

Experimental Study of the Formation of β - and γ -Phase Isotactic Polypropylene and Estimation of the Phase Composition by Wide-Angle X-Ray Scattering

Introduction

Isotactic polypropylene (iPP) is a polymorphic material which can crystallise in three crystalline forms, denoted as α (monoclinic), β (hexagonal) and γ (triclinic). During high undercooling, a mesophase, usually called 'smectic', is formed instead of a crystalline phase. The most common crystal modification, formed under normal processing conditions for commercial grades of iPP, is the monoclinic α form, first identified by Natta and Corradini [1]. The following lattice parameters were determined for monoclinic α -iPP: $a=6.65\text{\AA}$, $b=20.96\text{\AA}$, $c=6.50\text{\AA}$, $\beta=99.8^\circ$. The α -iPP reveals the lamellar branching of crystallographic origin which is a unique feature in polymer crystallography. Such branching leads to the formation of two populations of lamellae, of radial (mother) and tangential (daughter) orientation in spherulitic growth [2,3], and also results in the formation of so-called 'cross-hatched' lamellar morphology within the spherulites. Due to this specific morphology α -spherulites may demonstrate either a positive or mixed optical character [2]. Many experimental observations demonstrated that the α modification is thermodynamically the most stable crystalline form of iPP.

The two other known crystalline forms of isotactic polypropylene are the β and the γ modifications. iPP's ability to crystallise in the β form was discovered by Keith et al. in the late 1950s, [2] while the γ form was discovered by Addink & Beintema in 1961 [4]. It was found that these thermodynamically less favourable crystal forms can only be obtained by special modification of the crystallisation conditions (e.g. by the use of specific nucleants and crystallisation in the presence of shear). Temperature gradients can

Abstract

Isotactic polypropylene (iPP) can crystallise in several crystalline forms. The study of crystallisation methods promoting the formation of β - and γ -crystalline forms of iPP is described. In order to obtain samples which crystallise preferentially in the β form, two calcium salts of pimelic and suberic acids, reported as specific nucleating agents of β crystals, were synthesised and their efficiency was tested. The blends of iPP with a concentration of nucleant within a range of 0.1-0.3 wt.% were prepared and tested. The obtained samples, presumably containing β -form crystals, were investigated by differential scanning calorimetry (DSC) and wide-angle x-ray scattering (WAXS) in order to evaluate their phase composition. Ca-pimelate was found to be a more effective and stable nucleant than Ca-suberate, yielding a β -modification content of over 95% at the nucleant concentration of 0.2 wt.%. At the same concentration, the Ca-suberate yielded 93% of β modification. Samples of plain iPP homopolymer were also crystallised isothermally at high pressure in order to obtain γ -form crystals. To optimise the phase structure, the temperature and time of crystallisation were varied. X-ray measurements revealed that the samples crystallised at the pressure of 200 MPa demonstrated the content of γ modification of 100% for a crystallisation temperature of 190°C and a crystallisation time longer than 2h. Specimens containing a mixture of γ and α crystals were obtained when applying a crystallisation temperature below 190°C.

Key words: isotactic polypropylene, β -modification, γ -modification, WAXS.

promote the β phase [5-13], while crystallisation at high pressure leads to formation of the γ phase [4] or a chain structure, such as the incorporation of comonomer units [14].

The crystal structure of β -modification of iPP was established by Meille et al. [15], who found a trigonal unit cell with parameters $a=b=11.01\text{\AA}$, $c=6.5\text{\AA}$, $\alpha=\beta=90^\circ$ and $\gamma=60^\circ$, containing three isochiral 3_1 helices. The arrangement of lamellae in β -spherulites is radial. No traces of a 'cross-hatched' structure were detected. This results in the formation of well-individualised negatively birefringent spherulites, which can be easily distinguished from α -spherulites. The β -phase is thermodynamically metastable and recrystallises to the α form on heating. The β -form spherulites grow sporadically under the usual conditions for crystallising plain iPP, where the α form is dominant. The amount of β -phase usually does not exceed a few percent. However, many research groups developed several special crystallisation techniques which allowed higher levels of the β form in solidified polypropylene to be obtained, such as the temperature gradient method [5], shear-induced crystallisation [11-13] or the use

of specific nucleating agents [6-10]. Numerous experimental studies [e.g. 6-10] led to the discovery of very effective specific nucleants promoting crystallisation in the β -form. The most efficient nucleants allow materials containing the nearly pure β form to be obtained, with only a few percent of α -phase. According to recent knowledge, two groups of substances can be specified as the most effective β nucleants. The first group consists of aromatic ring compounds such as γ -quinacridone, anthracene, phenothiazin, triphenodithiazine and phenanthrene [6-7]. The second group contains certain salts (e.g. Ca or Zn) of dicarboxylic acids [8-10]. It is worth mentioning that these calcium salts of pimelic and suberic acids show very high activity without any of the drawbacks (such as change of colour, low activity, instability) demonstrated by many other nucleants [9].

It was found that iPP-based materials rich in β -phase show very interesting mechanical properties, more advantageous than conventional α -iPP. In particular, the impact strength, toughness and whitening under tensile deformation of β -iPP markedly exceed those of α -iPP [10, 11, 16-18].

The γ -phase of iPP, though first mentioned some decades ago [4], were more extensively studied only much later. Brückner & Meille [19] determined the triclinic unit cell with lattice parameters of $a=6.55\text{\AA}$, $b=21.57\text{\AA}$, $c=6.55\text{\AA}$, $\alpha=97.4^\circ$, $\beta=98.8^\circ$, $\gamma=97.4^\circ$. The overall architecture of the γ -iPP crystal structure is very unusual, in that the chain axes in the crystal are not aligned in one direction as in other polymer crystals. Instead, the chains form alternating layers of parallel chain ordering. The angle between the chain direction in two consecutive layers is approximately 80° . This is the same angle as that observed between the mother and daughter lamella of the α -iPP [19-22]. The γ -form is usually not present in plain iPP crystallised under normal conditions. The preferred crystallisation in the γ -form was found in some copolymers of propylene [14], or on the crystallisation of plain iPP homopolymer under high pressure [4,19-21]. With the increase in pressure, crystallisation of both the α - and γ -forms was observed. At crystallisation pressure of 200 MPa, the γ -phase predominate [23]. Crystallisation at the pressure of 200 MPa or higher results in crystallisation in the γ -form exclusively.

Although the crystallographic structure of the γ -form was already established, there is no data in the literature concerning the mechanical properties of this modification, nor the mechanical performance of materials containing this modification as a major crystalline form.

The aim of the work reported in this paper was to find effective methods of preparing bulk samples of iPP homopolymer primarily containing β - and γ -form crystals. Preparation of such samples was a necessary step for subsequently studying the mechanical properties of these crystalline modifications of iPP, and for finding the micro-mechanisms active during the deformation of the crystals of these forms.

Experimental

Materials and sample preparation

The material used in this investigation was a commercial-grade isotactic polypropylene homopolymer, Malen - P, F-401 ($M_w = 297,200$, $M_n = 56,400$, isotacticity index $> 95\%$) provided by Basell-Orlen Polyolefins (Poland).

The calcium salts of two dicarboxylate acids for use as specific β -nucleating

agents of iPP were synthesised from suberic acid (*Octanedioic acid*, $M = 174.20\text{ g/mol}$, $mp = 142-144^\circ\text{C}$, soluble in alcohol) and pimelic acid (*Heptanedioic acid*, $M = 160.17\text{ g/mol}$, $mp = 103-105^\circ\text{C}$, soluble in water and alcohol), both supplied by Sigma-Aldrich (Germany).

The calcium salt of pimelic acid (Ca-Pim) was synthesised according to the following procedure: an equimolar amount of CaO dispersed in water was added to the aqueous solution of a pimelic acid while stirring at the temperature of 10°C . The calcium salt of suberic acid (Ca-Sub) was synthesised according to a different procedure: an equimolar amount of NaOH dissolved in water was added to a diluted solution of a suberic acid in ethanol while stirring, at room temperature, to produce the sodium salt. Next, an equimolar amount of CaCl_2 solution was added to the (Na-Sub) solution to obtain the calcium salt.

In both procedures, the Ca-salts precipitated from solution as a fine white powder. The collected powders were filtered, washed with ethanol, and then dried in vacuum at room temperature [8]. The dry powders were milled to reduce grain size.

The average size of particles was reduced in this way to approx. $0.5\ \mu\text{m}$. The powders obtained of both salts were used as specific β -nucleating agents. They were mixed in the desired proportions with molten iPP at 200°C using the Plasticorder mixer (Brabender, Germany). The mixtures containing from 0.1 to 0.3 wt.% of the nucleating agent were prepared. The nucleated samples were compression-moulded at 190°C to the form of 4 mm-thick plates and then crystallised non-isothermally by fast cooling in iced water. Isothermal crystallisation experiments within the temperature range of $127-133^\circ\text{C}$ were also performed.

γ -phase samples were prepared by crystallisation of plain iPP at high pressure using a specially designed crystallisation cell, shown in Figure 1, made of ultra high-strength steel (30HGSNA type, withstanding the pressure up to 1000 MPa, at a temperature up to 320°C) and a computer-controlled loading frame of a tensile testing machine (Instron 1114, capacity 5T).

A cylindrical channel of 9.5mm in diameter along the axis of the cell was made with very high diameter accuracy and high surface smoothness. Tungsten carbide

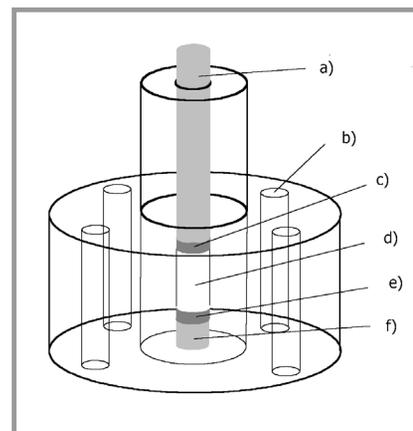


Figure 1. Schematic view of a high-pressure crystallisation cell.

ride rods, tightly matching the diameter and smoothness of the channel in the cell, were used as plungers to compress the sample placed between them. The high precision fitting of the cell and plungers, as well as the use of several stacked brass and Teflon seals, provided high tightness of the cell and prevented any leakage of the highly pressurised polymer melt out of the cell. The cell was equipped with load and strain gauges, as well as four main electrical heaters (1000W of total power) connected to the temperature controller. Two additional heaters, placed between the plungers and the loading frame and connected to separate temperature controllers, were used to eliminate an axial temperature gradient. The temperature control of the material inside the cell was better than 0.2°C .

After filling the cell with a polymer sample and mounting the seals and plungers, the assembled cell was pressed up to a load two times higher than that which gave the desired working pressure (200 MPa), in order to tighten all the brass seals. Next, the force was reduced to a pressure of 200 MPa inside the cell. The load was continuously adjusted to keep that pressure constant over the course of the entire crystallisation process. The computer control of the loading frame allowed the constant pressure to be maintained inside the cell with an accuracy of 0.1 MPa. The pressurised cell was heated up to a temperature of 248°C in order to melt the iPP sample inside ($T_m = 242^\circ\text{C}$ at $p=200\text{ MPa}$ [19]). The molten polymer was kept at this temperature for 3 minutes. The temperature was then reduced to the desired crystallisation temperature, set within the range of $170^\circ\text{C}-200^\circ\text{C}$. Depending on the crystallisation temperature, the sample was kept under these con-

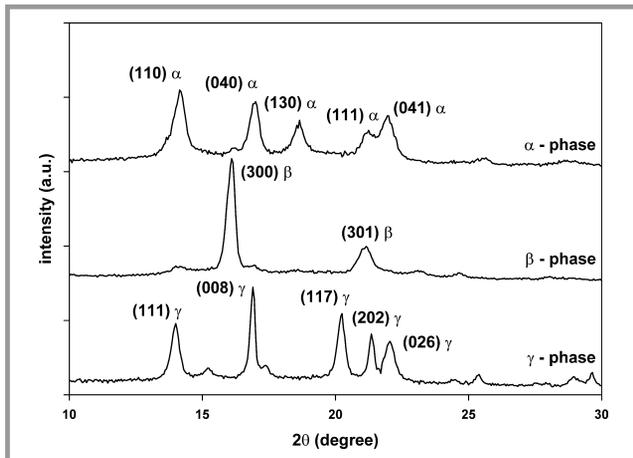


Figure 2. X-ray diffractograms of α -, β - and γ -phases of isotactic polypropylene ($\text{Cu K}\alpha$ radiation). The main peaks are indexed.

ditions for 1 to 4 hours to allow complete crystallisation. After that, the cell was cooled down to room temperature and then unloaded. Finally, the crystallised specimen was pushed out from the cell in the form of a rod of 9.5 mm in diameter and between 3 to 20 mm high.

Characterisation

DSC. Thermal analysis of the samples was carried out with the TA 2920 DSC apparatus (Thermal Analysis), indium calibrated. Specimens of 5 to 8 mg were cut out from the core of the crystallised samples. The overall crystallinity level was estimated on the basis of the heat of melting the sample which was recorded during heating from room temperature to 200°C with the heating rate of 10°C/min, assuming the heat of melting of 100% crystalline β and γ -iPP of $\Delta h_f = 199$ J/g and $\Delta h_f = 181.2$ J/g, respectively [24,25].

WAXS. A computer-controlled X-ray diffractometer (DRON, Russia) coupled to a sealed-tube source of filtered $\text{CuK}\alpha$ radiation, operating at 50kV and 30mA (Phillips PW 3830, the Netherlands) was used for the acquisition of 2θ scans. The step of 0.05° was used in all scans. The disc-shaped specimens of approximately 2 mm thickness were cut out from the core of the crystallised samples.

SEM. In order to prepare specimens for microscopic observations, an internal surface of the sample was first exposed by cutting with an ultramicrotome (Tesla BS 490A, the Czech Rep.) equipped with a freshly prepared glass knife. The resultant microtomed surface was then etched over 2 hours at room temperature with an etching mixture of 0.7 wt./vol. % of KMnO_4 dissolved in a 5:4:1 vol./vol. mixture of 95% sulphuric acid, 85% phospho-

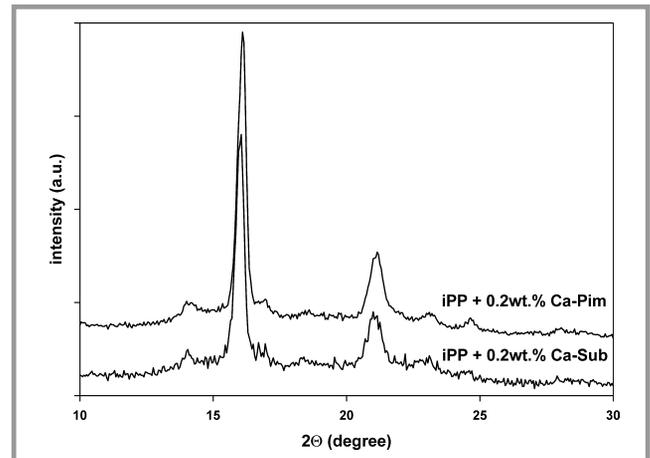


Figure 4. X-ray diffraction scans of iPP nucleated with 0.2 wt.% of calcium suberate (lower curve) and calcium pimelate (upper curve).

ric acid and distilled water, according to the procedure developed originally by Olley et al. [26]. To improve etching, the mixture was placed in an ultrasonic bath running periodically for a short time during process. Next, the etched samples were washed subsequently in diluted sulphuric acid, concentrated hydrogen peroxide, distilled water and pure acetone, all under ultrasonic agitation. The dried specimens were mounted on microscopic stages and coated with fine gold layer by ion-sputtering (JEOL JFC-1200, Japan). Finally, they were examined with the scanning electron microscope (JEOL JSM-5500LV, Japan) working in high vacuum mode and with an accelerating voltage of 10kV.

Results and discussion

β -phase

Figure 2 presents X-ray diffractograms of α , β and γ crystal modifications of iPP. As can be seen, each crystal form shows distinctive diffraction peaks. The position and size of the peaks characteristic for a given modification allow its easy identification, especially in the case of α and β forms, since the two distinct peaks characteristic for the (300) and (301) crystal planes of β -phase are separated from the peaks of the main planes of the α -phase, i.e. (110), (040) and (130). This feature allows the determination of the actual phase composition of any sample containing crystallites of both α and β modifications. However, the calculations require the separation of the crystalline peaks, amorphous halo and background, since they have finite width and overlap with their tails in the experimental curve.

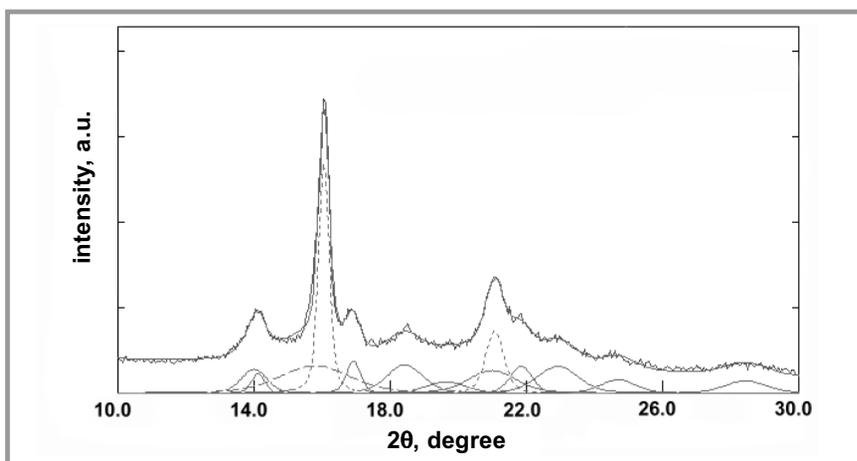


Figure 3. Example result of peak separation procedure. The nucleated sample of iPP contains both α and β crystal modification. The peaks of α modification are drawn with α solid line, β with a short-dash line and the amorphous halo with a long-dash line. The calculated fraction of β modification in the presented specimen [nucleated] with 0.1 wt.% of Ca-Pim, $K_\beta = 0.82$.

From the diffraction intensities characteristic of the separated peaks, the relative content of the β -form in the specimen can be estimated in terms of the $K\beta$ parameter, calculated according to the equation proposed by Turner-Jones et al. [27]:

$$K_{\beta} = \frac{I_{\beta(300)}}{I_{\beta(300)} + I_{\alpha(110)} + I_{\alpha(040)} + I_{\alpha(130)}} \quad (1)$$

where $I_{\alpha(110)}$, $I_{\alpha(040)}$ and $I_{\alpha(130)}$ are the intensities of the strongest peaks of the α -form attributed to the (110), (040) and (130) planes of the monoclinic cell, located at $2\theta=14.2^{\circ}$, 17° and 18.8° respectively, while $I_{\beta(300)}$ is the intensity of the main (300) diffraction peak of the hexagonal β -form, located at $2\theta=16.2^{\circ}$. The $K\beta$ can take values from the range between 0 and 1 for samples containing from 0 to 100% contribution of β modification to the crystalline phase. This parameter is only an estimate of the actual phase composition, because only the highest diffraction peaks are taken into account instead of all the crystalline reflections. However, since the other experimental peaks either contain contributions from both modifications or are much lower than those considered, an error of estimation of the phase composition using the $K\beta$ parameter is relatively low. This parameter, although not very precise, is commonly used in the literature. Thus, calculating it is useful to compare the efficiency of the various nucleating agents or the crystallisation procedures tested.

Figure 3 presents an example of curve-fitting and separation of diffraction peaks (iPP nucleated with 0.1 wt. % Ca-pimelate). A custom-written computer program was employed for the peak separation procedure. The shape of peaks was approximated with a superposition of Gauss and Lorentz peaks with respective weights of 0.7 and 0.3. The separated peaks were plotted in this Figure along with their superposition and the experimental diffraction curve, which demonstrate the good quality of the fits obtained. A similar decomposition of experimental curves into separate peaks was performed for samples containing various amounts of the two nucleants tested. On the basis of the calculated peak intensities, the respective values of the $K\beta$ parameters were calculated using equation (1). The results obtained are presented in Table 1. Additionally, the overall crystallinities, as determined from the DSC melting data, are also reported.

Table 1. Phase composition and crystallinity of β -nucleated iPP.

	Nucleant content (wt.%)	$K_{\beta}^{(1)}$	$X_c^{(2)}$ (wt.%)
iPP with Ca-Sub	0.1	0.85	42.1
	0.2	0.93	44.2
	0.3	0.93	41.8
iPP with Ca-Pim	0.1	0.82	43.1
	0.2	0.95	46.0
	0.3	0.96	42.7

⁽¹⁾ calculated with eq. (1)

⁽²⁾ calculated from DSC data.

The data presented demonstrates that both the Ca-suberate and Ca-pimelate appeared to be very effective nucleating agents, both promoting crystallisation mainly in the β -modification. With the increase in amount of the nucleating agent, the overall crystallinity does not change substantially, while the content of β modification noticeably increases. This increase is stronger in the range of low concentration of the nucleant, 0.1-0.2 wt.%. When its concentration is increased further to 0.3 wt.%, only a minor increase of β modification content can be noted. This indicates that the optimum concentration of nucleating agent is around 0.2wt.%. For this concentration the amount of β phase reaches 93% to 95%, depending on the type of nucleant. This is also illustrated in Figure 4, presenting diffraction scans of samples containing 0.2 wt% of Ca-pimelate and Ca-suberate respectively. It can be seen that the curves almost entirely show features which are typical for β -modification, with only minor marks of the α phase.

The results obtained indicate that the Ca-pimelate appears more effective than the Ca-suberate – the maximum fraction of the β phase was 0.96 for the Ca-pimelate, but only 0.93 for the Ca-suberate of the same nucleant concentration. Furthermore, it must be noted that each sample prepared with the Ca-pimelate gave reproducible results, which demonstrates the good stability of this compound in addition to its high efficiency.

γ -phase

The diffraction curves characteristic for the α - and γ -crystal modifications of iPP were presented above in Figure 2. A comparison of both curves demonstrates that most of the peaks characteristic for both phases are located at nearly the same positions, which makes identifying crystalline phases in the particulate specimen and calculating its phase structure very difficult. The only difference between these diffraction curves can be found in the 2θ range of $18-21^{\circ}$, where we can find

two well-separated diffraction peaks of the (130) plane of the α modification ($2\theta=18-19\text{s}$) and the (117) plane of the γ modification ($2\theta=19.2-20.5\text{s}$). By comparing the intensities of these peaks, the actual fraction of the α and γ phase in the specimen can be estimated. The fraction of the γ modification in the crystalline component is then calculated from the following equation:

$$K_{\gamma} = \frac{I_{\gamma(117)}}{I_{\gamma(117)} + I_{\alpha(130)}} \quad (2)$$

where $I_{\gamma(117)}$ and $I_{\alpha(130)}$ denote the intensities of the respective diffraction peaks of the γ and α modifications respectively. The parameter $K\gamma$ can vary within the range of 0-1 for samples containing from 0 to 100% contribution of the γ modification.

The phase composition of the iPP samples obtained by isothermal crystallisation at high pressure was evaluated on the basis of the diffraction curves determined by using the above equation (2). The samples were crystallised isothermally at various temperatures and times under the constant pressure of 200MPa. These conditions of crystallisation were pre-selected on the basis of literature data [23]. Figures 5a and 5b show the representative diffraction curves determined for the samples obtained within the range of 2θ angle of interest ($18-24^{\circ}$). It can be seen that all the samples primarily contain the γ crystalline form, with only traces of the α form.

On the basis of the experimental curves, the parameter $K\gamma$ was determined for every specimen. In order to increase the precision of estimation, the actual area of the respective peaks was determined using the peak separation procedure. For this purpose, the PeakFit program (Jandel, Germany) was used. Table 2 summarises the calculated values of the $K\gamma$ parameter for the samples studied. The overall crystallinity of the samples, determined from the DSC data, are also presented in this table.

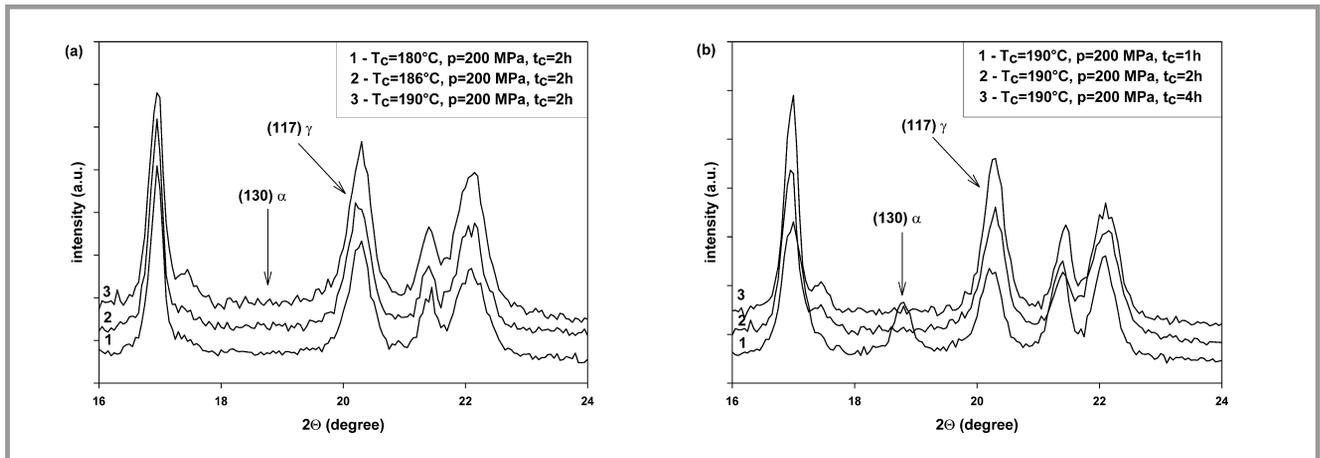


Figure 5. Diffraction patterns obtained for samples crystallised at a constant pressure of 200 MPa and various temperature and time, indicated in the plots. The positions of characteristic peaks of α and γ phase are marked with arrows.

The data presented in Table 2 demonstrates that the overall crystallinity of the samples crystallised at elevated pressure are noticeably higher than the crystallinity of samples crystallised at atmospheric pressure (*cf.* Table 1). This increase is due to the lower undercooling in crystallisation proceeding at an elevated pressure ($\Delta T=52-62^\circ\text{C}$) as compared to the samples crystallised non-isothermally at atmospheric pressure ($\Delta T\sim 70^\circ\text{C}$). All the samples exhibit a very high proportion of γ -modification crystals, ranging from 82 to 100%. The actual phase composition appears to depend on the temperature of crystallisation, increasing together with the increase in temperature (over the same time of crystallisation). Furthermore, the data shows that the crystallisation of the γ -phase at elevated pressure proceeded very slowly; samples crystallised at the same temperature of 190°C for 1, 2 and 4 hours exhibit fractions of γ crystals of 0.82, 0.98 and 1.00 respectively. When a shorter time was applied the crystallisation was probably not completed at the high-pressure stage, and the α crystals observed in the sample probably formed upon the cooling of the sample and the release of the pressure. When the time of crystallisation was long enough (4h), the

sample crystallised entirely in the γ modification, and no α crystals were observed. We can also point out that in the diffraction pattern of the specimens crystallised over 2 and 4 hours, one smaller peak characteristic of the γ -modification can be recognised. This is the (115) plane located at $2\theta=17.2^\circ$. This peak was too low to be distinguished in the samples containing a lower fraction of the γ -phase. This additionally confirms the high phase purity of the specimens crystallised at 190°C and 200 MPa by at least 2 hours.

Morphology of β and γ -phase

Supplementary SEM observations of the samples investigated were performed to reveal their morphology. The representative micrographs obtained for the iPP samples containing a majority of the β or γ modification are presented in Figures 6b and 6c. For comparison, the micrograph of the α -iPP specimen obtained under conventional crystallisation conditions is also presented in Figure 6a.

The examination of micrographs revealed that all the specimens show spherulitic morphology. Spherulites of the β form are much smaller than those

observed in the reference sample of α -iPP. This results from enhanced primary nucleation due to the presence of the nucleation agent. In fact, the traces left by the nucleant particles can frequently be observed in the centres of the β spherulites. Further, the spherulites of γ modification grown at high pressure are smaller than the α spherulites in the reference sample, which indicates enhanced primary nucleation under such crystallisation conditions.

Another feature of the structures observed is that the spherulites of both the β - and γ -modifications do not show any of the ‘cross-hatched’ lamellar morphology characteristic for the α -phase. The lamellae in spherulites of the β -form grow straight almost radially from the spherulites’ centres (*cf.* Figure 6b). In the central part of numerous spherulites, the characteristic feature of lamellae branching extensively from the central sheaf-like nucleus part of the spherulite can be clearly observed. This leads to the entire volume of the spherulite being completely filled by lamellae, and their further radial growth with only little branching.

The lamellar morphology found in γ spherulites (Figure 6c) is different from that observed in the α or β spherulites. They frequently show ‘feather-like’ structures with a central stem formed by a bunch of long and relatively straight lamellae, and plenty of shorter ‘daughter’ lamellae branching sideways from this stem. These daughter lamellae frequently make an angle of about 80° with respect to the central stem and each to the other. A similar morphology was previously observed by Meille et al.[19].

Table 2. Phase composition and crystallinity of samples crystallised under pressure of 200 MPa.

Temperature of crystallisation ($^\circ\text{C}$)	Time of crystallisation (h)	$K_\gamma^{(1)}$	$X_c^{(2)}$ (wt.%)
180	2	0.94	55.2
186	2	0.96	57.6
190	2	0.98	54.3
190	1	0.82	53.2
190	2	0.98	54.3
190	4	1.00	57.6

⁽¹⁾ calculated from equation (2)

⁽²⁾ estimated from the heat of melting (DSC)

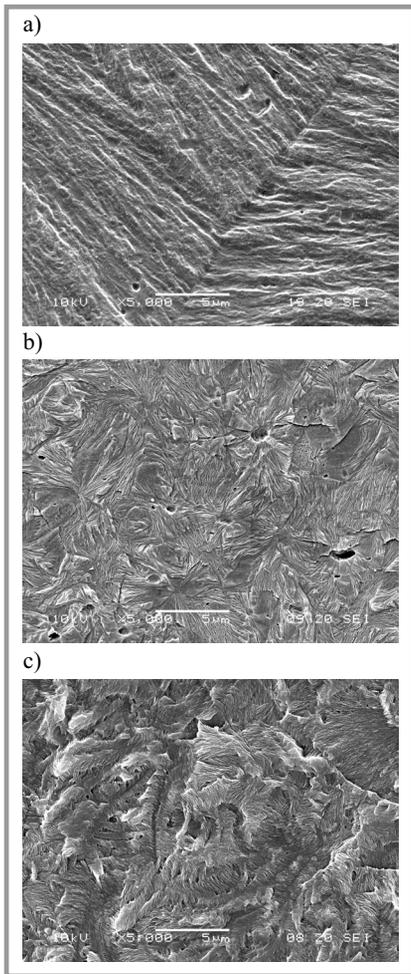


Figure 6. SEM micrographs of α , β and γ -phase isotactic polypropylene: (a) α -spherulites, (b) β -spherulites and (c) feather-like structure in γ -iPP spherulite. All samples were etched prior to examination. Scale bar shown on each micrograph.

Conclusions

The experimental work reported in this communication allowed the production of bulk samples of iPP in which the major crystalline phase was either of the β or γ modification.

The preferential crystallisation of the β modification was induced in iPP by a selective nucleating agent. Of the two tested nucleants, the Ca salt of pimelic acid (Ca-Pim) appeared the more effective and stable one, yielding up to 96% of the β modification within the crystalline phase.

Crystallisation at an elevated pressure of 200 MPa allowed samples containing nearly pure γ modification to be obtained. When the isothermal crystallisation under this pressure was long enough, highly crystalline samples containing the γ form exclusively and no traces of other crys-

tal modifications of iPP were obtained. Samples containing a mixture of the major γ -phase and a relatively small fraction of the α -phase were obtained at crystallisation temperatures lower than 190°C.

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