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Plastics — Determination of fracture toughness of films and thin sheets — Essential work of fracture (EWF) method

Plastiques — Détermination de la ténacité à la rupture des films et feuilles minces — Méthode du travail essentiel de rupture (EWF)

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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical behavior*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Fracture occurs under plane stress displaying gross ductility in many practical applications of polymeric materials in which they are used as thin sheets or films (e.g. packaging and coatings). It is inappropriate to adopt thicker test specimens, which are generally used in fracture tests, to measure the fracture toughness in such cases. Thicker test specimens suppress crack tip ductility and bring about a change in stress state which does not occur in practice. The essential work of fracture (EWF) method, described in this document, provides toughness measurement under plane stress. The method which is relatively simple is based on a suggestion by Broberg[1], further developed first by Cotterell, Reddel and Mai for metals[2],[3] and then by a series of workers[4]-[9] for ductile polymers. More recent reviews on this method are given in References [10], [11], [12].

The method assumes that the overall energy associated with fracture can be partitioned into two components: the essential work necessary to create new surfaces in the so-called fracture process zone, and the non-essential work dissipated for the plastic deformation in the surrounding volume, the process zone.

The essential work of fracture has been shown to be a material property, i.e. independent of the specimen geometry, for a given sheet thickness[13],[14], when the condition of full yielding of the specimen ligament before the onset of crack propagation is fulfilled. In this case, the essential work of fracture is a parameter that gives an intrinsic material property dependent only on thickness and therefore useful in product design. However, the condition of full yielding of the ligament is usually difficult to verify without specific instrumentation, not commonly available in every laboratory.

Even if this condition is not fulfilled, the EWF test method can still be applied to determine the essential work and non-essential work of the fracture energy, which are repeatable and reproducible parameters useful in the development of new materials, in quality control and interlaboratory comparisons.

This document describes the EWF method independently of the verification of the full ligament yielding condition.

Plastics — Determination of fracture toughness of films and thin sheets — Essential work of fracture (EWF) method

1 Scope

1.1 This document specifies the principles and the method for determining the fracture toughness of polymeric films and thin sheets in the crack opening mode (mode I) under plane stress conditions. The essential work of fracture (EWF) method is based on the use of double edge notched tensile (DENT) specimens.

1.2 The method is suitable for use with films or thin sheets, of thickness not greater than 1 mm, made of ductile polymeric materials, in which fracture propagation is stable (crack growth is always driven by the external applied force). If, at any time during the test, brittle fracture occurs, with fast crack propagation driven by the elastic energy stored in the specimen, the sample is not suitable for the application of the present test method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dates 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*

ISO 4593, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning*

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

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

ISO 16012, *Plastics — Determination of linear dimensions of test specimens*

3 Terms, definitions and symbols

3.1 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1.1
initial distance between the grips
$\boldsymbol{L_g}$
distance between the grips before the beginning of the test

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

Note 2 to entry: See Figure 1.

3.1.2
gauge length
$\boldsymbol{L_0}$
initial distance between the grips L_g (3.1.1) when the displacement is measured by the change in the distance between the grips during the test

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

3.1.3
extensometer gauge length
$\boldsymbol{L_{0e}}$
gauge length (3.1.2), set equal to the ligament length *b*, when the displacement is measured by an extensometer

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

3.1.4
displacement
x
increase in the *gauge length* L_0 (3.1.2), or in the *extensometer gauge length* L_{0e} (3.1.3), occurring from the beginning of the test

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

3.1.5
test speed
v
rate of separation of the gripping jaws

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

3.1.6
overall fracture energy
$\boldsymbol{W_f}$
energy measured by the area under the force-displacement curves

Note 1 to entry: It is expressed in millijoules (mJ).

3.1.7
specific fracture energy
$\boldsymbol{w_{sf}}$
ratio of the *overall fracture energy*, W_f (3.1.6) to the minimum cross-section area of the specimen

Note 1 to entry: The minimum cross-section area of the specimen is given by the ligament length, *b*, times the thickness, *h*.

$$w_{sf} = W_f / hb$$

Note 2 to entry: It is expressed in kilojoules per square metre (kJ/m^2).

3.1.8
shape factor
β
dimensionless geometrical factor accounting for the shape of the plastically deformed zone around the fracture zone, the volume of which is proportional to hb^2

3.1.9
essential work of fracture
w_e
specific energy to create new surface

Note 1 to entry: It is expressed in kilojoules per square metre (kJ/m^2).

3.1.10
non-essential work of fracture
w_p
energy per unit volume dissipated for plastic deformation in the volume around the fracture zone

Note 1 to entry: It is expressed in megajoules per cubic metre (MJ/m^3).

3.1.11
maximum stress
σ_{max}
maximum force, F_{max} divided by the minimum cross-section area of the specimen given by the ligament length, b, times the thickness, h

$$\sigma_{max} = F_{max}/hb$$

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

3.1.12
average maximum stress
σ_m
average of the maximum stress, σ_{max} (3.1.11) values obtained on the 25 specimens used for the essential work of fracture, w_e (3.1.9) determination

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

3.2 Symbols

h	specimen thickness, expressed in millimetres (mm)	See Figure 1.
B	specimen width, expressed in millimetres (mm)	See Figure 1.
b	un-cracked ligament length, expressed in millimetres (mm)	See Figure 1.
L	specimen length, expressed in millimetres (mm)	See Figure 1.
W_s	energy necessary to create new surfaces, expressed in millijoules (mJ)	
W_{pl}	energy dissipated for plastic deformation in the volume around the fracture zone, expressed in millijoules (mJ)	
F	force	
F_{max}	maximum force, expressed in Newton (N)	
x_{Fmax}	displacement at maximum force, expressed in millimetres (mm)	

4 Principle

The principle of the experimental technique is to prepare a series of double edge-notched tensile specimens (see Figure 1) having the same thickness (h), width (B) and length (L) and varying ligament length (b). Specimens are extended along their major longitudinal axis at constant displacement rate up to fracture. The overall fracture energy W_f is measured from the relevant force-displacement traces. This energy is supposed to be made of two additive components: $W_f = W_s + W_{pl}$. The first component (W_s) is the energy necessary to create new surfaces and thus proportional to the fractured area (hb). It can therefore be expressed as ($W_s = w_e\, hb$), where w_e is the essential work of fracture. The second component (W_{pl}) is the energy dissipated for plastic deformation in the volume around the fracture zone. This volume can be expressed as $\beta\, hb^2$, β being a shape factor. Thus, W_{pl} can be expressed as ($W_{pl} = w_p\, \beta\, hb^2$) where w_p is the non-essential work of fracture (i.e. the energy per unit volume dissipated for plastic deformation). Accordingly, the specific energy to fracture ($w_{sf} = W_f\,/hb$) can be written as shown in Formula (1):

$$w_{sf} = w_e + w_p \beta b \tag{1}$$

where the essential work of fracture, w_e, and the product βw_p shall then be determined from a least squares linear regression to w_{sf} versus b experimental data.

To obtain a valid value of w_e, some limitations shall be taken into consideration:

— plane stress conditions shall prevail: this limits the minimum acceptable ligament length;

In this document, a fixed minimum ligament of 5 mm is specified for all considered specimen thicknesses.

NOTE The requirement of plane stress conditions generally limits the minimum acceptable ligament length to at least 5 times the thickness (see Reference [12]). In this document, for the maximum considered thickness of 1 mm (see 1.2), the requirement gives a minimum ligament length of 5 mm. For thinner films smaller ligament lengths can be made, however this gives rise to difficulties in specimen preparation and handling. Therefore, a fixed value of 5 mm for the minimum ligament length b_{min} is adopted in the present document.

— no edge effects: this condition limits the minimum notch length. This limits in turn the maximum ligament length;

— full yielding of the specimen ligament before crack onset: this last requirement ensures that the fracture mechanism is the same irrespective of ligament length and that w_e is a material property, i.e. independent of the specimen geometry, for a given sheet thickness. However, as already stated in the Introduction, this document will not consider the verification of this condition. If the condition is not verified, w_e is not a geometry independent material property but, nevertheless, the method provides a useful, repeatable and reproducible characterization of fracture toughness. This last limitation, therefore, will not be taken into consideration in this document.

5 Apparatus

The testing machine shall be in accordance with ISO 7500-1 and ISO 9513, and shall meet the specifications given in 5.1 and in 5.2.

5.1 Force measurement system, in accordance with class 1 as defined in ISO 7500-1 in the relevant range of forces.

5.2 Extensometer, in accordance with ISO 9513, class 1. The accuracy of this class shall be attained in the strain range over which measurements are being made.

For the measurement of the displacement, the use of an extensometer is preferred. Non-contact extensometers or low drag-force contact extensometers can be used. If low drag-force contact extensometers are used, ensure that the force applied to the specimen by the extensometer in the test direction does not exceed 2 % of the maximum force F_{max} measured on the specimen having the smallest ligament length b (see 5.4 and 6.2.3).

When using an extensometer, the gauge length L_{0e} shall be set equal, for each specimen, to the relevant nominal value of the ligament length b (see 6.2.3) and shall be perpendicular to the ligament plane; its centre shall correspond with the centre of the specimen.

If suitable extensometers are not available, the displacement shall be measured by the change in the distance between the grips during the test (grip separation). The initial distance between the grips L_g shall correspond, for all the specimens, to the gauge length L_0 (see 3.1.2 and 3.1.3).

Extension measurements using the crosshead displacement shall be corrected for the compliance of the machine. If the machine is equipped with built in routines for compliance correction, these shall be applied.

When the displacement is measured by the change in distance between the grips, the overall fracture energy W_f includes both the plastic energy dissipated in the region surrounding the fracture zone and the viscoelastic energy dissipated far from the fracture zone. Instead, when using an extensometer and a gauge length L_{0e} equal to the ligament length b, only the plastic energy involved in the fracture process zone is considered in the evaluation of the overall fracture energy, W_f[15].

The value of the essential work of fracture, w_e, is not influenced by the displacement measurement method (extensometer or grip separation), but the value of the product βw_p, (the shape factor times the non-essential work of fracture) will be overestimated when the displacement is measured by the change in distance between the grips.

5.3 Tensile testing machine, capable of maintaining the test speed, *v*, required by the present procedure (see 7.1), i.e. 10 mm/min, with the tolerance of ±20 %.

5.4 Vernier caliper and thickness gauge

All dimensions shall be measured in accordance with ISO 16012.

Width and length of the specimens shall be measured with an accuracy of 0,05 mm.

The thickness, *h*, shall be measured by a dead weight thickness gauge according to ISO 4593.

The thickness, *h*, of each specimen shall be measured (after notching) along the ligament with an accuracy of 1 % of the nominal thickness or 0,001 mm, whichever is greater. Readings every 5 mm of ligament length shall be made and the average value shall be used as the value of the specimen thickness *h*.

The ligament length shall be measured by means of a vernier caliper, by placing the tips of the caliper jaws as close as possible to the two notch tips. The ligament length shall be measured with an accuracy of ±0,05 mm.

The lengths of the two notches, measured by means of a vernier caliper as the distance between each notch tip and the nearest specimen border, shall be equal within 0,5 mm

The two notch tips shall lie on a plane perpendicular to the longitudinal axis of the specimen. Maximum permissible deviation from perpendicularity, measured as the maximum distance between the two planes, perpendicular to the longitudinal specimen axis, each containing one of the notch tips, shall be 2 % of the ligament length *b*.

5.5 Optical microscope

Notch tip radius is required to be smaller than 10 µm (see 6.2.5). This requirement shall be checked by means of an optical microscope using a magnification of 200X or higher. Annex B (see Figure B1) gives some examples on how to perform this verification.