Enhanced cyclic fracture mechanics tests to examine morphological and molecular results on slow crack growth in contemporary PE pipe grades

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ABSTRACT

Three different polyethylene pipe grades and three different lots of one PE pipe material were investigated with fracture mechanics procedures under cyclic and impact loads. The cyclic tests with Cracked Round Bars allowed a ranking of the different PE pipe grades and lots concerning crack resistance as a function of failure time as well as of crack initiation time. The ranking corresponded to the expectations based on the molecular and morphological properties of the materials. Concerning lot-to-lot variations, in particular, a correlation to crystallization kinetics, melt flow rate and density measurements could be established.

KEYWORDS: Fracture Mechanics, Structural Integrity, Hydrogen Embrittlement, Ultrasonics

1.0 INTRODUCTION

Improvements in the polymerization process of polyethylene (PE) over the last two decades have led to polyethylene high density (PE-HD) pipe materials (PE80, PE 100, PE 100+) with bimodal distribution of the molecular mass and specific placement of the short chain branches on the high molecular mass fraction being responsible for an extraordinary long-term performance of these materials [1 - 15]. From the scientific literature it is well known that crack propagation properties of PE are extremely sensitive even to small changes in molecular and morphological parameters (average molecular mass and molecular mass distribution, side chain length and concentration, details of crystallinity) [16 - 32]. Consequently, lot-to-lot variations in the materials crack resistance can not be excluded. The currently used methods (particularly EN ISO 9080: Determination of the long term hydrostatic strength of thermoplastic materials in pipe form by extrapolation; ISO 16770: Full Notch Creep Test – FNCT; ISO 16241: Pennsylvania Notch Test – PENT) to assure the required service life of up to 50 years for PE pipe materials are not suitable for quality assurance procedures, because they are extremely time consuming and consequently expensive as well [33 - 47]. Therefore, the main objective of this work, was to enhance the test sensitivity of the cyclic tests with Cracked Round Bars (CRB) and to proof its applicability for a quick quality assurance method which is able to assess different molecular and morphological effects, as well as lot to lot variations on slow crack growth (SCG), which is widely accepted to be the critical failure mechanism of pipes for long-term failure under internal pressure [48, 67].

2.0 BACKGROUND

One way to accelerate SCG even in higher performance PE-HDs and at room temperature is the application of fatigue loads. In recent years the authors could establish a procedure based on cyclic tests with CRB, to characterize the fatigue crack growth (FCG) behavior of PE-pipe materials within less than a week at 23 °C by measuring the failure times at different initial loads [68 – 76]. Currently they were able to improve the method by using extensometers and detecting the crack initiation time, which gives another reduction of testing time of about 50 % [77 - 83].

3.0 EXPERIMENTAL

All investigations were performed on 3 materials and 3 lots of one commercially available PE 100 (MRS 10) pipe material (PE100.1; PE100.2-a, -b, -c; PE100.3). The material classification PE 100 is established from internal pressure tests on pipes and means that pipes with a hoop stress of 10 MPa at 23 °C have a durability of 50 years. FCG testing was conducted using a servo-hydraulic closed-loop testing machine (MTS Systems GmbH, D) under sinusoidal load at a frequency of 10 Hz and an R-ratio (Fmin/Fmax) of 0.1 at 23 °C. Cracked Round Bars (CRB) with a length, L, of 100 mm and a diameter, D, of 14 mm (Fig. 1) were machined from 15 mm thick compression molded plates. Razor blades were used to pre- crack the specimens. Three extensometers were placed on the CRBs in

peripheral direction and in distances of 120 °. The crack initiation times and the failure times were documented as described in [24 - 46]. Based on a linear regression in a double logarithmic scale the investigated formulations were compared. Impact tests were conducted on a conventional pendulum machine (Ceast, I) at 23 °C. The Charpy specimens were machined according to EN 10450 with a cross section of 10 × 10 mm and a length of 55 mm; the relative notch length a/W was 0.35 (the final pre-crack in the specimens were produced with a razor blade) [47 – 59]. The span of the sup- ports was defined with S=4×W, 40 mm. The tests were performed with an instrumented 15 Joule pendulum at a velocity of 1.5 m/s. The energy data (total fracture energy) was calculated from the load-displacement traces. Based on experiences in [60 - 76] the different materials and lots were also evaluated by their isothermal crystallization behavior at 124 °C. Samples of 7 mg were weighed and sealed in aluminum-light crucibles (40 µl) prior insertion into the DSC equipment (Mettler Toledo, CH) [77 – 83]. In addition, the results from the above described testing methodologies were compared with molecular and morphological properties characterized by infrared spectroscopy (IR), size exclusion chromatography (SEC), rheological experiments, melt flow rate measurements (MFR) according to ISO 1133 (5 kg - 190 °C) and density measurements according to DIN 53479-A [14 – 29].

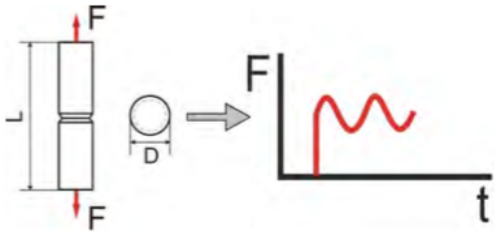

Fig. 1 Cracked Round Bar (CRB) specimen and fatigue loading mode.

4.0 RESULTS & DISCUSSION

In Table 1 the results of the infrared spectroscopy for the different PE100 grades are shown. The materials differ in commoner type (PE100.1 and PE100.3: hexane, PE100.2: butane) and concentration, with PE100.1 showing the highest concentration of short chain branches [30 – 42].

Table 1 Co-monomer type and concentration of the PE100 grades.

Material [-]	Co-monomer	
	Type	Concentration [1/1000 C]
PE100.1	Hexene	2.81
PE100.2	Butene	1.41
PE100.3	Hexene	1.91

In Table 2 and Fig. 2 the results of the size exclusion chromatography for the different PE100 grades are shown. PE100.1 and PE100.2 have a bimodal distribution of the molecular mass. PE100.3 is modified for injection molding and shows an unideal distribution. The width of the molecular mass distribution characterized by the polydisparity (PD) is also clearly higher for PE100.1 and PE100.2 [43 – 57].

Table 2 Molecular mass of the PE100 grades.

Material [-]	M_w [kg/mol]	M_n [kg/mol]	PD [-]
PE100.1	287	9	32
PE100.2	271	9	30
PE100.3	198	10	20

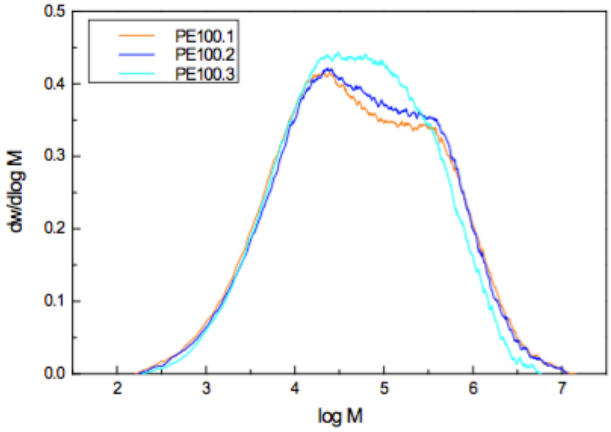

Fig. 2 Cracked Round Bar (CRB) specimen and fatigue loading mode.

In Table 3 and Fig. 3 the results of the rheological experiments for the different PE100 grades are shown. The data are evaluated by the intersection of the storage shear modulus G' and the loss shear modulus G''. A shift of this crossover to lower angular frequencies gives evidence to a higher weight average molecular mass (+MM) and a shift to lower modulus values is a hint to a broader molecular mass distribution (+MMD) [9]. The results correspond to those of the SEC with the highest molecular mass and broadest molecular mass distribution for PE100.1 followed by PE100.2 and PE100.3 [56 – 64].

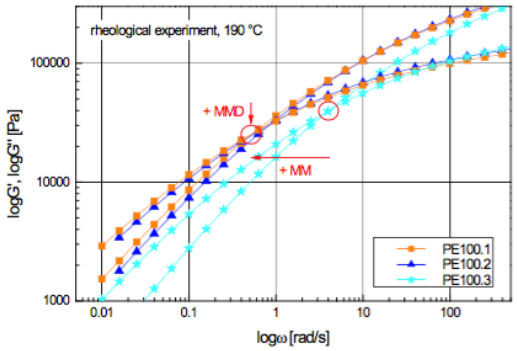

Fig. 3 Loop of storage and loss modulus as a function of the angular frequency of the PE100 grades.

Table 3 Crossover values of the PE100 grades.

Material [-]	Crossover ω_C [rad/s]	G_C [Pa]
PE100.1	0.57	26241.41
PE100.2	0.89	31266.03
PE100.3	4.05	39944.03

In Table 4 the results of the MFR and density measurements are shown. The differences in MFR between PE100.1 and PE100.2 are only minor whereas the MFR of PE100.3, which is modified for injection molding, is much higher. The values of the density increase from PE100.1 to PE100.3.

Table 4 MFR and density values of the PE100 grades.

Material [-]	$MFR_{190/5}$ [g/10min]	Density ρ [g/cm³]
PE100.1	0.204	0.9607
PE100.2	0.202	0.9615
PE100.3	0.626	0.9627

In Fig. 4 the results of the fatigue tests with the CRB specimens for the different PE100 grades are shown. Ac- cording to the expectations based on the molecular and morphological properties PE100.1 (high concentration of hexane, high average molecular mass and bimodal distribution) shows the highest failure times followed by PE100.2 and PE100.3, that gives clearly lower cycles to failure. The crack initiation times give the same ranking, so a reduction of testing times of about 50 % is possible.

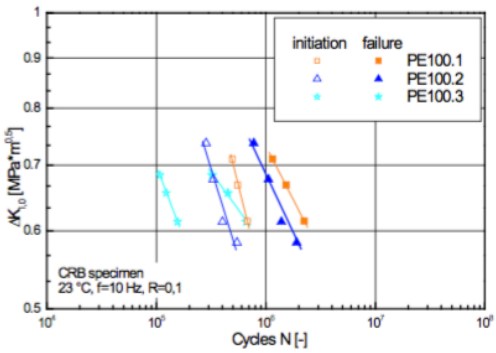

Fig. 4 Ranking of the PE100 grades based on FCG experiments with CRB specimens.

In Fig. 5 and Fig. 6 the results of the impact tests with Charpy specimens are depicted. Looking at the fracture surfaces of the three different grades in Fig. 5 it is obvious that at these test conditions only PE100.2 and PE100.3 show rapid crack growth – RCP whereas PE100.1 shows ductile failure. In case of PE100.2 and PE100.3 on the fracture surface of the Charpy specimens' different areas can be distinguished [65 – 76]. After the initial notch a stress whitened "thumbnail" marks, what is assumed to be stable craze growth during loading. It is the breakdown of craze extension to initiate RCP. Distinctive features appear at each verge of the RCP surface: shear lips, indicating ductile drawing from the surface. Finally, a ductile hinge develops (this is typically for impact bend specimens) [77 – 83]. The heavily notched ligament is deformed by a combination of craze growth and gross plastic deformation until the specimen can escape between the supports [19 – 31]. The fraction of the different areas on the whole fracture surface is very sensible not only to variations of testing conditions (temperature, velocity, etc.), but also to molecular and morphological variations [10]. Although different mechanisms are responsible for FCG and RCP, the ranking based on total fracture energy in the RCP tests (Fig. 6) is similar to the ranking from the fatigue tests. According to the expectations based on the interpretation of the fracture surface the total fracture energy of PE100.1 is much higher than that of the other two grades [32 – 47].

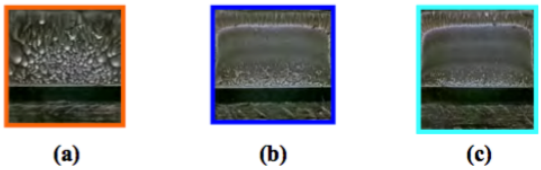

Fig. 5 Fracture surfaces of the Charpy specimens of the PE100 grades tested with an instrumented 15 Joule pendulum at 23 °C and 1.5 m/s; (a) PE100.1, (b) PE100.2, (c) PE100.3.

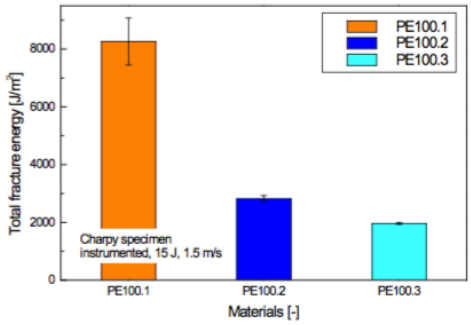

Fig. 6 Ranking of the PE100 grades based on instrumented impact tests with Charpy specimens at 1.5 m/s.

In Table 5 the results of the infrared spectroscopy for the different PE100.2 lots are shown. Of course, the types of the co-monomers are the same for all lots, however, the concentrations are slightly different, with the highest values for PE100.2-a [48 – 63].

Table 5 Co-monomer Type and Concentration of the PE100.2 lots.

Material [-]	Co-monomer	
	Type	Concentration [1/1000 C]
PE100.2-a	Butene	1.41
PE100.2-b	Butene	1.16
PE100.2-c	Butene	1.31

In Table 6 and Fig. 7 the results of size exclusion chromatography for the different PE100.2 lots are shown. All lots have the typical bimodal distribution of the molecular mass. The values of the weight average molecular mass Mw and the number average molecular mass M_n are only slightly different and do not allow any differentiation [64 – 76].

Table 6 Molecular values of the PE100.2 lots.

Material [-]	M_w [kg/mol]	M_n [kg/mol]	PD [-]
PE100.2-a	271	9	30
PE100.2-b	264	9	29
PE100.2-c	271	9	30

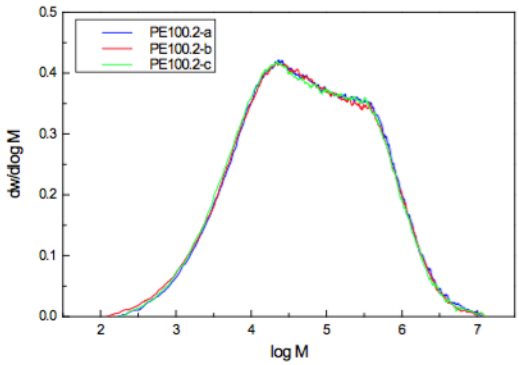

Fig. 7 Distributions of the molecular mass of the PE100.2 lots.

In Table 7 the results of the rheological experiments for the different PE100.2 lots are shown. Here a distinct variation of the crossover points can be detected. Clearly lower values for frequency and modulus of the crossover for PE100.2-a indicate a higher weight average molecular mass and a broader distribution of molecular mass [1 – 13].

Table 7 Crossover values of the PE100.2 lots.

Material [-]	Crossover	
	ω_C [rad/s]	G_C [Pa]
PE100.2-a	0.89	31266.03
PE100.2-b	1.02	33064.32
PE100.2-c	1.03	32362.35

In Table 8 the results of the MFR and density mea- surements for the different PE100.2 lots are shown. Re- flecting the results from SEC and rheology the values of MFR and density increase from PE100.2-a to PE100.2-c [14 – 29].

Table 8 MFR and density values of the PE100.2 lots.

Material [-]	$MFR_{190/5}$ [g/10min]	Density ρ [g/cm³]
PE100.2-a	0.202	0.9615
PE100.2-b	0.235	0.9631
PE100.2-c	0.251	0.964

In Fig. 8 the isothermal crystallization peaks at 124 °C are compared for the three lots of PE100.2. It is evident that the peak of the curve of PE100.2-b is shifted to higher heat flow values and shorter crystallization times. Moreover, slightly higher crystallization enthalpies could be found for this lot. So again also in this test the lots of PE100.2 were different in their behavior. The results reflect the different concentrations of butane and therewith the different concentrations of side chains in the three lots [30 – 44].

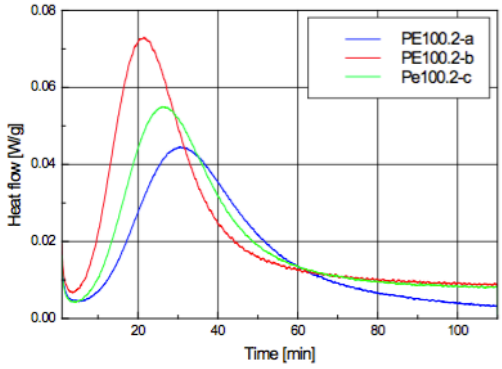

Fig. 8 Isothermal crystallization behavior of the PE100.2 lots at 124 °C.

In Fig. 9 the results of the fatigue tests with the CRB specimens for the different PE100.2 lots are shown. In accordance with the expectations from the molecular and morphological properties PE100.2-a (highest molecular mass and highest concentration of short chain branches) gives the highest failure times and crack initiation times followed by PE100.2-b and PE100.2-c, that show only minor differences. Comparing this ranking with the results from MFR and density measurements, the findings in others, could be proven, that a better crack growth resistance with- in different lots of one grade is accompanied by lower MFR and density values [45 – 59].

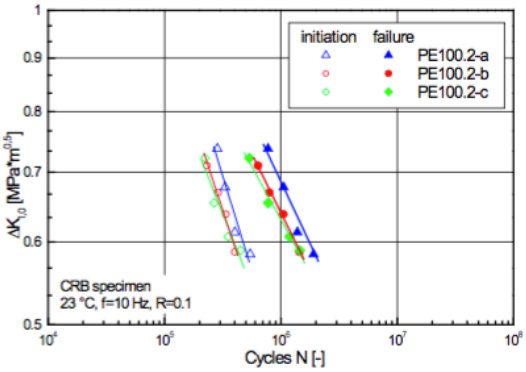

Fig. 9 Ranking of the PE100.2 lots based on FCG experiments with CRB specimens.

In Fig. 10 the results of the impact tests with Charpy specimens are depicted. The fracture surfaces of the three different lots show the same typical features of RCP like PE100.2 in Fig.5. The total fracture energy decreases from PE100.2-a to PE100.2-c, so that also for lot to lot differences ranking with regard to RCP was similar to FCG [60 – 76].

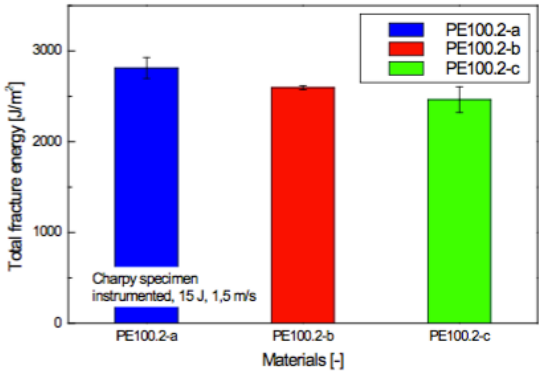

Fig. 10 Ranking of the PE100.2 lots based on instrumented impact tests with Charpy specimens at 1.5 m/s.

4.0 CONCLUSION

Differences in the slow crack growth resistance were measured for three PE 100 pipe materials and three lots of one grade under fatigue loads. The same ranking was found in fracture mechanics impact tests. The ranking could be clearly explained by a systematic investigation of the molecular and morphological parameters of the materials using infrared spectroscopy, size exclusion chromatography, rheological experiments, melt flow rate and density measurements. Consistent with common knowledge molecular mass, concentration and length of short chain branches are the key parameters in determining the crack growth resistance of PE-HD pipe grades. Concerning lot- to-lot variations, in particular, the ranking in the fatigue tests correlated with the ranking in crystallization kinetics, melt flow rate and density measurements. Although especially the latter two characterization techniques are quick and inexpensive tests usually used in quality assurance they can give a picture of the long term performance of lots of one grade when done thoroughly.

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