

CRACK TIP DEFORMATION AND TOUGHNESS IN POLYPROPYLENES

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ABSTRACT

In-situ measurements were done to get information about crack tip deformation processes in different polypropylenes. For the measurements a conventional fracture mechanics test arrangement for three point bending tests coupled with a stereo microscope and a camera was used. In this way a directly correlation of load and deflection with crack extension and crack tip opening displacement is possible and crack resistance curves (R-curves) can be achieved. On principle the method is a single specimen method.

The influence of ethylene content and elastomer content on the crack opening displacement at the crack initiation point is demonstrated on different polypropylene (PP) materials: homopolymers, random copolymers, blends and heterophasic copolymers.

KEYWORDS

fracture mechanics, physical crack initiation, single specimen method, polypropylene

INTRODUCTION

The knowledge of the physical crack initiation process is the basis for the quantification of deformation determined fracture processes. It is generally well known that the fracture process is initiated by the crack tip blunting and stretch zone formation. The crack initiation occurs by opening the crack tip after exceeding a critical deformation. The blunting process depends on stress state, microstructure, testing velocity and temperature. Therefore, polymers show no unique crack tip blunting and initiation behaviour by reason of their structural variety and their specific critical behaviour which depends on testing velocity and temperature.

Crack tip blunting and initiation processes in polymers are a combination of crazing, local shear deformation and voiding, which depends on the structure, the material state and the testing conditions. For this reason crack initiation processes of polymers are not inevitable in accordance with the classical stretch zone concept [1,2]. Different investigations about crack tip blunting and crack tip deformation processes are done, e.g. in [3-6] for amorphous and in [3,7-13] for semicrystalline polymers. The process of transition from a blunted to a growing stable crack is controversial discussed for quite some time. In principle two mechanisms of crack tip deformation processes are provable. Both are influenced by the molecular structure. Results of different investigations exist which include problems of stretch zone determination on fracture surfaces [14-17].

EXPERIMENTAL

Materials

Different polypropylene (PP) materials were investigated: homopolymers, polyethylene (PE)/PP random copolymers, heterophasic copolymers with different interparticle distances A , and PP/EPR (ethylene propylene rubber) blends with different EPR content (Table 1). The average particle diameter of the elastomeric phase in the blends ($\approx 2 \mu\text{m}$) and in the heterophasic copolymers ($\approx 1.5 \mu\text{m}$) are comparable. Additionally the EPR phase of the heterophasic copolymers includes lamellas of crystalline PE [18,19].

TABLE 1
INVESTIGATED PP MATERIALS

term	material	description
PP 1, 2	homopolymers	polypropylene
RaCo 1, 2	random copolymers	4 mol.-% and 8 mol.-% ethylene
Blend 1, 2	PP/EPR blends	PP 1/EPR = 85/15 and 80/20
HeCo 1, 2	heterophasic copolymers	matrix PP 2, $A = 2.12 \mu\text{m}$ and $A = 1.38 \mu\text{m}$

In-situ testing

For the in-situ tests a conventional fracture mechanics test arrangement for three-point bending tests was coupled with a stereo microscope and a camera. Uniform time-scale enables direct correlation between the load-time and deflection-time signals and the crack extension as well as the crack tip opening displacement. Injection moulded single edge notched bend specimens with the dimensions $80 \times 10 \times 4 \text{ mm}^3$ were used. The specimens were notched with a razor blade. To prevent shear lips side-grooves (radius $r = 0.1 \text{ mm}$) were mill-cut. Measurements were done using a support span of 40 mm and a strain rate of 0.001 s^{-1} .

The crack tip opening displacement (CTOD) δ and the stable crack growth Δa were taken from the recorded videos, J values were determined from the measured load-deflection curves (Eqn. 1) according to [20]:

$$J = \eta_{el} \frac{A_{el}}{B(W-a)} + \eta_{pl} \frac{A_{pl}}{B(W-a)} \left\{ 1 - \frac{(0.75\eta_{el} - 1)\Delta a}{(W-a)} \right\}, \quad (1)$$

where A_{el} and A_{pl} are the elastic and the plastic part of generally deformation energy and η_{el} and η_{pl} are geometry functions.

The in-situ investigation allows the separation of the crack tip blunting process and the crack growth process. The blunting line was fitted using a linear equation (2). Crack growth curve was fitted with the help of a power law (3).

$$J = c \Delta a \quad (2)$$

$$J = C_1 (C_3 + \Delta a)^{C_2} \quad (3)$$

The crack initiation point was determined visually during analysing the video. This results were compared with results from δ - Δa curves, where the intersection of blunting line and crack growth curve was defined as crack initiation point.

For the investigation of deformation behaviour and damage in the crack tip region sections of about $5 \mu\text{m}$ were microtomed at $-100 \text{ }^\circ\text{C}$ using a MICROM microtome and a glassy knife. The investigated specimens were both fully unloaded and fixed in several bended states. The crack tip region of the latter ones was fixed with an embedding resin and cutting was done after curing of the resin. Microscopical observations were done at a ZEISS Axiolab Pol using crossed polars and a SEM PHILIPS XL 30.

RESULTS

Typical results of the measurements are shown in Fig. 1. Received load-deflection curves were normalized to the effective area, which depends on the actual crack length. The homopolymer breaks unstable at a crack length of about 0.6 mm whereas the modified materials do not break under the testing conditions chosen. Analysing the recorded video the crack initiation points were defined (see the arrows in Fig. 1). Crack initiation takes place below the maximum of the curves, but clearly above the linear region.

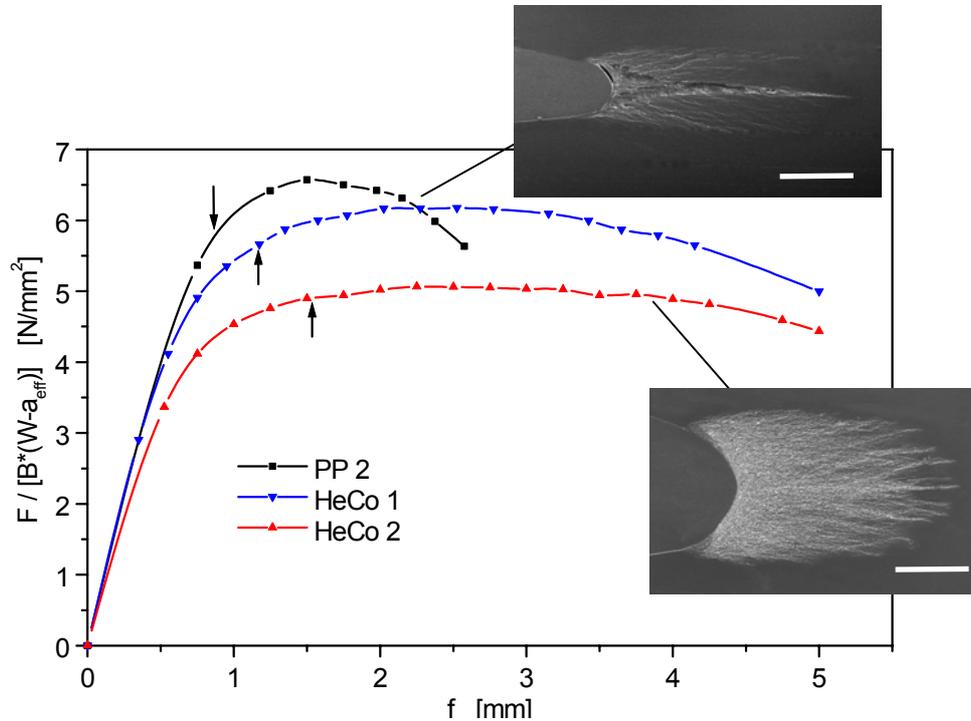


Figure 1: Normalized load vs. deflection for several PP-copolymers and the PP-homopolymer. Arrows indicate the visually defined crack initiation point. Scale bar length: 1 mm.

From the in-situ determined δ values and crack lengths δ - Δa curves can be plotted and crack initiation values were determined [21]. This single specimen method allows the determination of fracture mechanics values without relaxation and blunting effects, which is the main advantage in comparison with the partial unloading method. Furthermore the crack lengths are measured, not calculated.

CTOD at the in-situ determined crack initiation point depends on morphological and structural parameters (Fig. 2). For the heterophasic copolymers there is a critical value at a interparticle distance of about 2 μm .

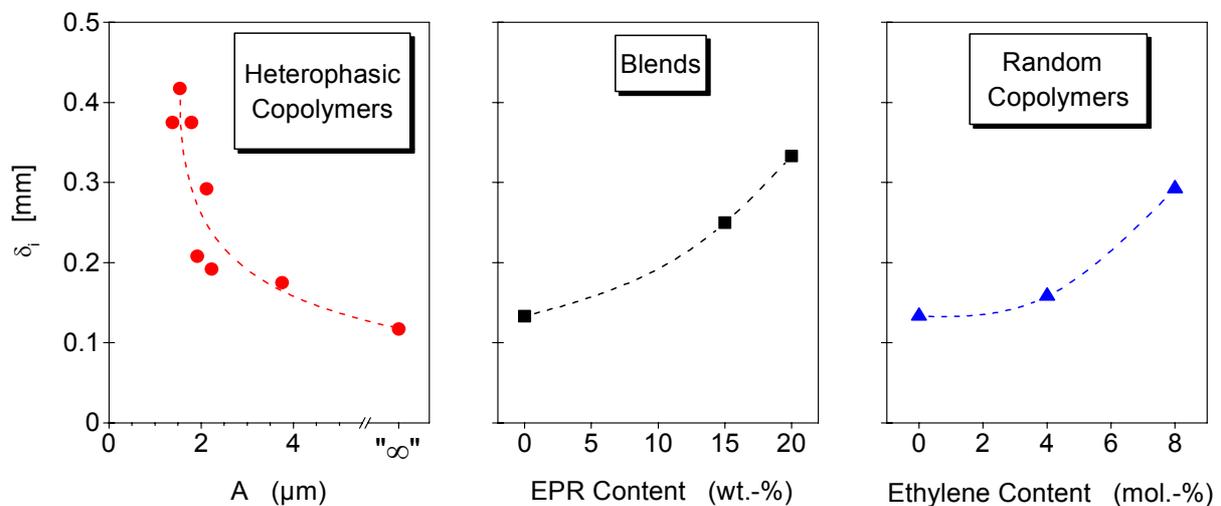


Figure 2: CTOD at the in situ determined crack initiation point δ_i in dependence on structural parameters.

Undergoing this value leads to a strong increase of δ_i –values. In dependence on EPR- and ethylene content the crack initiation values increase. Critical values are not able to determine due to the less number of investigated materials.

In conventional fracture mechanics tests the CTOD at crack initiation can be determined from δ - Δa curves as the intersection of the blunting line and the crack growth curve. Therefore the comparison of the CTOD values defined from δ - Δa curves with the visually defined ones (Fig. 3) is of a special interest. This procedure serves to check the experiment. For most of the investigated materials there is a good agreement between them.

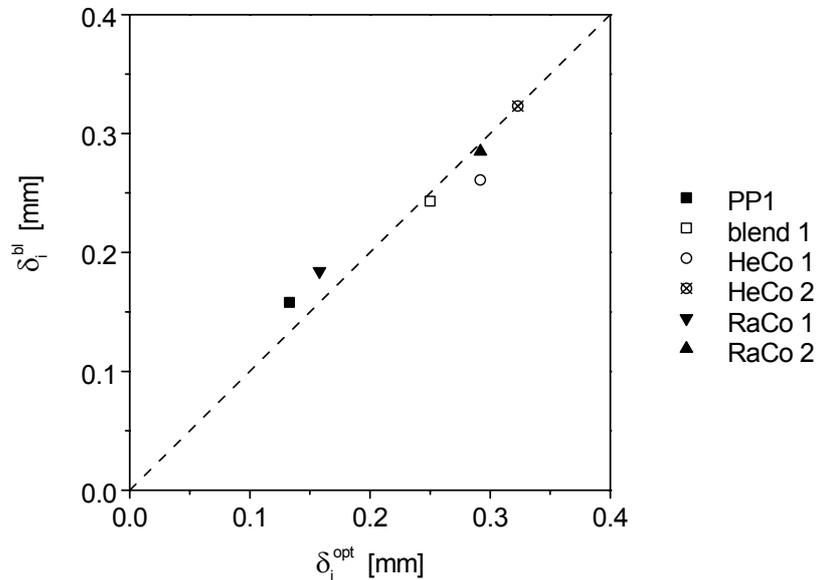


Figure 3: CTOD from δ - Δa curve δ_i^{bl} in dependence on visually defined CTOD at crack initiation point, δ_i^{opt} .

Figure 4 demonstrates that for polymers the elastic and viscoelastic deformation parts of general CTOD are very high. That means, that measurement of stretch zone dimensions on fracture surfaces can lead to an underestimation and blunting lines determined with this method are also underestimated. The quantity of the “error” in the first place depends on material and loading conditions.

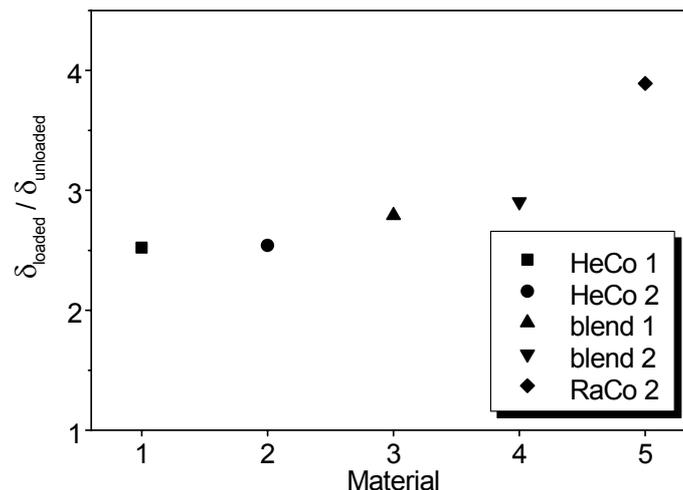


Figure 4: Ratio of the loaded and of the fully unloaded specimen for several PP-materials at a deflection of 5 mm.

The fracture process of the investigated PP- materials is demonstrated in Figure 5. In the first stage of blunting which corresponds to a seeming linear material behaviour in the load-deflection diagrams first damage occurs in front of the crack tip. For the homopolymers (Fig. 5/1a and b) and random copolymers deformations are of craze-like type. The whole blunting process (Figs. 5/1c and 5/2a) takes place without

translation of the crack tip and includes beside the seeming linear material behaviour a not neglectable non-linear part (see Fig. 1, position of the crack initiation points). The process is more a stretching in tension direction than a curving. Strong, large craze-like deformations are visible not only in the centre but also at the sharp edges between the blunted tip and the flanks of the initial razor notch (Figure 5/1c). The pre-crack flanks control shape of the large middle craze. The crack initiation is characterized by a more “gradual” than an abrupt transition from blunting to growing. The shape of the crack tip does not change clearly. The moving crack tip is not very sharp. Figures 5/1d and 5/2b show growing cracks in the different materials.

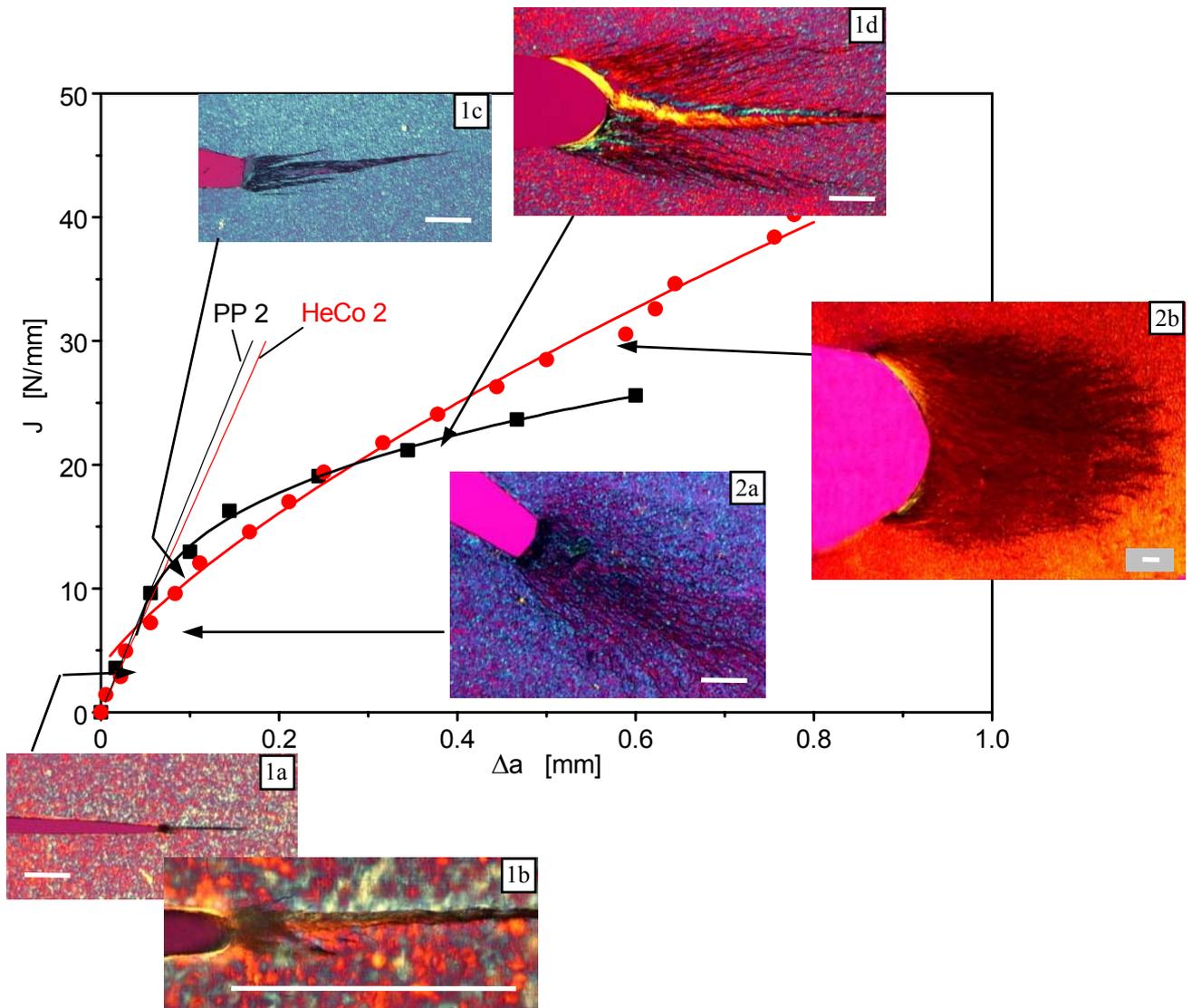


Figure 5: J-Δa curves of a PP-homopolymer and a heterophasic copolymer. Deformation states in the crack tip region: (1 a-d) homopolymer; (2 a,b) copolymer; scale bar length: 200 μm.

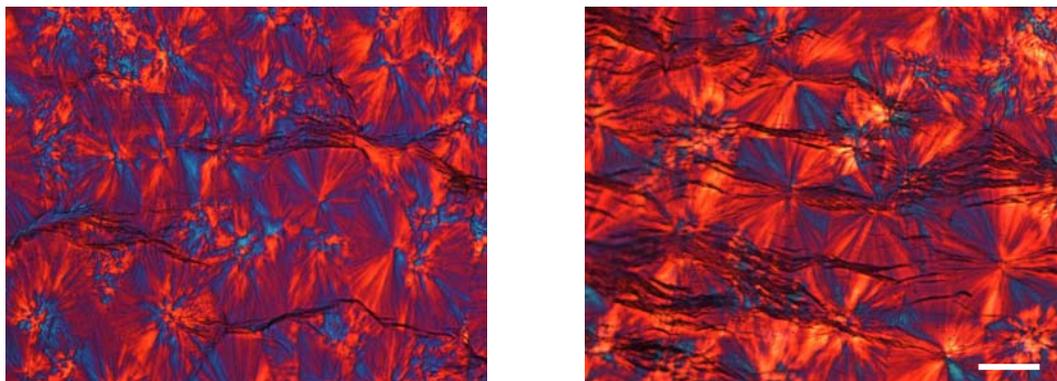


Figure 6: Crazing in the damaged region in front of the crack tip (scale bar length 50 μm)

Craze and microcrack paths in front of the crack tip are trans- and interspherulithic, which can be seen in Figure 6. For the elastomer modified materials the shape of the damaged region in front of the crack tip is different compared to the homopolymers (Figures 5/2a and b). Its shape is circular like and in the SEM failure of the rubber/matrix interphase can be seen. So-called “croids” are forming [18].

CONCLUSIONS

In-situ testing using a stereomicroscope is a suitable method to get information about deformation behaviour in front of the crack tip. It can be used as a single specimen method because the crack front of the side-grooved specimens is nearly linear, not round shaped.

Crack opening displacement at the visually defined crack initiation point was in good agreement with the CTOD determined from intersection of blunting line and fitted δ - Δa curve. The obtained crack initiation values depend on the ethylene content and on the elastomer content for the random copolymers and the blends or heterophasic copolymers respectively. For all materials crack initiation takes place without a clear change of tip shape. No stretch zone could be proved on fracture surfaces.

In homopolymers and random copolymers there is a crazed region in front of the crack tip in the elastomer modified materials, i. e. blends and heterophasic copolymers, however “croiding” was found.

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