INTERNATIONAL STANDARD

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Plastics — Determination of resistance to environmental stress cracking (ESC) —

Part 6: Slow strain rate method

Plastiques — Détermination de la fissuration sous contrainte dans un environnement donné (ESC) —

Partie 6: Méthode à vitesse de déformation lente

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22088-6 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

ISO 22088 consists of the following parts, under the general title *Plastics — Determination of resistance to environmental stress cracking (ESC)*:

— *Part 1: General guidance*

— *Part 2: Constant tensile load method* (replacement of ISO 6252:1992)

— *Part 3: Bent strip method* (replacement of ISO 4599:1986)

— *Part 4: Ball or pin impression method* (replacement of ISO 4600:1992)

— *Part 5: Constant tensile deformation method* (new test method)

— *Part 6: Slow strain rate method* (new test method)

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Plastics — Determination of resistance to environmental stress cracking (ESC) —

Part 6:
Slow strain rate method

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1 Scope

This part of ISO 22088 describes a procedure for assessing the environmental stress cracking (ESC) susceptibility of polymeric materials in chemical environments by slowly increasing the strain applied to a tensile specimen at a constant rate.

It is applicable to test specimens prepared by moulding and/or machining and can be used to assess the relative ESC susceptibility of a material exposed to different environments or the relative ESC susceptibility of different plastics exposed to a specific environment.

This is essentially a ranking test and is not intended for the provision of design data.

The principle advantage of the test compared with the test methods described in Parts 2 to 5 of ISO 22088 is the rapidity with which the ESC susceptibility of a particular polymer/environment combination can be assessed.

2 Normative references

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

ISO 527-2, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics*

ISO 22088-1, *Plastics — Determination of resistance to environmental stress cracking (ESC) — Part 1: General guidance*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 22088-1 and the following apply.

3.1
crosshead displacement
CHD
distance the crosshead has moved from the start of the test

3.2
crosshead speed
CHS
distance travelled by the crosshead, CHD, divided by the time from the start of the test

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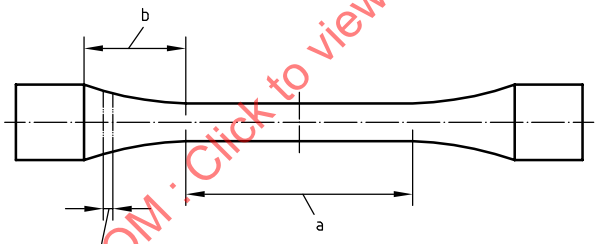
3.3
length of parallel-sided section of specimen
l_1
length of the narrow parallel-sided section in the middle of the specimen (see Figure 1)

3.4
area of parallel-sided section
A_1
cross-sectional area of the narrow parallel-sided section of the specimen (see Figure 1)

3.5
length of tapered region
l_2
overall length of the tapered/non-parallel-sided region at one end of the specimen (see Figure 1)

3.6
length increment
Δl_2
length of the sections into which the tapered/non-parallel-sided regions are divided in order to calculate the effective gauge length (see Figure 1)

3.7
area of incremental section in tapered section
A_2
average cross-sectional area of one of the incremental sections into which the tapered/non-parallel-sided regions of the specimen are divided (see Figure 1)



a Narrow parallel-sided section of the specimen, of length l_1 and cross-sectional area A_1.

b Tapered/non-parallel-sided region of the specimen, of length l_2.

c Incremental sections (of no more than 1 mm in length) into which the tapered region is divided, of length Δl_2 and cross-sectional area A_2.

Figure 1 — Schematic diagram of the specimen showing the relevant dimensions

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3.8
effective gauge length
l_0
length of the specimen under strain, taking into account the contributions made by both the narrow parallel-sided section and the tapered sections at each end of the specimen

NOTE The grips are assumed to extend up to the beginning of the tapered region of the specimen. If gripped further back, allowance for the displacement in the wider parallel-sided section of the specimen will have to be made. The effective gauge length is given by:

$$l_0 = \left(l_1 + 2A_1 \sum \frac{\Delta l_2}{A_2} \right)$$

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3.9
stress
σ
force measured by the load cell divided by the initial cross-sectional area, A_1

3.10
strain
ε
distance, d, the crosshead has moved during the test, divided by the effective gauge length, l_0 (see Annex A)

3.11
strain rate
$\dot{\varepsilon}$
strain, ε, in the specimen divided by the time from the start of the test

4 Principle

The test involves subjecting a specimen to an increasing strain at a constant crosshead displacement rate while it is exposed to a specified test medium. The tests are conducted under tension at relatively low strain rates to enhance the influence of the test medium on the specimen. The development of crazes causes the strain to be taken up locally at the crazes such that the stress is reduced compared to an inert environment.

The focus of the test is to identify craze initiation, which is associated with the departure of the stress-strain curve in the test medium from that in air (see Figure 2). The departure stress or departure strain tends to be a very repeatable and reproducible parameter but the time to failure, if indeed the specimen fails within the range of displacement of the machine, can be highly variable and does not provide a useful basis for ranking the performance of plastics exposed to different fluids.

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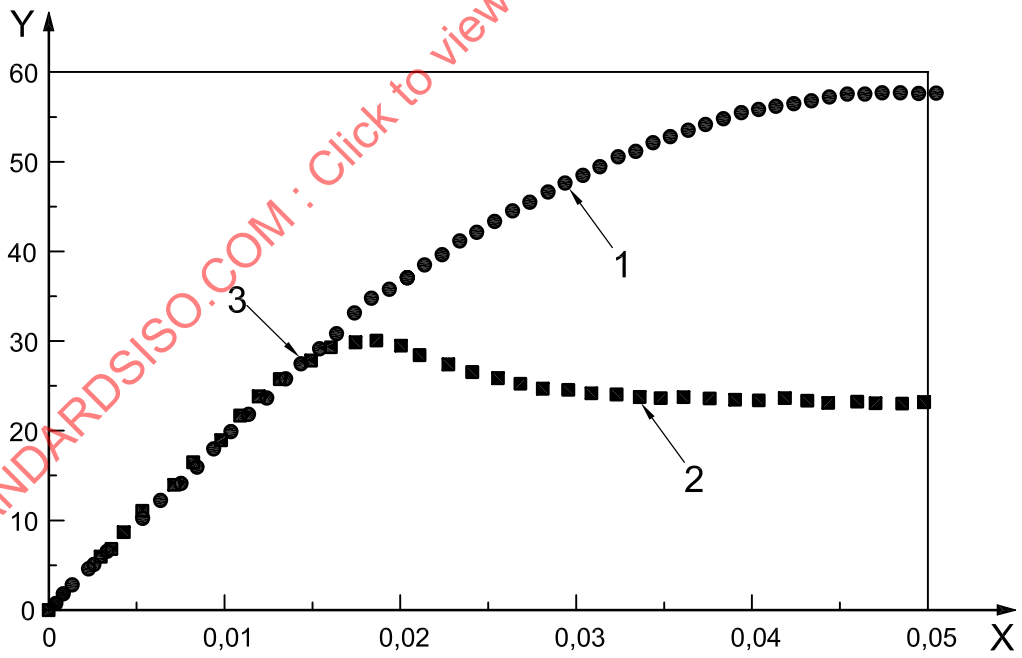

Key
X strain
Y stress (MPa)
1 specimen tested in air
2 specimen tested in test medium
3 departure point

Figure 2 — Typical stress-strain plot showing difference in stress-strain profile for material exposed in air and in the test medium

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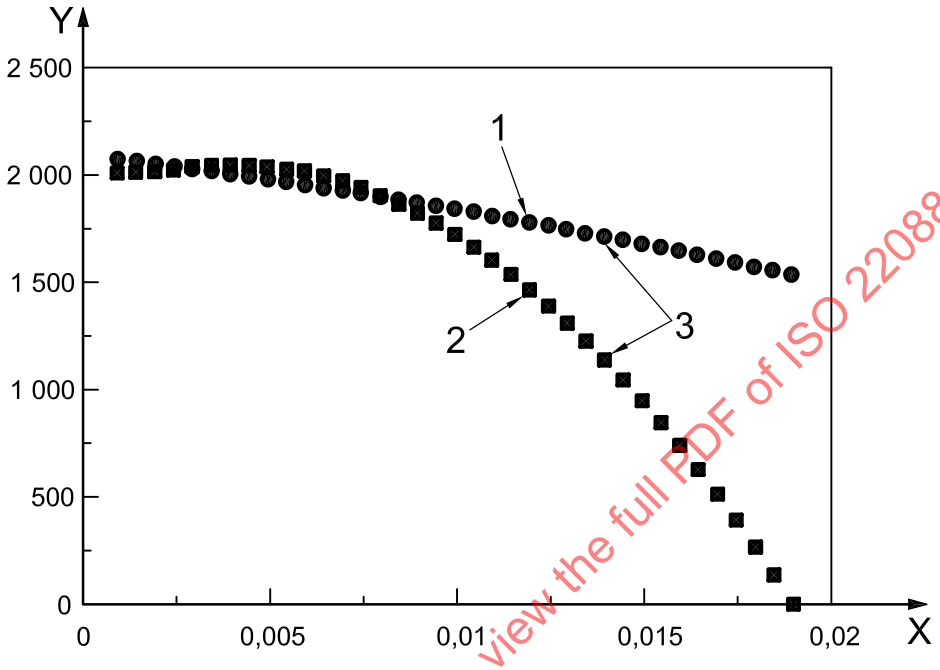
The definition of departure depends on the perspective by which the stress-strain curves are viewed and the "noise" level of the parameters. To avoid subjective judgement, and based on extensive measurements of a wide range of plastic-fluid pairs [1] to [4], departure has been defined as occurring when the derivative of the stress-strain curve (see 9.3) obtained in the test medium falls to 75 % of the derivative of the curve obtained in air. This accounts better for those systems for which initial stiffening occurs due to environment exposure (see Figure 3).



Key

X strain
Y tangent modulus (MPa)
1 specimen tested in air
2 specimen tested in test medium
3 departure point

Figure 3 — Typical plot showing the tangent modulus of the stress-strain curves as a function of strain (departure is defined as the point at which the derivative of the curve obtained in the test medium falls to 75 % of the derivative of the curve obtained in air)

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5 Apparatus

5.1 Tensile-testing machine, capable of producing a constant displacement rate, repeatable to ± 2 %. The initial strain rate that has been used most frequently in the slow strain rate testing of plastics is $9 \times 10^{-6}\ s^{-1}$. Care shall be taken to ensure that the apparatus subjects the specimens only to forces parallel to their longitudinal axis and not to bending or twisting forces.

The load shall be measured using a load cell accurate to 1 % and crosshead displacement shall be measured using a displacement transducer accurate to 0,4 %.

The load and displacement shall be recorded throughout the test, at intervals of not more than 10 min, using a data-logger.

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5.2 **Grips with ripple contact**, for gripping the specimens in such a way that no slippage occurs during the test.

5.3 The specimen and grips are enclosed in a **test chamber** which shall be inert to the extent that it has no influence on the test fluid and hence the results.

6 Conditioning and test conditions

6.1 Conditioning

Unless otherwise agreed by the interested parties, the specimens shall be stored under controlled conditions for at least 24 h at (23 ± 2) °C and (50 ± 10) % relative humidity.

6.2 Test temperature

Unless otherwise agreed by the interested parties, the temperature shall be maintained at (23 ± 2) °C.

6.3 Test medium

The test medium used shall represent that used in service or be prepared from analytical reagent grade chemicals.

7 Test specimens

7.1 Test specimens shall be produced using the dimensions quoted for the type 1BA tensile specimens in ISO 527-2, unless there are specific reasons for doing otherwise.

7.2 Unless otherwise agreed by the interested parties, the machined edges of the specimen shall be dry-ground progressively using 400 grit, 600 grit, 800 grit and 1 200 grit abrasive papers.

7.3 Prior to testing, the specimens shall be cleaned in an ultrasonic bath with distilled water for 1 min and then gently dried with a tissue.

7.4 Specimens shall be visually inspected for evidence of damage before they are used. If damage, such as scratch marks or embedded grit, is observed, the specimen shall not be used unless the surface is reground.

7.5 Measure the width and thickness of the parallel-sided gauge length of the specimen using a micrometer or travelling microscope accurate to ± 0,025 mm. Take three readings along the gauge length and use the minimum values of thickness and width to calculate the cross-sectional area of the gauge length.

8 Procedure

8.1 Set the crosshead speed on the tensile-testing machine to obtain the required strain rate in the specimen. The initial strain rate that has been used most frequently in the slow strain rate testing of plastics is 9×10^{-6} s^{-1}.

8.2 The required crosshead speed (CHS) can be calculated as follows:

$$\mathrm{CHS} = \dot{\varepsilon} l_0$$

where

$\dot{\varepsilon}$ is the strain rate;

l_0 is the gauge length as defined in Annex A.

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8.3 Insert the specimen in the grips of the tensile-testing machine, ensuring that the specimen is axially aligned and completely enclosed within the environmental chamber.

8.4 Load the specimen rapidly (< 30 s) to a small load (< 100 N) just sufficient to take up any slack in the machine.

8.5 Fill the environmental chamber with the chemical under investigation to immerse the specimen.

8.6 Record the initial load and displacement, then switch on the data-logger.

8.7 Start the drive motor on the tensile-testing machine.

8.8 Allow the test to run until the specimen fractures or necks.

8.9 Inspect the specimen to ensure that it has not slipped or broken in the grips. If slippage or fracture has occurred within the grips, record this and discard the results.

8.10 Unless otherwise agreed by the interested parties, conduct at least one repeat test.

8.11 To provide reference data, carry out the test in air in the same way as in the test medium. Unless otherwise agreed by the interested parties, the relative humidity of the air shall be (50 ± 10) %.

9 Expression of results

9.1 The departure stress is determined by comparing the stress-strain curve in the environment with that in air.

9.2 Fit both stress-strain curves with third-order polynomials to the peak stress. If the correlation factor, R^2, is < 0,99, use a higher-order polynomial. This provides a convenient method to fit the data:

$$\sigma = b\varepsilon + c\varepsilon^2 + d\varepsilon^3$$

9.3 Determine the derivative to obtain the tangent modulus of each polynomial at each stress:

$$\frac{\mathrm{d}\sigma}{\mathrm{d}\varepsilon} = b + 2c\varepsilon + 3d\varepsilon^2$$

9.4 Divide the derivatives (tangent moduli) obtained from the test conducted in the chemical environment by the derivatives obtained in air at each stress level. The departure stress is then defined as the stress at which this value equals to 0,75.

$$\left(\frac{\mathrm{d}\sigma}{\mathrm{d}\varepsilon}\right)_{\text{chem}} \Big/ \left(\frac{\mathrm{d}\sigma}{\mathrm{d}\varepsilon}\right)_{\text{air}} = 0{,}75$$

9.5 Record the value of the departure stress and all coefficients obtained for the polynomial fitted to the data.

9.6 The aggressivity of fluids or combinations of fluids on the polymer can be compared by normalizing the value of the departure stress relative to the maximum stress in air to obtain an ESC index:

$$\text{ESC index} = \frac{\text{Departure stress in environment}}{\text{Maximum stress in air}}$$

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10 Test report

The test report shall include the following information:

a) a full description of the test material from which the specimens were taken, including composition, processing/service history and product type;

b) the orientation, type and size of the test specimens and their surface preparation;

c) the test medium used, including its chemical composition, temperature and pressure where appropriate;

d) the test conditions, including the strain rate and the initial load and strain applied to the specimen;

e) the individual test results and their arithmetic mean;

f) the date of testing.

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