thermoscientific

Analysis of Polymer Materials using Benchtop Thermo Scientific ARL EQUINOX 100 Diffractometer

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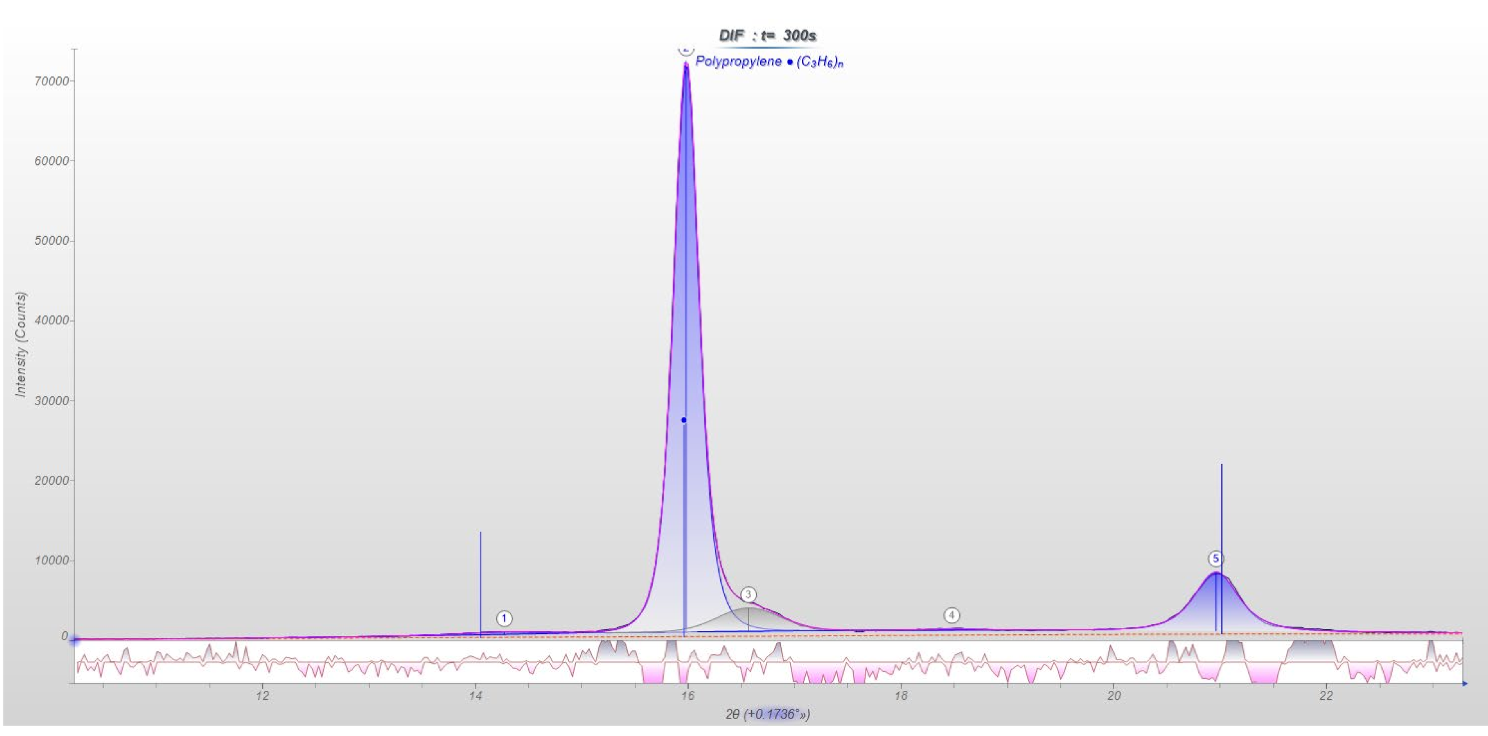
INTRODUCTION

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 polymer materials microstructures like in common metal alloys (e.g. steel) are observable which also influence the mechanical properties of these materials. Adding additives or pigments to polymer matrices is a well-known application and allows to tune e.g. optical or physical properties. 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 for QC/QA or research related applications.

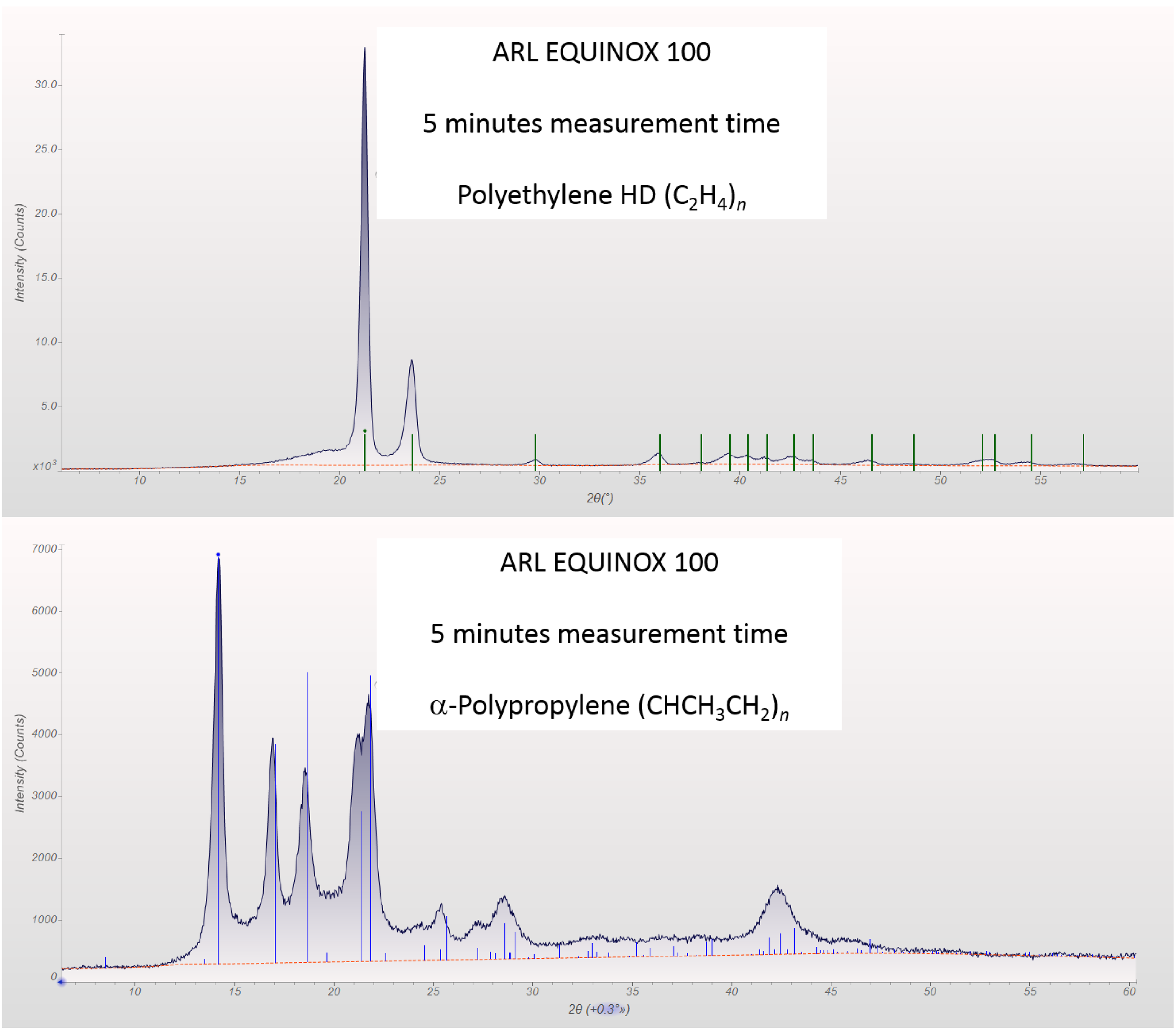
THERMO SCIENTIFIC ARL EQUINOX 100

POLYMORPHISM AND CRYSTALLITE SIZE



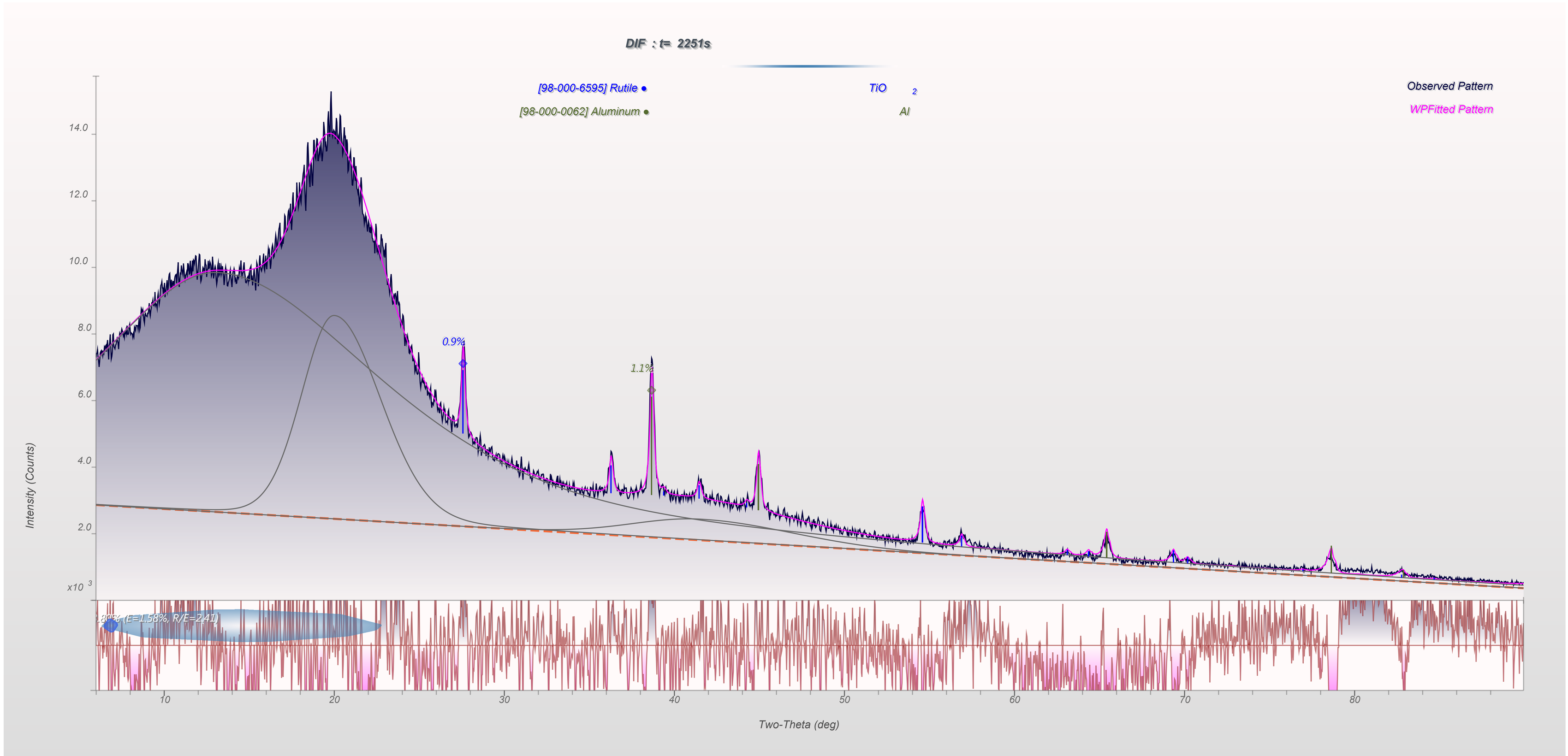
Comparing the data to ICDD PDF4+ Organic database clearly yields predominantly β-PP (blue) and traces of α-PP (grey) as results, whereas the domain sizes differ between β-PP (33 nm) and α-PP (10 nm). The intergrowth structure of α- and β- PP is crucial for the properties of the resulting material which is widely used in industrial applications.

IDENTIFICATION OF TYPE AND POLYMORPHISM



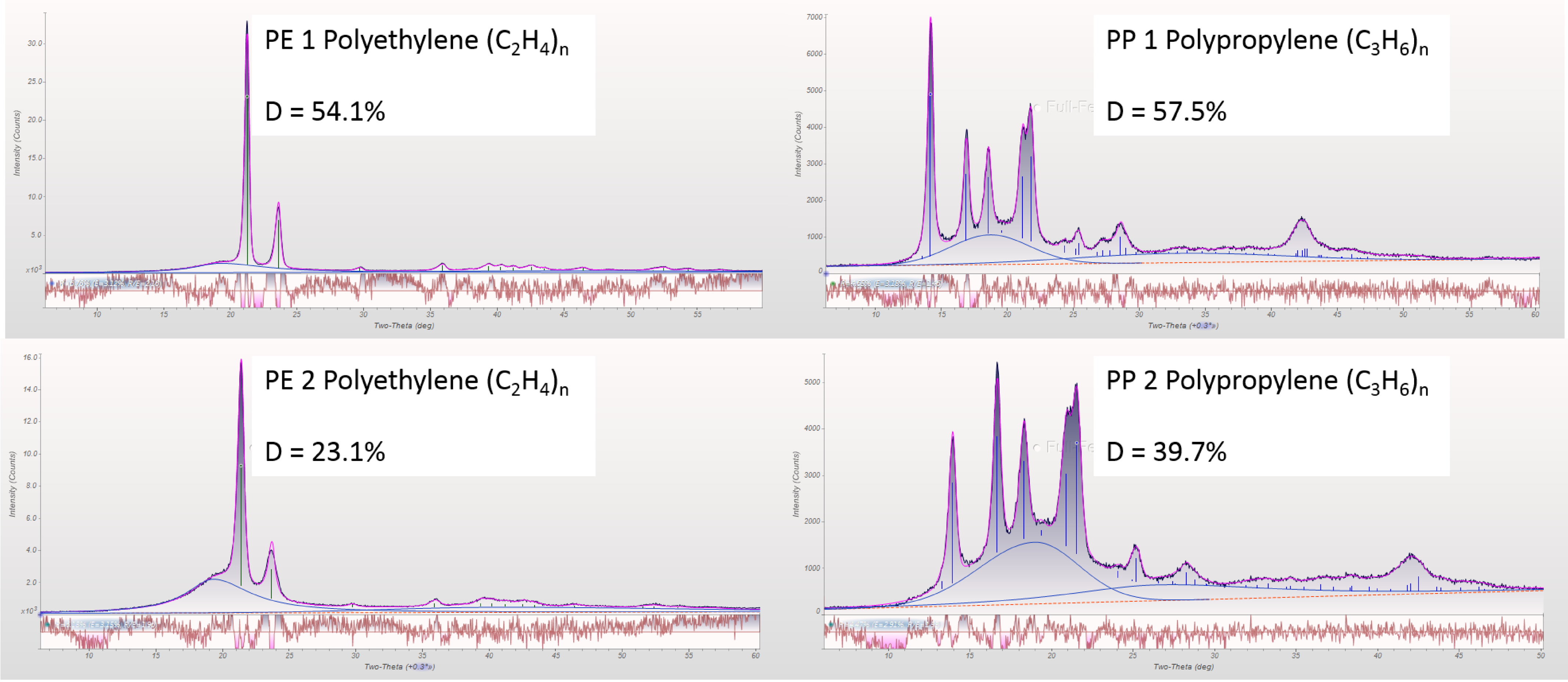
Comparing the data to ICDD PDF4+ Organic database clearly yields high density Polyethylene (HD PE) and Isotactic Polypropylene as results.

ADDITIVES AND PIGMENTS



Using XRD on a resin sample with embedded Al and TiO_2 (Rutile) particles allows to quantify an amorphous content of 98% (XRF:98%) , 0.9% TiO_2 (XRF: 0.74%) and 1.1% Al (XRF: 1.16%) by the means of Whole Pattern Rietveld refinement with MDI JADE 2010 which offers a standardless method for quantification of amorphous content.

DEGREE OF CRYSTALLINITY (DOC)



Comparing the data to ICDD PDF4+ Organic database clearly yields high density Polyethylene (HD PE) and Isotactic Polypropylene as results. Qualitative phase analyses clearly reveal Polyethylene (PE) and Polypropylene (PP) type materials. 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. For the calculation of the amorphous content (in w%) a density of 0.85 g/cm3 was used. This method yields unambiguous results for PE and PP samples with high and low D values.

INSTRUMENTATION AND EXPERIMENTAL

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.

The ARL EQUINOX 100 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

For XRD measurements, sheet samples of PE (Polyethylene) and PP (Polypropylene) were measured in transmission geometry for 5 min whereas the resin sample was measured 38 min using Cu-Kα radiation. The quality of the data obtained with benchtop ARL EQUINOX 100 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.

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