



Effect of liquid oilfield-related media on slow crack growth behavior in polyethylene pipe grade materials



Paul Franz Schoeffl*, Reinhold Walter Lang

Johannes Kepler University Linz, Altenberger Straße 69, 4040 Linz, Austria

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ABSTRACT

Polyethylene, as non-polar material, shows a high affinity especially to liquid non-polar aromatic and aliphatic hydrocarbons, and liquid hydrocarbons (LHC) to a certain extent migrate into the bulk material by sorption, leading to material plasticization (i.e., drop in modulus and yield stress). This paper aims to study the crack growth mechanism and failure behavior of commercial pipe grade materials when exposed to deionized water or LHC (90/10 wt% i-octane/toluene) under the simultaneous application of cyclic loads. The results of the cyclic crack growth experiments with three PE 100 pipe grades, using cracked round bar (CRB) specimens and performed at two different temperatures (35 °C and 60 °C), are compared in terms of the specimen lifetimes, and the micro-modes and kinetics of failure by referring to concepts of fracture mechanics. Most importantly, while crack advance is preceded by crack-tip crazing in water, shear yielding takes place at crack-tips in the LHC environment.

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1. Introduction

Since the end of the 1950s, several procedures have been developed to estimate the lifetime of pressurized polyethylene (PE) pipes accounting for aqueous media [1–7] or (gaseous) hydrocarbon media [8–12]. While the latter standards for gaseous hydrocarbon media also take account for exposure to gas condensates at least to a certain extent by a nominal reduction of mechanical strength values, no such procedures and standards for pipe testing are currently implemented and available accounting for slow crack growth (SCG) behavior under liquid hydrocarbon (LHC) environment. And yet, in practical oilfield applications such pipes are frequently exposed to a variety of different LHCs. In terms of any effects on pipe performance and lifetime, LHCs based on non-polar aromatic and aliphatic molecular structures with small molecular dimensions are expected to be most critical, as such LHCs more easily migrate into PE, thereby affecting the materials yield and crack growth behavior via plasticization effects.

In terms of pipe performance and potential failure modes, three prime mechanisms are known to control long-term failure of pressurized pipes [3]. At high pressure respectively stress levels and correspondingly short failure times, pipe failure usually occurs by larger-scale yielding, which is controlled by the time and

temperature dependent yield stress of the pipe material (Region I of pipe failure). At intermediate pressure levels and related to longer pipe lifetimes, pipe failure frequently involves the mechanisms of crack growth initiation and slow crack growth prior to ultimate failure by through-thickness cracks (Region II of pipe failure). Finally, a third regime of failure is sometimes observed, particularly at high test temperatures and even lower stress levels than in Region II, in which failure occurs nearly stress independent and is controlled by large-scale molecular degradation accompanied by material embrittlement (Region III of pipe failure).

Applying this general failure scheme to pipes in oilfield applications experiencing maximum temperatures of about 60 °C, the failure regimes Region I and Region II are of practical importance. As far as Region I failures are concerned, there is at least one standard [10] for polyolefin pipe materials dealing with the effect of LHC exposure on mechanical properties such as modulus, yield stress and tensile strength along with corresponding strain values. And yet, this procedure, while providing a first indication on LHC uptake in polyolefins (up to exposure times of more than 100 days) along with the influence on short-term mechanical properties, is still of limited value since long-term mechanical properties under combined and superimposed mechanical and environmental (LHC exposure) loading are not covered. In terms of lacking knowledge even more critical is the absence of any procedures and investigations addressing Region II failure under superimposed mechanical stresses and LHC environments at service relevant (elevated) temperatures.

* Corresponding author.

E-mail addresses: paul.schoeffl@jku.at (P.F. Schoeffl), reinhold.lang@jku.at (R.W. Lang).

When comparing the situation to pressurized water pipes, a vast body of literature has been generated on the overall failure behavior of PE pipes [1–4,13,14] as well as on the micro-mechanisms of failure in the various pipe failure regimes [3,4,15–20]. As a result of continuous improvements of pipe grade PE materials regarding their crack growth resistance over the last decades (e.g. PE 63, PE 80, PE 100, PE 100-RC), numerous test procedures have been proposed and implemented specifically dealing with SCG failure as observed in Region II in a pressurized pipe failure curve. Common to all these procedures is that they aim to more directly address the phenomenon of SCG at a laboratory specimen level and perhaps to accelerate the experimental test times of pressurized pipe tests according to ISO 9080 [6] and ASTM D2513 [9] of up to 9 000 h and 10 000 h, respectively, by at least one magnitude. Some of these techniques are included in relevant standards and thus have become mandatory for material qualifications and rating. For example, in the US according to a PE 4710 classification (ASTM D2837) [7] a minimum lifetime of 500 h in the Pennsylvania Notch Test (PENT; 2.4 MPa, 80 °C in water; ASTM F1473) is required [7,21]. In Europe and other countries analogous requirements for a PE 100-RC (enhanced resistance to slow crack growth) classification according to PAS 1075 [22] exist that also include evidence for minimum lifetimes of 8 760 h (1 year) in the water pressurized Notched Pipe Test (NPT; ISO 13479) [23] in addition to meeting the MRS requirement for PE 100 (10 MPa; 50 years at 20 °C) according to ISO 9080 [6].

Moreover, further experimental techniques have been proposed and are described in the literature that allow for material comparisons and rankings in terms of their SCG resistance. These test procedures include the Full Notched Creep Test (FNCT) which is performed under static loads with a pre-notched specimen immersed in an environmental stress cracking (ESC) liquid at temperatures up to 80 °C (ISO 16770) [24]. On the other hand, methods to characterize SCG behavior via fatigue testing and without any ESC detergents have been developed [4,16,18,25,26] and more recently resulted in efforts to standardize a (cyclically loaded) cracked round bar (CRB) test [27].

Since there is currently no information available on the effects of LHC environments on the SCG behavior of PE pipe grade materials reflecting service relevant conditions, the prime objectives of the present paper are:

- To establish a test procedure based on the CRB technique that allows for the characterization of the SCG behavior of PE pipe materials under service relevant conditions in which mechanical loads, liquid environments (e.g. water, LHC) and predefined environmental temperatures are simultaneously applied and superimposed.
- To perform a first set of systematic investigations utilizing this test procedure to study any effects of such superimposed loading conditions for various commercial PE 100 grades on the SCG kinetics and the corresponding micro-mechanisms of failure.

2. Methodology and experimental

2.1. Materials and specimen preparation

For this investigation, three commercial PE types were selected as model materials to cover a range of behavior in terms of their crack growth resistance. Two of the materials were of the PE 100 type, provided by two different suppliers and designated PE 100-1 and PE 100-2, respectively. The third PE grade was designated as PE 100-RC according to PAS 1075 [22], as it exhibits enhanced resistance to slow crack growth in FNCT and NPT tests. All materials were first compression molded to plaques of 15 mm thickness (300 × 300 × 15 mm). The CRB specimens for the SCG experiments

were manufactured according to ONR 25194 [27] and circumferentially notched with a razorblade and an initial nominal notch length of 1.5 mm.

2.2. Selection of liquid environments and specimen pre-conditioning

To cover an adequate range of service relevant liquid exposure conditions, in a first step, representative liquid environments needed to be defined. Hence, deionized water was selected to reflect conditions of injection water lines, but also as general reference environment. To simulate worst case oilfield and gas condensate exposure conditions, an LHC reference environment was defined. Bearing in mind that nearly all of the constituents in crude oil are lipophilic, similar to polyethylene, attractive interactions exist on a molecular level allowing for the solubility of hydrocarbons in the amorphous domains of the polymer. Moreover, low hydrodynamic radii (molecular dimension) of the LHCs are proposed to exacerbate the test conditions. In a gas condensate analysis conducted by OMV E&P GmbH (Gänserndorf/A) from a representative gas well, the BTEX content reflecting small aromatic constituents (Benzene, Toluene, Ethylbenzene and Xylene) was found to be between approx. 10- and 12 wt%. Based on such considerations, a mixture of 90/10 wt% i-octane/toluene (90/10 i-octane/toluene) was selected as LHC reference environment. This environment also accounts for the fact that crude oils and gas condensates in general contain both, aliphatic and aromatic constituents.

The importance of the proper selection of the LHC environment rests upon two basic phenomena involving various physical and/or chemical mechanisms being associated with the exposure of PE compounds to LHCs. First, LHCs are well known to physically act as plasticizer in PE, thus reducing the materials modulus and yield strength, with both properties being of structural relevance to pipes. Furthermore, LHCs also may again physically extract certain additives such as antioxidants from the bulk compound by leaching, thereby affecting the longer-term thermo-oxidative stability and the chemical aging behavior (molecular degradation) of the compound. Prior to SCG testing and in order to perform the SCG tests under equilibrium (steady state) conditions right from the start and to avoid initial transient effects, all CRB specimens to be tested in the LHC environment were preconditioned (immersed) at the designated mechanical test temperature in the respective environments until saturation (mass constancy). For this purpose, CRB samples were first weighed on a KERN PLS 1200-3A precision scale and then immersed in LHC in 500 ml wide mouth glass bottles (DURAN GLS 80) with an aluminum cap on the inside of the PP-plug. These glass bottles were then placed in heating oven (BINDER FED 53) under atmospheric pressure at the desired temperature. The weight gain of the specimens was recorded periodically

Table 1
Material designation and test program.

Material code	Media	T (°C)	Tests performed
PE 100-1_w_60	Deionized water	60	$t_f(\Delta K_{I,ini}); \sigma_{ys}$
PE 100-1_90-10_60	90/10 i-octane/ toluene	60	$t_f(\Delta K_{I,ini}^{app}); \sigma_{ys}; \Delta m_{max}$
PE 100-2_w_35	Deionized water	35	$t_f(\Delta K_{I,ini}); E'(T); \sigma_{ys}$
PE 100-2_w_60	Deionized water	60	$t_f(\Delta K_{I,ini}); E'(T); \sigma_{ys}$
PE 100-2_90-10_35	90/10 i-octane/ toluene	35	$t_f(\Delta K_{I,ini}^{app}); E'(T); \sigma_{ys}; \Delta m_{max}$
PE 100-2_90-10_60	90/10 i-octane/ toluene	60	$t_f(\Delta K_{I,ini}^{app}); E'(T); \sigma_{ys}; \Delta m_{max}$
PE 100-RC_w_60	Deionized water	60	$t_f(\Delta K_{I,ini}); \sigma_{ys}$
PE 100-RC_90-10_60	90/10 i-octane/ toluene	60	$t_f(\Delta K_{I,ini}^{app}); \sigma_{ys}; \Delta m_{max}$

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