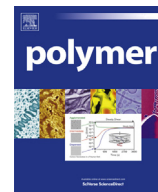




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## Influence of trisamide-based additives on the morphological and mechanical properties of isotactic polypropylene

M. Kersch<sup>a</sup>, L. Pischke<sup>a</sup>, H.-W. Schmidt<sup>b, c</sup>, V. Altstädt<sup>a, c, \*</sup>

<sup>a</sup> Polymer Engineering, University of Bayreuth, Universitätsstraße 30, 95447 Bayreuth, Germany

<sup>b</sup> Macromolecular Chemistry I, University of Bayreuth, Universitätsstraße 30, 95447 Bayreuth, Germany

<sup>c</sup> Bayreuther Institut für Makromolekülforschung (BIMF), Germany

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### ABSTRACT

The influence of two different trisamide-based additives on the morphological and mechanical behavior of isotactic polypropylene (PP) is investigated. Morphological investigations by wide-angle X-ray scattering (WAXS) show that only 0.04 wt.-% of one of the additives is enough to induce 80% beta modification in the crystalline part of the polymer. The materials are investigated with quasistatic (tensile testing) as well as short-time dynamic (Charpy impact strength) methods and for the first time fatigue crack growth measurements are performed on alpha- and beta-nucleated PP. In all cases a much higher toughness of the material containing the beta-nucleating agent (*beta NA*) can be observed. The crack growth propagation rate is one order of magnitude smaller with the use of the beta-nucleating agent compared to the alpha polymer. Scanning electron microscopy (SEM) pictures are used to support the correlation of the mechanical behavior with the morphological changes.

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### 1. Introduction

Isotactic polypropylene (PP) is a widely used commodity plastic due to its good combination of good mechanical properties and low price. To even further expand the possible applications of this material, an interest in toughening PP by either adding modifiers like rubbers [1] or modifying the crystal structure of the polymer has been increasing over the past years [2]. PP is a polymorphic material and can crystallize in different modifications: a monoclinic alpha phase, a trigonal beta phase, an orthorhombic gamma phase and a smectic mesophase [3]. Under normal processing conditions, mainly alpha-PP is obtained [4–6]. This alpha modification is thermodynamically stable and consists of a so called “cross hatched” lamellar morphology with radial lamella that are tangentially crosslinked. This hinders deformation leading to higher strength and stiffness and a more brittle behavior compared to the beta phase [3,7,8]. The thermodynamically metastable beta phase is usually not found under normal processing conditions, but can be induced by shear [9–13], temperature gradient [14,15] or by adding special beta-nucleating agents [13,16–19]. Beta spherulites

consist of only radial lamella without tangential crosslinks and thus enable plastic deformation [7,20–23]. The unstable gamma modification that can be obtained under high pressures, as well as a smectic mesophase that can be achieved by high cooling rates, are industrially not relevant [7]. The influence of the presence of a beta phase on quasistatic and short-time dynamic mechanical behavior of PP has already been investigated by some groups [2]. However to the best of our knowledge, no investigation have been done so far to study the influence of the beta modification in the case of cyclic loading of a material with an initial crack.

The easiest and industrial most relevant method to obtain a high amount of beta phase is clearly the use of nucleating agents. They can be easily added to the polymer without requiring much change in the production process. However, most nucleating agents that can induce the beta modification have some drawbacks. Even additives that do not influence the visual properties of the polymer, most additives have disadvantages such as the need for good dispersion. A new class of additives that overcomes this problem is the group of highly efficient, soluble nucleating agents based on benzene-trisamides, which was discovered in 2005 [24]. Since then, different groups have studied the influence of a number of these nucleating agents on the thermal behavior and optical properties of PP. Some of these nucleating agents act as alpha-nucleating agents and can improve the optical properties by 24% [24]. However it was also found that some of these additives can

\* Corresponding author. Polymer Engineering, University of Bayreuth, Universitätsstraße 30, 95447 Bayreuth, Germany. Tel.: +49 921557471; fax: +49 921557473.

E-mail address: [altstaedt@uni-bayreuth.de](mailto:altstaedt@uni-bayreuth.de) (V. Altstädt).

induce the beta modification in PP. Even though it is known that these nucleating agents exhibit a strong concentration dependent behavior [24], only a fixed concentration (0.15 wt.-%) was investigated so far regarding its influence on the crystallization temperature and beta phase content. Also no work has been done so far to investigate the influence of these additives on the mechanical properties of PP.

## 2. Experimental

### 2.1. Materials

PP (Moplen HP500V, MFR: 120 g/10 min) was kindly supplied as pellets by LyondellBasell. The nucleating agents used in this study were a commercially available alpha-nucleating agent (Irgaclear XT386, BASF SE) (*alpha NA*) and an experimental nucleating agent 1,3,5-Tris(2,3-dimethylcyclohexyl-carbonylamino)benzene (*beta NA*), synthesized at the Department of Macromolecular Chemistry I, University of Bayreuth. The influence of both additives on the optical properties of PP has already been reported [24]. In addition, it has been reported that *alpha NA* results in a complete alpha modification of PP whereas 0.15 wt.-% of *beta NA* resulted in 74% of beta modification [24]. An investigation of different concentrations has not been performed yet.

### 2.2. Sample preparation

In order to study the influence of different concentrations of the nucleating agents on the nucleation of the polymer and the amount of induced beta phase, small circular samples with additive concentrations from 0.01 to 1.0 wt.-% were prepared. PP pellets were pulverized, mixed with 1 wt% of additive in a glass bottle and dry blended for 24 h with a rotating mixing equipment. The powder was afterwards compounded under nitrogen atmosphere in a co-rotating twin-screw microcompounder (DSM Xplore) at a rotational speed of 40 rpm and a temperature of 240 °C for 5 min. The melt was discharged and filled directly into the injection molding piston. The different concentrations were prepared by subsequently diluting the initial nucleating agent concentration with neat PP. Circular plates with 25 mm diameter and a thickness of 1 mm were injection molded in a DSM injection molding machine using a conic mold. The process was carried out at 230 °C with a mold temperature of 20 °C and an injection pressure of 4 bar.

Higher amounts of selected concentrations were then compounded in a co-rotating twin screw extruder (Brabender DSE 20/40). Along the length of the barrel, the temperature was gradually increased from 140 °C to 240 °C, resulting in a final melt temperature of 240 °C at the die. The screw speed was 90 rpm. Tensile test and compact tension specimens were obtained by injection molding with an ENGEL e-max 200/50 injection molding machine with a mold temperature of 30 °C.

### 2.3. Thermal analysis

The crystallization temperatures and the degree of crystallinity of the samples were determined using a Mettler Toledo DSC/SDTA 821e DSC. The measurements were performed over a temperature range of 0–250 °C at a heating rate of 10 K/min under nitrogen atmosphere. The degree of crystallinity was determined using following equation [3]:

$$X_c = \frac{\Delta H_m}{\Delta H_{m,0}} \quad (1)$$

with  $\Delta H_m$  as the measured melting enthalpy and  $\Delta H_{m,0}$  as the melting enthalpy of a 100% crystalline PP (207 J/g) [1].

### 2.4. X-ray diffraction analysis (WAXS)

The crystal modification of the samples (tensile test specimen) was studied using a Seifert ID 3000 X-ray diffractometer operating at a voltage of 40 kV and a current of 30 mA. Using nickel-filtered Cu K $\alpha$  radiation (1.54 Å), the diffraction pattern was recorded with a step size of 0.05° between  $2\theta = 8^\circ$  and  $30^\circ$ . The amount of beta-modification was determined using the Turner-Jones method (eq. (2)). [25]

$$k = \frac{H\beta_1}{H\beta_1 + (H\alpha_1 + H\alpha_2 + H\alpha_3)} \quad (2)$$

with  $H\beta_1$  as the height of the beta peak at  $2\theta = 15.8^\circ$  and  $H\alpha_1$ ,  $H\alpha_2$  and  $H\alpha_3$  as the heights of the three strong alpha peaks at  $2\theta = 13.85^\circ$ ,  $16.6^\circ$  and  $18.2^\circ$ .

### 2.5. Optical microscopy

In order to investigate the skin-core structure of the injection molded samples, thin sections (7  $\mu$ m) were cut from the samples perpendicular to the mold filling direction with a microtome and investigated with a polarized light microscope.

### 2.6. Tensile testing

Tensile tests were performed according to DIN EN ISO 527 using a universal testing machine from Zwick GmbH & Co. KG (Z020). The module of the materials was determined at a speed of 1 mm/min whereas tensile strength and elongation at break were determined at a speed of 50 mm/min. For all materials at least six samples were tested.

### 2.7. Charpy impact strength

Determination of the Charpy impact strength was performed according to DIN EN ISO 197-2/eA with an impact pendulum from Zwick (RKP 5113). Notched samples (type 1 according to DIN EN ISO 197-2/eA) were prepared from the parallel section of the tensile test specimens. For all materials at least six samples were tested.

### 2.8. Fatigue crack growth

For determination of the fatigue crack growth behavior, compact tension (CT) specimens were used. Testing was performed based on ISO 15850/ASTM E647. The CT specimens were loaded dynamically in tension–tension mode with a frequency of 1 Hz using a servo-hydraulic testing machine (IST Hydropuls MHF). The applied load was sinusoidal, with a steadily increasing stress amplitude  $\Delta K$ , and a minimum-to-maximum load ratio  $R = 0.1$ . A sharp crack was initiated with a razor blade before the measurement. The fatigue crack growth rate was determined in accordance to the method described by Saxena and Hudak [26].

### 2.9. Scanning electron microscopy (SEM)

The fracture surfaces of the samples tested for fatigue crack growth were investigated with a field emission scanning electron microscope (LEO 1530) with an accelerating voltage of 3 kV. The surfaces were sputtered with platinum before the investigation.

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