CRACK INITIATION BEHAVIOUR OF PP-MATERIALS

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ABSTRACT

To get information about the deformation processes at the crack tip, in situ measurements were done using a miniaturized bending device and a stereomicroscope. The influence of ethylene content and elastomer content on the crack opening displacement at crack initiation point is demonstrated on different polypropylene (PP) materials: random copolymers, blends and heterophasic copolymers.

Crack resistance curves can be achieved from results of the in situ measurements. This shows, that the used experiments are suitable as a single specimen method.

INTRODUCTION

The knowledge of the physical crack initiation process is the basis for the quantification of fracture processes determined by deformation. It is generally well accepted that the crack tip blunting and stretch zone formation occur at first. The crack initiation occurs by opening the crack tip after exceeding a critical deformation on the crack tip. The blunting process depends on stress state, microstructure, testing velocity and testing 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.

So, the crack tip blunting and crack initiation processes of polymers are of a complex nature. They 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]. A multitude of investigations about crack tip blunting are done, e.g. in [3-6] of blunting in amorphous and in [3,7-13] of crack tip deformation processes in semicrystalline polymers.

The process of transition from a blunted to a growing stable crack is controversial discussed at present.

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/ethylene propylene rubber (EPR) blends with different EPR content (see Table 1). The average particle diameter of the elastomeric phase in the blends (about 2 µm) and in the heterophasic copolymers (about 1,5 µm) is comparable. Additionally the EPR phase of the heterophasic copolymers includes lamellas of crystalline PE [18, 19].

TABLE 1

INVESTIGATED PP MATERIALS

name	material	description
PP 1	homopolymer	
PP 2	homopolymer	
RaCo 1	random copolymer	4 mol.-% ethylene
RaCo 2	random copolymer	8 mol.-% ethylene
Blend 1	PP/EPR	PP 1/EPR = 85/15
Blend 2	PP/EPR	PP 1/EPR = 80/20
HeCo 1	heterophasic copolymer	A = 2.12 µm, matrix PP 2
HeCo 2	heterophasic copolymer	A = 1.38 µm, matrix PP 2

In situ testing

To observe the fracture processes a KAMMRATH & WEISS miniaturized bending device and a stereomicroscope were used. The bending device was modified to increase constraint. Video equipment consisting of CCD camera, monitor and recorder was connected to the stereomicroscope.

Specimens with a length of 35 mm, a width W of about 5 mm and a thickness B of 4 mm were cut from the centre of injection moulded specimens (80x10x4 mm^3). The specimens were notched with a razor blade. To prevent shear lips sidegroves (radius r = 0.1 mm) were mill-cut. Measurements were done using a support span of 24 mm and a testing velocity of 1.5 mm/min, which is the maximum speed of the miniaturized bending device. Testing arrangement can be seen in Figure 1a. The crack front of the side groved specimens is nearly linear (Figures 2 a and b). This is a requirement if the method should be used as a single specimen method. There are necking effects in some materials.

The recorded videos of the crack tip deformation processes were analyzed afterwards. Figure 1b shows the determination of crack tip opening displacement (CTOD) and stable crack growth (Δa). J-integral values were determined from measured load-deflection curves (Eqn. 1) according to [20]:

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

where A_{el} and A_{pl} are elastic and 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. Blunting line was fitted using linear equation (2). Crack growth curve was fitted with the help of a power law (3).

$$J = c\,\Delta a \qquad (2)$$

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

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

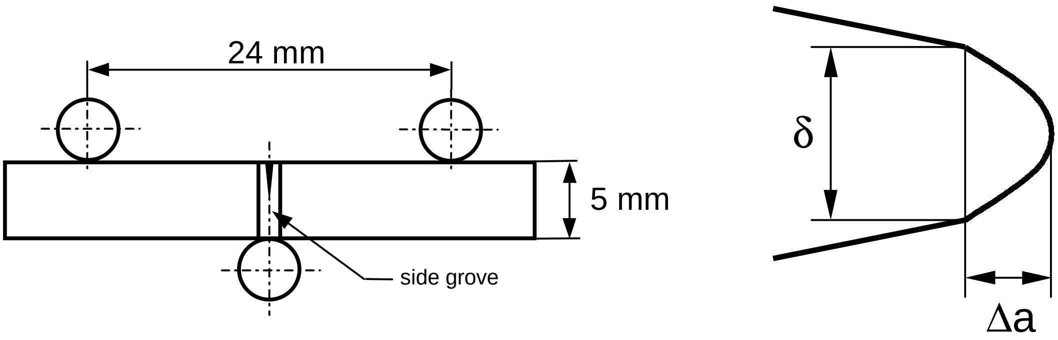

Figure 1: In situ testing: specimen dimensions and testing arrangement (a); Determination of crack opening displacement δ und crack growth Δa from side view (b)

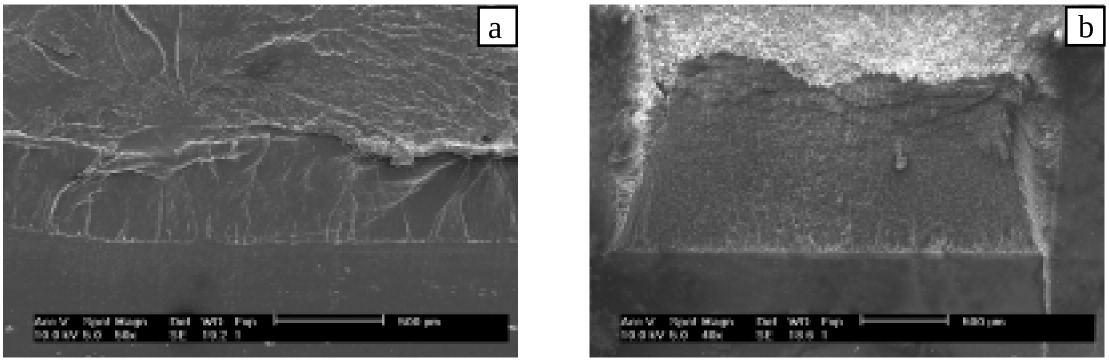

Figure 2: Fracture surfaces of PP-homopolymer (a) and heterophasic copolymer (b)

Polarization microscopy

For the investigation of deformation behaviour and damage in the crack tip region sections of about 5 µm were microtomed from the tested specimens at - 100 °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. After hardening of the resin they could be cut with the microtome. Microscopical observations were done at a ZEISS Axiolab Pol using crossed polars.

Scanning electron microscopy

Besides light microscopy SEM investigations were done on fracture surfaces and cross sections of the crack tip region. For the investigations of fracture surfaces, loaded specimens were cooled to cryogenic temperatures and then broken or cut. Cutting of both fully unloaded and fixed specimens were done using the microtome. To prevent electrostatical effects a thin gold layer was sputtered on the specimen surface. The SEM was of PHILIPS XL 30 type.

RESULTS

Typical results of the measurements are shown in Fig. 3. Received load-deflection curves were normalized to the effective area, which depends on the actual crack length. Analyzing the recorded video the crack initiation points were defined (see the arrows in Fig. 3). Crack initiation takes places below the maximum of the curves, but clearly above the linear region.

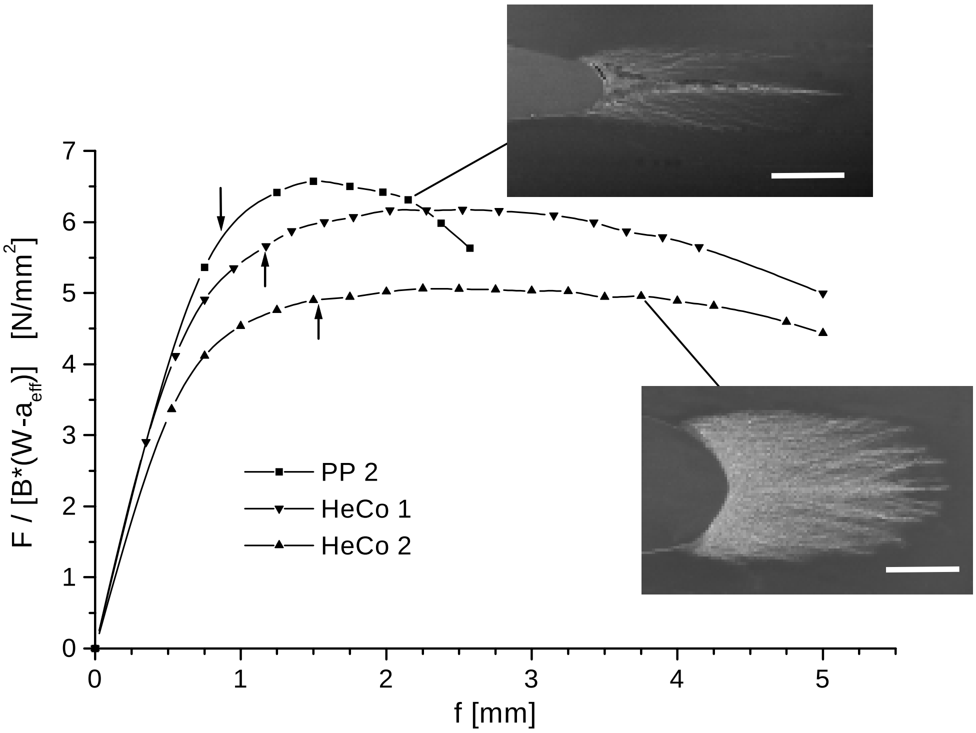

Figure 3: Normalized load vs. deflection for several PP-copolymers and the PP-homopolymer. Arrows indicate visually defined crack initiation point. Scale bar length: 1 mm.

From the in situ determined CTOD values and crack lengths δ-Δa curves were determined, which are plotted exemplarly in Figure 4. Blunting and stable crack growth were seen. The slopes of the blunting lines are only slightly influenced by the morphology. The slopes of the crack growth curves depend on the interparticle distance. The homopolymer breaks unstable at a crack length of about 0.6 mm whereas the modified materials do not break at the testing conditions chosen.

Note, that all results received from respective one specimen, so it is a single specimen method. One advantage to the partial unloading method is that the relaxation and blunting effects which are problematic for the evaluation of R-curves and the corresponding fracture mechanics values in partial unloading tests are considered. Furthermore the crack lengths are measured, not calculated.

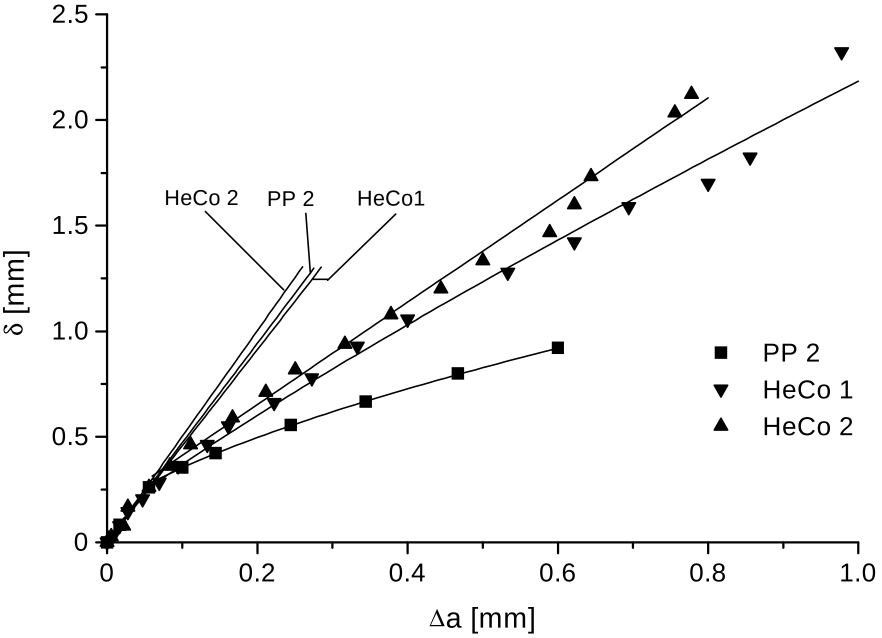

Figure 4: δ - Δa curves determined from the in situ measurements for several heterophasic copolymers and the homopolymer.

CTOD at the in situ determined crack initiation point depends on morphological and structural parameters (Figure 5). For the heterophasic copolymers there is a critical value at a interparticle distance of about 2 µm. 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.

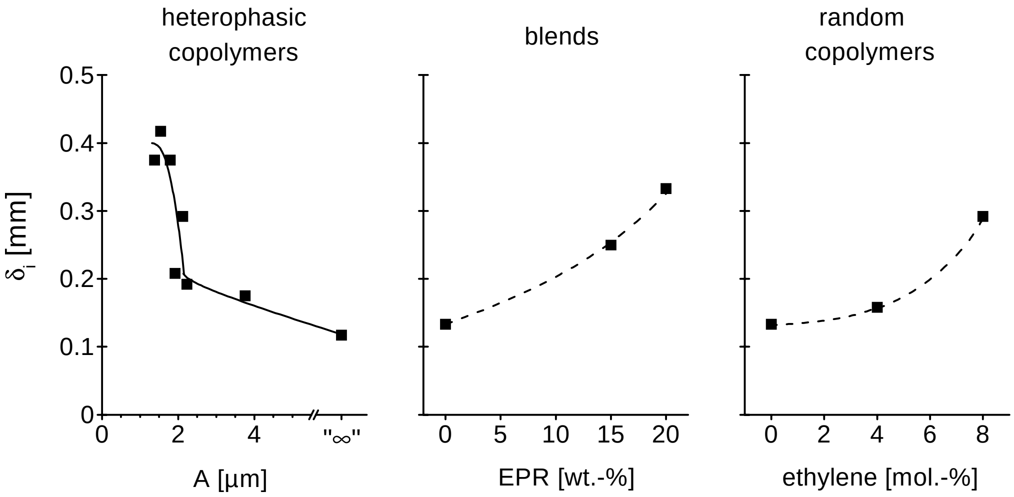

Figure 5: CTOD at the in situ determined crack initiation point in dependence on morphological or structural parameters respectively.

Normally CTOD at crack initiation point is determined from δ-Δa curves as the intersection of blunting line and crack growth curve. Therefore the comparison of the CTOD values defined from δ-Δa curves with the visually defined ones (Figure 6) is of a special interest. This procedure serve to check the experiment. For most of the investigated materials there is a good agreement between them.

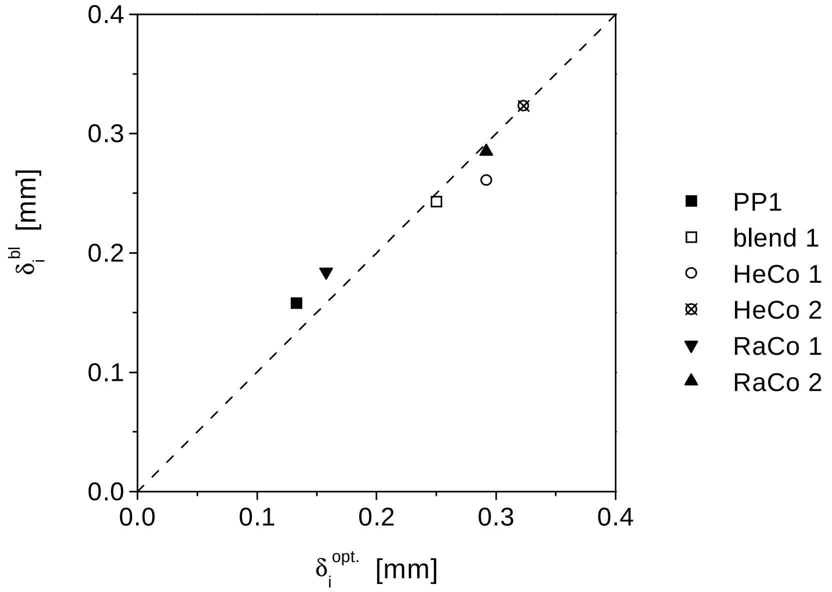

Figure 6: CTOD from δ-Δa curve δ_i^{bl} in dependence on visually defined CTOD at crack initiation point, δ_i^{opt}.

Figure 7 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.

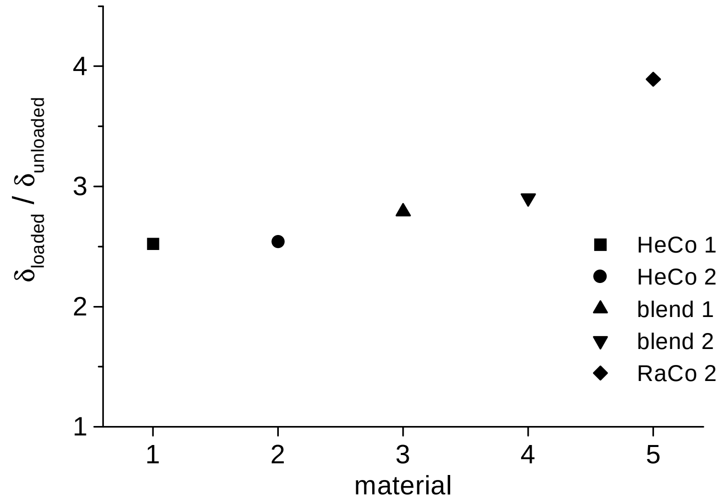

Figure 7: Ratio of the loaded and of the fully unloaded specimen for several PP-materials at a deflection of 5 mm.

The fracture process of the investigated PP- materials is demonstrated in Figure 8, which shows J-Δa curves and micrographs of thin sections of the crack tip region. 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

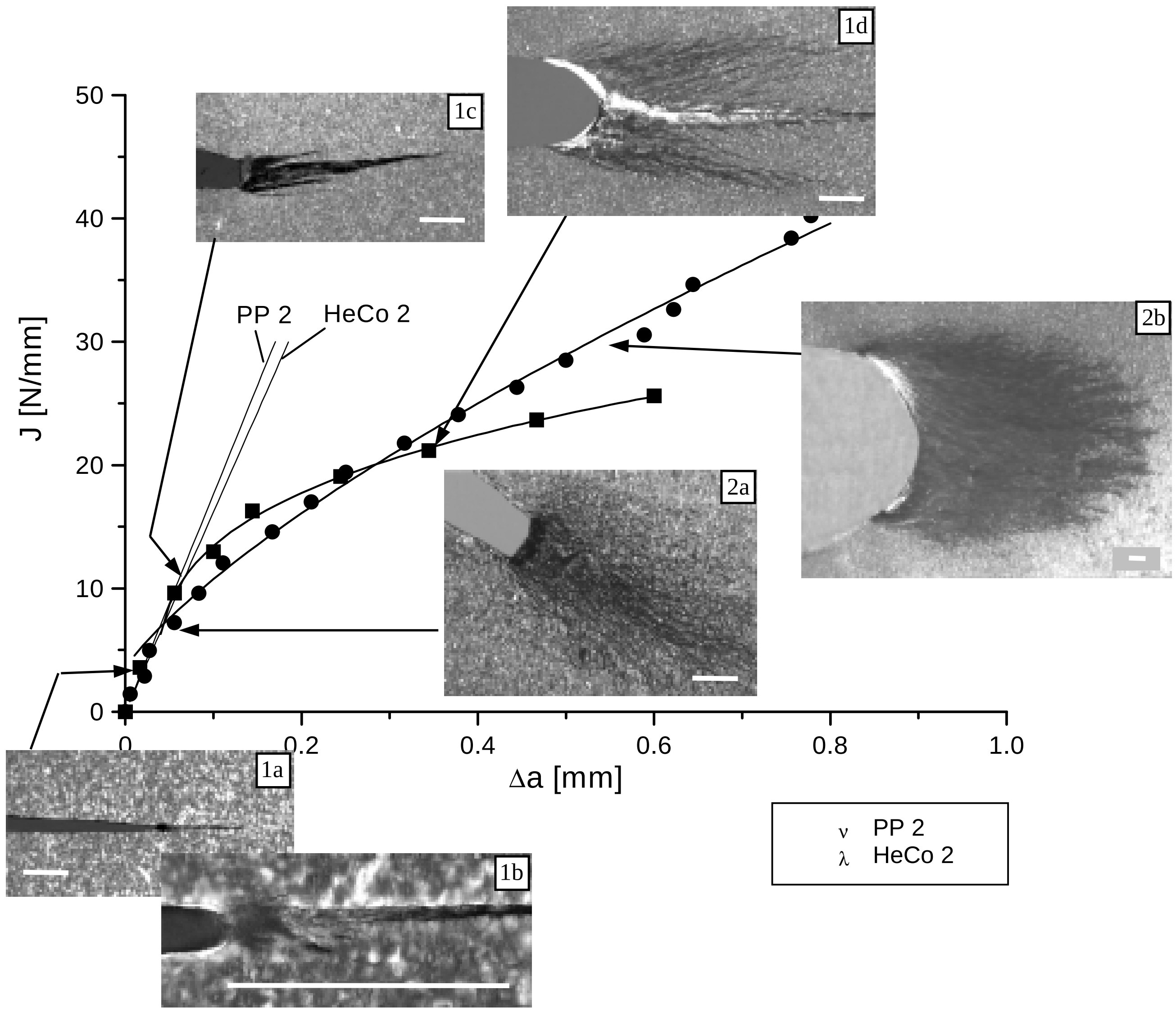

Figure 8: J-Δa curves of a PP-homopolymer and a heterophasic copolymer. Deformation states in the crack tip region: (1 a-d) homopolymer; (2 a,b) copolymer; scale bar length: 200 µm.

tip. For the homopolymers (Figure 8/1a and b) and randomcopolymers deformations are of crazelike type. The whole blunting process (Figures 8/1c and 8/2a) takes place without translation of crack tip and includes beside the seeming linear material behaviour a not neglectable non-linear part (see Figure 3, position of the crack initiation points). The process is more a stretching in tension direction than a curving. Strong, large crazelike deformations are visible not only in the centre but also at the sharp edges between blunted tip and flanks of initial razor notch (Figure 8/1c). Shape of the large middle craze is controlled by the precrack flanks. 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 8/1d and 8/2b show growing cracks in the different materials.

Craze or microcrack paths in front of the crack tip respectively are trans- and interspherulithic, which can be seen in Figure 9.

For the elastomer modified materials the shape of the damaged region in front of the crack tip is different compared to the homopolymers (Figures 8/2a 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.

SEM investigations of fracture surfaces show that no stretch zone can be proved at the end of initial crack for all investigated materials (see Figure 2).

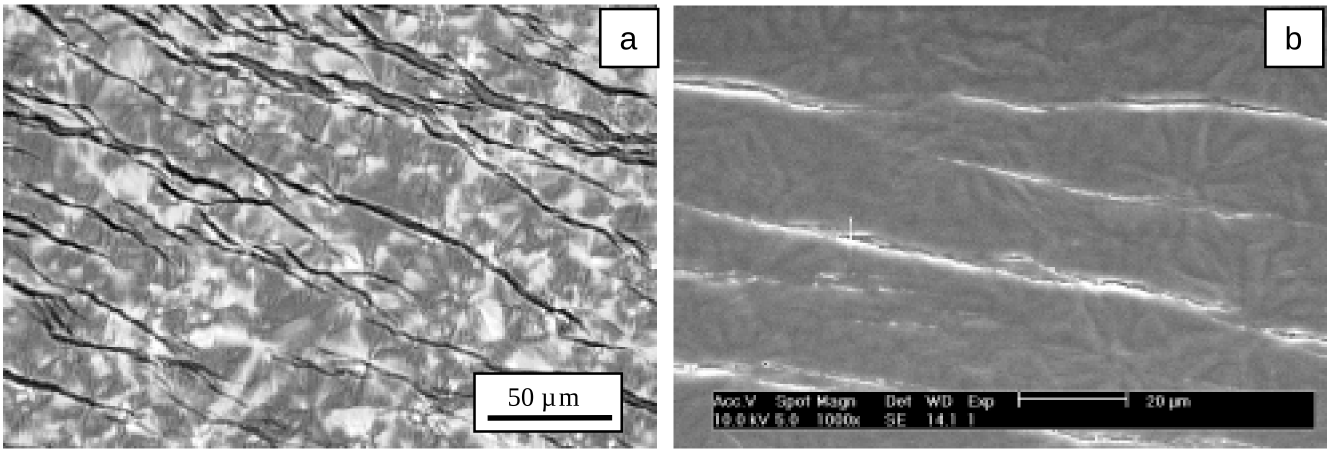

Figure 9: Crazing in the damaged region in front of the crack tip (material PP 2)
(a) optical micrograph of thin slide; (b) SEM image of cross section

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 groved 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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