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# POLYMER TESTING

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# Prediction of the remaining lifetime of polyethylene pipes after up to 30 years in use

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## ARTICLE INFO

## ABSTRACT

Plastics pipes made of polyethylene (PE) play an outstanding role in gas and water supply. While for modern pipe grades typical lifetimes of 50 years are taken for granted and service times of 100 years are discussed, pipes made of PE with a lower performance have been used for decades. As the repair and rehabilitation of existing pipe systems involve immense costs, the question of their qualitative condition has to be considered. In this paper, four different pipes used in the gas and water distribution in Austria with an age up to 30 years have been investigated. After a morphological and mechanical study, particular attention was paid to material stabilization, which is essential for long-term applications. Fracture mechanics tools have been used to gain information on the resistance to crack initiation and slow crack growth. Furthermore, a fracture mechanics extrapolation procedure has been applied to predict the remaining lifetime of the pipes. The results have indicated that all the pipes investigated are still in a very good condition and are likely to be sufficiently safe to remain in use.

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## 1. Introduction

Pressurized plastics pipes have been used successfully for several decades and, especially, pipe systems made of polyethylene (PE) are widely used in fuel gas and water supply as well as in sewage systems [1–3]. In 2007, the worldwide demand for pipes made of polyethylene with high density (PE–HD) was about 3.7 million tons and a growth rate of six percent per year up to 4.9 million tons is expected in 2012 [4]. Based on results from internal pressure tests, the standard extrapolation method described in EN ISO 9080 [5] classifies these pipe grades by their minimum required strength (MRS) to ensure service times of at least 50 years. Nowadays, modern materials with the classification PE 100 (MRS = 10 MPa) and above are available. Knowledge of the long-term failure behavior

is essential for the lifetime and safety assessment of these pipes, and the commonly accepted failure mechanisms thereof are characterized by crack initiation and slow crack growth (SCG) [1,6–10]. To gather information on the resistance to these failure mechanisms, modern methods of linear elastic fracture mechanics (LEFM) are used.

In long-term applications of plastics pipe systems, the ageing behavior of the material has to be considered as well. Physical or chemical ageing may change material properties by influencing the morphology (crystallinity) and molecular mass (crosslinking or degradation), both of which certainly have an effect on the mechanical properties of the pipes and on the resistance to crack initiation and SCG.

Modern PE pipe grades seem to guarantee a lifetime of 50 years and more. However, pipes made of PE grades with lower performance have been used for gas and water transportation for decades and, as the repair and rehabilitation of buried pipes is associated with high technical effort and immense costs, the reliability of these pipe systems is of substantial interest. Unfortunately, there are

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only a few scientific publications which focus on the investigation of the remaining lifetime of used pipe systems [11–13]. The objective of this paper is to conduct an extensive study of four different gas and water supply pipes which have been in use for up to 30 years in Austria with an emphasis on a systematic investigation of the structure, morphology and stabilization of the pipe materials. Furthermore, fracture mechanics tests have been performed in order to evaluate the resistance to crack initiation and SCG. Based on an extrapolation procedure by means of cyclic fatigue tests, a fracture mechanics prediction of the remaining lifetime has been made.

## 2. Background

The failure mechanisms of internally pressurized pipes can be divided into three characteristic regions [1] depending on the load level, schematically shown in Fig. 1. Failure region A appears at a relatively high hoop stress $\sigma_{hoop}$ and at relatively short times $t_f$, and is dominated by ductile failure along with large scale plastic deformation. Usually, plastics pipe systems are designed to operate below region A. With a decreasing $\sigma_{hoop}$, a transition knee into the quasi-brittle region B is passed and the failure is characterized by creep crack growth initiation, creep crack growth, and only small scale crack tip plasticity. It is generally accepted that this failure region determines the lifetime of long-term applications [1,6–10]. The brittle failure region C is nearly load-independent and is a result of large scale material ageing and polymer degradation [14].

Crack initiation and slow crack growth heavily depend on the molecular structure and morphology of the material such as the molecular mass, the molecular mass distribution, the positioning, the concentration and the length of short chain branches, and the crystallinity. With improvements in the polymerization process of PE and controlled variation of these material parameters, the raw material suppliers were able to achieve a significant increase in the resistance to crack initiation and slow crack growth [15,16]. During the service time of buried pipes over several decades, changes in the morphology can lead to physical

ageing. However, sufficient and effective material stabilization inhibits molecular degradation and oxidation processes. The concentration and the type of the stabilizer may significantly affect the lifetime of pressurized pipes in the quasi-brittle and in the brittle failure regions. Likewise, the concept of local crack tip ageing can be used to explain crack growth mechanisms depending on different stabilizer systems [17].

To investigate the quasi-brittle failure region B in highly crack-resistant modern PE pipe grades, a number of accelerated laboratory tests using fracture mechanics considerations have been developed; among which are the Notched Pipe Test (NPT), the Pennsylvania Notch Test (PENT) and the Full Notch Creep Test (FNCT) [9,18]. Besides these test methods, fatigue tests with cyclic loads are used for accelerated characterization of the resistance to crack initiation and SCG, and a number of studies prove the correlation of fatigue and SCG in high density PE as well as in medium density PE [19–23]. Especially, fatigue tests with circumferentially cracked round bar (CRB) specimens show a high potential for quick material ranking [24,25]. By measuring the kinetics of fatigue crack growth at different loading ratios, an extrapolation to the kinetics of creep crack growth at static loading is possible [27–30].

The crack growth kinetics da/dt for static loading is usually shown in a double logarithmic diagram as a function of the stress intensity factor (SIF) $K_I$, and often results in a S-shaped relationship (see Fig. 2). The region of stable crack growth can be described by the equation by Paris and Erdogan Paris law, Equation (1) [31], in which A and m are parameters depending on the material, the temperature and the loading conditions.

$$\frac{da}{dt} = A \cdot K_I^m \tag{1}$$

When knowing the crack kinetics under static loading conditions (creep crack growth, CCG) and the material constants of the Paris law, a fracture mechanics lifetime prediction is possible. By integrating Equation (1), the time for the creep crack growth $t_{CCG}$ from an initial starter defect with size $a_{ini}$ to a final defect size $a_f$ can be calculated (Equation (2)). The overall failure time $t_f$ consists of the sum
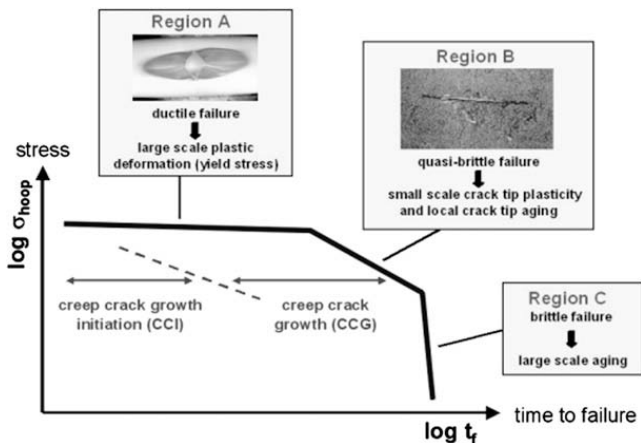


**Fig. 1.** Schematic illustration of the failure behavior of pressurized PE pipes [1].
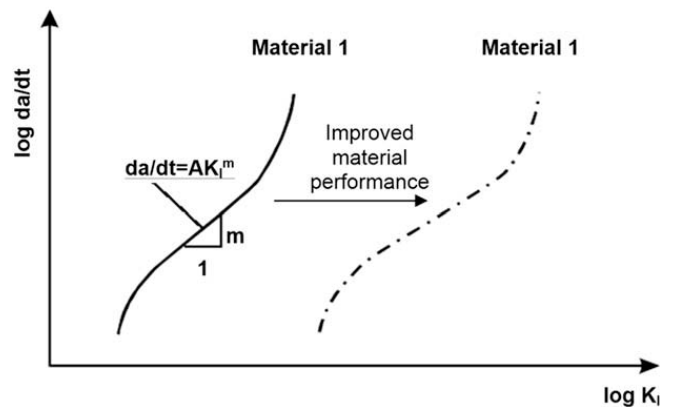


**Fig. 2.** Schematic illustration of the crack growth kinetics da/dt as a function of the stress intensity factor $K_I$ [26].

of the time until the crack initiation $t_{ini}$ and $t_{CCG}$ (Equation (3)). The evaluation of $t_{ini}$ was not part of this study, which means that all calculated lifetime predictions in this paper are on the conservative side as they only take an already growing crack into account.

$$t_{CCG} = \frac{1}{A} \cdot \int_{a_{ini}}^{a_f} \frac{1}{K_I^m} \cdot da \qquad (2)$$

$$t_f = t_{tot} \approx t_{in} + t_{CCG} \qquad (3)$$

## 3. Experimental

Four different pipes with unknown polyethylene pipe grades and of different ages (1976–1988) have been investigated: a black water pipe from 1988 (DN160 SDR11) and three yellow pipes transporting gas from 1987 (DN110 SDR11), 1981 (DN160 SDR11), and 1976 (DN160 SDR17). Since the origin of the materials of the old pipes was unknown, two typically available polyethylene pipe materials with hexene comonomer from the early 1990s, GM5010T2 (Hoechst AG, 1991) for a high density polyethylene (PE 80-HD) and Finathene 3802B (Atofina, 1993) for a medium density polyethylene (PE 80-MD), have been used as a reference in their virgin form. Their density was measured by means of density gradient columns (Davenport, UK) across the entire wall thickness (ISO 1183–2). The melt flow rates MFR 190/5 and MFR 190/21.6 (ISO 1133) were determined with a Zwick 4106/4103 (Zwick, GER). An elemental analysis was conducted by Wavelength Dispersive X-ray Fluorescence (WDXRF) with a Bruker S4 pioneer (Bruker AXS GmbH, GER). The molecular structure was investigated by means of gel permeation chromatography (GPC) with an Alliance GPC 2000 (Waters Corp., USA) with trichlorbenzole as a solvent. Infrared (IR) spectroscopy and Fourier transform-IR-spectroscopy (FTIR) were carried out with a Bruker Vertex 70 (Bruker AXS GmbH, GER).

For the morphological characterization, specimens were taken from inner, middle, and outer positions of the pipe wall with a microtome (Jung, GER). The temperature-dependent storage modulus and the loss factor were determined by means of dynamic mechanical analysis (DMA) with a DMA/SDTA861e (Mettler Toledo GmbH, CH) at a heating rate of 3 K/min in a displacement controlled mode. The results were compared to specimens from a modern PE 100 pipe (Hostalen CRP 100, Basell Polyolefine GmbH). The crystallinity was measured by dynamic scanning calorimetry (DSC) with a DSC822 (Mettler Toledo GmbH, CH) at a heating rate of 10 K/min. The oxidation induction time (OIT) was measured near the inner pipe wall position with a DSC of the type 2920 (TA Instruments, USA). Additives were detected by high performance liquid chromatography (HPLC) with an Agilent Technologies Series 1200 (Agilent Technologies, USA).

For the estimation of residual stresses in the circumferential direction, ring segments of 80 mm in length were prepared. In the axial direction, strips with a thickness of 5 mm and a length of 250 mm were prepared [3,32,33]. To correlate the deformation of these specimens with residual stresses, the creep modulus under bending conditions (ISO 899–2) was measured with a Zwick 010 (Zwick, GER).

The resistance to crack initiation and slow crack growth was determined by full notch creep tests (FNCT) and cyclic fatigue tests on CRB specimens. For the FNCT (ISO 16770), specimens with a cross-section of $10 \times 10$ mm were milled out of the pipes and an initial crack of 1.6 mm was inserted with a razor blade. The tests were performed at a temperature of 80 °C in a medium of 2% aqueous Arkopal N110. The CRB specimens of the old pipes were milled and drilled out with a diameter of 10 mm and a circumferential initial crack of 1 mm in depth was inserted with a razor blade. The cyclic fatigue tests were executed on a servo-hydraulic closed-loop testing system MTS Table Top (MTS Systems GmbH, GER) with sinusoidal loading at a frequency of 10 Hz at 23 °C. All tests were performed at an R-ratio (minimum/maximum load) of 0.1. To compare the results of the FNCT and the fatigue tests of the old pipe materials, two typical pipe grades of those times (compression-molded plates) were also tested: Finathene 3802B (Atofina, 1993) for a medium density polyethylene (PE 80-MD) and Daplen CE4664 (Borealis Polyolefine GmbH) for a high density polyethylene (PE 80).

For the prediction of the remaining lifetime, an extrapolation methodology was applied with a procedure based on material-specific and specimen-specific compliance having been used for the direct measurement of the crack kinetics [24,26–30,34]. To develop a compliance calibration curve, the material-specific compliance ($\Delta C$) was measured with CRB specimens with different initial crack lengths $a_{ini}$ between 0.0 and 4.0 mm. Following the determination of the $\Delta C$ calibration curve, the crack kinetics could be measured in a single CRB fatigue test. For the measurement of $\Delta C$, a larger diameter of the CRB specimens was necessary. Only the walls of the pipes from 1981 and 1988 were thick enough to prepare CRB specimens with a diameter of 13.8 mm. The initial crack length of 1.5 mm was inserted with a razor blade. For the lifetime prediction procedure, the crack kinetics was measured at different $R$-ratios ($R = 0.1$, 0.3, and 0.5) and was afterwards extrapolated to $R = 1.0$, which represents static loading conditions. From this "synthetic" CCG curve, the relevant material constants A and m were obtained. To predict the remaining lifetime of the pipes, the stress intensity factor for a pressurized pipe with an initial defect on the inner pipe surface was chosen from literature [35] and the time for the creep crack growth $t_{CCG}$ was calculated with Equation (2).

## 4. Results and discussion

Table 1 summarizes some basic material properties of the old pipes. Values from virgin PE–HD and PE–MD are also listed in this table as reference values. While for the yellow gas pipes the density was found below 945 kg/m³, the black water pipe from 1988 shows the highest density with 947.5 kg/m³. Compared to the two reference materials, the density of all the pipe materials was lower than the virgin material. The melt flow rates are in a typical range for PE–MD of this time. However, the value for the pipe from 1988 is comparable to PE–HD. The pipe from 1988 has the

**Table 1**
Basic properties of old pipes compared to typical pipe materials.

|  | 1988 | 1976 | 1981 | 1987 | GM5010T2, 1991 | Finathene 3802B, 1993 |
|---|---|---|---|---|---|---|
| Color | Black | Yellow | Yellow | Yellow | Black | Black |
| Pipe dimension | 160SDR11 | 160SDR17 | 160SDR11 | 110SDR11 | Pellets | pellets |
| Density 23 °C [kg/m$^3$] | 947.5 | 944.9 | 940.4 | 942.2 | 954 | 949 |
| MFR 190/5 [g/10 min] | 0.47 | 1.42 | 1.05 | 0.84 | 0.43 | 0.95 |
| MFR 190/21.6 [g/10 min] | 9.21 | 29.37 | 22.3 | 16.9 | 12.7 | 18.2 |
| MFRI 21.6/5 | 20 | 21 | 21 | 20 | 30 | 19 |
| $M_w$ [g/mol] | 190000 | 111800 | 114000 | 185000 | 334000 | 218000 |
| $M_n$ [g/mol] | 14051 | 15900 | 13950 | 10500 | 14000 | 21000 |
| PDI | 13.5 | 7 | 8.2 | 17.7 | 23.6 | 10.6 |

highest weight average molecular mass $M_w$. The molecular mass of the pipes from 1976 and 1981 is very similar, while the values of the pipes from 1988 and 1987 are comparable. However, for all old pipes, $M_w$ is lower than the molecular mass of the reference materials, which could be a result of polymer degradation or crosslinking (crosslinked molecules have not been detected in GPC). The ratio of $M_w$ and the average molecular weight $M_n$ represent the polydispersity index (PDI) as a characteristic indicator of the broadness of the molecular mass distribution. While the pipes from 1976 and 1981 show a relatively narrow distribution, the pipes from 1988 and 1987 have a broader one.

The results of the FTIR in Table 2 show that the molecular structure of the old pipe materials did not significantly differ from the values of the reference materials. All materials could be identified as chromium-based catalyst PE with hexene for the pipe materials from 1988, 1976, and 1981 and butene for the pipe material from 1987 as comonomer. Likewise, the density of short chain branches is within a typical range.

To characterize the thermomechanical properties of the old pipes, DMA was used to investigate the temperature-dependent storage modulus E′ and the damping tanδ. With respect to the processing history, samples near the inner and outer pipe wall surface and the middle of the pipe wall were tested. Fig. 3 shows the results for the youngest pipe from 1988 and the oldest pipe from 1976. In both materials, the specimen from the outer pipe wall position shows a lower E′ across the entire temperature range than specimens from the middle and inner pipe position. This is an effect of the cooling process during the extrusion, due to which the crystallization on the outer pipe surface is retarded by a higher cooling rate. A similar behavior was found in the damping behavior, in which an increase in tanδ from the inner pipe wall position to the outer pipe wall position was noticed. Depending on the pipe wall position, the values of E′ at a temperature of 23 °C in the pipe from 1988 varied between 700 and 950 MPa. In the pipe from 1976, the storage modulus was slightly lower, between 670 and 870 MPa. These values are within a typical range for PE pipe materials and can be compared to the result of a compression-molded plate of a modern PE 80-MD (E′ = 1100 MPa at 23 °C). The results for the old pipes from 1987 and 1981 are very similar to the data discussed, yet for a better illustration their curves have not been included in Fig. 3.

The crystallinity $\alpha$ was investigated with DSC at three different pipe wall positions near the inner and the outer surface and at the middle of the pipe wall. The results in Fig. 4 again show the influence of the cooling of the pipe during processing. For all pipes, the lowest $\alpha$ was found at the outer pipe wall position. With an increasing distance from the cooled surface, the material had more time to crystallize. While for the pipe materials from 1976, 1981, and 1987 $\alpha$ varies between 46 and 51%, the crystallinities for the pipe from 1988 are slightly higher (52–59%). The increase of $\alpha$ from the outer to the inner pipe wall position is in agreement with the results from the DMA, according to which materials with a higher crystallinity result in a higher storage modulus and lower damping.

Residual stresses in plastics materials highly depend on their processing history. The faster the product (e.g. the pipe) has been cooled, the higher are the internally frozen stresses in the component. The residual stresses of the old pipes were measured in two directions, circumferentially and axially. The results summarized in Fig. 5 have been compared to previous experiments [32], in which a typical

**Table 2**
Molecular structure of the old pipes compared to typical pipe materials.

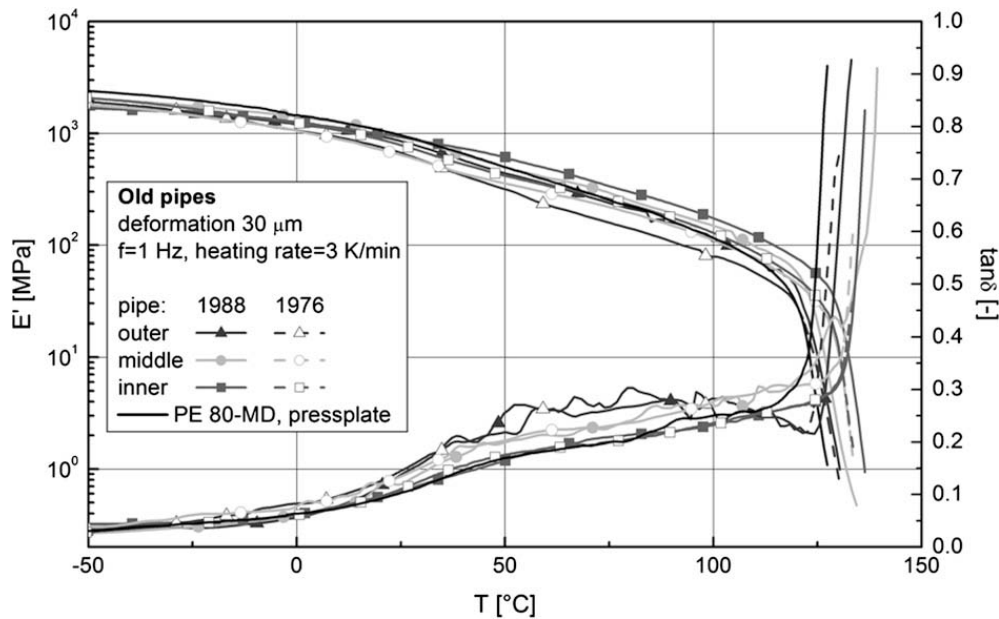|  |  | 1988 | 1976 | 1981 | 1987 | GM5010T2, 1991 | Finathene 3802B, 1993 |
|---|---|---|---|---|---|---|---|
| CH$_3$ | /100 C | 3.8 | 8.5 | 8.1 | 8.6 | 4.3 | 9.1 |
| CO | /100 C | 0 | 0 | 0 | 0 | 0 | 0 |
| —– | /100 C | 0.03 | 0.04 | 0.07 | 0.1 | 0.12 | 0.08 |
| —= | /100 C | 1.02 | 0.9 | 0.98 | 1.19 | 1.6 | 1.04 |
| =< | /100 C | 0.1 | 0.14 | 0.16 | 0.16 | 0.21 | 0.19 |
| Comonomer |  | 1.1% C6 | 3.6% C6 | 3.2% C6 | 1.4% C4 | C6 | C6 |

**Fig. 3.** Storage modulus $E'$ and loss factor tan$\delta$ at different pipe wall positions as a function of the temperature for the pipes from 1988 and 1976 and the compression-molded plate from PE 80–MD.

range of residual stresses in the circumferential direction $\sigma_{r,c}$ was found between approx. 1.9 and 5.0 MPa and in the axial direction $\sigma_{r,a}$ between approx. 1.5 and 6.4 MPa. The amount of residual stresses for all pipes, except for the pipe from 1981, was within this typical range between 3 and 4 MPa. The lowest values with $\sigma_{r,c} = 1.9$ MPa and $\sigma_{r,a} = 1.6$ MPa were found for the pipe from 1981, which may suggest a relatively low cooling rate during the processing. Not enough material had been left from the pipe from 1988 which is why the residual stresses in the axial direction could not be measured.

To study the effectiveness of the material stabilization and the extent of material ageing, the old pipes were investigated with OIT, additive analysis and IR-spectroscopy. Apart from the general stabilizer consumption, it can be expected that stabilizers are eluted especially near the inner pipe wall under the influence of media so that samples near the inner pipe wall position were tested. The OIT's measured

at 210 °C in an oxygen atmosphere (see Table 3) vary between 15 and 36 min. Although the pipes have been in use for up to 30 years, the materials measured still show a distinct resistance of more than 15 min. The findings of the additive analysis summarized in Table 3 complement the results from the OIT, as packages of classical phenolic and phosphitic antioxidants were detected in all materials, even in the oldest pipe from 1976. In the material from 1981, the antioxidants may be included in the unknown peak.

Fig. 6 shows the results of the IR-spectroscopy for samples near the inner pipe wall position of the old pipes and the PE 80-MD reference material, with the transmission $T$ being a function of the wave number $\nu$. As this method is also useful for material identification, typical characteristics of PE could be seen such as hydrocarbon bands at wave numbers of 2925, 2850, and 1470 cm$^{-1}$. Typical ageing products are carbonyl groups with bonds at wave numbers between 1700 and 1740 cm$^{-1}$. The measurements of the old pipes show only small peaks in the range of 1735–1740 cm$^{-1}$, which matches aldehyde and
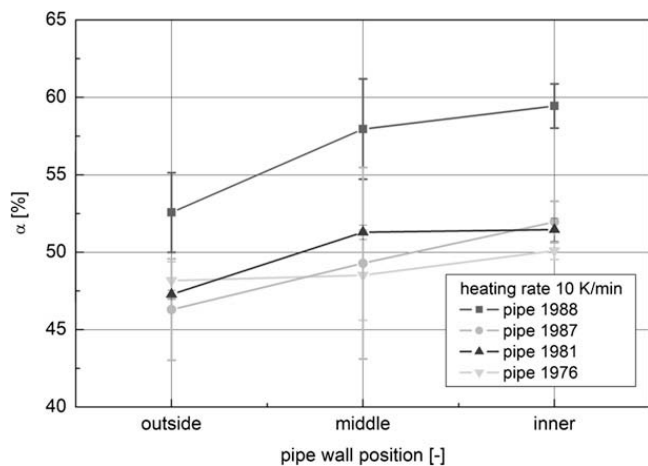


**Fig. 4.** Crystallinity $\alpha$ depending on the pipe wall position for the pipes from 1988, 1987, 1981, and 1976.
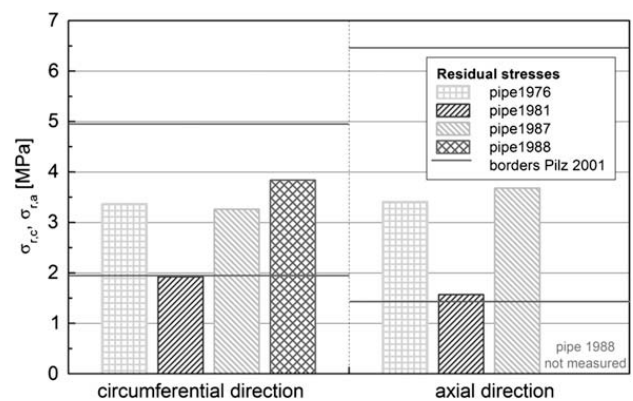


**Fig. 5.** Residual stresses in the circumferential and axial direction for the pipes from 1988, 1987, 1981, and 1976 compared to reference data [32].

**Table 3**
Oxidation–induction times and additive analysis of old pipes.

|  | Values in [ppm] | 1988 | 1987 | 1981 | 1976 |
|---|---|---|---|---|---|
| OIT [mm] |  | 15 | 36 | 15 | 25 |
| Phenol | IRGANOX 1010 | 35 | 730 | – | – |
| Phenol | IRGANOX 1076 | – | 90 | – | – |
| Phenol | IRGANOX 1330 | 350 | – | – | – |
| Phenol | BHT | – | – | – | 85 |
| Phosphite | IRGAFOS 168 | – | 825 | – | – |
| Phosphite | IRGAFOS 168OX | 890 | 285 | – | – |
| Phosphite | ULTRANOX 626 | – | – | – | 150 |
| UV-absorber | Chimasorb 81 | – | – | – | 1790 |
|  | Unknown peak | – | – | 1535 | – |
| UV (HALS) | TINUVIN 770 | – | – | 1290 | – |

ester groups as products of restricted material ageing. The indications for all materials at wave numbers between 1500 and 1700 $cm^{-1}$ and 3000 and 3500 $cm^{-1}$ could be assigned to the stabilizer systems found in the additive analysis.

The results presented so far indicate that the old pipes are still in very good condition. Unfortunately, an exact identification of the origin PE type was not possible. The melt flow rates, the weight average molecular mass $M_W$, and the crystallinities indicate that the materials of the pipes from the years 1976, 1981, and 1987 are most likely to be PE–MD pipe grades, which were widely used for gas pipes at the respective times. The higher density and crystallinity of the pipe from 1988 rather matches PE–HD pipe grades, which are typically used for water pipe applications. All materials show a relatively low $M_W$ compared to the reference materials. The DMA results show that the stiffness and damping behavior of all the old pipes is in the region of virgin polyethylene. The different crystallinities, depending on the pipe wall thickness, indicate that no post-crystallization at the outer pipe wall position has taken place and the residual stresses have obviously not been released over the years either. It can be

concluded from these results that the mechanical material properties of the PE pipes have been almost unaffected. The investigation of the ageing of the old pipes shows that there is only a slight amount of carbonyl groups which indicate ageing. However, distinct OIT values and clearly detectable amounts of stabilizer systems found in the additive analysis ascertain that the pipes are sufficiently stable to stay in service, even after 30 years in use.

Fracture mechanics tests are typically used to rank pipe grades by their resistance to crack initiation and SCG. Fig. 7 shows the results of the FNCT at 80 °C in 2% Arkopal N110 for the old pipes and for a PE 80 reference material (Daplen CE4664), with the failure times $t_f$ being plotted as a function of the stress level $\sigma_0$. The shortest failure times by far, which were lower than 10 h, were found for the oldest pipe from 1976. With decreasing pipe age, $t_f$ also increased correspondingly. The pipes from 1987 and 1988 are nearly the same age; however, $t_f$ for the pipe from 1987 is the highest around $\sigma_0 = 4$ MPa. The time interval between the pipe in the worst and in the best condition is about a decade. In this test, all failure times were shorter than the time of the PE 80 reference material.

A possibility to obtain an accelerated material ranking is to conduct fatigue tests with cyclic loads on CRB specimens. On the one hand, the major advantage of this method is that the tests are performed at an ambient temperature of 23 °C, which is much closer to the application temperature of real pipes than is the case with internal pressure tests, FNCT, or other fracture mechanics tests. On the other hand, fatigue tests on CRB specimens are carried out without any additional stress-cracking liquids. Fig. 8 shows the failure times $t_f$ of the old pipes in the cyclic fatigue tests at $R = 0.1$ at a temperature of 23 °C as a function of the initial stress intensity factor difference $\Delta K_I$ and the initial maximum ligament stress $\sigma_{max,ini}$, respectively. For comparison, the PE 80 material (Daplen CE4664) was tested again as well as the PE 80-MD reference pipe grade. The shortest failure times were determined for the oldest pipe from 1976. The
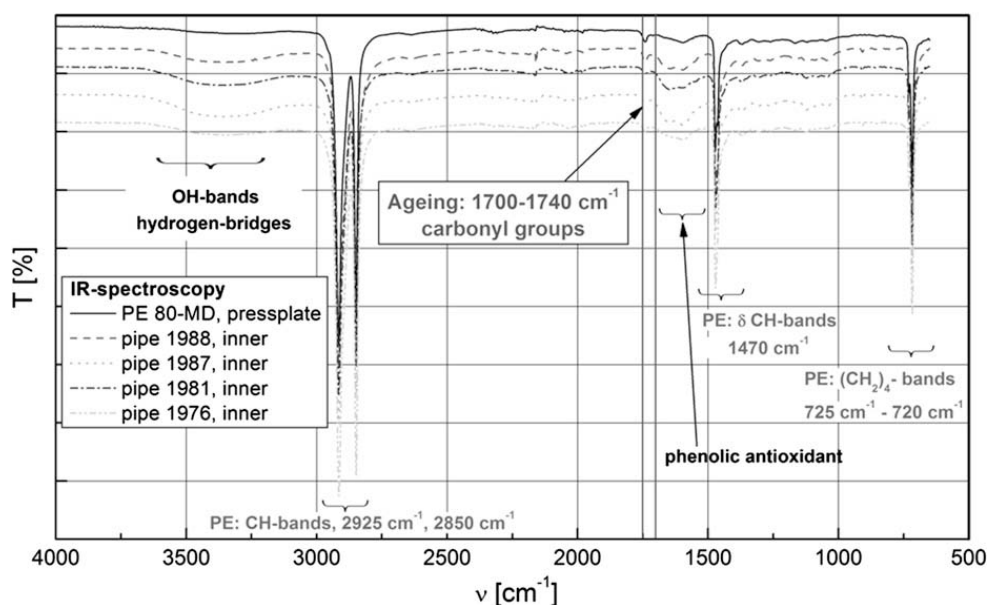


Fig. 6. Infrared spectroscopy for the pipes from 1988, 1987, 1981, 1976, and the compression-molded plate from PE 80–MD.
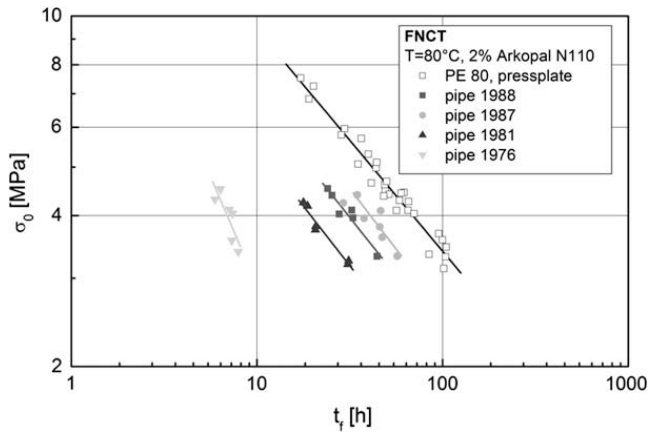
**Fig. 7.** Failure time $t_f$ as a function of the stress level $\sigma_0$ in FNCT for the pipes from 1988, 1987, 1981, and 1976 and the compression-molded plate from PE 80.

failure time $t_f$ increased with decreasing pipe age and at stress levels of $\sigma_{max,ini} = 8$ MPa the youngest pipe from 1988 showed the best performance, what is in contrast to the FNCT results at 4 MPa. In previous studies, fatigue tests on CRB specimens proved to differ from one PE type to another and even within the same pipe grade [24,25]. The results in Fig. 8 show that the best PE–MD pipe from 1987 quite well matches the virgin PE 80-MD reference material. The pipe from 1988, identified as a PE–HD type, even had slightly longer failure times than the PE 80 reference material, which could be attributed to different processing histories. In the GPC measurements, the pipes from 1976 and 1981 were identified as the materials with the lowest molar mass, which is of course the main reason for their reduced crack growth resistance. However, it could also be noticed that the PE–MD materials have a lower gradient than the PE 80 pipe from 1988 and the PE 80 reference material, which implies a crossover of the failure times with decreasing loads, which is an indication of a higher crack growth resistance of PE–MD at low stress levels.

Compared to the FNCT with failure times up to 60 h, the testing times of the cyclic CRB tests between a few hours up to a day emphasize the potential of this approach as a quick ranking method [24,25]. Although the rankings of both methods are comparable, the FNCT is performed at
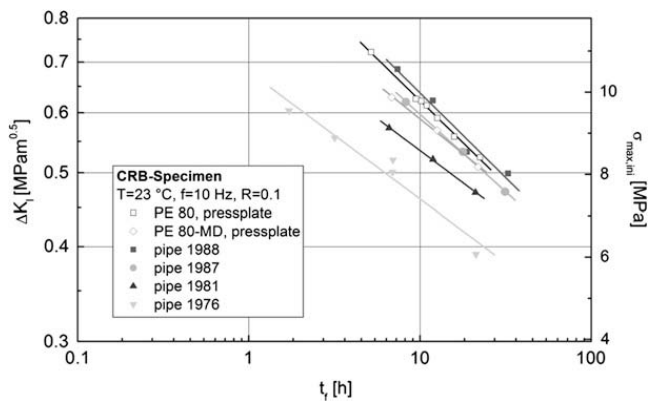
elevated temperatures far beyond the applied service temperatures and the specimens are embedded in an aggressive stress-cracking liquid. Regarding the different rankings of the pipe from 1987 and the pipe from 1988, the question has to be raised as to whether the same micro-mechanics effects are responsible for crack initiation and SCG in the FNCT as in the fatigue tests at 23 °C.

A vital advantage of cyclic CRB tests is the possibility to gather deeper information on crack initiation and crack kinetics. Based on a fracture mechanics extrapolation procedure [27,30,34], a prediction of the creep crack growth (CCG) is possible with cyclic tests at different $R$-ratios. If the crack kinetics under static loading conditions is known, a fracture mechanics lifetime prediction of components is possible. Fig. 9 shows the compliance calibration curve for the materials of the pipes from 1981 and 1988, with the difference of the compliance $\Delta C$ being a function of the crack length $a_c$. For the pipe from 1988, the calibration curve is clearly lower than for the pipe from 1981, which indirectly reflects the higher stiffness of the PE–HD material in the pipe from 1988. For the mathematical fitting of the data, a second order polynomial function was used and then transformed to express the crack length $a_c$ of a single CRB test as a function of $\Delta C$, which enables the determination of the crack kinetics da/dt.

In Fig. 10, the crack kinetics da/dt as a function of the maximum stress intensity factor $K_{I,max}$ for the two pipe materials is shown for $R$-ratios of 0.1, 0.3, and 0.5. With an increasing $R$-ratio, the kinetic lines are shifted to the right side to higher stress intensity factors. The kinetics of the pipe from 1981 has a steeper slope than the one of the pipe from 1988. Although the kinetics for the pipe from 1981 is slightly faster than the kinetics of the pipe from 1988, at lower stress intensity factors the lines seem to cross one another, which proves the failure times in Fig. 8, in which the crack resistance of PE–MD is higher at lower loads. While the slope of the material from 1981 is nearly constant at all $R$-ratios, it gets slightly steeper for the pipe material from 1988. The "synthetic" CCG curve at $R = 1.0$ was developed with an exponential extrapolation and the material constants were $A = 6.41 \times 10^{-8}$ and $m = 5.02$ for the pipe material from 1981 and $A = 4.49 \times 10^{-8}$ and $m = 4.68$ for the pipe from 1988.
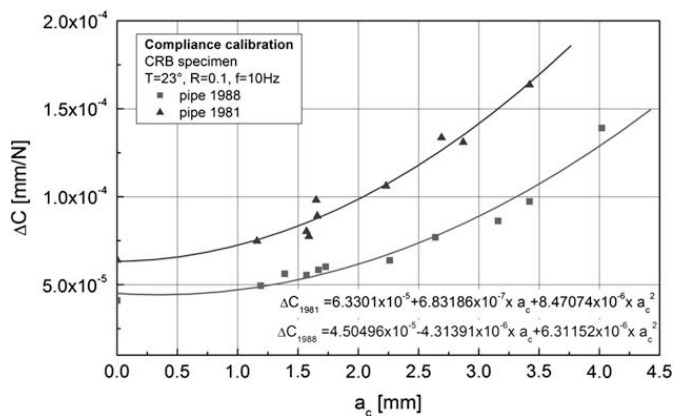


**Fig. 8.** Failure time $t_f$ as a function of the stress intensity factor $\Delta K_I$ of cyclic fatigue tests on CRB specimens at $R = 0.1$ for the pipes from 1988, 1987, 1981, and 1976 and the compression-molded plates from PE 80 and PE 80–MD.



**Fig. 9.** Compliance calibration curves $\Delta C$ as a function of the crack length $a_c$ for the pipes from 1981 and 1988.
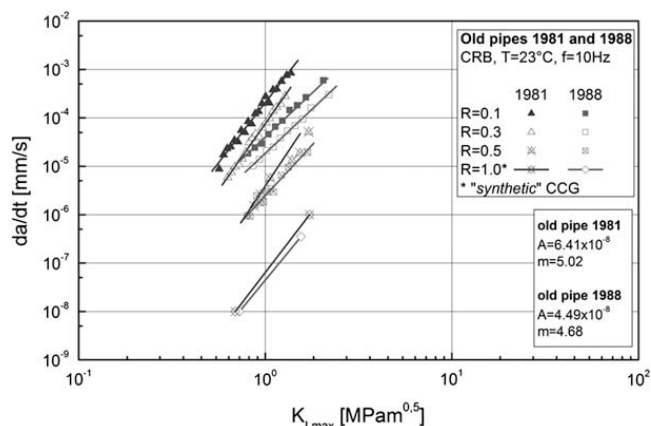
**Fig. 10.** Crack kinetics of the materials of the pipes from 1981 and 1988 at $T = 23°C$ at $R = 0.1$, 0.3, and 0.5 and extrapolated "synthetic" creep crack growth (CCG) curves ($R = 1.0$) to obtain the material constants A and m.

The remaining lifetime to achieve an overall service time of 50 years for the pipe from 1981 is 23 years, and 30 years for the pipe from 1988. For calculating the stress intensity factor in an internally pressurized pipe with defects on the inner pipe surface, different solutions are available [34,36]. For this study, the stress intensity factor by Murakami [35] was used. Fig. 11 shows the predicted remaining lifetimes of the pipes from 1981 and 1988, calculated with Equation (2) and the material constants A and m from the "synthetic" CCG curves. The results show a very high dependency on the size of the initial defect. A typical defect size $a_{ini}$ for the initiation of quasi-brittle cracks was found to be between 100 and 400 μm [1,37,38]. Using a hoop stress level of $\sigma_v = 8$ MPa (MRS in a PE 80 pipe grade) as a reference, both pipes seem to remain intact for the required service time of 50 years and even longer. Considering that the crack initiation time is neglected in this calculation, these pipes can be expected to remain in good order should they stay in use.
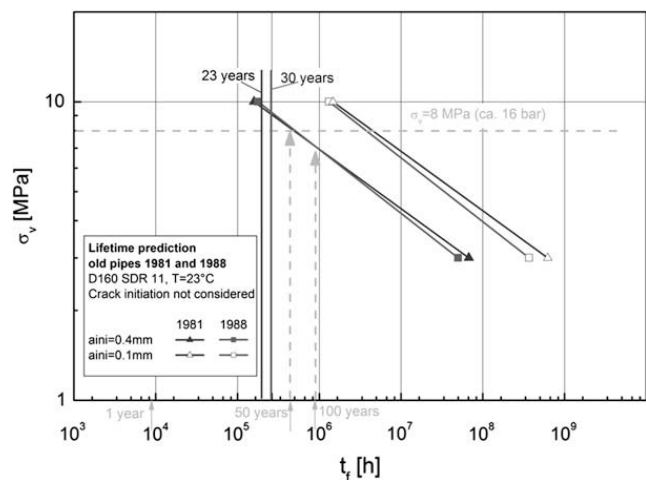


**Fig. 11.** Predicted remaining lifetime of the pipes from 1981 and 1988 at $T = 23 °C$ for estimated initial defect sizes $a_{ini}$ between 0.1 and 0.4 mm. Failure times $t_f$ as a function of the hoop stress $\sigma_v$ only include crack propagation time; crack initiation time has not been considered.

## 5. Conclusions

Four different used polyethylene pipes for gas and water supply with an age of up to more than 30 years have been investigated with respect to molecular and morphological changes. Furthermore, special focus has been put on the characterization of the remaining resistance to slow crack growth. It was not possible to identify the original materials by means of extensive molecular, morphological and fracture mechanics characterization, yet it could be found that the materials were still in a very good condition. The molecular analysis did not show any significant polymer degradation as an indication of ageing of the materials. Moreover, the thermomechanical behavior delivered comparable values to the reference materials. The absence of post-crystallization on the outer pipe wall and a still existing typical amount of residual stresses showed that the mechanical material properties have been almost unaffected. Distinct OIT values and clearly detectable amounts of stabilizers in the pipes ensure a sufficient stability for a continuation of the service. However, the fracture mechanics investigations indicated a decrease in failure times along with an increase in the age of the pipes. Using an extrapolation concept to predict the creep crack growth by means of fatigue tests, a prediction of the remaining lifetime was made for two pipes. Although the time for the crack initiation had not been considered, both pipes showed sufficient stability to prevent a quasi-brittle failure to remain in good order for 50 years.

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