RANKING OF PE-HD PIPE GRADES BY FATIGUE CRACK GROWTH PERFORMANCE

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ABSTRACT

It is well known that resistance against slow crack growth is important for the lifetime of pressurized polyethylene (PE) pipes. Thus several methods have been proposed to evaluate the long-term performance of PE using fracture mechanics. It is generally believed that this leads to results more quickly compared to internal pressure tests. In the presented research work a method is introduced which uses fatigue loading of cracked round bar (CRB) specimens, to characterize crack growth resistance. It is shown that in the brittle failure regime typical fibrillated fracture surfaces where present and that the failure time gives an indication on the slow crack growth performance of PE pipe materials. The method was applied to several commercially available PE pipe materials and a ranking was established. In addition some crack growth tests were performed at 80 °C; it turned out that although some materials behave almost similar at room temperature, there is a considerable difference at 80 °C.

INTRODUCTION

Pressurized polyethylene (PE) pipes have been used successfully for about 50 years, primarily in fuel gas and water supply systems. Consequently substantial experience concerning the failure behavior and the fitness for purpose of PE piping systems are available. Among other things, it is well known that crack initiation followed by creep crack growth (CCG) is the most important long-term failure mechanism, which is reflected by numerous publications dealing with this topic (1-6). Moreover current discussions about trench-less installations techniques and point loads emphasize the importance of SCG for the long term performance of PE piping systems (7, 8).

Internal pressure tests on pipe specimens are the traditional way to determine the long-term properties of PE pipes. Unfortunately this method is expensive and very time-consuming, especially when information on the CCG behavior is needed (3). Due to this, strong efforts have been put into the development of fracture mechanics tests, which are able to simulate the CCG behavior of pipes in a laboratory test. Thus making it possible to obtain information on the long-term behavior of PE pipes within a reasonable time-frame. CCG tests evaluated according to Linear Elastic Fracture Mechanics (LEFM), the Full Notch Creep Test (FNCT), the Pennsylvania Notch Test (PENT), the Notched Pipe Test (NPT) and the Cone Test, among others, were introduced and are widely used throughout the industry as well as the scientific community (5, 6, 9-15).

Parallel to the development of these methods the influence of the molecular structure on crack growth was investigated. The average molecular mass, the distribution of the molecular mass, the type of short chain branches and in particular the distribution of the short chain branches were identified as the most important parameters (16-18). Specific improvements in the polymerization process of PE followed and the development of third generation resins in particular has led to outstanding CCG resistance. This was achieved by a bimodal distribution of the molecular mass and the specific placement of the short chain branches on the high molecular mass fraction. Even with the above mentioned fracture mechanics methods testing of these materials exceeds practicable time-frames. Consequently quicker test methods are needed to evaluate the long-term behavior of modern PE pipe materials.

One possibility to propagate cracks even in high performance PE pipe materials by pure mechanical means, is to apply cyclic loads and characterize the fatigue crack growth (FCG) behavior. Despite the differences in loading conditions, investigations on PE showed, that the micro-mechanism of crack growth have at least qualitative similarity and rankings based on FCG generally correlate well with those obtained for CCG (1, 6, 15, 19-24). Furthermore, it is possible to predict CCG from FCG experiments by extrapolation (21, 22, 25) and currently major efforts are directed towards calculating the lifetime of PE piping systems based on FCG test results (26).

Already 15 years ago first reports about FCG tests on round circumferentially notched bar (CRB) specimens were published in Japan (24, 27); later some work was also done in Europe (19, 28, 29). In these studies the failure behavior and failure mechanism were studied under different testing parameters and it was concluded that FCG tests on CRB are able to characterize the crack growth resistance of PE. Advantages of this type of specimen are its simple geometry which can be manufactured from compression molded plates as well as pipes easily. Moreover a very well defined plane strain condition prevails along the whole notch and the stress in the remaining ligament is of equal height as in pressurized pipes.

Although several papers have been published, hardly any information is available wether FCG test using CRB specimens are capable of establishing a ranking with respect to the crack growth resistance of PE pipe materials. Thus in this work - after preliminary tests on one PE 80 and one PE 100 material, including analysis of fracture surfaces as well as the initiation period - a methodology was proposed to characterize the crack growth resistance of these materials efficiently. Afterwards a ranking of several commercially available PE pipe materials was established and compared with result from the FNCT, which is widely used in Europe.

EXPERIMENTAL

Materials

All investigations in this study were performed on commercially available PE-HD pipe materials with a minimum required strength (MRS) of 8 MPa (MRS 8 or PE 80) and 10 MPa (MRS 10 or PE 100), respectively. In addition also a PE material cross-linked by peroxide (PEX-G) was included in this investigation. Some characteristic material properties are summarized in Table 1.

With the exception of PEX-G (pipe: 160 x 14,6 mm) from all materials compression-molded plates with a thickness of 10 mm and 15 mm were manufactured. Subsequently FNCT specimens (10 x 10 x 100 mm) were milled from the plates and notched by pressing fresh razor blades to a depth of 1,6 mm into each side of the specimen. Furthermore CRB specimens (D = 14 mm; L = 100 mm) were turned from the plates and in case of of PEX-G from the pipe, respectively; a notch with a depth of 1,5 mm was induced by razor blades.

Table 1: Properties of the investigated materials from data sheets (ρ: density; MFR: melt flow rate; M_n and M_w: number and weight average molecular mass; SCB: number of short chain branches; E: Young's modulus; σ_y: yield stress; MRS: minimum required strength).

Material-Code	Color	ρ [g/cm³]	MFR (190 °C/5 kg) [g/10min]	M_n [kg/mol]	M_w [kg/mol]	SCB [1/1000 C]	Comonomer	E [N/mm²]	σ_Y [N/mm²]	MRS [MPa]
PE80-A	black	0,955	0,50	16	290	4	hexene	1000	22	9,0
PE80-B	black	0,948	0,92	15	190	5,5	hexene	700	18	8,7
PE80-C	yellow	0,940	0,92	15	190	5,5	hexene	700	18	8,3
PE100-D	black	0,960	0,25	8	365	3,8	butene	1100	25	10,8
PE100-E	black	0,959	0,29	14	261	2,5	hexene	1100	26	10,8
PE100-F	black	0,959	0,45	7,5	230	-	butene	1400	26	10,5
PEX-G *	yellow	0,958	-	-	-	-	butene	-	-	-
PE80-H	black	0,953	0,80	9	200	3 - 5	butene	900	22	9,7
PE80-I	black	0,956	0,50	9,5	225	3 - 5	butene	1000	23	9,8
PE100-J	black	0,957	0,30	10	250	3 - 5	butene	1050	24	10,8

* properties of the compound befor cross-linking

Full Notch Creep Test

The FNCT can be described as a tensile constant load test, measuring the failure time of notched specimens (10 x 10 x 100 mm) at elevated temperatures in a surface active environment (s. Fig. 1). In this investigation the experiments were carried out according to ISO 16770 (13) at 80 °C with 2 wt-% Arkopal N110 in deionised water. A test apparatus designed by the Polymer Competence Center Leoben GmbH was used. After the test the exact ligament area of every specimen was determined using a stereo-microscope (Wild - M3Z, Switzerland) and taking into account the applied load, the stress in the ligament area was calculated. For each material the failure time, t_f, was recorded at several different stress levels. For data presentation the failure times were plotted versus ligament stress, σ_0, in a double logarithmic diagram.

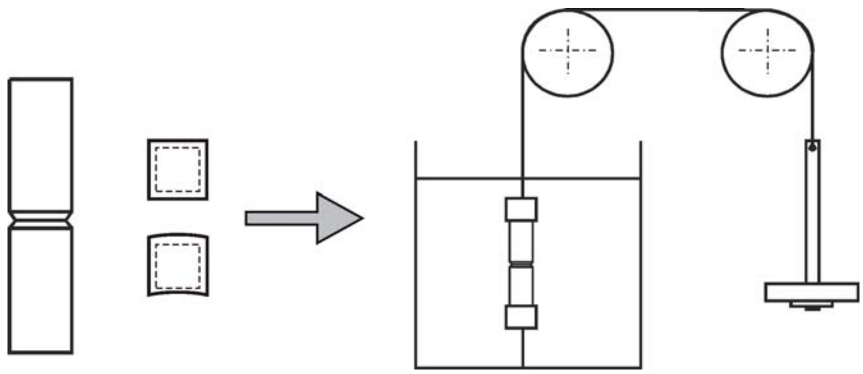

Fig. 1: FNCT specimen and schematic drawing of test setup.

Fatigue Crack Growth Experiments

FCG experiments on CRB-specimens were performed using a servo-hydraulic closed-loop testing machine (MTS 858 Table Top, MTS Systems GmbH, Berlin, Germany); a sinusoidal load at a frequency of 5 Hz and a ratio of minimum load F_{min} to maximum load F_{max} of 0,1 was used (s. Fig. 2a). The tests were conducted at 23 °C as well as 80 °C. The experiments were evaluated using LEBM (30, 31). Failure times, t_f, at different initial stress intensity factor ranges, $\Delta K_{I,0}$, (index I stands for opening mode or pure tensile loading conditions) were measured. ΔK_I was calculated for the CRB specimens according to the equation from Benthem and Koiter (32, 33):

$$\Delta K_I = \frac{\Delta F}{\pi \cdot b^2} \cdot \sqrt{\frac{\pi \cdot a \cdot b}{R}} \cdot f\left(\frac{b}{R}\right) \qquad (1)$$

$$f\left(\frac{b}{R}\right) = \frac{1}{2} \cdot \left(1 + \frac{1}{2} \cdot \left(\frac{b}{R}\right) + \frac{3}{8} \cdot \left(\frac{b}{R}\right)^2 - 0{,}363 \cdot \left(\frac{b}{R}\right)^3 + 0{,}731 \cdot \left(\frac{b}{R}\right)^4\right) \qquad (2)$$

where ΔF is the difference of F_{max} and F_{min}, R is the radius of the specimen, b the radius of the remaining ligament and a the crack length (s. Fig. 2b).

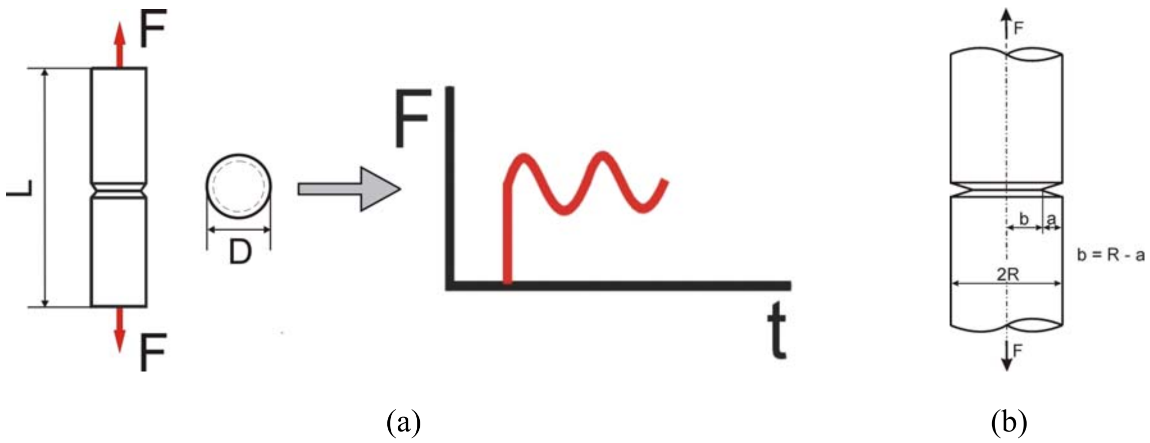

Fig. 2: (a) CRB specimen and fatigue loading conditions and (b) CRB specimen with variables used in Equations 1 and 2.

Crack lengths were monitored during the tests with the aid of a travelling microscope equipped with a linear variable transducer for displacement measurements. Furthermore the crack opening displacement, COD, was recorded using a strain gage. The results were presented in double logarithmic diagrams where t_f was plotted vs. $\Delta K_{I,0}$. Additionally the stress in the ligament of the specimens at the start of the test, σ_0, is given in these diagrams.

RESULTS AND DISCUSSION

Test Method Development for Fatigue Crack Growth Test

At the beginning of the investigations FCG experiments were performed on PE80-A and PE100-D in order to develop and optimize the test methodology. As expected PE100-D showed longer failure times compared to PE80-A. At high loads ductile failure was observed

(s. Fig. 3). Below a certain level the failure mechanism changed and in the second part of the diagram crack initiation followed by crack growth led to the failure of the specimens. Similar to other creep rupture tests, in each regime the failure times were located on straight lines in a double-logarithmic diagram. Furthermore it can be seen in Fig. 3, that the FCG tests in CRB specimens have a high reproducibility; the scatter of the results was smaller than ±15 %.

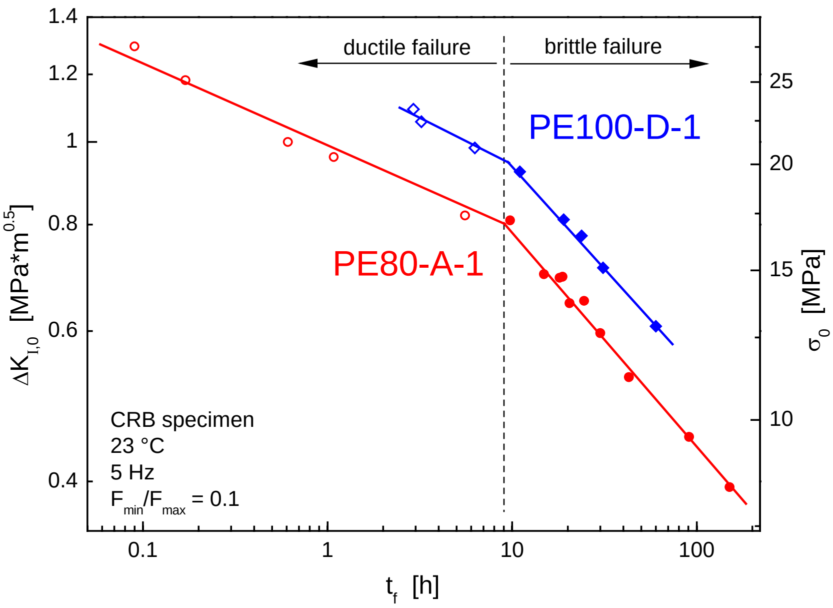

Fig. 3: Fatigue crack growth behavior of PE80-A compared with PE100-D.

The ductile/brittle-transition for PE80-A was around $\Delta K_{I,0} \approx 0.8$ MPa*m$^{0.5}$ and for PE100-D approximately $\Delta K_{I,0} \approx 0.95$ MPa*m$^{0.5}$. Later it turned out that these were typical values for all investigated PE 80 and PE 100 materials, respectively. Typical fracture surfaces for both failure modes are presented in Fig. 4.

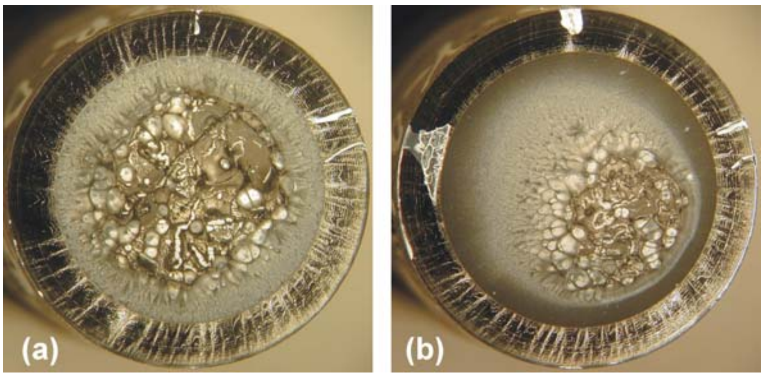

Fig. 4: Fracture surfaces of PE100-D after fatigue loading of CRB specimens (23 °C, 5 Hz, $F_{min}/F_{max} = 0.1$): (a) ductile failure at $\Delta K_{I,0} = 1.09$ MPa*m$^{0.5}$, (b) brittle failure due to crack initiation and crack growth at $\Delta K_{I,0} = 0.61$ MPa*m$^{0.5}$

The investigations using optical microscopy were supported by SEM analyses of fracture surfaces. When brittle failure due to crack growth occurred, a typically fibrillated fracture surface with micro voids, similar to failures in real pipes, was found (s. Fig. 5b and 5c).

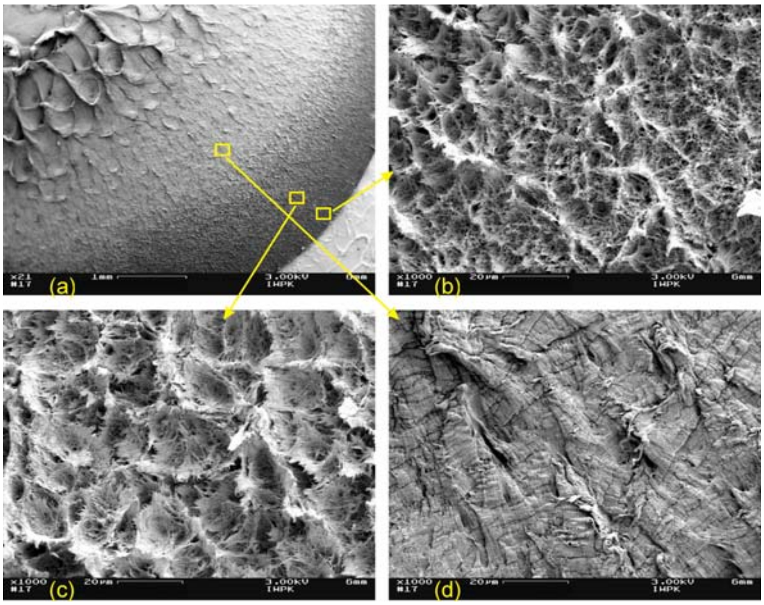

Fig. 5: REM investigation of brittle fracture surface of a CRB specimen (PE100-D; $\Delta K_{I,0}$ = 0.61 MPa*m$^{0.5}$, 23 °C, 5 Hz, F_{min}/F_{max} = 0.1): (a) overview, (b) 0.1 mm after crack initiation (ΔK_I = 0.62 MPa*m$^{0.5}$), (c) 0.5 mm after crack initiation (ΔK_I = 0.68 MPa*m$^{0.5}$) and (d) 2 mm after crack initiation (ΔK_I = 0.93 MPa*m$^{0.5}$).

As already mentioned, in addition to the failure times COD was evaluated for several tests. In Fig. 6, by way of example, COD_{max} as well as ΔCOD (= COD_{max} - COD_{min}) is plotted versus the number of cycles for one test. Typically the first step in the curve marks the point of crack initiation (neglecting the creep component of COD_{max} at the beginning of the test). Furthermore the curve shows, that after the initiation a step wise crack propagation mechanism prevailed, until finally fracture occurred within a few cycles.

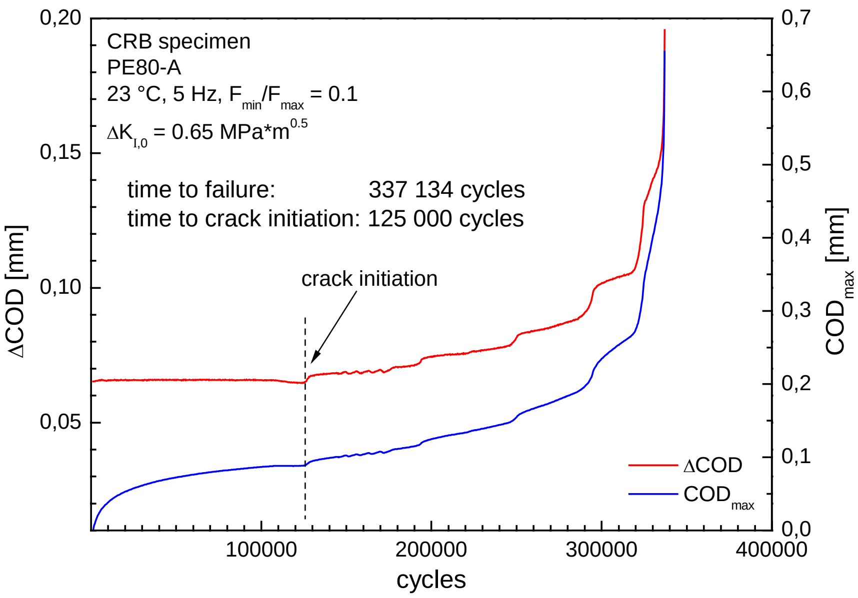

Fig. 6: ΔCOD and COD_{max} of a CRB specimen (PE80-A, 23 °C, F_{min}/F_{max} = 0.1, ΔK_I = 0.93 MPa*m$^{0.5}$).

Evaluating the initiation times, t_i, as a function of $\Delta K_{I,0}$ it was found, that in a double logarithmic diagram, similar to the failure times, they are located on a straight line (s. Fig. 7). However, the slope of the regression line was higher compared to the failure times. This can be explained by the fact, that at high loads, near the ductile/brittle-transition, soon after the crack initiation final ductile failure occurs. Whereas at lower loads crack growth can be observed for a longer period before ductile fracture takes place.

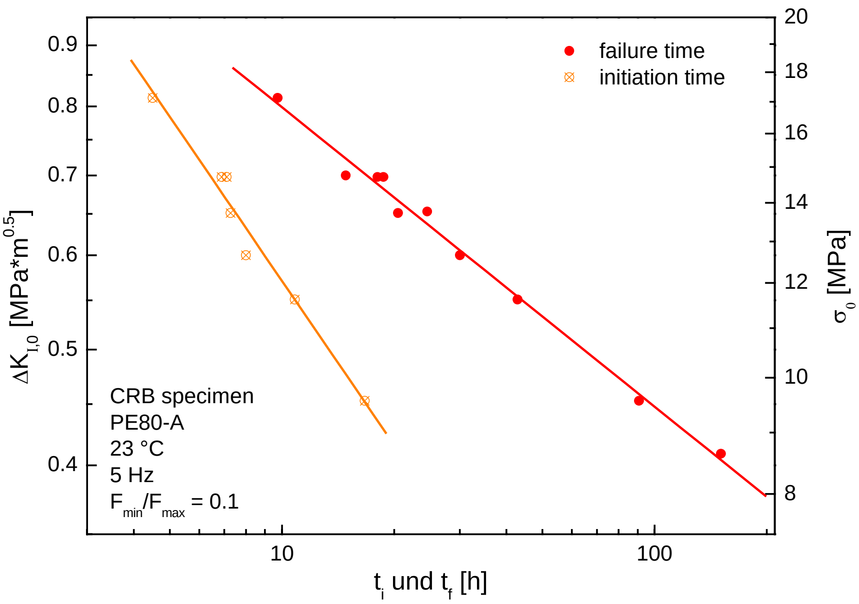

Fig. 7: Crack initiation and failure times of PE80-A at different initial loads.

The determination of crack initiation times is not always as straight forward as pictured in Fig. 6 and was sometimes connected with uncertainties resulting in a scatter of at least ±25 %. For example when, as a result of asymmetric crack growth, crack initiation did not happen at the position of the strain gage, the time for crack initiation could not be identified from COD data. Due to this reasons crack initiation data were not used for material comparison in this work. For future research it is planed to implement a method for detailed acquisition of COD values all around the notch with at least 3 strain gages. This should make it possible to identify crack initiation more reliable.

Based on these preliminary tests a method was established, that can be used to compare different PE pipe materials with regard to their crack growth behavior. It was suggested to test at least 3 specimens of each material and to compare the failure times at different stress levels in the range of 0.55 to 0.75 MPa*m$^{0.5}$ for PE 80 and 0.65 to 0.85 MPa*m$^{0.5}$ for PE 100 (this corresponds to a ligament stress σ_0 of 11.6 to 17.9 MPa). On the one hand these initial loads guarantee brittle failure by crack growth and on the other hand offer the possibility to characterize the FCG behavior of PE pipe materials within 3-5 days.

Ranking of materials using fatigue loaded crack round bars

Following the preliminary tests a ranking of 10 different materials was established. In Fig. 8 $\Delta K_{I,0}$ as well as σ_0 is plotted versus the failure time. As expected the cross-linked PE type had the best FCG resistance, followed by PE 100 which was superior to PE 80. Also within the PE 80 and especially within the PE 100 materials it was possible to distinguish between the different grades.

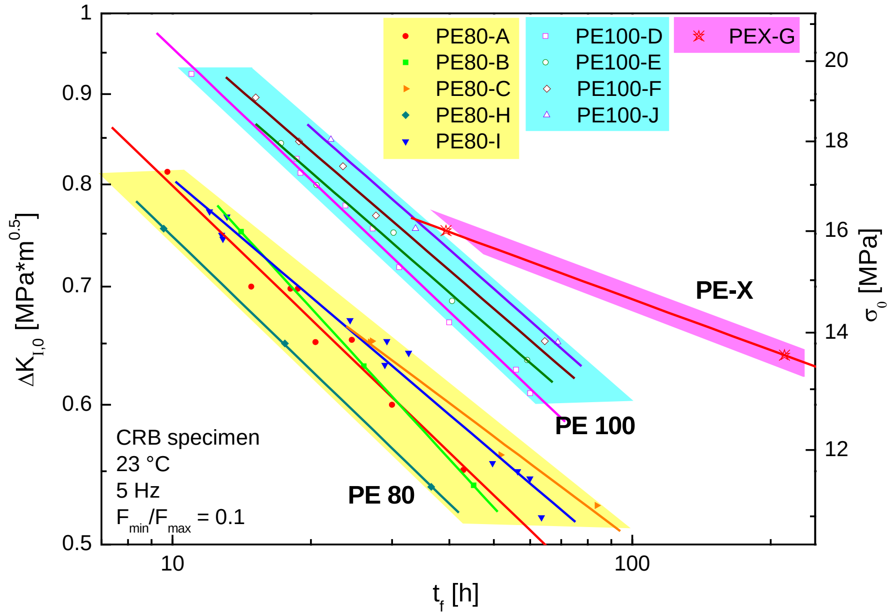

Fig. 8: FCG failure times of the investigated materials at various levels of $\Delta K_{I,0}$.

Comparison of fatigue crack growth with FNCT

Although the discussion about the FNCT is rather controversial, it gives an indication of the CCG behavior, when applied correctly (10, 34). In the presented research work, for 6 materials comparative values were generated to provide a ranking (s. Fig. 9). The same ranking as with the FCG experiments on CRB specimens was found (s. Fig. 8 and 9).

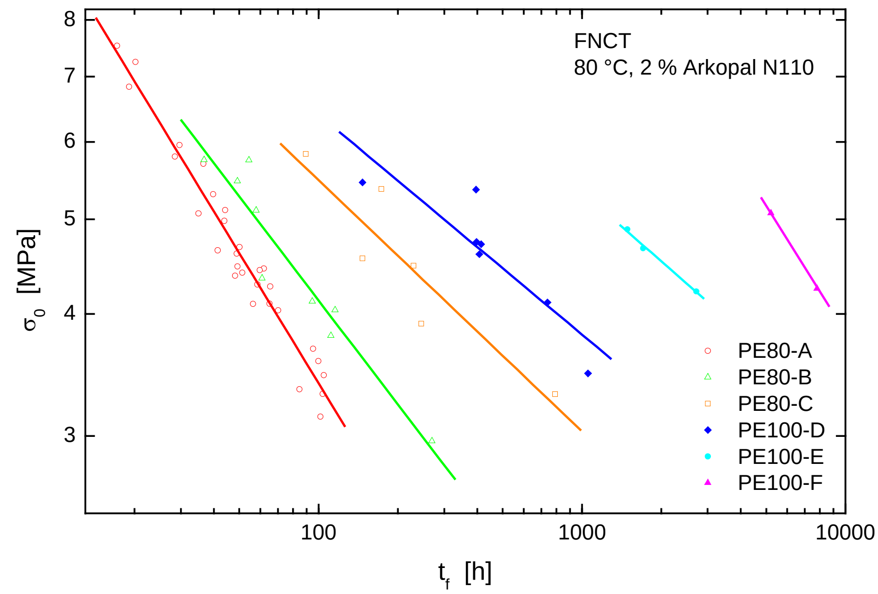

Fig. 9: FNCT failure times of the 6 investigated materials.

Nevertheless, it was obvious that the differences between the materials are a lot smaller in the fatigue experiments compared to the FNCT. On the one hand this might be caused by the different loading conditions, and on the other hand it is also possible that the differences in crack growth behavior between the materials are smaller at room temperature (CRB cyclic test) than at 80 °C (FNCT).

To get further information on this topic, FCG tests at 80 °C were conducted for PE80-A, PE80-B and PE80-C. At initial loads higher than 0.3 MPa*m$^{0.5}$ all three materials showed ductile failure within one hour. At $\Delta K_{I,0}$ values below 0.3 MPa*m$^{0.5}$ slow crack growth was observed, however, testing times became rather long. The results are presented in Fig. 10 and show that although the materials behave almost similar at room temperature, there is a considerable difference at 80 °C. This difference had the same ratio as it was found in the FNCT. About the reason for this finding, it can only be speculated at this point. Maybe the relaxation of internal stresses as well as post crystallization at 80 °C may effect the results (18, 22, 35, 36). Moreover it is possible that the at 80 °C the activation energies for fibril rupture or the building of plastic zones change.

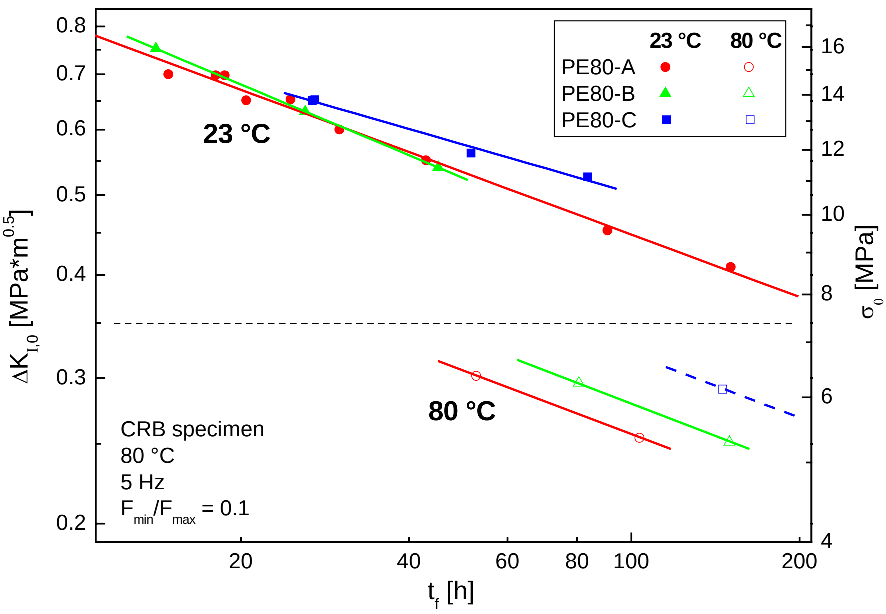

Fig. 10: Comparison of FCG results at 23 °C with results at 80 °C.

In the literature evidence can be found that not in any case material behavior at 23 °C can be predicted from tests at 80 °C using linear extrapolation. For example Ifwarson (3) showed that Arrhenius lines are not parallel for all PE-materials and moreover are not always straight lines, thus making different rankings at 23 °C and 80 °C conceivable. At the moment material development and optimization is based on tests at 80 °C and it is believed that their performance at 80 °C can be transferred to 23 °C and service temperature, respectively. The results presented here, however, suggest that crack growth behavior at 23 °C can not implicitly be predicted by tests at 80 °C.

Conclusions

The results of this investigation show that it is possible to use FCG experiments on CRB specimens to compare materials concerning there crack growth behavior. It was shown that a similar crack growth mechanism as in real pipes prevailed and the ranking of different materials correlated well with existing methods (e.g. FNCT). The biggest advantage of the FCG experiments is that it is possible to reduce testing times considerably so that even modern PE pipe materials can be characterized within a few days at room temperature.

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REFERENCES

1. M.B. Barker, J.A. Bowman and M. Bevis, *Journal of Materials Science*, 1983, 18 1095-1118
2. R.W. Lang, A. Stern and G. Doerner, *Die angewandte makromolekulare Chemie*, 1997, 247 131-137
3. M. Ifwarson, *Kunststoffe*, 1989, 79 (6), 525-529
4. K. Sehanobish, A. Moet, A. Chudnovsky and P.P. Petro, *Journal of Materials Science Letters*, 1985, 4 890-894
5. X. Lu and N. Brown, *Polymer Testing*, 1992, 11 309-319
6. C. Grein, C.J.G. Plummer, H.H. Kausch, Y. Germain and P. Béguelin, *Polymer*, 2002, 43 3279-3293
7. J. Hessel, *3R international - Special Plastics Pipes*, 2001, 40 4-12
8. D. Gueugnaut, F. Berthier, A. Darut and A. Constantinescù, *in proc. Plastics Pipes XII*, Baveno, Italien, 2004
9. W.L. Bradley, J.B. Slay, R.A. Self, D. Register and M. Lamborn, *in proc. Plastics Pipes X*, Göteborg, Schweden, 1998
10. M. Haager, G. Pinter and R.W. Lang, *in proc. ANTEC 2004*, Society of Plastics Engineers, Chicago, Illinois, USA, 2004
11. ASTM F 1473, 1997, Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resins
12. ISO/FDIS 13480, 1997, Polyethylene Pipes - Resistance to Slow Crack Growth - Cone Test Method
13. ISO 16770, 2004, Plastics - Determination of Environmental Stress Cracking (ESC) on Polyethylene (PE) - Full Notch Creep Test (FNCT)
14. ASTM F 1474, 1993, Standart Test Methode for Slow Crack Growth Resistance of Notched Polyethylene Plastic Pipe.
15. G. Pinter, M. Haager, W. Balika and R.W. Lang, *Plastics Rubber and Composites*, 2005, 34 (1), 25-33
16. R.K. Krishnaswamy and Q. Yang, *in proc. ANTEC 2005*, Society of Plastics Engineers, Boston, Massachusetts, USA, 2005
17. V. Stephenne, D. Daoust, G. Debras, M. Dupire, R. Legras and J. Michel, *Journal of Applied Polymer Science*, 2001, 82 916-928
18. N. Brown, X. Lu, Y. Huang, I.P. Harrison and N. Ishikawa, *Plastics, Rubber and Composites Processing and Applications*, 1992, 17 (4), 255-258
19. W. Balika, G. Pinter, B. Choi and R.W. Lang, *in proc. ANTEC 2004*, Society of Plastics Engineers, Chicago, Illinois, USA, 2004
20. M. Haager, W. Zhou, G. Pinter and A. Chudnovsky, *in proc. ANTEC 2005*, Society of Plastics Engineers, Boston, Massachusetts, USA, 2005
21. M. Parsons, E.V. Stepanov, A. Hiltner and E. Baer, *Journal of Materials Science*, 2000, 35 2659-2674
22. F. Van der Grinten and P.W.M. Wichers-Schreur, *Plastics Rubber and Composites Processing and Applications*, 1996, 25 (6), 294-298

23. G. Pinter, W. Balika and R.W. Lang, *Temperature-Fatigue Interaction* (L. Remy and J. Petit, ed.), Elsevier Science Ltd. and ESIS, 2002, 267-275
24. H. Nishimura and I. Narisawa, *Polymer Engineering and Science*, 1991, 31 (6), 399-403
25. W. Balika and R.W. Lang, *in proc. Plastics Pipes XI*, München, Deutschland, 2001
26. R.W. Lang, G. Pinter, W. Balika and M. Haager, *in proc. Plastics Pipes XIII*, Washington DC, USA, 2006
27. H. Nishimura, A. Nakashiba, M. Nakakura and K. Sasai, *Polymer Engineering and Science*, 1993, 33 (14), 895-900
28. C.J.G. Plummer, A. Goldberg and A. Ghanem, *Fracture of Polymers, Composites and Adhesives II* (B.R.K. Blackman, A. Pavan and J.G. Williams, ed.), Elsevier Ltd. and ESIS, 2003, 3-14
29. V. Favier, T. Giroud, E. Strijko, J.M. Hiver, C. G'Sell, S. Hellinckx and A. Goldberg, *Polymer*, 2002, 43 1375-1382
30. A.J. Kinloch and R.J. Young, *Fracture Behaviour of Polymers*, Applied Science Publ., 1983
31. T.L. Anderson, *Fracture Mechanics - Fundamentals and Application*, CRC Press Inc., 1991
32. M. Scibetta, R. Chaouadi and E. Van Walle, *International Journal of Fracture*, 2000, 104 145-168
33. J.P. Benthem and W.T. Koiter, *Method of Analysis and Solutions of Crack Problems* (G.C. Sih, ed.), Noordhoft International Publisching, 1973, 131-178
34. F.L. Scholten, D. Gueugnaut and F. Berther, *in proc. Plastics Pipes XI*, Munich, Germany, 2001
35. S. Choi and L.J. Broutman, *Polymer(Korea)*, 1997, 21 (1), 93-102
36. R.K. Krishnaswamy, *Polymer*, 2005, 46 (25), 11664-11672