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FRACTURE OF NOTCHED POLYPROPYLENE SPECIMENS UNDER CREEP CONDITIONS

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SUMMARY: The kinetics of slow crack growth of isotactic polypropylene (Vestolen PP P 9022) was observed in single-edge-notched specimens under a constant tensile load and at a temperature of 95 °C. The notch opening under different stress levels was measured as a function of time. Two types of specimens (perpendicular and parallel to the extrusion direction) were examined. The development of the processing zone and the crack advance was observed after unloading at surfaces introduced along and across the crack plane. Optical and scanning electron microscopy was used for these observations. Morphologies of the processing zone as a function of loading time and stress level were assessed. The relationships between (i) stress and time to rupture for ductile and brittle region and (ii) minimal notch opening rate $\dot{\delta}_0$ and stress intensity K_{I0} were expressed quantitatively. A pronounced effect of the orientation on the crack propagation was established. The time to break of specimens taken along the extrusion direction was about 20 % higher than that of the specimens taken in the cross direction.

Introduction

Polypropylene (PP) has many critical applications such as pipes or geomembranes in landfills and containers, which undergo low stresses in long-term service. Under these conditions, PP fractures in a brittle mode after a period of time, so-called "long-time brittle failure". The long-time mechanism of failure is controlled by the crack initiation and by a slow crack growth.

The same type of failure was observed in polyethylene around room temperature and under low stresses [1]. Brown and his co-workers [2] estimated that the fracture process of PE under plane strain conditions consists of sequences of micro-events: (i) generation of microcraze, (ii) increase in size of the macrocrazes to form coarse fibrils, (iii) extension of fibrils and their fracture. We assumed that the lifetime of PP would be controlled by the same sequence of events.

The applications mentioned above require polypropylenes with very long lifetimes. Then the necessary standard hydrostatic-pressure tests [3], currently used either as a quality control test or as an introduction test for a new resin, take a very long time.

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Brown and Lu introduced an accelerated test method [4] which measures the slow crack growth behaviour of PE. The method involves a constant load test on a notched specimen under plane strain conditions in air at various temperatures. This method simulates the same fracture process which occurs during the long-time failure of PE.

The aim of this paper was: (1) to apply this method to PP, (2) to estimate experimental conditions for the brittle mode of failure, (3) to describe the kinetics of this process by the crack opening displacement (COD) as a function of time and by the rate of COD as a function of fracture mechanics parameter K_I, (4) to estimate time to failure (t_f) and time for crack initiation (t_b) as a function of the applied stress.

Experimental

Polypropylene homopolymer Vestolen PP P 9022 from Hüls AG was used for this investigation. Its physical and mechanical properties are given in Table 1.

Table 1. Physical and mechanical properties of Vestolen PP P 9022

Parameter	
Melt index (21 N, 230 °C)	0.39 g/10min
Melt index (49 N, 230 °C)	1.50 g/10min
Density	920 kg/m^3
Yield stress	32 MPa
Tensile strain at yield	20 %
Tensile strain at break	> 50 %

Fracture behaviour

The specimens (50×25×10 mm) were prepared from extruded plaques parallel and perpendicular to the extrusion direction. They were supplied by the University of Erlangen-Nürnberg (Department of Plastics Technology, Demonstration Centre for Fibre Composites).

For evaluation of the "long-time brittle failure", the method according to Brown [4] was used. The geometry of the specimens was SENT (single-edge-notch tensile specimen). The specimens were 25 mm thick so that their fracture, with the aid of 1-mm side grooves, was almost entirely plane strain. The notch depth was 3.5 mm in order to minimise the failure time but not to produce an excessive creep on the remaining ligament. The notch was made by indenting a fresh razor blade into the specimen at a constant speed of 330 μm/min. Such specimen geometry is also desirable because of the stress intensity analysis.

The kinetics of the failure process was observed in a range of constant nominal stresses (4.5, 4.0, 3.5, 3.1, 3.0, 2.8, 2.5 and 2.0 MPa) and at 95 °C. The COD was measured with an optical microscope. The COD was viewed in the middle of the notch and measured in two positions, (i) at the surface of the specimen (notch opening displacement) and (ii) in the tip of the notch (COD). The error of the measurement of the COD was about 2 μm.

The testing conditions are important for the following reasons: (i) the temperature 95 °C is usually the highest temperature for testing PP because above 95 °C, significant morphological changes occur and (ii) it is possible to estimate the upper stress limit producing the same type of brittle fracture which occurs at room temperature under long-time service conditions.

The kinetics of brittle fracture process in PP was measured until a defined time (2 h, 0.2 t_f, 0.5 t_f, and 0.8 t_f, where t_f is the time to failure at a chosen stress level) and over a range of stresses (3.5, 3.3, 3.0 and 2.8 MPa).

Tensile modulus and yield stress over the range of temperatures and testing speeds were measured. The time dependences of modulus and yield stress were estimated by the standard superposition procedure [5].

The FEM (finite element method) was used to describe the elastic-plastic behaviour in the neighbourhood of the notch tip.

Morphological characteristics

Small crack advances during the test were observed on freeze-fracture surfaces after unloading. The specimens were chilled and then broken by a Charpy hammer. The changes in shape and size of the processing zone were observed in the mid-plane transversal to the notch plane and parallel to the crack growth direction. This surface was prepared by a slow blade cutting of the specimen.

Results and discussion

The plots of notch opening displacement versus logarithm of time for different stress levels are presented in Fig. 1 (specimens oriented along the extrusion direction).

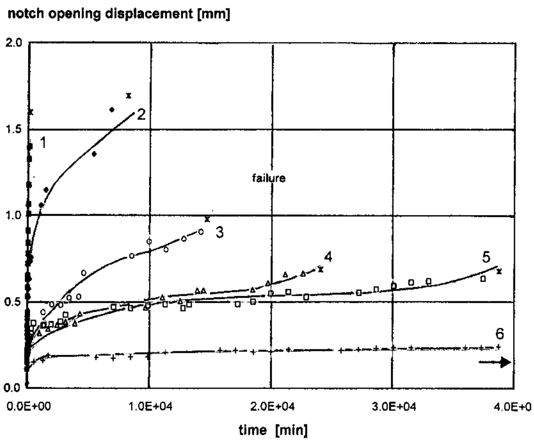

Fig. 1

Notch opening displacement versus time for different nominal stress levels

σ (MPa): *1* 4.5, *2* 4.0, *3* 3.5, *4*, 3.0, *5* 2.5, *6* 2.0

The examples of transversal and crack plane view of the fracture surfaces are shown in Fig. 2.

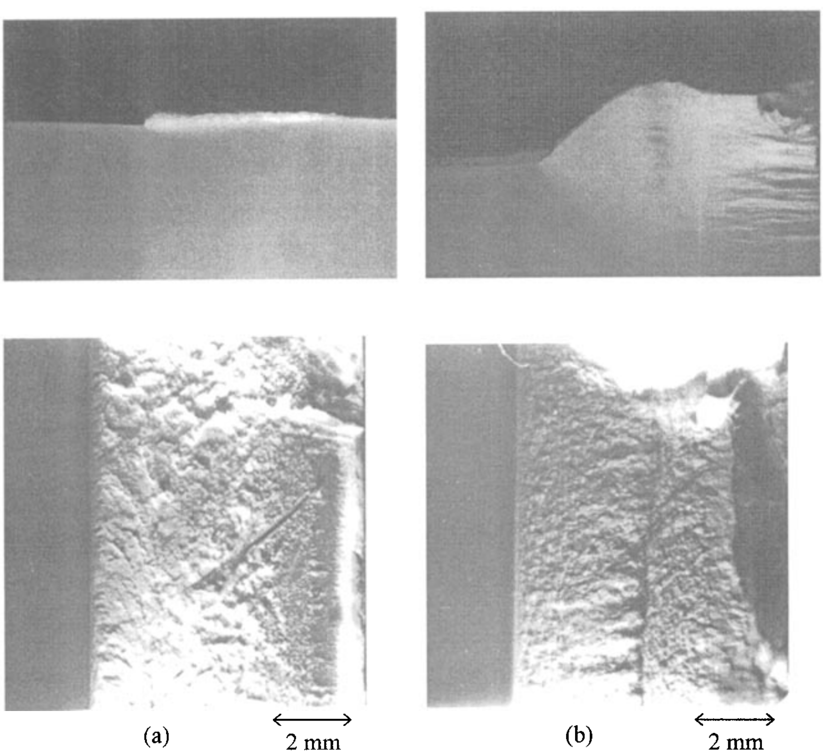

Fig. 2. Optical microphotographs of transversal cut surfaces and fracture surfaces for specimens loaded onto stress level (a) 3.5 MPa, (b) 4.5 MPa

The effect of stress on the time to failure is presented in Fig. 3.

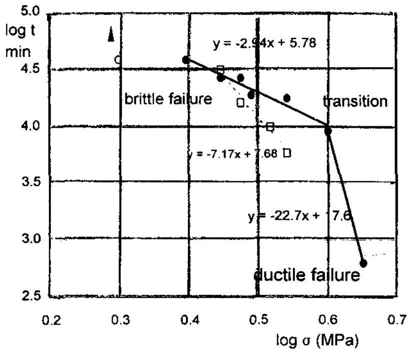

Fig. 3

Life-time curves for parallel (●) and perpendicular (□) specimens

Three failure modes were observed: ductile, transitional and brittle. The time-to-failure (parallel specimens) depends on the stress in the ductile region as $t_f = 4 \cdot 10^{17} \cdot \sigma^{-2.3}$ and that in the brittle region varied as $t_f = 6 \cdot 10^5 \cdot \sigma^{-2.9}$. The upper stress limit of the brittle region is 3.5 MPa. The failure shows features of transitional mode at the stress level of 4.0 MPa. Above this level, only ductile failures occur. As expected, the time to failure $t_f\|$ of parallel specimens differs from that of $t_f\perp$ of perpendicular specimens (Fig. 3) due to molecular orientation.

The kinetics of brittle failure process was estimated from the morphological observation of unloaded specimens (Fig. 4) which were before loaded over the stress range (2.8, 3.0, 3.3, 3.5 MPa) and time range (2 h, 0.2 t_f, 0.5 t_f, 0.8 t_f).

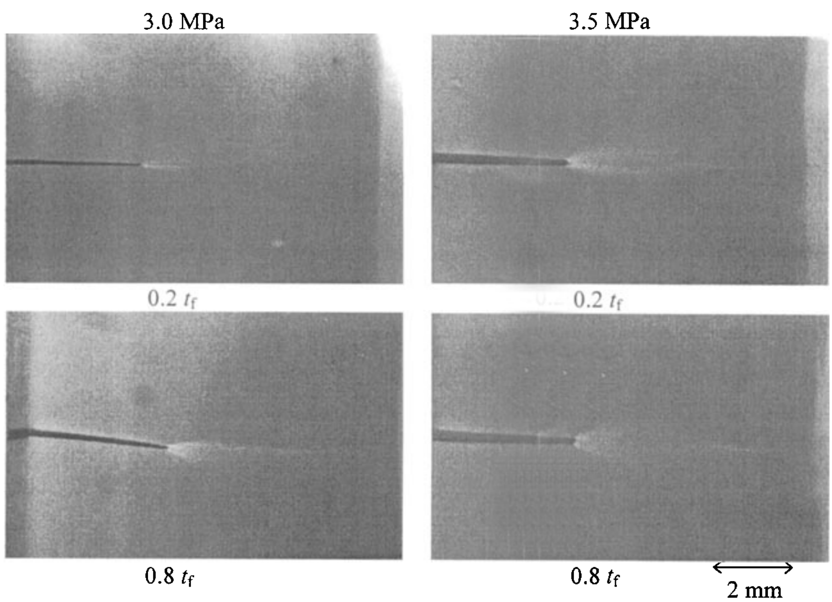

Fig. 4. Optical microphotographs of transversal cut surfaces for specimens loaded onto stress levels 3.0 and 3.5 MPa for different portions of their life-time at a given stress level

Time dependences of notch and crack opening displacement were also measured (Figs. 5,6).

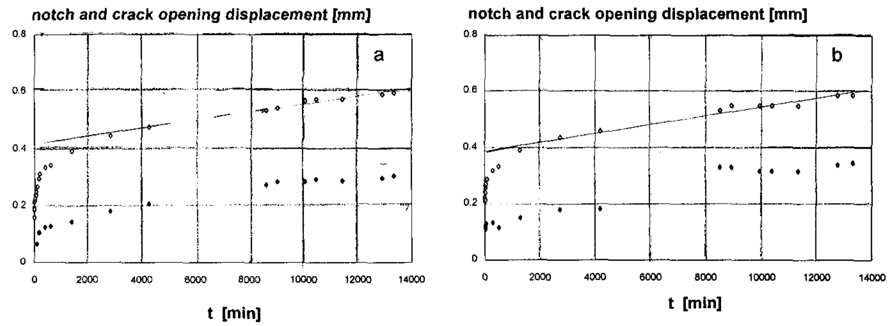

Fig. 5. Examples of time dependence of COD in Vestolen PP P9022
(a) parallel, (b) perpendicular (PENT test, 3.0 MPa, 0.5 t_B). ◇ notch, ◆ crack

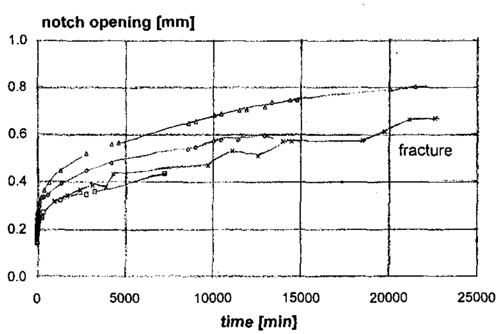

Fig. 6

An example of COD-time dependence for a set of Vestolen P 9022 specimens loaded onto stress level of 3.0 MPa for different time periods (PENT test, parallel)
○ 2 h, □ 0.2 t_f, ◇ 0.5 t_f, Δ 0.8 t_f, x t_f

After the observation of the freeze fracture and transversal-cut surfaces (Fig. 4), we concluded that no crack advance occurs until the time of 0.8 t_f and over the stress range of 2.8, 3.0, 3.3, and 3.5 MPa. It is clear that the kinetics of this process is driven mainly by the advance of the processing zone and not by the advance of the crack as was observed in PE [2]. The advance of the processing zone correlates with the drop in yield stress estimated by superposition measurements. The minimum slope of the COD vs. time curve represents the rate of stable brittle failure process.

The finite element method (FEM) procedure was used to describe the shape and advance of the plastic zone. The input data for calculation were time-dependent modulus of elasticity and yield stress (Figs 7 and 8). The results are presented for 3.0 MPa and time t = 30 s and $t = 10^6$ s (Fig. 9). The theoretical (Fig. 9) and experimental (Figs 4 and 10) shapes of the plastic zone differ considerably. The only possible explanation is a combination of two failure mechanisms in the tested PP, (i) generation and growth of crazes and (ii) generation and advance of the plastic zone.

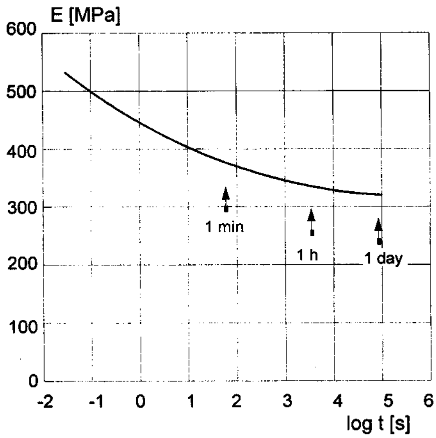

Fig. 7. The generalised curve of tensile modulus of elasticity for PP at 95 °C

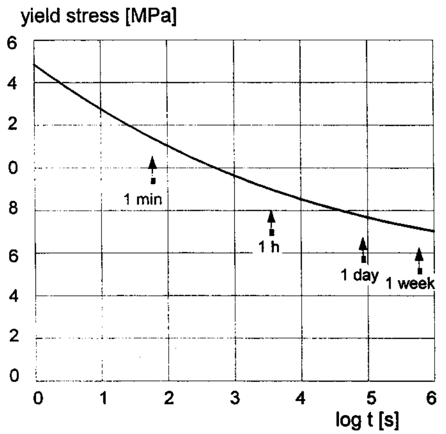

Fig. 8. The generalised curve of yield stress for PP at 95 °C

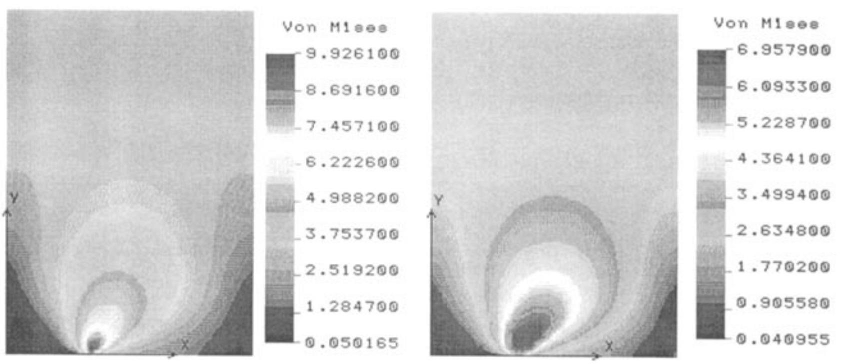

Fig. 9. The shape of plastic zones in SENT specimen for two different times of loading, 30 and 10^6 s. (Computed with the FEM using the von Mises criterion)

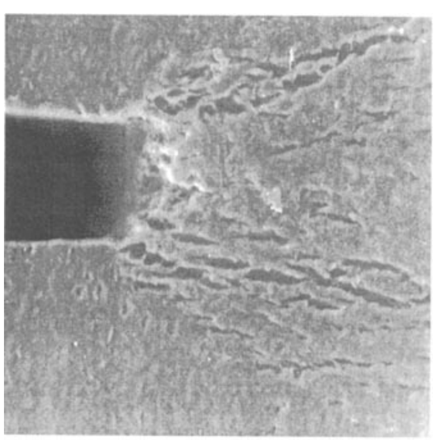

Fig.10

SEM microphotograph of transversal cut surfaces for specimens loaded onto stress level 3.0 MPa for the 0.5 t_f

The most convenient fracture parameter used in linear elastic fracture mechanics (LEFM) is stress intensity factor K_I. For the type of failure observed in PP, a more convenient fracture parameter for describing this type of fracture process would be the elastic-plastic fracture parameter, J-integral. Nevertheless, we tried to find a correlation between minimal rate of notch opening $\dot{\delta}_0$ and K_{I0} (for the initial crack length a_0). For a single specimen geometry and single type of loading used in our investigation, the stress intensity factor is given by $K_I = Y \cdot \sigma \cdot a_0^{1/2}$ where Y is geometrical factor, s is applied stress and a_0 is the crack length. The geometrical factor for a specimen with a rectangular cross-section is given by

$$Y = \sqrt{\pi \cdot \left[1.12 - 0.231 \cdot (a_0 / W) + 10.55 \cdot (a_0 / W)^2 - 21.72 \cdot (a_0 / W)^3 + 30.39 \cdot (a_0 / W)^4\right]}$$

where W is the width of the specimen (W = 10 mm and a_0 = 3.5 mm).

We found a good correlation between K_{I0} and $\dot{\delta}_0$ (Fig. 11). This dependence can be described by equation $\dot{\delta}_0 = A_1 \cdot K_{I0}^{5.43}$.

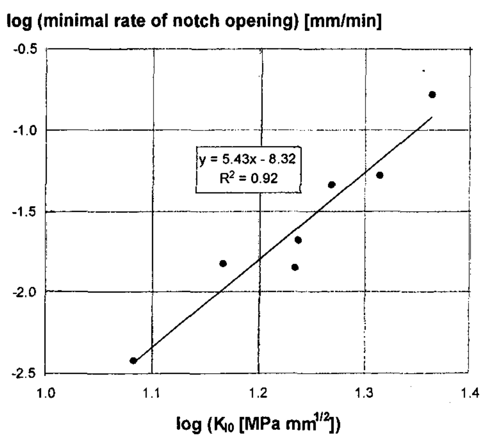

Fig.11

Correlation between the minimal rate of COD (representing the rate of the failure process) and stress intensity factor K_{I0} for parallel PP specimens

Conclusions

– The present study explored the possibility of using the accelerated test method introduced by Brown and Lu for polypropylene.

– The measurement over a range of stresses enabled estimation of the upper limit for the brittle mode of failure at the highest test temperature T = 95 °C. For Vestolen PP P 9022, this parameter is: σ = 3.5 MPa.

– The failure process in PP was described by the dependences of notch and crack opening displacements vs. time and by morphological observations.

– The anisotropy of extruded plaques was confirmed.

– The most important parameters in the process of brittle failure are: (i) the minimum rate of notch opening and (ii) time to failure.

– A good correlation between the fracture parameter K_{I0} and $\dot{\delta}_0$ was found. Nevertheless, the kinetics of failure in PP in the brittle region is driven mainly by a combination of advance of the plastic zone at the notch tip and growth of crazes. This behaviour was confirmed from fracture surfaces and from FEM calculation.

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