to reduce the cost and improve the quality.

Figure 2: Diffraction patterns of PE and PP with 5 min measurement time



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