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Test equipment

# Yielding and crack growth testing of polymers under severe liquid media conditions

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ABSTRACT

Several techniques for testing under superimposed mechanical and environmental loading are described in terms of test devices and test arrangements implemented on conventional mechanical test equipment (universal screw-driven test machines or electro-dynamic test machines). As to the application of mechanical loads, monotonic, static and cyclic tests are covered, including standard tensile tests and fracture mechanics based experiments. The former case emphasizes the determination of liquid environments on modulus, yield and post-yield behavior using conventional dumbbell specimens of the 5A Type (ISO 527) or notched pipe ring tensile (NPR-T) specimens. The latter methods are designed to obtain fatigue life and crack growth kinetics data using cracked round bar (CRB) or compact type (CT) specimens. Selected examples of material characterization for various loading conditions in air, water and liquid hydrocarbon environments are described and discussed with respect to predict material behavior under simultaneous mechanical and environmental loads.


## 1. Introduction

The structural performance profile and the mechanical behavior of polymeric materials are known to depend strongly on the mechanical loading conditions (e.g., loading rate, monotonic vs. static vs. cyclic loading) and environmental conditions (e.g., temperature, various gaseous or liquid environments). This is also particularly true for structural design and safety relevant ultimate mechanical properties such as the materials yield stress and crack growth resistance [1–6]. Not surprisingly, numerous test methods have been proposed in the literature and have been standardized to cover the effects of specific test environments or media exposure conditions on the mechanical properties of polymers [7–12]. In many of these methods, environmental exposure of test specimens and mechanical testing are conducted consecutively, and are thus separated (i.e. specimens are immersed into a liquid environment over a certain time or until saturation in a first step, and are subsequently transferred to the mechanical test equipment and tested in a second step). However, it is well known that superimposing mechanical loads and exposure to specific environmental conditions may lead to a very different mechanical material response [1,7–9,13–16].

As this latter situation also reflects conditions experienced by polymeric parts and components in real service, there is a great need for test methods that adequately account for such superimposed mechanical and environmental loading combinations, particularly under aggressive chemical service environments (e.g. liquid hydrocarbons in oilfield applications). While a number of techniques for superimposed mechanical-environmental testing are described in the literature (e.g. Full Notch Creep Test (FNCT) in stress cracking “test” detergents [12], Pennsylvania Notch Test (PENT) in water [17], linear elastic fracture mechanics (LEFM) based creep crack growth tests in water [7,8]), they frequently are designed for rather specific

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loading conditions (e.g. static loading) and environmental conditions which do not necessarily cover the range of service environments of interest. Of course, to more closely reflect service conditions, many details of superimposed mechanical-environmental testing will need to be adapted to cover the specifics of the type of mechanical loading and the environment of practical relevance.

Hence, a main objective of the present paper is to describe a comprehensive test approach and test methodology that allows for superimposed mechanical and liquid environmental loading under various modes of loading (monotonic, static, cyclic) at different temperatures based on widely used standardized test methods for characterizing the material behavior typically in air. As alluded to above, rather than being totally novel and by utilizing commercial tensile and fatigue test frames in combination with modern optical devices for strain and crack length measurements, the test approach and the test techniques described in this paper build on a variety of techniques described and used by others before. Nevertheless, the set-up described in this paper offers a high flexibility and numerous advantages for superimposed mechanical-environmental testing regarding variations in the loading mode, the test temperature and the liquid environment. The focus of the present paper is on the mechanical material response in terms of two "ultimate" performance profiles. In one case, the conventional engineering stress/strain behavior is characterized with a particular focus on the yield and post-yield regime, in the other fracture mechanics tests under cyclic loads are performed to obtain information on the materials crack growth and failure behavior under superimposed environmental loading. The specifics of the investigations performed in terms of selecting polymeric materials and specimens, on the one hand, and liquid environments and test temperatures on the other, were defined for the case of oilfield applications of polyethylene (PE) pressure pipes.

## 2. General test system requirements

Using commercial test systems, a basic premise of the superimposed mechanical-environmental test methods to be designed and implemented was to extend existing mechanical measurement and control technology for monotonic tensile loading and cyclic loading to be capable of simultaneous media exposure. The monotonic testing mode should allow data generation for typical engineering stress/strain diagrams using a conventional screw-driven universal testing machine. The cyclic loading mode, on the other hand, is to be applied to fracture mechanics based crack growth experiments, again using an existing electrodynamic test facility.

Moreover, the test systems should meet the following main general requirements:

- capability to be used with, and hence withstand, a wide range of liquid exposure media (incl. corrosive and reactive chemical liquids),
- cover a wide test temperature range from below 0 °C to above 100 °C,
- avoidance of transient effects by ensuring sorption equilibrium conditions,
- transparent environmental containments for the immersion of specimens while being tested to allow for the application of overall and local deformation measurement devices such as optical transducers or picture correlation systems,
- fulfillment of all safety requirements for operating with hazardous liquids (prevention of media release to the laboratory environment in liquid and vapor form, explosion protection),
- functional and ergonomic design accounting for easy and safe specimen installation.

In addition, several more specific requirements should be met for monotonic or static tensile tests and the cyclic fracture mechanics tests. For the monotonic/static tensile tests these requirements are:

- tensile testing of various specimen configurations including standardized dumbbell specimens (according to ISO 527 [18] especially multi-purpose specimens of the type 1A and 1B and small size specimens of the type 5A) and notched pipe ring tensile (NPR-T) specimens (analogous to ASTM D2290 [19]) up to a pipe diameter of DN40),
- specimen clamping devices accommodating the various specimen configurations and for use in combination with the specific (liquid) media environments,
- tensile testing under monotonic (deformation rate range $5 \times 10^{-4}$ to $1.5 \times 10^{3}$ mm/min and static/creep loading conditions up to 10 days).

For the cyclic fracture mechanics tests the requirements include:

- fatigue crack growth testing of various specimen configurations including cracked round bar (CRB; according to ONR 25194 [20]) and compact type (CT; according to ASTM E647 [21]),
- self-aligning specimen test fixtures accommodating the various specimen configurations and for use in combination with the specific (liquid) media environments,
- fatigue testing at frequencies ranging from $10^{-3}$ Hz to $10^{2}$Hz and at R-ratios from 0.05 to 0.9

## 3. Specific test systems and testing devices

A main feature of any superimposed mechanical environmental test is to achieve stable "equilibrium state" conditions in terms of the interaction of a specific polymeric material and the exposure to a given environment throughout the entire mechanical test duration. This usually implies a two-step procedure, in which the material (specimen) is exposed to the defined environment (e.g. immersed in a liquid) in a first pre-conditioning step until an equilibrium state of medium uptake by sorption corresponding to the conditions of subsequent testing (i.e. test temperature) is reached. In a second step, specimens are then transferred to the mechanical test device which is equipped with a specimen containment that again ensures such stable environmental conditions in terms of media composition and temperature during mechanical testing. To avoid any transient effects, it is essential that the time for specimen transfer from the pre-conditioning containment to the test machine media containment is kept short (to minimize any media desorption) and some time to again achieve equilibrium conditions (typically up to one hour) is allowed for, prior to applying mechanical loads.

The specific test systems used in this study for superimposed environmental and monotonic/static or cyclic loading conditions are shown in Fig. 1. The photographs illustrate the respective test systems equipped with the test machine media containments and the specimen fixture devices along with the required test control units. For monotonic/static testing, a screw-driven universal testing machine of the type ZwickRoell Z005 (Zwick GmbH & Co. KG, Ulm, D) and for cyclic testing, an electro-dynamic test system of the type Instron E3000 (Instron, Norwood, MA/USA) were equipped with the environmental control units described below.

### *3.1. Environmental test containments, temperature control and measurement tracking*

For the superimposed mechanical-environmental test stage, and depending on the test to be performed (monotonic/static versus cyclic) and the specimen type (configuration and geometry), special environmental containments were designed allowing for media exposure under temperature control while performing the mechanical test. A basic prerequisite for the construction of the environmental containments was the resistance to a wide range of corrosive and aggressive chemical media while simultaneously meeting the requirements for optical tracking of measurements. Due to its outstanding chemical resistance and durability, the sidewalls of the media containments were made of Duran glass. In order to avoid any media evaporation of circulating aggressive fluids to the test laboratory environment and to eliminate contamination of heat control devices (e.g. thermostat, hoses), the environmental containment was conceived as double-walled glass tube. This design allows for indirect heating and cooling in the outer wall (heating and cooling jacket) via a circulating non-aggressive and transparent heat transfer fluid while the environmental (chemically aggressive) test fluid remains in the inner part of the tube where the specimen is positioned. For temperature control, an external thermostat (Huber CC-K6-NR, Peter Huber Kältemaschinenbau GmbH; Offenburg, D) with integrated heating and cooling function (−25°C to 200 °C) was connected to the outer wall of the double-walled environmental containment. Water or water/glycol can be used as heat transfer fluid, depending on the required test temperature.

In order to trace specimen deformation during the performed experiments, the transparency of the test machine media containments allowed for optical tracking of displacement and crack growth. Therefore, a high speed camera system (HXG 40, Baumer GmbH, D) has been installed and implemented combined with a powerful NI cRIO 9074 (real time) triggering and control unit and picture processing software (National Instruments, Austin, TX/USA). For optical calibration purposes, a calibration grid (Edmund Optics, Barrington, NJ/USA) was used to account for distortion effects due to different refraction indices.

### *3.2. Test arrangement and test devices for monotonic/static testing*

The mounting fixtures for the monotonic/static environmental containment are shown in Fig. 2 and consist of a

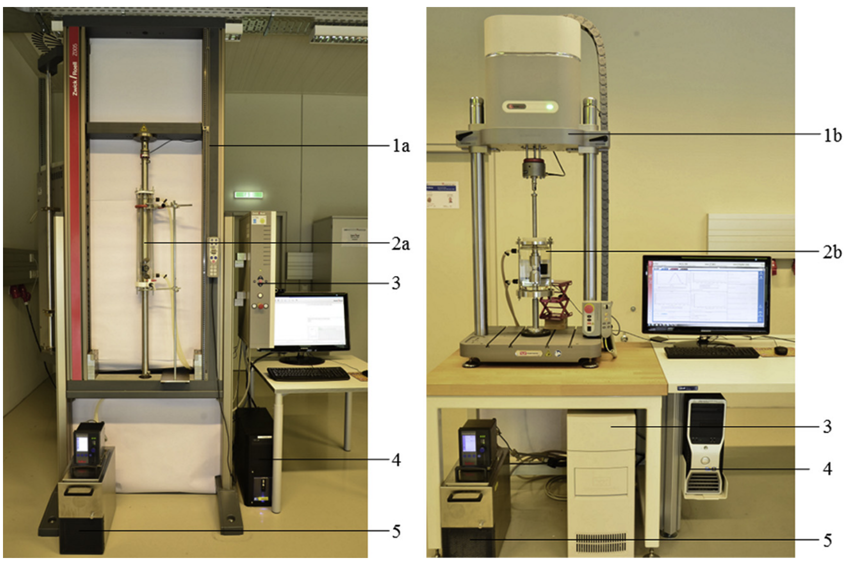


**Fig. 1.** Images of test systems for superimposed mechanical-environmental testing; monotonic/static loading (left) and cyclic loading (right); (1a – universal screw-driven testing machine, 1b – electro-dynamic test machine, 2a – monotonic/static environmental containment, 2b – cyclic environmental containment, 3 – control unit, 4 – computer, 5 – thermostat).

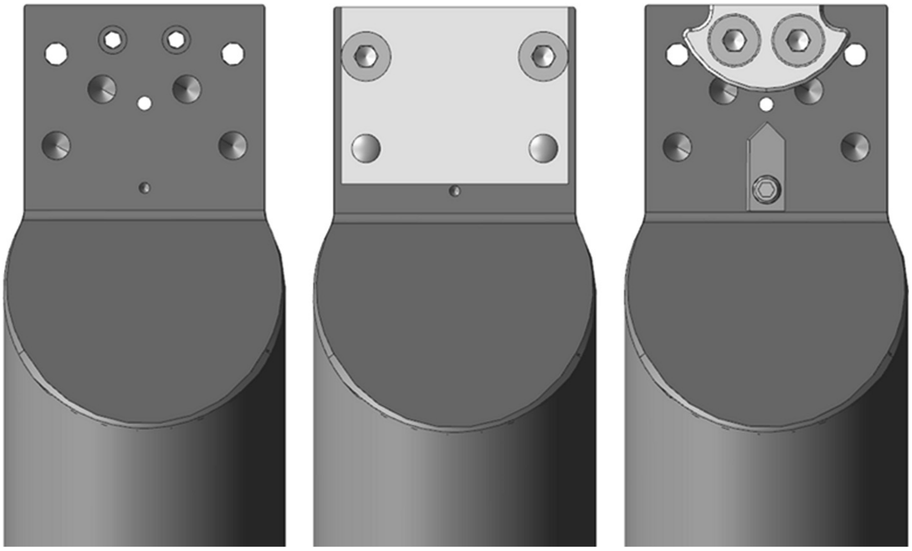


**Fig. 2.** Specimen mounting (a): multi-purpose base platform (b) ISO 527 dumbbell type configuration (c) NPR-T sample configuration.

multi-purpose base platform which can be used for various fixture systems depending on the specimen type to be tested. To allow for sample characterization with almost arbitrary geometry, the multi-purpose base platform contains tapped holes in the flat section at one end of each rod onto to which appropriate tailor made specimen fixture may be mounted. High accuracy in the cylindrical shape of the rods along with a polished surface (Ra~0.1) is needed for the simplified sample exchange described below. All components of the specimen mounting fixtures (including screws and other parts) were made of highly alloyed stainless steel to resist aggressive chemicals.

The tubes of the double-walled environmental media containments were manufactured from Duran glass. The size of the tubes was specified to achieve up to 500 % strain when testing 5A specimens, leading to an overall height for the glass tube media containment of 500 mm, with an outer and inner diameter of 90 mm and 63 mm, respectively. The converging upper and lower ends of the double-walled glass containment were capped with coaxially bolted end-plates made of polytetrafluoroethylene (PTFE), as shown in Fig. 3. Both end-plates contained a concentric bore and a milled groove for a coaxial sealing with the glass containment. The diameter of the bore of the upper end-plate was 1.5 mm larger than the platform rod diameter to guarantee free movement of the upper platform rod attached to the load cell (and the test machine loading crosshead) while simultaneously keeping any evaporation of the test fluid at a low level. The centric bore in the lower end-plate was manufactured with a diameter of just 0.1 mm larger than the diameter for the lower platform rod and, additionally, contained a milled grove for the insertion of a wiper seal. The vertical position of the environmental containment was fixed with a stand and can be vertically shifted for installation or replacement of specimens on the one hand and for conducting a test on the other hand. To empty the containments at the end of a test series, the lower end-plate was equipped with a screwed-in drainage tap (pos 11 in Fig. 3).

### *3.3. Test arrangement and test devices for cyclic testing*

For cyclic testing under liquid environments, similar basic design principles were applied for the temperature controlled environmental glass media containment as in the monotonic/static case. However, numerous details of the design had to be adapted to account for the different test machine periphery and attachments, the different specimen configurations and geometries (i.e. CRB specimens and CT-specimens), and the rather different specimen mounting devices (including specimen insertion and replacement).

The test arrangements produced and implemented for testing CRB specimens and CT specimens are illustrated in Fig. 4. The overall height of the glass tube media containment was 200 mm. To account for both larger (e.g. CT, WOL, AT) and smaller specimens (e.g. CRB), and in order to keep similar proportions with regards to avoiding temperature gradients, two types of media containments (different in size and mounting devices) were developed. The outer/inner diameters of the containment were 65/40 mm for CRB specimens and 150/114 mm for CT specimens. In sharp contrast to monotonic/static testing, both end-plates (pos. 6 and 15 in Fig. 4) are made of highly alloyed stainless steel, with the lower end-plate being firmly connected to the bottom of the cyclic test machine. To install and remove specimens, the upper end-plate was conceived with a wide bore (bore diameter of 30 mm for CRB specimens and of 90 mm for CT specimens). Moreover, to install and remove fixture devices (including specimens), another removable PTFE cover plate (pos. 5 in Fig. 4) was connected in a centered position on top of the upper end-plate. To minimize media evaporation, this cover plate has a bore diameter of only 4 mm larger than

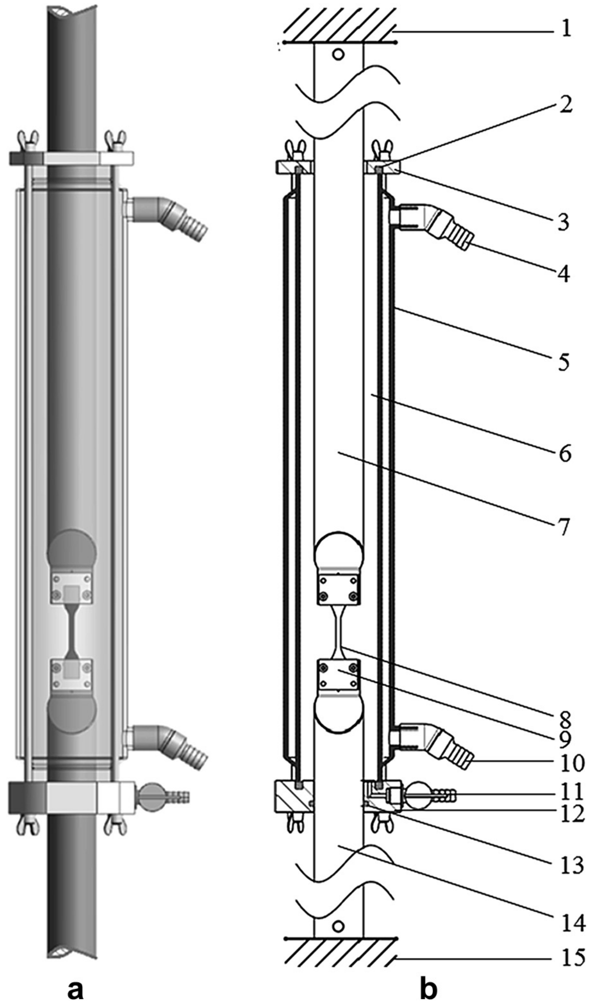


**Fig. 3.** (a)–(b): Schematic illustration of monotonic/static testing device for 5A specimen configuration (1 – crosshead/load cell, 2 – coaxial-sealing, 3 – upper PTFE plate, 4 – thermo-fluid inlet, 5 – Duran glass, 6 – test media, 7 – upper platform rod, 8 – 5A specimen, 9 –clamping, 10 – thermo-fluid outlet, 11 – tap, 12 – lower PTFE plate, 13 – wiper sealing, 14 – lower platform rod, 15 – connection to machine).

the extensional rod diameter and contains a sealing ring at its bottom.

For specimen installation/removal, threaded sleeves for CRB-type specimens and pin-hole clevis-grips for CT specimens were designed and produced. The specimen mounting devices were constructed with a self-aligning ball lug fixture (pos. 2 and 3 in Fig. 4) with the ball end screwed into an extension rod (reaching outside the containment) in the upper mounting device and directly screwed into the specimen fixture at the lower device. Both screwed in ball ends of the upper and lower specimen fixture were connected to the cyclic testing machine by hooking into appropriate lugs at the load cell (upper lug, mounted at the actuator) and at the center of the bottom end-plate (lower lug), respectively. Due to the self-aligning design of this clamping system, any bending of the specimen (e.g. by asymmetric crack growth) is reduced to a minimum.

## 4. Materials, specimens and environmental conditioning

### 4.1. Materials and specimens

As a model material, a commercial PE type (PE 100 polyethylene pipe grade) was selected for all investigations of this paper. For monotonic/static experiments, 5A dumbbell specimens were used, as illustrated in Fig. 5. The dumbbell specimens were punched from 300 × 300 × 2 mm compression molded plaques. NPR-T specimens were manufactured from 32 × 3 mm pipes, cut into segments of 15 mm length and prepared with two perpendicular notches with a radius of 20 mm at each side.

For cyclic experiments, the PE 100 pipe grade material was first compression molded to plaques of 15 mm thickness (300 × 300 × 15 mm). From these plaques, CRB specimens and CT specimens were machined according to ONR 25194 [20] and ASTM E647 [21], respectively. The corresponding specimen configurations are depicted in Fig. 6 along with information on essential specimen dimensions.

Both specimen types were pre-cracked with a razorblade cut of a nominal depth of 1.5 mm. In the case of CRB specimens, a circumferential cut was directly introduced at the specimen center. In the CT specimens, the razor blade pre-crack was introduced at the tip of the V-shaped pre-notch with a nominal notch length of 20 mm (amounting to an initial total crack length measured from the load line of 11.5 mm).

### 4.2. Liquid environments and specimen pre-conditioning

The liquid media selected for this study were specified according to requirements of PE pressure pipes for oilfield applications where such pipes are frequently exposed to a variety of different liquid hydrocarbons (LHC). To simulate worst case oilfield and gas condensate exposure conditions, a mixture of 90/10 wt% i-octane/toluene (90/10 i-octane/toluene) was selected as LHC reference environment. Deionized water was used as an environment to reflect conditions of injection water lines, and also as a general reference environment.

Prior to performing the monotonic/static or cyclic test experiments, all specimens to be tested in the LHC environments were first pre-conditioned (immersed) at the designated mechanical test temperature in the respective environments until saturation (mass constancy). For this purpose, all specimens 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 a heating oven (BINDER FED 53) under atmospheric pressure at the desired temperature. The weight gain of the specimens was recorded periodically by weighing specimens immediately when the surface appeared dry after removal from the (immersion) bottles. The specimens to be tested in the deionized water environment were conditioned in deionized water at the designated test temperature, but for two hours only since PE is not prone to absorbing water.

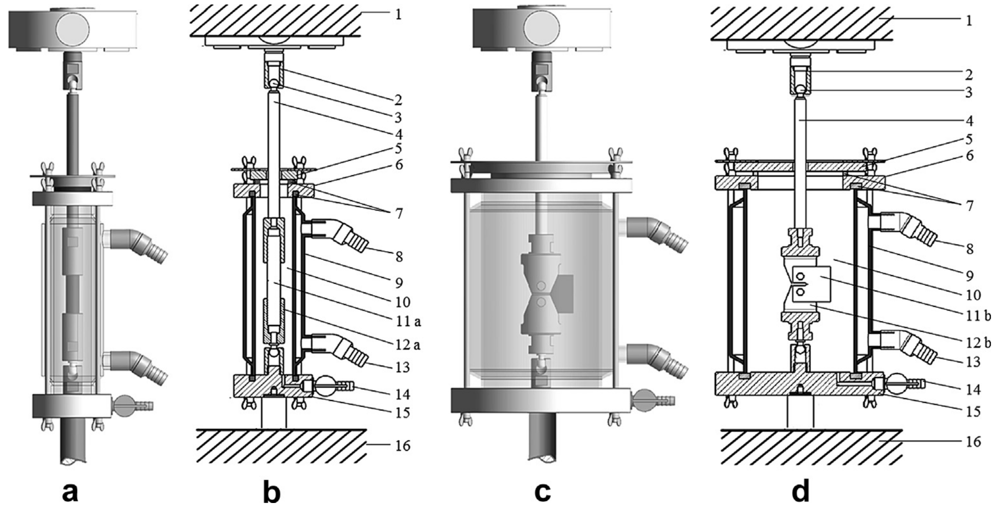


**Fig. 4.** Schematic illustration of cyclic testing devices for (a)–(b) cracked round bar (CRB) specimen and (c)–(d) compact type (CT) specimen configuration (1 – actuator/load cell, 2 – hook-in lug, 3 – screwed-in ball, 4 – extensional rod, 5 – cover plate, 6 – upper stainless steel plate, 7 – sealing, 8 – thermo-fluid outlet, 9 – Duran glass, 10 – test media, 11 a – CRB-specimen, 11 b – CT – specimen, 12 a – threaded sleeves, 12 b– clevis grips, 13 – thermo-fluid inlet, 14 – tap, 15 – lower stainless steel plate, 16 – connection to machine).

## 5. Experimental results and discussion

### 5.1. Monotonic testing

A first test series was performed to investigate the difference in the monotonic stress-strain behavior of LHC pre-conditioned specimens which were subsequently mechanically tested in air or a superimposed LHC environment. The specimen configuration for these test series was of the 5A type. Pre-conditioning and testing was performed at 60 °C with an LHC uptake in the saturated pre-condition state amounting to about 7.5 wt %. The tensile tests were also performed at 60 °C and at a strain rate of $10^{-3}$ $s^{-1}$. For comparison and reference purposes, non-conditioned specimens were also tested in an air environment with the other testing parameters kept equal to those of the pre-conditioned specimens.

The results of these test series are shown in Fig. 7 in terms of nominal (engineering) stresses versus nominal strains (determined from clamping length and crosshead displacement). As expected, due to the plasticization effect of LHC uptake, the specimens pre-conditioned in 90/10 i-octane/toluene exhibit a significant drop in yield stress and in the post-yield behavior compared to the non-conditioned specimens tested in air. The yield stress drop from about 11.7 MPa for non-conditioned specimens to about 7.7 MPa for the pre-conditioned specimens amounts to about 35 %. Most notably, however, when testing pre-conditioned specimens in air or in the superimposed LHC environment, the stress-strain curves for these two test conditions are essentially identical up to the yield-point but then clearly separate over the entire deformation range (i.e. over the entire post-yield deformation regime), with specimens tested in air revealing higher stress levels at equivalent nominal strains. It should be mentioned that equivalent phenomena were observed when testing NPR-T specimens (taken from pipe sections) in the same environmental conditions.

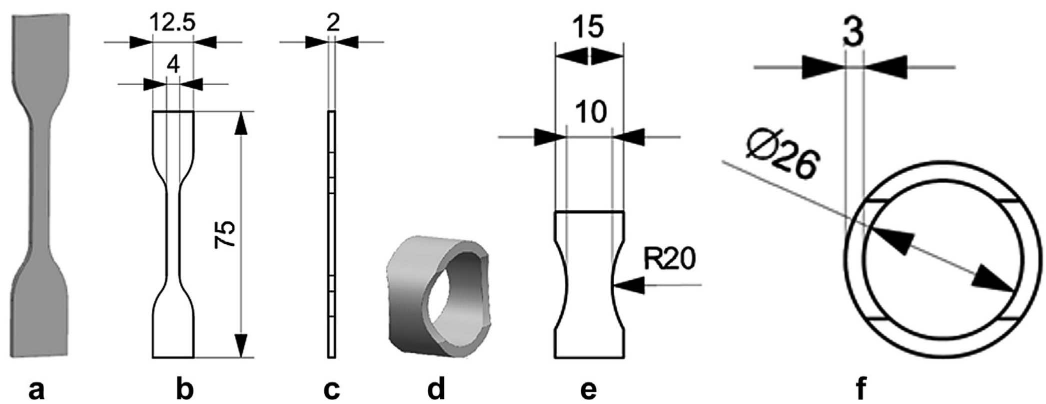


**Fig. 5.** Specimen configurations for monotonic/static test experiments; left (a–c): 5A specimen; right (d–f): Notched pipe ring tensile (NPR-T) specimen.

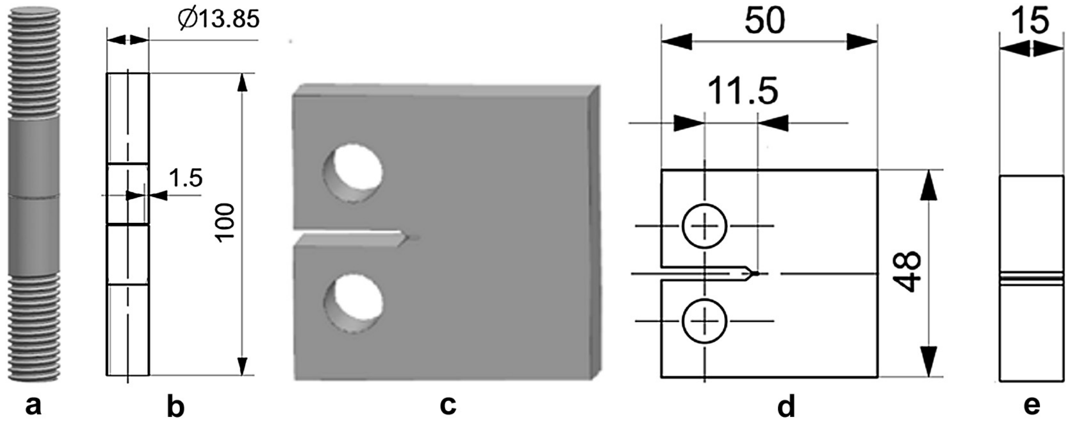


**Fig. 6.** Specimen configurations for cyclic test experiments; left (a–b): Cracked round bar (CRB) specimen; right (c–e): Compact type (CT) specimen.

To explain the effects depicted in Fig. 7, it should be pointed out that the specimens were exposed to the test temperature at 60 °C in air for about 5 minutes to minimize LHC desorption prior to starting the test by mechanical loading. The corresponding temperature equilibration time for the LHC tensile test was about two hours to ensure a homogeneous test temperature in the entire environmental test containment. The total time-to-yield for the tests on pre-conditioned specimens subsequently tested in air, amounted to about 10 minutes, the remaining time from reaching the yield strain to the maximum strain achieved in the test of about 600 % (i.e. post-yield regime) amounts to about 200 minutes. This indicates that significant desorption measurably affects the mechanical performance and must be accounted for in such mechanical tests that run longer than about 10 minutes. This time controlled desorption process of course depends on the test temperature and may also be affected by the volume change of the bulk material taking place in the pre-yield and post-yield regime. These results, of course, highlight the importance of superimposed environmental mechanical testing versus simply subsequently testing pre-conditioned specimens in air even for short-term monotonic tests. Moreover, they underscore the indispensable necessity for performing any meaningful static or cyclic long-term tests on environmental effects on polymer mechanical behavior under superimposed loading conditions.

Another series of experiments was performed to investigate the limitations of the test system in terms of environmental/thermal/mechanical stability and the achievable test duration in LHC. For this purpose, tests were performed covering nearly the entire strain rate range specified for the universal tensile test equipment used (i.e. $10^{-1}$ $s^{-1}$ to $10^{-6}$ $s^{-1}$). These tests were again carried out on pre-conditioned specimens of the 5A type, in this case at a pre-conditioning and test temperature of 35 °C and 60 °C. The results are shown for the various strain rate experiments by plotting the yield stress as a function of time-to-yield in Fig. 8, indicating a time scale of these experiments ranging from a few seconds to more than 200 hours. The test conditions were found to remain totally stable in this test time range.

Regarding the test results, a characteristic knee was found in the yield stress vs. time-to-yield curves which is known to also occur in tests in air [22–25]. For these tests in air, this knee has been related by these authors to a

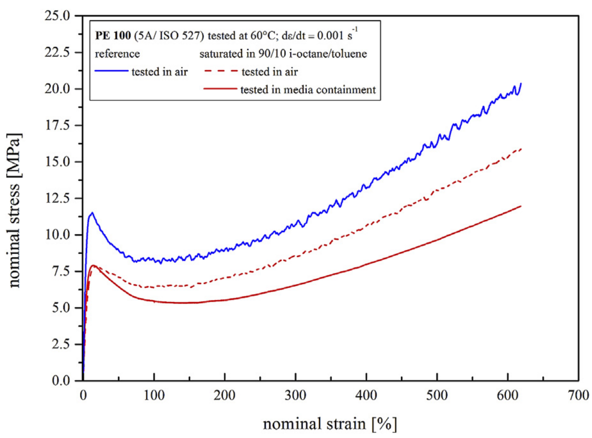


**Fig. 7.** Nominal stress/nominal strain curves of PE 100 comparing LHC pre-saturated 5A specimens tested in LHC or in air, and a non-conditioned specimen tested in air as reference.

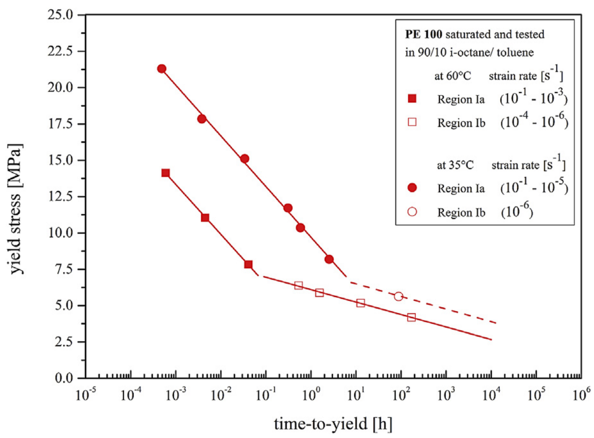


**Fig. 8.** Yield stress of saturated PE 100 specimens tested in LHC environment as a function of strain rate and temperature.

change in the micro-mechanisms governing time-dependent yield deformation, and the two straight line sections of the curve can be ascribed to Ia and Ib type failure mode. Apparently, this phenomenon also occurs in tests under LHC environments, albeit at lower stress levels, due the above mentioned plasticization.

### 5.2. *Cyclic testing*

The cyclic failure behavior of PE 100 CRB specimens during liquid media exposure is depicted in Fig. 9, comparing experimental results obtained from tests at 35 °C in water and in 90/10 i-octane/toluene along with fracture surface analysis via scanning electron microscopy (SEM) and optical microscopy (OM). For microscopy, an optical stereo microscope of the type OLYMPUS SZX 16 (Olympus Corp., Tokyo/JP) and a SEM of the type JEOL 6400 (JEOL Ltd., Tokyo/JP) were used. The data are plotted in terms of the initial cyclic stress intensity factor range $\Delta K_{I,\ in}$ ($f = 10$Hz, $R = 0.1$) vs the time-to-failure. To also account for the growth of "blunt" cracks, the Y-axis is designated as $\Delta K_{I,\ in}$ apparent.

The failure curves for the tests in water exhibit a more or less pronounced knee, indicative of the transition from mostly ductile failure (designated as Type I failure) to quasi-brittle failure via the stable growth of a sharp crack (Type II-sc). Compared to the water experiments, the failure lines for tests performed in the LHC environment are at least initially shifted towards lower $\Delta K_{I,in}$ values. According to fracture surface analysis, the observed failure mechanism in LHC is governed by blunt crack growth (Type II-bc; crack-tip shear yielding mechanism), with the failure lines exhibiting significantly lower slopes than those observed for Type II-sc failure (crack-tip crazing mechanism) in water. Most remarkably, a cross-over is observed in the lifetime data obtained for 90/10 i-octane/toluene and water at around 100 hours ($3.7 \times 10^6$ cycles), with the time-to-failure of specimens tested in the LHC environment exceeding the corresponding values of specimens tested in water. Further results along with details of the experimental methodology and data reduction procedure, as well as an interpretation of the phenomena observed, are described elsewhere [26,27].

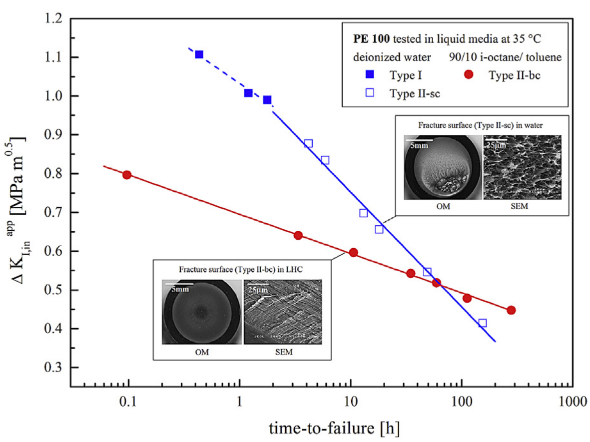


**Fig. 9.** Failure times of PE 100 CRB specimens tested at 35 °C in water and LHC, respectively.

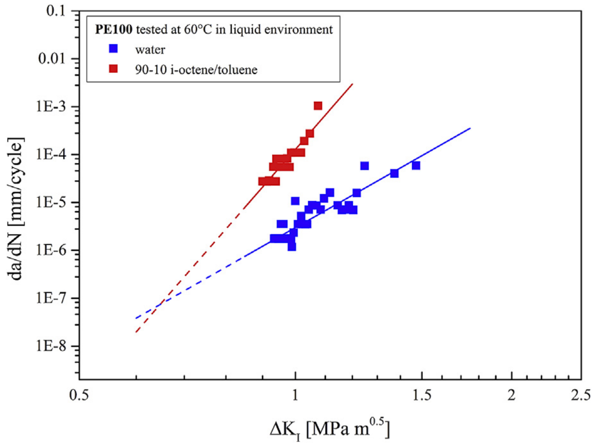


**Fig. 10.** Crack growth kinetics of PE 100 CT specimens tested at 60 °C in water and LHC, respectively.

Finally, an example of results of a fatigue crack growth experiment with CT-specimens allowing for a data reduction in terms of the cyclic crack growth rate (da/dN) vs. the cyclic stress intensity factor range ΔK is depicted in Fig. 10. The CT specimens were tested at 60 °C during liquid media exposure in water and in 90/10 i-octane/toluene. Tests in both environments were performed starting at initial $\Delta K_I$ levels of ca. 0.9 MPa $m^{0.5}$. The crack growth rates of CT specimens pre-conditioned and tested in LHC were found to be significantly higher, and exhibit a steeper slope, than specimens tested in water. By extrapolation of the different crack growth kinetics curves for water and for LHC, a cross-over occurs at $\Delta K_I$ levels of approx. 0.6 MPa $m^{0.5}$, indicating superior crack growth resistance of LHC saturated specimens at low stress intensity factor levels. This extrapolated cross-over may be interpreted as corresponding to the cross-over in the lifetime curves illustrated in Fig. 9. Again, a more in-depth coverage of this methodology and the merits of linear elastic fracture mechanics (LEFM) in environmental crack growth testing are provided separately [27].

## 6. Summary and outlook

Accounting for the increasing demand of mechanical material data under specific liquid environmental conditions, a test systems approach was conceived and practically implemented that allows for superimposed mechanical and environmental loading in a flexible and comprehensive manner. Due to the fact that this approach builds on already existing test methods combined with commonly used and commercially available mechanical test equipment (universal screw-driven test systems and electro-dynamic test systems), the test results obtained can easily be compared to other (existing) data sets. Furthermore, all tests in this study were performed with standardized specimen configurations and geometries and, therefore, no new specimens with unknown or undefined mechanical loading states had to be introduced. Numerous details of the test devices (test arrangement, environmental containments, etc.) are described and discussed, along with data generated under various test conditions. The results clearly show that superimposed mechanical-environmental testing compared to testing pre-conditioned specimens in air is of importance for the behavior in the yield and post-yield regime, and particularly for static long-term and fatigue testing. Moreover, different crack growth rates and, especially, different failure mechanisms during fatigue testing (crack-tip crazing in water vs. crack-tip shear yielding in liquid hydrocarbons) underline the significance of testing under simultaneous mechanical and environmental loading.

While the present paper had its focus on superimposed mechanical-environmental test techniques for typical laboratory specimens (i.e. specimen level) under uniaxial mechanical loads (although multi-axial stresses may exist locally at notch or crack-tips), further work will be reported shortly on pressurized pipe tests (component level) under long-term multi-axial overall stresses to compare and contrast some of the time dependent failure properties obtained on the specimen level and on the pipe level.

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