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Polyethylene (PE) materials for piping systems — Determination of Strain Hardening Modulus in relation to slow crack growth — Test method

Matériaux polyéthylène (PE) pour systèmes de canalisations — Détermination du module d'écrouissage en relation avec la propagation lente de fissures — Méthode d'essai

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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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*.

Introduction

Resistance to slow crack growth is related in general to the lifetime of polyethylene and thus, the lifetime of polyethylene products, e.g. pipes and fittings. The slow crack growth behaviour can be regarded as a combination of yield stress and the capability of disentanglement of tie molecules as reported by Kramer and Brown.[3],[6],[7] The disentanglement capability of a polymer will determine its resistance against slow crack growth.

The strain hardening modulus of a polymer is a measure of the disentanglement capability of the tie molecules of this polymer and is an intrinsic property. The strain hardening modulus of polyethylene is obtained from a stress-strain curve above the natural draw ratio. The stress-strain curve of a compression moulded sample is relatively easily obtained using a tensile test apparatus equipped with an optical extensometer. The test time of the strain hardening modulus is a consequence of the speed of tensile testing and is therefore constant for all measurements and independent of the slow crack growth property of the tested material itself.

The strain hardening modulus value allows discrimination between materials. It has been demonstrated that the strain hardening modulus corresponds very well with several environmental stress cracking test methods for high density polyethylene.[4],[5],[8]

Polyethylene (PE) materials for piping systems — Determination of Strain Hardening Modulus in relation to slow crack growth — Test method

1 Scope

This International Standard specifies a method for the determination of the strain hardening modulus which is used as a measure for the resistance to slow crack growth of polyethylene.

The strain hardening modulus is obtained from stress-strain curves on compression moulded samples. This International Standard describes how such measurement is performed and how the strain hardening modulus shall be determined from such a curve. Details of the required equipment, precision, and sample preparation for the generation of meaningful data are given.

This International Standard provides a method that is valid for all types of polyethylene, independent from the manufacturing technology, comonomer, catalyst type, that are used for pipes and fittings applications.

NOTE This method could be developed for materials for other applications.

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 527-1, *Plastics — Determination of tensile properties — Part 1: General principles*

ISO 7500-1, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 9513, *Metallic materials — Calibration of extensometer systems used in uniaxial testing*

3 Terms and definitions

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

NOTE The symbols and their terms and definitions, as given below, are in line with ISO 527-1 and/or ISO 16241.

3.1
gauge length
l_0
initial distance between the gauge marks on the central part of the test specimen

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

3.2
thickness
h
smaller initial dimension of the rectangular cross-section in the central part of a test specimen

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

3.3
width
b
larger initial dimension of the rectangular cross-section in the central part of a test specimen

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

3.4
test speed
v
rate of separation of the gripping jaws

Note 1 to entry: It is expressed in millimetres per minute (mm/min).

3.5
length
l
distance between the gauge marks on the central part of the test specimen at any given moment

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

3.6
stress
σ
normal force per unit area of the original cross-section within the *gauge length* (3.1)

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

3.7
stress at yield
σ_y
stress at the *strain at yield* (3.10)

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

3.8
true stress
σ_{true}
draw ratio (3.11) multiplied with the normal force per unit area of the original cross-section within the *gauge length* (3.1)

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

3.9
strain
ε
increase in *length* (3.5) per unit original length of the gauge

Note 1 to entry: It is expressed as a dimensionless ratio, or as a percentage (%).

3.10
strain at yield
yield strain
ε_y
first occurrence in a tensile test of strain increase without a stress increase

Note 1 to entry: It is expressed as a dimensionless ratio, or as a percentage (%).

3.11
draw ratio
λ
length (3.5) per unit original length of the gauge

Note 1 to entry: It is expressed as a dimensionless ratio, or as a percentage (%).

3.12
strain hardening modulus
<G_p>
slope of the Neo-Hookean constitutive model between a true *strain* (3.9) of 8 and up to the point of maximum *stress* (3.6), but not above 12

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

4 Principle

Test specimens cut from compression moulded sheet are subjected to a tensile test at 80 °C. The stress-strain curve is obtained sufficiently beyond the natural draw ratio. The strain hardening modulus is determined from the slope of this curve in the area after the natural draw ratio.

5 Apparatus

5.1 Tensile-testing machine, complying with ISO 527-1 and capable of maintaining a test speed of (20 ± 2) mm/min.

5.1.1 Load cell, which shall comply with Class 1, as defined in ISO 7500-1. The load cell shall be able to accurately measure forces in the range of 40 N for 0,30 mm thick samples and 120 N for 1,0 mm thick samples.

5.1.2 Extensometer, which shall comply with Class 1, as defined in ISO 9513. The traverse displacement shall not be used as a measure of strain. For a 0,30 mm thick specimen, a non-contact extensometer is preferred.

5.1.3 Temperature chamber, to control the temperature at (80 ± 1) °C.

5.1.4 Clamps, remote operation to enable closing and opening without the need to open the temperature chamber is recommended.

5.2 Devices for measuring the thickness and width of the test specimens.

5.2.1 Thickness shall be measured with a device with an accuracy of 0,005 mm and a device with a contact dimension of less than the width of the parallel specimen section (4,0 mm).

5.2.2 Width shall be measured with a device with an accuracy of 0,01 mm. Care shall be taken not to change the width of the test specimen by deformation. Therefore, it is recommended to use a microscope to measure width to avoid deforming the test specimen.

5.3 Punch knife, in accordance with 6.1 shall be used.

6 Test specimens

6.1 Test specimen geometry and dimensions

The test specimen geometry as shown in Figure 1 shall be used. A large clamping area of the specimen to avoid slippage in the clamps is required to accurately measure the strain hardening regime.

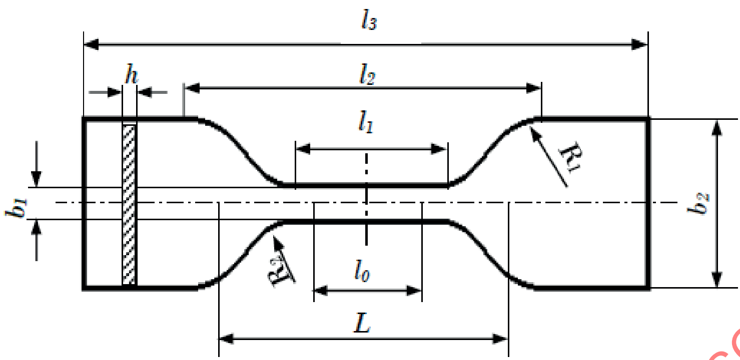


Figure 1 — Test specimen

Table 1 — Dimensions of test specimens

Dimension		Size mm
L	Start length between clamps	30,0 ± 0,5
l_0	Gauge length	12,5 ± 0,1
l_1	Length of narrow parallel sided portion	16,0 ± 1,0
l_2	Length between the parallel portions of the clamp area	46 ± 1,0
l_3	Minimum overall length[a]	70
R_1	Radius	10,0 ± 0,5
R_2	Radius	8,0 ± 0,5
b_1	Width at narrow parallel sided portion	4,0 ± 0,1
b_2	Width at ends	20,0 ± 1,0
h	Thickness	0,30 + 0,05/-0,03 or 1,0 ± 0,1

[a] A greater overall length may be necessary to ensure that only the wide end tabs come into contact with the machine grips, thus avoiding “shoulder breaks”.

6.2 Test specimen preparation

The PE granules shall be compression moulded into sheets of 0,30 mm or 1,0 mm thickness according to the moulding conditions given in Table 2, but in case of dispute, the 0,30 mm thickness shall be used.

Table 2 — Conditions for compression moulding of sheet

Thickness mm	Moulding temperature °C	Average cooling rate[a] °C/min	Preheating time[b] min	Full pressure MPa	Full-pressure time min
0,30 or 1,0	180	15 ± 2	5 to 15	5	5 ± 1

[a] Demoulding temperature <40 °C.

[b] Preheating pressure to equal contact pressure.

After compression moulding, annealing of the sheets shall be carried out by conditioning the sheet for 1 h in an oven at a temperature of (120 ± 2) °C and slowly cooled down to room temperature by switching off the closed temperature chamber with an average cooling rate of less than 2 °C/min. During this operation, free movement of the sheets shall be allowed.

NOTE As cooling will take a long time, it is advised to anneal the samples at the end of the day and allow cooling of the samples overnight in the temperature chamber.

Five test pieces shall be punched from the pressed sheets using a die for the specimen geometry specified (see 6.1). The punching procedure shall be carried out in such a way that no deformation, crazes, or other irregularities are present on the test pieces.

The thickness of the samples shall be measured at three points of the parallel area of the specimen. The lowest measured value for thickness of these measurements shall be used for data analysis (see Clause 8).

Apply the gauge marks on each test specimen equidistant from the midpoint keeping the gauge length, l_0.

7 Test procedure

— Measure the exact dimensions of width (b) with an accuracy of 0,01 mm and of thickness (h) with an accuracy of 0,005 mm of each individual test specimen.

— Condition the test specimens for at least 30 min in the temperature chamber at a temperature of (80 ± 1) °C prior to starting the test.

— Clamp the test piece in the upper clamp. The clamps shall be chosen to avoid damage and slippage of the test piece.

— Close the temperature chamber.

— After reaching the temperature of (80 ± 1) °C, clamp the test piece with the lower clamp

— The sample shall remain between the clamps for at least 1 min before the load is applied and measurement starts.

— Apply a pre-stress of 0,4 MPa with a speed of 5 mm/min.

— During the test, measure the load and elongation sustained by the specimen.

— Extend the test specimen at a constant traverse speed of 20 mm/min Data points shall be collected from λ = 8,0 until λ = 12,0 or breakage for analysis of the data. If the sample breaks before λ = 8,5, the sample shall be rejected.

At least five specimens shall be tested. If slippage of the test bar takes place in the clamps, the test result shall be discarded and the test repeated.

8 Data analysis

The draw ratio, λ, is calculated from the length, l, and the gauge length, l_0, as shown by Formula (1).

$$\lambda = \frac{l}{l_0} = 1 + \frac{\Delta l}{l_0} \qquad (1)$$

where

Δl is the increase in the specimen length between the gauge marks.

The true stress, σ_{true}, is calculated according to Formula (2) which is derived on the assumption of conservation of volume between the gauge marks.

$$\sigma_{true} = \lambda \cdot \frac{F}{A} \qquad (2)$$

where

F is the measured force (N).

It is important that the initial cross section, A, shall be determined for each individual test bar.

The Neo-Hookean constitutive model is represented by Formula (3) and is used to fit and extrapolate the data from which $<G_p>$ (MPa) for $8 < \lambda < 12$ is calculated (see Annex A).

$$\sigma_{true} = \frac{<G_p>}{20} \cdot \left(\lambda^2 - \frac{1}{\lambda} \right) + C \qquad (3)$$

where

C is a mathematical parameter of the constitutive model describing the yield stress extrapolated to $\lambda = 0$.

Accuracy of fit of data (R^2) greater than 0,9 shall be achieved.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 18488:2015;

b) complete identification of the material tested including type, source, manufacturer's designation, and test piece (manufacturer, method of manufacture, production date);

c) minimum thickness of each test specimen;

d) mean width of each test specimen;

e) number of the test specimen tested;

f) the stress at yield for each test specimen;

g) the mean value and the standard deviation of the stress at yield for the total number of test specimens tested;

h) calculated strain hardening modulus for each test specimen;

i) the mean value and the variation coefficient of the strain hardening modulus for the total number of test specimens tested;

j) test temperature and any recorded variation;

k) statement as to whether any test specimens have been rejected and replaced, and, if so, the reasons and reasons for testing non-conforming specimens;

l) any factors which may have affected the results such as any incidents or any operating details not specified in this International Standard;

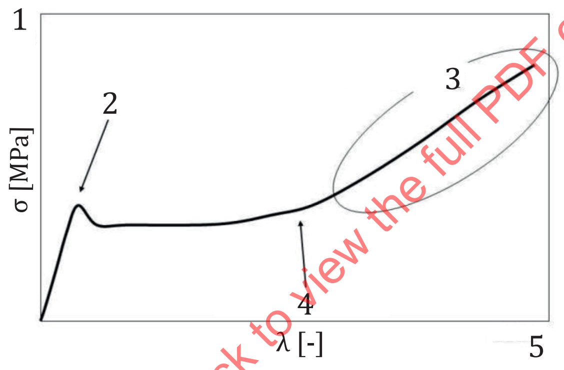
m) date of measurement.

Annex A
(informative)

Neo-Hookean constitutive model

A.1 General

The important role of the underlying molecular entanglement network has been described by Kramer and Brown[3],[6],[7] and is reflected by the strain hardening behaviour. It is proposed[4],[8] that the amount of strain hardening in a tensile test is a measure of fibril deformation resistance. The strain hardening modulus is obtained from a stress-strain curve above the natural draw ratio, notably at elevated temperature (80 °C). To that end, the α-transition of polyethylene is adopted to ascertain this condition. The strain hardening modulus, <G_p>, is determined using a Neo-Hookean constitutive model (see Clause 8).



Key

1 stress
2 yield
3 strain hardening
4 natural draw ratio
5 strain

Figure A.1 — Stress-strain curve

