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Polyethylene (PE) materials for piping systems — Determination of resistance to slow crack growth under cyclic loading — Cracked Round Bar test method

Matériaux polyéthylène (PE) pour systèmes de tuyauterie — Détermination de la résistance à la propagation lente de fissures sous un chargement cyclique — Méthode d'essai de la barre ronde fissurée

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Foreword

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The committee responsible for this document is ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, Subcommittee SC 5, *General properties of pipes, fittings and valves of plastic materials and their accessories — Test methods and basic specifications*.

This corrected version of ISO 18489:2015 incorporates the following corrections.

Formula (2) has been revised.

Formulae (A.1), (A.2) and (A.3) have been revised, as well as the corresponding explanation of symbols.

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Introduction

Knowledge about the resistance to long-term failure mechanisms as a result of crack initiation and slow crack growth (SCG) is important for the ranking and pre-selection of thermoplastic materials, especially for long-term applications such as pipes and fittings made of polyethylene. Several tests to determine the relevant failure mechanisms are available today where elevated temperatures and also the combination with stress cracking liquids are used to decrease the time frame for testing.

However, developments in modern raw materials have led to a significant increase of resistance of polyethylene to crack initiation and SCG so that testing with available methods exceeds practical time frames. Therefore, new acceleration methods, preferably at application relevant temperatures and without additional time reducing liquids, are required.

This test method achieves a significant decrease of testing time even at ambient temperatures of 23 °C. This is more relevant to the temperature range of many applications and testing at this temperature does not change the structural status of the polymer. Acceleration of material testing is achieved by the specimen geometry and the cyclic loading regime to result in completion of testing in a relatively short time.[2],[3],[4]

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Polyethylene (PE) materials for piping systems — Determination of resistance to slow crack growth under cyclic loading — Cracked Round Bar test method

1 Scope

This International Standard specifies a method to determine the resistance to slow crack growth (SCG) of polyethylene materials, pipes, and fittings. The test is applicable to samples taken from compression moulded sheet or extruded pipes and injection moulded fittings of suitable thickness.

This International Standard provides a method that is suitable for an accelerated fracture-mechanics characterization at ambient temperatures of 23 °C of different polyethylene grades, especially for PE 80 and PE 100 types for pipe applications.

NOTE This test method could be adapted for other thermoplastics materials by developing the procedure using different test parameters.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 2818, *Plastics — Preparation of test specimens by machining*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
crack length
a
depth of the crack at any time during a test measured from the specimen surface to the crack tip

Note 1 to entry: It is expressed in millimetres (mm).

3.2
cycle
N
smallest segment of a load-time or stress-time function which is repeated periodically

3.3
failure cycle number
N_f
total number of *cycles* (3.2) from the beginning until failure of the test specimen

3.4
frequency
f
number of *cycles* (3.2) within one second

Note 1 to entry: It is expressed in hertz (Hz).

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3.5
initial crack length
a_{ini}
measured depth of the crack from the specimen surface through the crack tip at the beginning of the test

Note 1 to entry: It is expressed in millimetres (mm).

3.6
initial ligament diameter
D_{ini}
inner diameter of the cylindrical specimen after notching

Note 1 to entry: It is expressed in millimetres (mm).

Note 2 to entry: It is calculated by

$$D_{ini} = D - 2 \cdot a_{ini}$$

3.7
load ratio
R
ratio of the minimum to the *maximum load* (3.9) in one *cycle* (3.2)

Note 1 to entry: It is calculated by

$$R = \frac{F_{min}}{F_{max}}$$

3.8
load range
ΔF
difference between the maximum and *minimum load* (3.10), in Newtons (N), in one *cycle* (3.2)

3.9
maximum load
F_{max}
highest value of the applied load, in Newtons (N), in one *cycle* (3.2)

3.10
minimum load
F_{min}
lowest value of the applied load, in Newtons (N), in one *cycle* (3.2)

3.11
notch distance
L_{min}
minimum distance from notch to clamping system

Note 1 to entry: It is expressed in millimetres (mm).

3.12
target initial crack length
a_{ini}^*
target depth of the crack from the specimen surface through the crack tip after notching

Note 1 to entry: It is expressed in millimetres (mm).

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3.13
target initial ligament diameter
D_{ini}*
target inner diameter of the cylindrical specimen after notching

Note 1 to entry: It is expressed in millimetres (mm).

3.14
target stress range
$\Delta\sigma_0$*
target difference between the maximum and minimum stress at the beginning of the test

Note 1 to entry: It is expressed in megapascals (MPa).

3.15
specimen diameter
D
diameter of the cylindrical specimen

Note 1 to entry: It is expressed in millimetres (mm).

3.16
specimen length
L
total length of the test specimen

Note 1 to entry: It is expressed in millimetres (mm).

3.17
stress range
$\Delta\sigma_0$
applied difference between the maximum and minimum stress after notching at the beginning of the test

Note 1 to entry: It is expressed in megapascals (MPa).

3.18
waveform
shape of the load-time curve within a single *cycle* (3.2)

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4 Principle

A cyclic tensile test with constant load range is imposed on a cylindrical specimen under suitable test conditions within the stress range where SCG is achieved. A circumferential notch is machined in the centre of the test specimen to enable crack initiation and SCG to final failure of the specimen. The number of cycles until final failure, N_f, is recorded as a function of the stress range, $\Delta\sigma_0$, at the initial crack length, a_{ini}. The specimen geometry ensures quick crack initiation and short testing times due to the high constraint and low plastics deformations along the crack tip.

NOTE Crack initiation can be monitored by the use of extensometers if required.[2]

5 Apparatus

5.1 Test machine

5.1.1 Loading system

The loading system shall be capable of imposing and recording a cyclic load varied by time between accurately defined limits (load control mode) with a specific waveform. Servo-hydraulically driven test

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machines with electronic control are generally suitable for this purpose. However, mechanically driven machines may also be used if they meet the requirements.

The cyclic load shall follow a sinusoidal waveform which is characterized by the maximum load, F_{max}, and the load ratio, R. The maximum and minimum loading values shall be constant during the entire test with an accuracy of ±1 %.

5.1.2 Load-cycle frequency

The load-cycle frequency (test frequency), f, shall not exceed 10 Hz with an accuracy of 1 %. The hysteretic heating at a frequency of 10 Hz usually causes no significant influence on N_f.[2] If extraordinary hysteretic heating at the crack tip is suspected, the frequency shall be reduced to 5 Hz or 1 Hz with an accuracy of 1 %.

5.1.3 Grips

The machine shall be equipped with grips suitable to clamp cylindrical specimens. It shall be ensured that the load distribution is in alignment with the specimen axis.

5.1.4 Temperature chamber

For testing at temperatures other than (23 ± 2) °C, the test machine shall be equipped with a suitable chamber that contains the environment and ensures complete immersion of the specimen. The chamber shall be constructed of materials which do not affect the environment and which are not affected by it. The temperature of the environment shall be controlled in order to maintain the test specimens within ±2 °C of the specified temperature.

5.2 Microscope

A microscope or an equivalent device shall be used to measure the initial crack length after the finished cyclic tests with an accuracy of 0,01 mm.

5.3 Notching apparatus

The device used shall be capable of producing a razor-sharp notch of accurately defined depth at the circumference of the cylindrical specimen. The notch tip radius produced shall be less than 10 µm. Usually, razor blades meet this requirement. A conventional lathe in combination with a razor blade tool may be suitable for this purpose. Bending of the razor blade shall be avoided and notching perpendicular to the specimen axis shall be ensured.

6 Test specimen

6.1 Specimen geometry and dimensions

The specimen configuration of the cracked round bar (CRB) specimen is shown in Figure 1. A cylindrical bar of length, L, and diameter, D, is prepared with a circumferential razor-sharp notch with a depth of a_{ini}* (target initial crack length) in the middle of the specimen resulting in a target initial ligament diameter, D_{ini}*.

Preferred specimen dimensions are L of 80 mm to 100 mm and D of 14 mm. The initial crack shall be perpendicular to the specimen axis with a target crack length of a_{ini}* of (1,50 +0,15/-0,0) mm. To avoid clamping effects at the crack tip, the notch distance, L_{min}, shall be at least 20 mm from the grips. To support specimen clamping, the specimen may be manufactured with, for example, M 14 × 1,0 metric fine thread.

NOTE Alternative dimensions can be used, but results are to be compared by the stress intensity factor range instead of the stress range (see Annex A),

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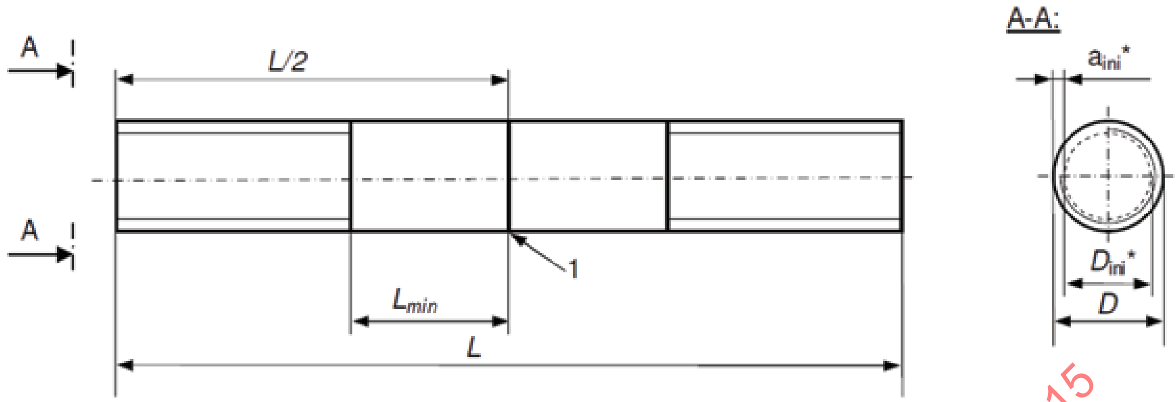

Key

L specimen length

L_{min} minimum distance between notch and clamping system

a_{ini}* target initial crack length

D_{ini}* target initial ligament diameter

D specimen diameter

1 razor-sharp notch

Figure 1 — Principal configuration of CRB test specimen with optional metric fine thread

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6.2 Test specimen preparation

The test specimens can be machined from compression moulded sheets or from pipe or fittings. Conditions for compression moulding of test specimens are given in Table 1. The use of different moulding conditions may affect the results. The specimens shall be machined in accordance with ISO 2818.

Table 1 — Conditions for compression moulding of test specimens

Thickness	Moulding temperature	Average cooling rate[a]	Preheating time[b]	Full pressure	Full-pressure time
mm	°C	°C/min	Min	MPa	Min
<10	180	15 ± 2	20	5	10
10 to 16	180	2 ± 0,5	45	10	25

a Demoulding temperature, <40 °C.

b Preheating pressure contact pressure.

6.3 Specimen notching

Specimens shall be notched at ambient temperature of (23 ± 2) °C with a notching apparatus according to 5.3. To avoid frictional heating which may lead to notch tip damage such as blunting and introduction of residual stresses, select a sufficiently low driving speed and feed. Favourable values for the specimen notching are a driving speed of 80 rpm up to 100 rpm and a razor blade feed of approximately 0,03 mm/rotation. Special attention shall be paid to notching perpendicular to the specimen axis. A razor blade shall be used for no more than 10 notches.

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6.4 Specimen conditioning

Unless otherwise specified, notched specimens shall be stored at condition 23/50, Class 2 according to ISO 291 prior to testing. Although the duration between notching and testing is not significant, it is recommended to precondition the specimens for a minimum of 24 h at this condition prior to testing.

7 Test procedure

7.1 Measurement of specimen dimensions

Before testing, the diameter, D, shall be recorded on both sides of the notch at a distance of 1 mm to 2 mm from the notch with an accuracy of at least 0,1 mm. An average of the two measurements shall be taken. Measuring the diameter directly at the notch shall be avoided.

7.2 Specimen mounting

Depending on the clamping system, the specimen shall be mounted into the test machine without inducing additional forces. Bending or twisting of the specimen shall be avoided. The distance from the grips to the notch shall be of a sufficient length, L_{min}, (see Figure 1) to avoid any clamping effects at the crack tip.

7.3 Test atmosphere

Test is preferably carried out at condition 23/50, Class 2 according to ISO 291, unless specified otherwise in a referring standard. In case of testing at elevated temperatures, the unloaded specimen shall be conditioned at the test temperature for at least 2 h after mounting in the test apparatus.

7.4 Calculation of test load

The test loads shall result in brittle failure by slow crack growth and specimens that fail in a predominantly ductile manner shall be excluded (see Figure 3). Specimens shall be tested with a load ratio, R, of 0,1 at different target stress range, $\Delta\sigma_0$*, levels between 10,5 MPa and 13,5 MPa depending on the density of the base polyethylene according to Table 2[3], unless specified otherwise in a referring standard. Target stress range of $\Delta\sigma_0$* above 13,5 MPa is to be avoided as this may result in ductile failure.

Table 2 — Typical stress ranges for cyclic CRB tests

Density of base polymer ρ kg/m^3	Target stress range $\Delta\sigma_0$* MPa
≤945	10,5 to 12,5
>945	11,5 to 13,5

A comparison of different materials is only allowed for the same specimen geometry and test parameters. If CRB specimens of different diameters to 14 mm are tested, the comparison of the results shall be made using the stress intensity factor range (ΔK_I) as described in Annex A.

At least four specimens shall be tested each at each different applied target stress range, $\Delta\sigma_0$*, regularly spread over the above specified load range. If an unknown material is tested, preliminary tests are recommended to ensure failure by slow crack growth and ductile behaviour is not occurring. The calculation of the maximum load, F_{max}, and the minimum load, F_{min}, shall be carried out according to Figure 2 and Formulae (1) to (3).

For product specifications, it should be possible to establish a single point test requirement using one load range.

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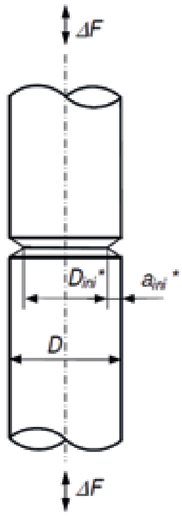

Key

ΔF load range

a_{ini}* target initial crack length

D_{ini}* target initial ligament diameter

D specimen diameter

Figure 2 — Calculation of F_{max} and F_{min} for cyclic CRB test

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$$\Delta F = F_{max} - F_{min} \tag{1}$$

$$F_{max} = \frac{D_{ini}{}^{2} \cdot \pi \cdot \Delta\sigma *}{4 \cdot (1 - R)} \tag{2}$$

$$F_{min} = F_{max} \cdot R \tag{3}$$

7.5 Load application

After mounting and conditioning of the specimen, the cyclic load shall be applied immediately with the defined load-cycle frequency without exceeding the maximum load, F_{max}. Static loading prior the test shall be avoided as this may cause creep at the notch tip of the test piece. Simultaneous to the load application, the load-cycle counter shall be started for data acquisition. The cyclic load shall be applied and recorded continuously until test piece failure.

7.6 Notch depth measurement

The accurate determination of the depth of the razor blade notch on the unloaded specimens before testing is difficult, therefore, the initial crack length, a_{ini}, shall be measured after completion of the cyclic test by analysing the created fracture surface. Different surface textures usually allow a clear distinction between the razor-sharpened notch and the fatigue crack initiated from the notch (see Figure 3). For this purpose, a microscope according to 5.2 or an equivalent device shall be used.

8 Data treatment

To evaluate the results, only specimens with predominantly brittle fracture shall be taken into account. Figure 3 shows typical microscope images of fracture surfaces of CRB specimens after testing. In both images, the relatively smooth ring-like surface from the initial notch, a_{ini}, is recognizable, which is a result of the specimen notching with a razor blade. The rough surface structure of the ductile failure

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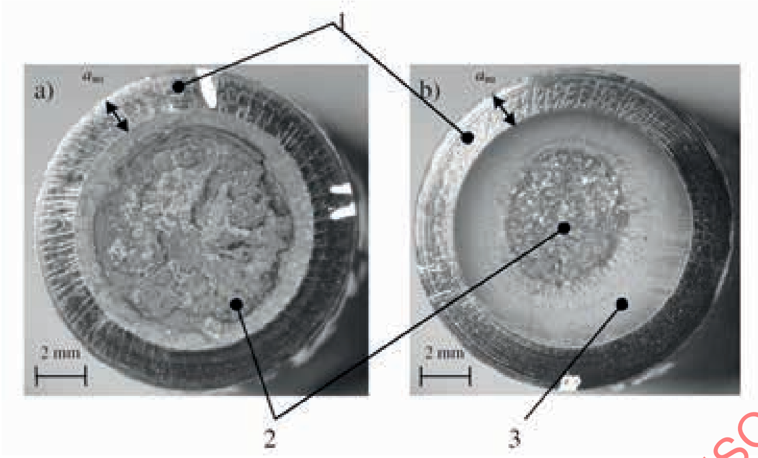
area [see Figure 3 a)] is significantly different from the brittle failure area [see Figure 3 b)]. In the second case, a relatively smooth surface typical of brittle failure was created by slow crack growth which changes to ductile at small ligament diameter. The specimen with ductile failure only [see Figure 3 a)] was tested at too high stress levels and shall be rejected for data evaluation.



Key

1 razor blade notch

2 area with ductile failure

3 area with brittle failure

Figure 3 — Typical fracture surfaces of CRB specimens after testing

To determine the stress range, $\Delta\sigma 0$, a calculation to correct the target stress range, $\Delta\sigma 0^*$, with a_{ini} measured at the fracture surface shall be carried out using Formula (4).

$$\Delta\sigma_0 = \frac{4 \cdot \Delta F}{\left(D - 2a_{ini}\right)^2 \cdot \pi} \qquad (4)$$

The failure cycle numbers, N_f, as a function of the applied stress range, $\Delta\sigma_0$, shall be displayed in a double logarithmic (log-log) diagram. In this diagram, the failure curve shall result in a linear correlation with an accuracy within at least 98 % confidence interval. If no clear linear correlation is recognizable, additional tests with suitable $\Delta\sigma_0$ shall be executed. The diagram shall also include information about the specimen configuration, test temperature, load-cycle frequency, and, R, ratio. An example of a diagram of $\Delta\sigma_0$ versus N_f is given in Figure 4.

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