

CHARACTERIZATION OF CRYSTALLINITY IN POLYPROPYLENE BY WIDE ANGLE X-RAY DIFFRACTION

J. H. Butler, D. J. Winter*, and R. B. Ortega*

ExxonMobil Chemical Company, Baytown, TX

**AMIA Laboratories, The Woodlands, TX*

The level of crystallinity in isotactic polypropylene (i-PP) is an important parameter, not just for establishing relationships between final product structure and mechanical strength, optical and thermal properties, but also for quality control and product specification. Consequently, an accurate, inexpensive and timely measurement of this quantity is needed. Today, solid state NMR remains the only generally accepted method for absolute measurement of the crystallinity index in polypropylene. As an alternative, the potential to extract quantified information such as the absolute degree of crystallinity, average crystallite size and the extent of crystal lattice disorder from wide angle x-ray diffraction (WAXRD) patterns of semicrystalline polymers has long been recognized. However, the exacting level of analysis required to do this presents a challenge for conventional x-ray diffractometers and data processing systems.

We have developed a method for analysis of WAXRD patterns from i-PP materials that uses an efficient 2-D x-ray detector system and applies sophisticated peak profile analysis methods. Measurements of percent crystallinity (%WC) are obtained by ratioing the integrated intensities of the separated crystalline diffraction peaks to the sum of the crystalline intensities plus the integrated amorphous contributions. Peak halfwidths reflect the amount of disorder within the crystallites, as peaks can broaden as a result of either crystallite size reduction or lattice distortion. Consequently, our measurement of full width at half maximum of the (110) peak (FWHM₁₁₀) serves as an indicator of crystallite perfection. In addition to the level of crystallinity and degree of order, it is shown that accurate measurements of the type and relative amounts of different crystalline phases, e.g. the relative ratio of gamma to alpha phase crystallinity (W_{γ}/W_{α}), can also be determined.

The objective of the present work is to present our methods for data acquisition and processing and compare them with historical analyses. The method is demonstrated on three sample sets: compression molded pads, injection molded parts and melt blown fabrics. Discrepancies are discussed within the context of relative comparisons among samples within a set, versus absolute values obtained from other measurement methods, as well as a rigorous mathematical analysis of the x-ray diffraction data.