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Fundamental studies on shear-induced nucleation and beta-phase formation in the isotactic polypropylene—effect of the temperature

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Abstract

The complex and incompletely understood phenomenon of shear-induced crystallization of polymers may be nowadays analysed via the in situ POM-shear stage methodology. In this research, the two main issues were investigated with the use of the Linkam CCS450 shear stage connected with POM microscope. It was found that the secondary nucleation in the tree well-known temperature regimes plays the greater role in the overall crystallization kinetics than the shear induced primary nucleation. Furthermore, it was found that the tendency towards β-phase formation in shear conditions is dependent on the temperature value during shear treatment. It may be concluded that the temperature is the key parameter in the primary and secondary nucleation process and beta-phase formation in the iPP melts.

Introduction

Since its discovery, the isotactic polypropylene (iPP) has been found to have many applications in various areas in view of its satisfactory properties and relative low material cost [1–3]. The discovery of stereospecific olefins polymerization catalysts enabled the efficient iPP synthesis on the industrial scale and its widespread use in man-made material technology, especially in the production of composites [4–16].

The observations of formation of different crystalline morphologies (e.g., spherulites or fibrils) were carried out as well as the crystallization kinetics (e.g., the issue of crystallization regimes) [17]. The last one is based on the Hoffman-Lauritzen theory and includes the dependence between the crystallites growth rate (G) and several factors, which are presented in Fig. 1. It can be defined by two different steps of nucleation: deposition of a critical nucleus (i) and later the deposition of stems adjacent to nuclei (g) [18, 19]. The secondary nucleation process may occur in three different crystallization regimes: regime I – where the $g \gg i$; regime II – where $g \sim i$ and regime III – where $g \ll i$ (Fig. 1) [19, 20].

Moreover, with the use of determined crystallites growth rate (G), it may be possible to prepare a curve, showing the values of K(g) – describing the crystallites growth—in three different regimes, where the key is the temperature value during isothermal crystallization.

It is impossible to recall the iPP without evoking the polymorphism phenomenon, it can be identified by different crystallographic forms: α, β and γ (moreover, the smectic form) [3, 21–23]. Polymorphism in isotactic polypropylene is our long-term research topic [22, 24, 25]. The α form is the most commonly observed form of isotactic polypropylene. The γ form of iPP is formed during high pressure crystallization as well as for short chain polymers. The β form of isotactic polypropylene can be obtained by providing suitable conditions for crystallization, such as high temperature gradients, the presence of shear forces or by using appropriate nucleating agents. The value of the equilibrium melting point for the β form is estimated to be around 150 °C. During the crystallization of the β form, it is possible to produce two types of spherulites. Subjecting to deformation above the elastic limit may convert the β form to the α form of an isotactic polypropylene [3]. The

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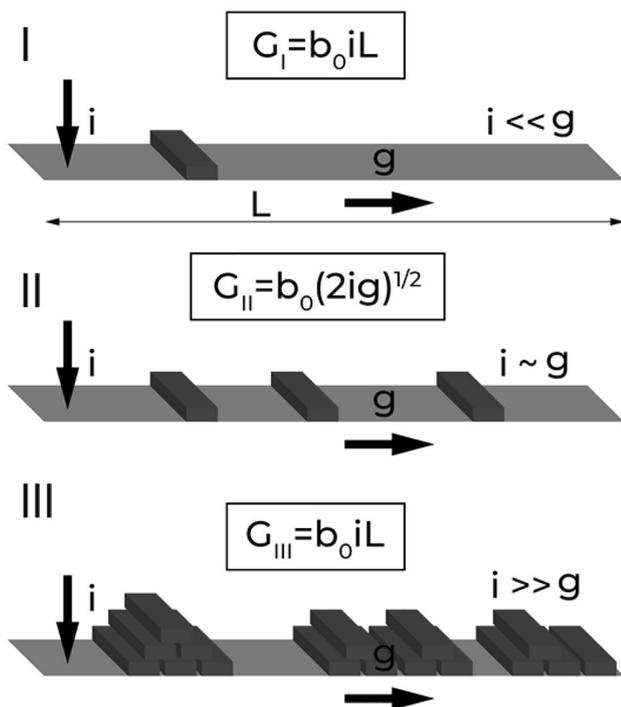
Fig. 1 The growth of crystalline structures in three different regimes

presence of the β form in the material may result in high impact strength and low stiffness of the material. So far, the mechanism of beta form formation has not been fully elucidated.

Several factors, e.g.: the undercooling ratio [26, 27], the effect of nucleating agents [28–31] and the shear forces influence [32–34] have a strong impact on creating the supermolecular structure in iPP, especially the β-form. In the processing of polypropylene with use of the most popular techniques, e.g., extrusion or injection molding, shear forces affect the polymer. It is related to direct contact between the polymer and the walls of mold or processing nozzle and between polymer matrix and the filler particles in composites (what may be called "local shearing") [35–38].

Presently used techniques allow observation of a formation of the polymer ordered structures in situ, directly during the shear forces treatment. There have been many attempts to do the research on polypropylene [39–51] and its composites [29, 52, 53], and also the polymer blends [54, 55] with use of Linkam CSS450, also called Oxford shear stage. Despite many results presented in the literature, the application of the Linkam system still enables one to make unique and interesting observations.

This study was focused on the polymorphism and crystallization kinetics in the iPP in case of shear induced nucleation in three different crystallization regimes by using different crystallization temperatures.

Experimental

Materials

In the current research, the commercial isotactic polypropylene Malen P S-702 (*iPP*) was used, with the MFR value 13.0 g/10 min, manufactured by Bassel Orlen Polyolefins, Poland. The iPP granules were dried in the convection dryer in the temperature 80°C for 12 h. Afterwards, with use of the hydraulic press (in the temperature 230°C and with the 5 ton pressure), the 300 μm thick iPP foils were prepared. The iPP films were used in the: structural (WAXS), thermal (DSC) and microscopic (POM) investigations.

The structural and thermal investigations

For the analysis of iPP structure in thin films, the WAXS analyze (Wide Angle X-Ray Scattering) was carried out with the use of a horizontal diffractometer (CuKα radiation with use of nickel filtering, $\lambda = 1.5418$ Å). The following parameters were applied: voltage 30 kV, anodic current 25 mA, angle range 2Θ: 10–30°, counting step (2Θ): 0.04°, counting time: 3 s, with remaining standard parameters. Performed WAXS studies were used in determining the fundamental parameters, like: maxima intensities and amount of β-iPP in overall crystalline phase in the material.

In order to validate obtained WAXS results, the DSC studies were performed using a Netzsch Differential Scanning Calorimeter, model DSC 200, in a nitrogen atmosphere. The samples were heated to 200 °C (at a heating rate of 10 °C /min) and kept at this temperature for 2 min. Then, the samples were quenched to 40 °C at the rate of 5 °C /min (using nitrogen flow). The cycle was repeated twice and the data from the second cycle were interpreted.

Polarizing optical microscope—in situ observations of crystallization

In the POM investigations the polarized optical microscope Axio Lab A1 (Zeiss, Germany), the ToupView camera and Linkam CSS 450 shear-hot stage (Linkam Scientific Instruments, UK) were used. The research was divided into three main parts:

I) observations of crystallization kinetics, II) the temperature dependence of shear nucleating ability and III) the formation of β phase of iPP.

In the first part of the research, in order to analyze the morphology and crystallization kinetics, shearing protocol "A" was used (Fig. 2). Polypropylene films were heated with 30°C/min gradient to 200°C. The material was kept at the temperature 200 °C for 300 s, to reduce the "crystalline
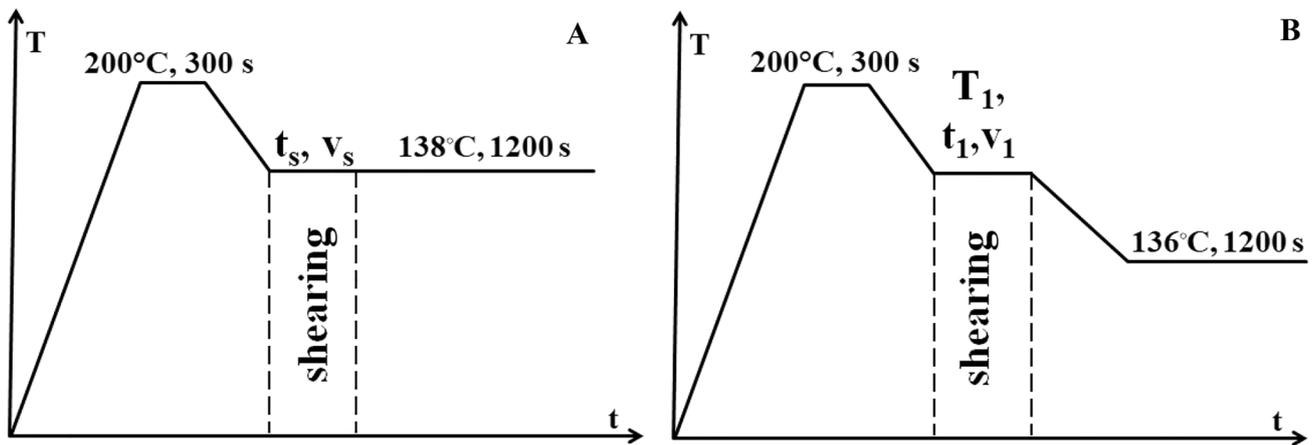
Fig. 2 The temperature and shearing protocols used: **A -** for the analysis of differences in spherulites structures morphology and spherulites diameters, **B** - for the observation of crystallization regimes

memory" phenomenon. Afterwards, the melt was cooled with 30°C/min gradient to the temperature 136°C. Subsequently, after the regulation of the gap between plates to 250 μm, the melt was treated with use of shear forces, which had specified shear rate v_s and shear time t_s (Table 1). The crystallization process was observed for 20 min with use of the POM technique.

In the second and third part of this study the shearing protocol "B" presented in Fig. 2 was used. After the melting in a temperature of 200°C for 300 s, the material was cooled with 30°C/min gradient to the temperature T_1. The used temperature T_1 values were: 154, 150, 145, 140, 138 and 136°C. Then the gap between plates was adjusted to 250 μm and the shear treatment was carried out (Table 1). After shear treatment the melt was cooled to 138°C and the isothermal crystallization of iPP was observed with the use of a POM microscope.

Furthermore, the observations of selective-melting of spherulites during heating have been carried out. The β phase of iPP melts in a temperature of about 150°C, and the α iPP melts in a higher value of temperature, about 160–170°C. The samples have been sheared with values of shear rate and shear time presented in Table 1. The order of operation was analogue, like in "B" shearing protocol, but after melting of iPP samples the behavior of spherulites in increasing temperature was observed. The crystallization of

Table 1 The shear and temperature parameters applied in current in-situ, hot-shear stage POM investigations

Shearing protocol	T_s [°C]	v_s [s^{-1}]	t_s [s]
A	138	1,5; 3,0; 4,5	10; 30
B	136, 138, 140, 145, 150, 154	1,5; 4,5	10

iPP samples in quiescent conditions (without shear treatment) was executed.

Interpretation of POM micrographs

Based on POM photographs, it was possible to set the very important parameter of crystalline structures growth. The spherulites growth rate (μm·s^{-1}), which defines the spherulites diameter increase during one second, was calculated on base of spherulites diameters measurements after 20 min of isothermal crystallization.
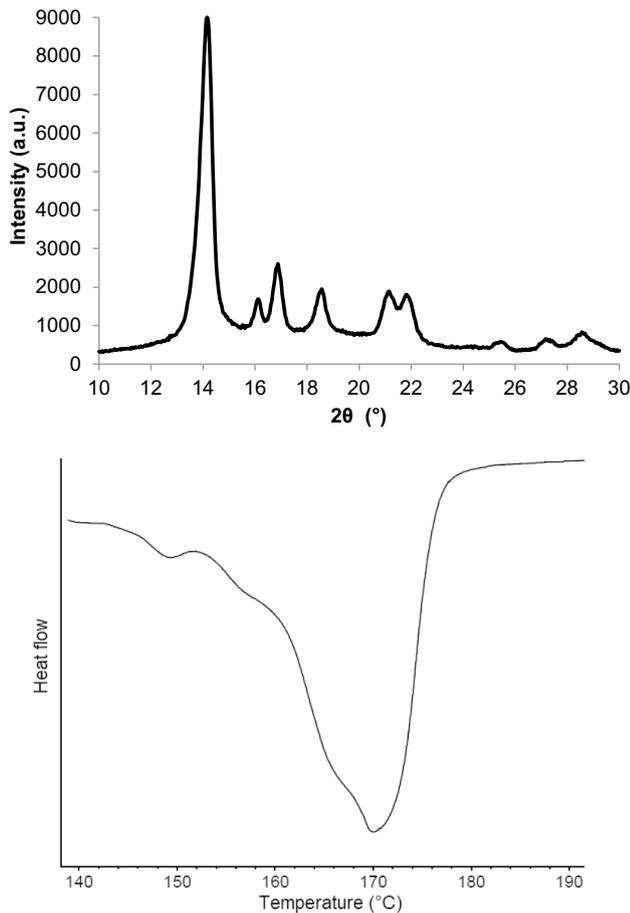
Results and discussion

The polymorphic structures of isotactic polypropylene

Figure 3 shows the WAXS curve prepared for iPP. It can be observed that there are a few significant differences in the supermolecular structure of examined polypropylenes. Only for the PP2 has the diffraction maximum at 2θ degree 16° been noticed, which suggests the β form of iPP presence in the material. It is an interesting phenomenon that the compression molding of that polymer induces the pseudohexagonal form nucleation. In the research of Garbarczyk and Paukszta [56] the formation of β-phase in compressed iPP has been analyzed with a presence of β-nucleating agents. It has been proved that the high pressure undermines the abilities of agents to nucleation. However, an intern slip between polymer layers (like in case of shearing model, when the fluid is divided into infinitely many and infinitely thin parallel layers) can probably induce a small amount of β-crystalls.

The amount of β-phase (k) in the crystalline phase of polypropylene has been determined through use of the

Fig. 3 The WAXS and DSC curves prepared for examined material

Spherulites growth rate and crystallites morphology after shearing

Figure 4 shows values of the spherulites growth rate and density nucleation measured for iPP, crystallized in various shear conditions. It is known from the literature that the kinetics of spherulites growth closely depends on the shear rate. it can be observed that the higher the shear rate is, the higher the growth rate of spherulites.
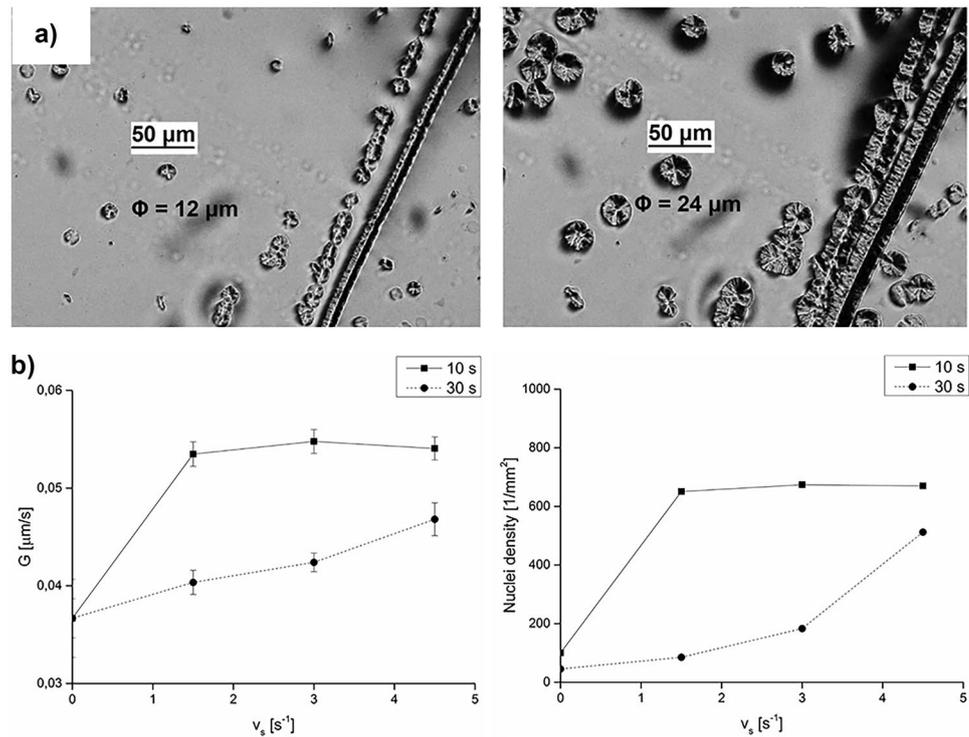
As can be seen in Fig. 4, in the iPP sheared for 10 s, a high increase of G can be found, from about 0,036 to 0,055 μm/s. However, in the melt sheared for 30 s, the difference in the G parameter value between iPP samples crystallized in quiescent conditions and sheared samples was lower. Moreover, the sharp increase in growth rate was observed between melt crystallized in quiescent conditions and sheared with rate $1.5 \, s^{-1}$, where the further growth of shear rate does not affect the crystallization kinetic significantly. The same correlation may be observed in case of nucleation density. Shear flow through preordering of iPP chains affects simultaneouslythe formation of stable nucleis (primary nucleation) as well as crystallization kinetics (secondary nucleation and crystallites growth).

Considering the data shown in Fig. 4, it can be concluded that for iPP the increase of shear time decreases the influence of shear forces on crystallization kinetics as well as nucleation process. The shear stress affected too long, what perhaps over-extends the polymer chains, which impedes the re-arrangement after cessation of shear forces and stable nucleis formation. In the research of Somani et al. [57] it has been concluded that during the treatment of melt with low values of shear rate, only smart parts of chains are able to orientate and crystallize. In the short-time treatment with high shear rate, the segments of macromolecules are oriented for too short a time for create a stable nucleis. The macromolecules orientation is induced more effectively in short-duration shear treatment at a high shear rate than that in long-time shear with low shear rate.

Except for the differences in crystallization kinetics, various morphology in shear-oriented crystallites may be observed. Figure 5a, b show the iPP samples, crystallized in quiescent conditions and after shear treatment.

As can be seen in Fig. 5, the iPP material shows a tendency to the creation of fibril-liked structures, also called fibrils crystallites in the samples sheared during 10 s and 30 s, with shear rates only $4.5 \, s^{-1}$. Pantani et al. [46, 58] considered the fibrilar morphology formation in sheared iPP melts. The conclusion was that above the concrete value of shear rate, the pre-formed crystallites can connect together into fibrilar structures as a result of flow. The critical shear rate value, presented in Pantani's work, was $\sim 0.46 \, s^{-1}$. Thus, in current research the critical value was exceeded and it may be concluded that the formation of fibrils depends

modified Turner-Jones equation [21], using performed WAXS results:

$$k = \frac{P_{\beta 1}}{P_{\beta 1} + P_{\alpha 1} + P_{\alpha 2} + P_{\alpha 3}}$$

where:

　$P_{\beta 1}$ – area of diffraction maxima coming from (300) β-form plane;

　$P_{\alpha 1}$ – area of diffraction maxima coming from (110) α-form plane;

　$P_{\alpha 2}$ – area of diffraction maxima coming from (040) α-form plane;

　$P_{\alpha 3}$ – area of diffraction maxima coming from (130) α-form plane;

　and the calculated value of k was about 0,015.

In Fig. 3 the results of the DSC analysis are presented. As can be seen, the low peak from β iPP phase melting in the temperature 152°C in PP2 material can be observed, so the β-phase presence in the material has been confirmed.

not only on chains ordering through shear strain but also on
the molecular weight of the polypropylene used as well as
the chains length.

The temperature regimes in case of shear-induced crystallization

The polymer chains, oriented by shear flow, after cessa-
tion of shear can return into the amorphous melt through
the shear-stress relaxation phenomena or may create the
energetically-stable nuclei. Figure 6 shows the spherulites
growth rate as a function of the shearing temperature. As
can be seen, there can be found an optimal, from the point
of view of spherulites diameters growth, temperature value
(138°C). It is connected with the occurrence of crystalliza-
tion process regimes.

In the research of Huo et al. [43] a conclusion was made
that in the temperature 142°C and higher, the abilities of

shear forces for crystallization inducing decreases with the
increase of shearing temperature. They are connected with
the competition between shear-induced nucleation and fur-
ther growth of spherulites and relaxation of polymer chains.
For iPP examined in this research, the temperature 138°C
can be called the "critical" value, above which the nucleat-
ing abilities of shear forces decrease with a further increase
in temperature. Consequently, it can be concluded that the
relaxation of macromolecules after cessation of shearing
plays a significant role in shear-induced crystallization issue,
in pure polymer melts and also in melts with an addition of
nucleating agents [59]. However, the issue of temperature
regimes should not be forgotten. Furthermore, a clear sepa-
ration between shear-induced nucleation and primary and
secondary homonucleation process in the polymer should
be ensured. Using the G data the plots $\log + \Delta E(2.3 R(T_c - T_\infty)$ versus $1/(T_c \Delta T)$ (Fig. 7) were prepared. The ΔE is an
activation energy (6270 J/mol, R = 8,31 J/mol·K, ΔT is an

Fig. 5 POM micrographs taken
after 20 min. of isothermal
crystallization at 138 °C: **a** in
quiescent conditions; **b** after
shearing ($v_s = 4.5 \text{ s}^{-1}$, $t_s = 10$ s)
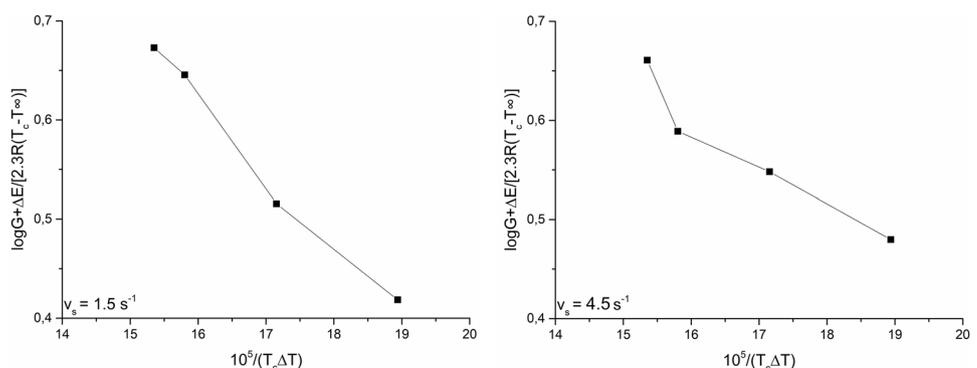
undercooling (T^0_m-T_c), $T_\infty = 231,2$ K and $T^0_m = 458,2$ K. It may be observed that the transition temperatures between various regimes are constant and depend only on the heat parameters and also do not depend on shear treatment. Thus, it may be concluded that the secondary nucleation process plays a greater role in overall crystallization kinetics than the primary, shear-induced nucleation.

## The presence of α and β iPP phase in sheared samples

To verify what kind of the crystallographic form of spherulites has been created through shear treatment, the method of selective-melting of iPP crystallites was carried out [60]. Observation of the melting of shear-induced spherulites in slow-increasing temperature allows to comment on the β and α form of iPP crystallites. After heating the crystallized

iPP film to the lower temperature value of 150–158°C, the disappearance of β-spherulites can be observed. Spherulites created form α iPP phase melts until at the higher temperatures, between 160–170°C. Figure 8a-c shows the spherulites behavior during heating of crystallized iPP films.

As can be seen in Fig. 8 and in Table 2, the melting of spherulites in the lower temperature of 158°C has been noticed only in the case of the melting of iPP samples crystallized after shear treatment.

Firstly, it should be emphasized that the shear forces induce a β phase of iPP formation [35, 38]. However, only for the samples sheared in the lower value of temperature T_s, 138°C, can the occurrence of β phase of iPP be noticed. In the research Huo et al. a conclusion can be found that above the concrete temperature value, the relaxation phenomena dominates over the shear-induced nucleation [43]. Thus, it can be said that the shear forces' ability for nucleation
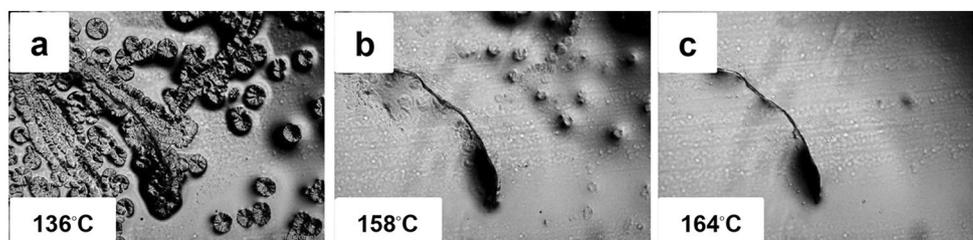
Fig. 6 Spherulites growth rate (G) after shear treatment in different shear temperature (T_s): **a** shear rate 1,5 s^{-1}, **b** shear rate 4,5 s^{-1}
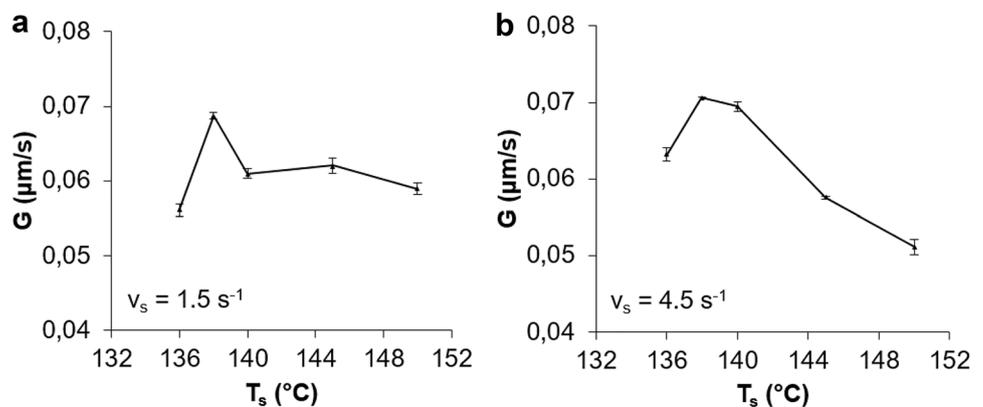
Fig. 7 The plots log + ΔE(2.3 R(T_c-T_∞) versus 1/(T_cΔT) determined for iPP sheared with shear rate appropriate 1.5 s^{-1} and 4.5 s^{-1}
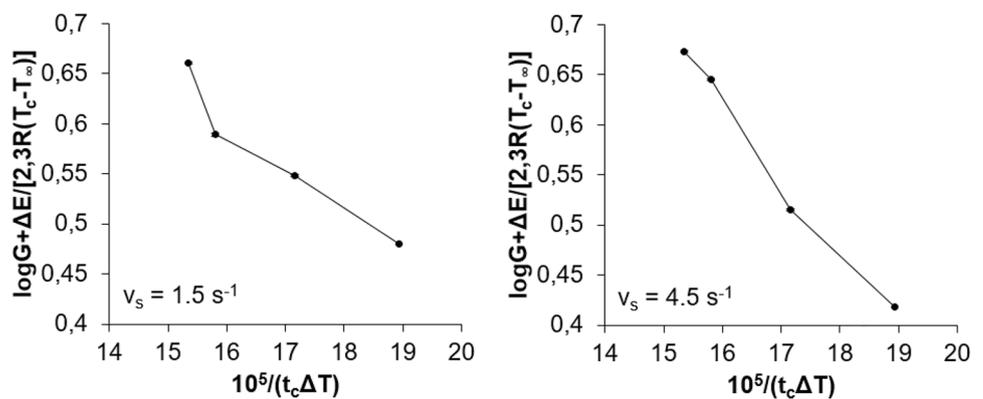
Fig. 8 The microscope images made during slow melting of shear-treated samples. Pictures a-c show iPP sheared in 138°C ($v_s = 4,5$, $t_s = 10$ s)

Table 2 Crystallographic forms of iPP observed in samples crystallized in different conditions

Shearing conditions			Crystallographic form of iPP present in shear-treated melt
T_s [°C]	v_s [s^{-1}]	t_s [s]	
154	4,5	10	α
		30	α
138	4,5	10	α and β
		30	α and β

decreases with increase of temperature. However, in our research only the growth of α spherulites in melt sheared in temperature 154°C can be observed. It should be highlighted why, in the higher temperature of shearing only, the α iPP form can be induced by shear forces. Moreover, the nucleis of β crystals created in the temperature higher than the value where the β-phase may occur, decompose immediately back to an amorphous phase or they reorganize on the line of β → α transition. The next possible reason for the described phenomenon is that in higher temperatures, wherein the nucleating abilities of shear forces are weaker, only the more energy-stable, monoclinic α form can be created. The shear forces in higher shear temperatures cannot make enough influence for chains, which is necessary for reorganization of them and creation of β crystals.

Conclusions

In this research, the shear-induced crystallization phenomenon was studied. It was focused on the kinetics crystallization of iPP as well as crystallites morphology and the occurrence of β-iPP. The formation of row nucleis and, in consequence, the fibrillar crystallites may be noticed only after shear treatment with shear rate over concrete, critical value (in current study higher than in the literature data, presented earlier).

The key parameter determining the shear-induced crystallization is a temperature value during shearing. It determines the crystallites morphology as well as crystallization kinetics. The shear rate and shearing time should not be neglected but from the viewpoint of the three crystallization regimes, the secondary homonucleation process is more important than the primary, shear-induced nucleation.

The influence of shear forces on crystallization kinetics and crystallites morphology (e.g., fibrils formation) is closely dependent on polypropylene type and processing characteristic. The shear forces may induce the β-iPP formation only in the appropriate temperature range. For samples sheared in the temperature higher than the melting temperature of β iPP value, the shear-induced β-crystallization

cannot be observed in contrast to the samples sheared in the lower temperature (138 °C). In higher temperatures, the abilities of shear forces to induce crystallization are weaker, and their abilities to creation β iPP nucleis practically do not occur.

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