INTERNATIONAL STANDARD

ISO 7765-2

First edition
1994-08-15

Plastics film and sheeting — Determination of impact resistance by the free-falling dart method —

Part 2:
Instrumented puncture test

Film et feuille de plastiques — Détermination de la résistance au choc par la méthode par chute libre de projectile —

Partie 2: Essai avec appareil de perforation

Reference number
ISO 7765-2:1994(E)

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.

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.

International Standard ISO 7765-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

ISO 7765 consists of the following parts, under the general title *Plastics film and sheeting — Determination of impact resistance by the free-falling dart method*:

— *Part 1: Staircase methods*

— *Part 2: Instrumented puncture test*

Annexes A and B of this part of ISO 7765 are for information only.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland
Printed in Switzerland

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ISO 7765-2:1994(E)

Plastics film and sheeting — Determination of impact resistance by the free-falling dart method —

Part 2:
Instrumented puncture test

1 Scope

1.1 The impact-penetration test described in the two parts of this International Standard is used for the assessment of plastic films and thin sheets (hereinafter referred to as films) under an impact stress applied at right angles to the plane of the film.

1.2 Part 1 of this International Standard can be used if it is sufficient to characterize the impact behaviour of the film by an impact-failure energy. Part 2 is used if a force-deformation or a force-time diagram, recorded at practically constant velocity of the striker, is necessary for characterization of the impact behaviour. This applies if:

— measured quantities derivable only from this diagram are required or

— only a small number of test specimens are available.

1.3 The test method is applicable to films of up to 1 mm thickness and makes it possible to compare impact-penetration forces, biaxial deformabilities and energy-absorption capacities of films. Furthermore, if required, the transition region between brittle and tough behaviour of the film under the conditions of testing can be determined by varying the temperature or the penetration velocity or the relative humidity (see also annex B).

NOTE 1 For thicknesses greater than 1 mm, ISO 6603-2 should be used.

1.4 The test results are comparable only if the conditions for preparation of specimens, their thickness and surfaces, and the test conditions are identical. Comprehensive evaluation of the reaction to impact stress requires that the determinations are made as functions of deformation rate and temperature for different material variables, such as crystallinity and moisture content.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 7765. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 7765 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 4593:1993, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning.*

ISO 6603-2:1989, *Plastics — Determination of multiaxial impact behaviour of rigid plastics — Part 2: Instrumented puncture test.*

ISO 7765-1:1988, *Plastics film and sheeting — Determination of impact resistance by the free-falling dart method — Part 1: Staircase methods.*

3 Definitions

For the purposes of this part of ISO 7765, the following definitions apply.

3.1 peak force, F_M: The maximum force exerted by the striker in the direction of impact during the test (see figures 1 to 3).

3.2 deformation at peak force, s_M: The deformation in the direction of impact at the centre of the test specimen corresponding to the peak force. For materials exhibiting a peak-force plateau, the deformation is taken at the centre of the plateau (see figure 1).

3.3 energy to peak force, W_M: The area under the force-deformation curve bounded by the origin, the peak force and the deformation at peak force (see figures 1 to 3).

3.4 total penetration energy, W_T: The total energy expended in penetrating the test specimen (see figures 1 to 3).

In contrast to the instrumented puncture test applied to test specimens made of brittle plastic (see ISO 6603-2), the force-deformation diagram of this test applied to film and sheeting frequently shows a clear point of first failure (failure point) indicated by a sharp drop in the force. If this is the case, and if the interested parties agree to use this point as a characteristic criterion, the following additional definitions may be used.

3.5 failure force, F_F: The force exerted by the striker in the direction of impact, measured at the failure point (see figures 1 and 2).

3.6 failure deformation, s_F: The deformation in the direction of impact at the centre of the test specimen, measured at the failure, point (see figures 1 and 2).

3.7 failure energy, W_F: The area under the force-deformation curve bounded by the origin, the failure force and the failure deformation (see figures 1 and 2).

NOTES

2 If the force-deformation diagram as measured during the test is influenced strongly by dynamic resonance effects, a mean curve may be used to obtain the values of the parameters defined in 3.1 and 3.4. This, however, is seldom the case when plastic film is tested.

3 When comparing films of slightly different thicknesses, it is advisable to relate F_M, F_F, W_M and W_F to the thickness d of the specimen. Though the normalized values F_M/d, F_F/d, W_M/d and W_F/d do not allow a physically exact comparison between film specimens of different materials, the thickness dependence of these normalized values is negligible for similar materials (those with the same amount of crystallinity and the same orientation) provided the thicknesses do not differ by more than a factor of 1,5.

4 Principle

The test specimen is penetrated normal to its plane by a striker at a nominally uniform velocity. The resulting force-deformation or force-time diagram is electronically recorded. The test specimen is firmly clamped during the test.

The force-deformation diagram obtained in these tests shows several features of the material's bahaviour under impact. For example, the fracture may be "brittle", "ductile", "tough" or characterized by initial damage or by crack initiation and propagation. In addition, dynamic effects may be present, such as load-cell/indentor resonance, specimen resonance and initial contact/inertia peaks (see figures 1 to 3).

In all cases care must be exercised in analysing these features because the operative mechanism and the trains of inference are not yet fully established, and are the subject of continuing research.

5 Apparatus

The apparatus consists of a mechanical test device for applying the test force, instruments for measuring the force and the deformation produced, and a thickness gauge.

5.1 Test device

The essential components of the test device are the energy carrier (normally a falling mass, but a pneumatically, hydraulically or spring-driven mass or a pendulum impact-testing device may also be used), the striker, and the clamping device consisting of the test specimen support and the clamping ring (see figures 4 and 5).

The apparatus shall permit the test specimen to be punctured at the centre at a nominally constant velocity, perpendicular to the specimen surface. The force exerted on the test specimen in the direction of impact and the deformation of the specimen in the direction of impact shall be measurable or derivable (see figure 4). Equipment suitable for this are falling-dart machines, pendulums long enough for the penetration path to be regarded as approximately straight, or high-speed tensile-testing machines with suitable attachments.

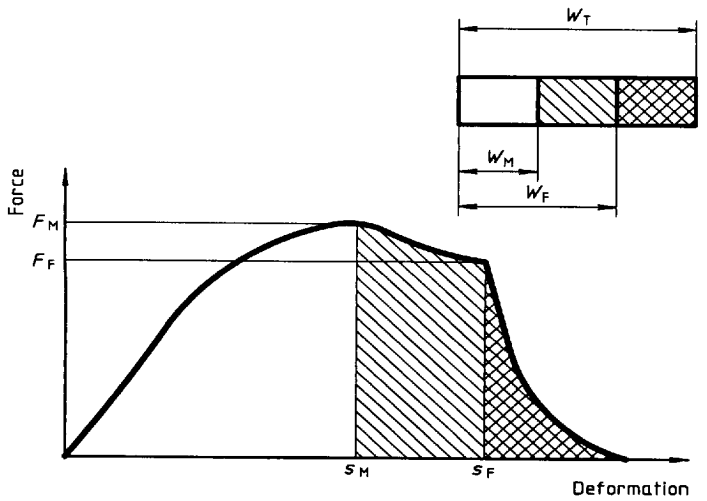


Figure 1 — Force-deformation diagram for very tough materials (schematic)

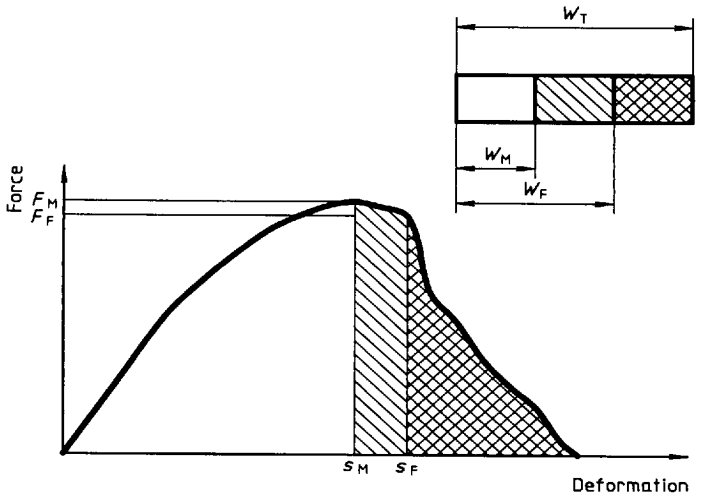


Figure 2 — Force-deformation diagram for tough materials (schematic)

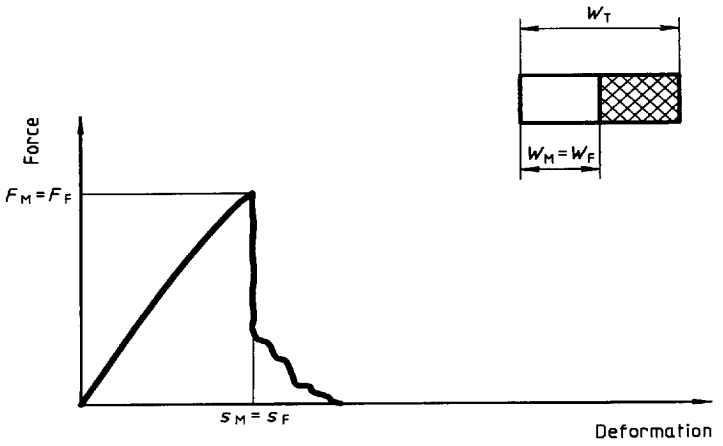
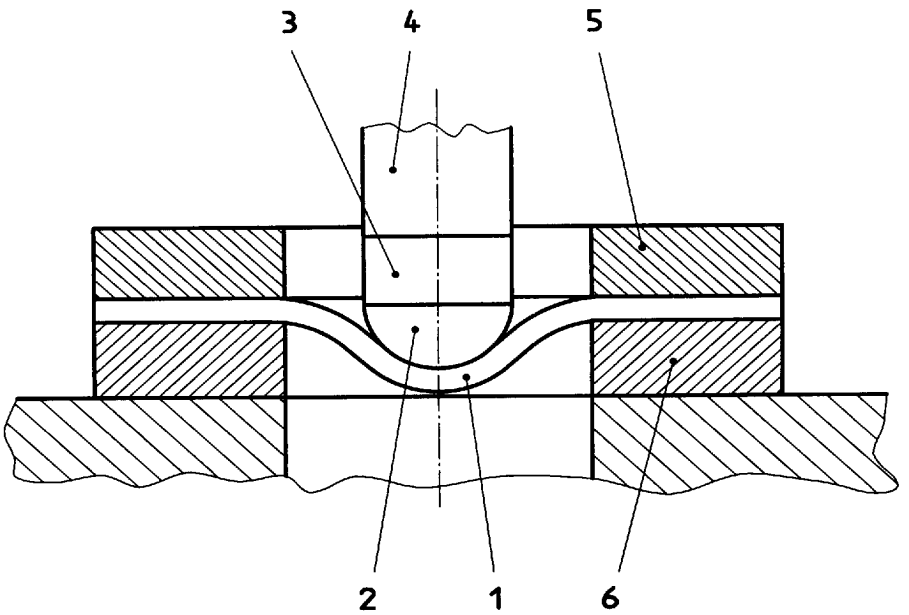


Figure 3 — Force-deformation diagram for brittle materials (schematic)



1 Test specimen
2 Hemispherical striker, diameter D_1
3 Load cell (preferred position)
4 Shaft
5 Clamping ring
6 Test-specimen support, inside diameter D_2

Figure 4 — Test apparatus (schematic)

Dimensions in millimetres

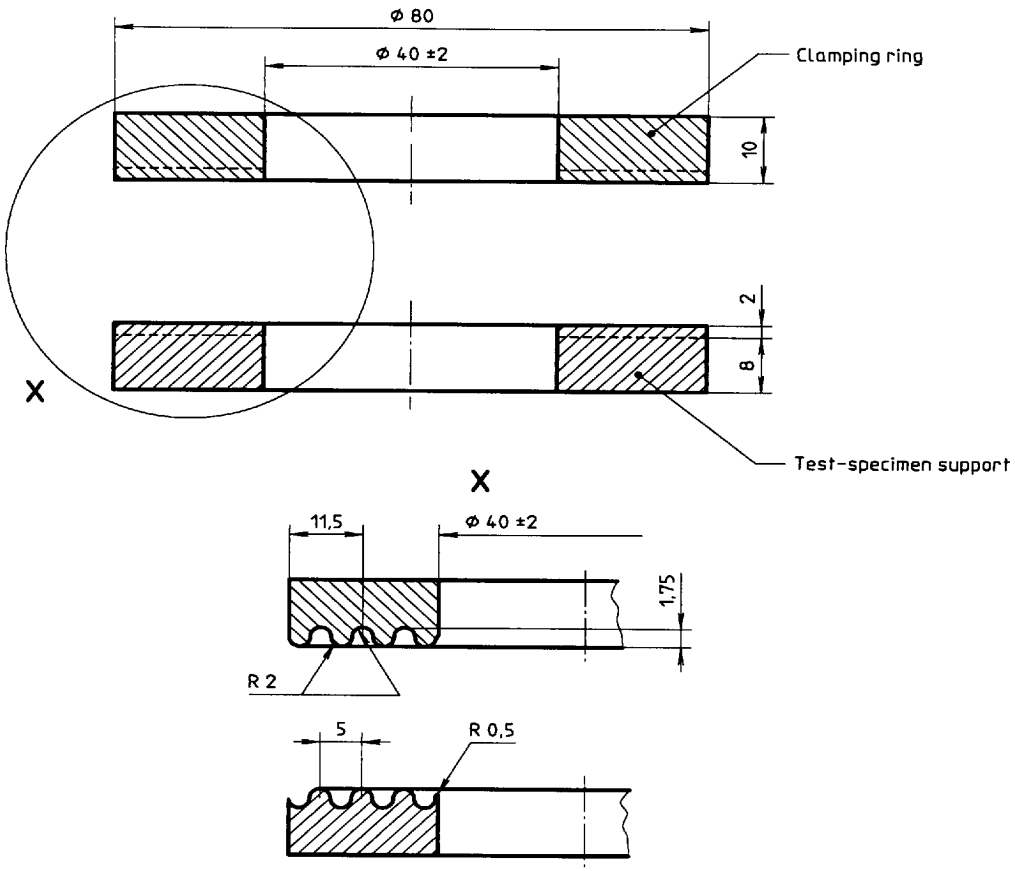


Figure 5 — Clamping device

5.1.1 Energy carrier

The impact energy (e.g. drop energy) available shall be large in comparison to the penetration energy absorbed W_T. This is because the influence of the test velocity (over the range of velocities used in the test) on the viscoelastic behaviour of plastics is relatively small; a decrease in the velocity of the striker of 20 % is acceptable.

This energy requirement is met by falling-dart machines if

$$m \geqslant 3W_T/gH_0$$

where

m is the falling mass, in kilograms;

g is the acceleration due to gravity (9,81 m/s^2);

H_0 is the height of fall, in metres;

W_T is the total penetration energy, in joules.

The falling-dart system used shall be capable of holding and releasing a weighted striker so that the striker falls constrained by a guide or guide(s). The fall shall be largely without friction or losses through windage. Any friction shall be taken into account in the calculations.

NOTES

4 In most cases, a weighted striker with a total mass m of 10 kg is sufficient.

5 A velocity-measuring sensor should be placed close to the point of impact to eliminate errors due to friction between the dart and the guides and to air resistance.

With hydraulically driven high-speed tensile-testing machines, any deviation of the velocity during impact shall be proved, e.g. by plotting the deformation-time curves and checking their slope.

5.1.2 Striker

The preferred striker has a polished, hardened, hemispherical striking surface with a diameter $D_1 = 20$ mm ± 0,2 mm. Alternatively, a striking surface of 10 mm ± 0,1 mm diameter may be used. The striker shall be constructed of steel.

The load cell on the striker shall be mounted as close as possible to the tip to minimize the effect of extraneous forces. An example is shown in figure 4.

The head of the striker may be powdered with talcum or lubricated with oil to reduce friction, provided that the interested parties agree on this procedure and use identical material. In some cases, this can reduce the statistical scatter of the results. It should be borne in mind, however, that lubricating the striker may influence the test results considerably.

The natural frequency f_n of the striker/load-cell assembly shall be higher than the value specified in 5.2.

5.1.3 Clamping device

The test-specimen clamping device shall have an inside diameter D_2 of 40 mm ± 2 mm. The clamping device shall be constructed in such a way that the circular specimen can be clamped flat and held securely during the test. Moreover, the clamping device shall not cause any radial pre-stretching of the specimen greater than 0,01 %. Both requirements can be met with manual or hydraulic clamping faces. Furthermore, placing a ring of fine emery paper on the specimen support has been found to be useful. Figure 5 shows a recommended clamping-device design.

5.2 Instruments for measuring force, specimen deformation and specimen thickness

The instruments for measuring force and deformation shall be capable of measuring the force and deformation to within 5 % of their maximum value.

EXAMPLE

If an electronic device has an accuracy of 0,4 % at full scale (FS) then, for a value of 20 % FS, the accuracy is 2 %.

5.2.1 Load cell

Because of the very short duration of the impact, only electronic load cells with a high natural frequency can be used. The shortest time interval $\Delta t_{F,min}$ which the device shall be required to measure shall be $\geqslant 5/f_n$, where f_n is the natural frequency of the striker/load-cell assembly.

For the bandwidth B_T of the amplifier train (direct-current or carrier-frequency amplifier) with a lower bandwidth limit of 0 Hz, the following applies by analogy:

$$B_T \geqslant 16/\Delta t_{F,min}$$

$$B_T = \left[\sum_{j=1}^{n} 1/B_j^2\right]^{-\frac{1}{2}}$$

where B_j is the individual bandwidth of the j^{th} amplifier stage.

NOTE 6 An example of such a measurement train is a piezoelectric load cell mounted between the striker and the shaft (see figure 4) and connected to a charge amplifier.

5.2.2 Device for measuring the specimen deformation

The deformation of the specimen in the direction of penetration can be determined directly with an electronic transducer, thus yielding a force-deformation curve. It is also possible to record a force-time curve and calculate the deformation in accordance with 7.4.

5.2.3 Thickness gauge

The instrument for measuring the thickness of the specimen shall fulfill the requirements of ISO 4593. It shall be capable of measuring the thickness d of the specimen to within 1 µm.

6 Test specimens

6.1 Sampling and preparation of test specimens

Sampling shall be in accordance with the instructions on the relevant product standard. If no such instructions are given, specimens should preferably be taken from film sheeting or from a piece of the film to be tested. The specimens shall be 80 mm ± 2 mm in diameter. The cut edges need not be of any particular quality. They shall be as uniform as possible over the whole of the width and taken at right angles to the machine direction of the film. Non-homogeneous edge strips of film rolls shall not be used. If a fairly large number of specimens is required in order to determine the temperature dependence of the measured values, the specimens for the entire test series shall be mixed before testing.

6.2 Number of test specimens

A minimum of five test specimens shall be tested (in the case of arbitration, 20 specimens are required). If the dependence of the measured values on temperature, relative humidity or other parameters is to be determined, five specimens per measurement point are generally sufficient, even in the case of arbitration. The number of test specimens required is doubled if the test result depends on the side from which the film is penetrated.

6.3 Conditioning of test specimens

The test specimens shall be conditioned as required by the specifications for the material concerned or as agreed upon by the interested parties. Otherwise, select the most appropriate set of conditions from ISO 291.

7 Procedure

7.1 Test atmosphere

The test shall be carried out in one of the standard atmospheres specified in ISO 291. If measurements are to be made at different temperatures or relative humidities, the test specimens shall be maintained under each set of test conditions until the results show no further change at that particular temperature or humidity. This conditioning time decreases at higher test temperatures.

7.2 Measuring the thickness

Determine the thickness d of each specimen in accordance with ISO 4593 to the nearest 1 µm, taking the average of three measurements at equidistant points on the circumference of a circle of radius 5 mm located at the centre of the specimen.

7.3 Clamping the test specimen

The specimen shall be clamped flat. The stress caused by clamping shall not result in an elongation (pre-stretch) of more than 0,01 % in the radial direction (see 5.1.3).

NOTE 7 The pre-stretch can be determined by means of a measuring microscope. The usual clamping device, however, always fulfills the above condition.

7.4 Impact-penetration test

The impact-penetration test is conducted with an impact velocity of 4,4 m/s ± 0,2 m/s, corresponding to a height of fall H_0 of 1 m. During the test, the speed shall not change by more than 20 % of its value on striking the specimen (see the conditions for the falling mass in 5.1.1).

During the test, the force-deformation diagram or force-time curve shall be recorded. The values of the parameters defined in clause 4 shall be taken from the curve or read from the recording instrument, e.g. a transient recorder. If a satisfactory deformation curve cannot be obtained because of resonance effects, then the impact velocity shall be reduced to 1 m/s.

NOTE 8 Although the impact velocity of 4,4 m/s for testing of plastic films, even those made of relatively brittle materials, is normally not too high, the velocity may be reduced if the interested parties agree.

If there is any reason to believe that the results will depend upon which side of the test specimen faces the striker, both sides shall be tested separately (see also 6.2).

8 Expression of results

For routine characterization purposes, the peak of the force-deformation curve shall be used to determine the test results. If it is clear from the force-deformation curve and/or other information that crack initiation has occurred in the test specimen, the corresponding point (failure point) on the force-deformation curve can also be used to determine the test results.

If the test measurements are in the form of a force-deformation curve, the force and deformation at the characterization point can be read directly from the curve. The corresponding energy values are determined (by planimetry or by other suitable methods, e.g. electronic integration) from the area under the curve.

Should results be in the form of a force-time curve, the deformation s, in metres, at the peak force or the failure force is given by the approximation (see annex A and ISO 6603-2:1989, annex C)

$$s \approx (v_0 - p/3m) \times t \qquad \ldots (1)$$

where

m is the falling mass, in kilograms;

v_0 is the impact velocity just before impact, in metres per second;

t is the time to peak force or the time to failure in seconds;

p is the impulse imparted up to the time of occurrence of the peak force or up to the time of failure, in newton seconds (the area under the force-time diagram), given by the equation

$$p = \int_0^t F(t)\,\mathrm{d}t \qquad \ldots (2)$$

NOTE 9 The exact calculation of the deformation s requires double integration:

$$s = -(1/m)\int_0^t\int_0^t F(t)\,(\mathrm{d}t)^2 + v_0 t \qquad \ldots (3)$$

The energy to peak force, W_M and the energy to failure, W_F, are given by the exact equations (4) and (5), respectively.

$$W_M = v_0\, p_M(1 - v_0\, p_M/4E_0) \qquad \ldots (4)$$

$$W_F = v_0\, p_F(1 - v_0\, p_F/4E_0) \qquad \ldots (5)$$

where

E_0 is the energy of the striker before impact, in joules;

p_M and p_F are calculated from equation (2) with an upper integration limit of t_M and t_F, respectively.

The mean, standard deviation and coefficient of variation of the parameters defined in clause 4 are calculated for each test series.

NOTE 10 Instead of a curve, or concomitantly with it, the values of the peak force and the deformation at peak force can be recorded electronically. This also applies to the energy to peak force and the total penetration energy after electronic integration.

9 Precision

The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added with the next revision.

10 Test report

The test report shall include the following information:

a) a reference to this part of ISO 7765;

b) the type, identification mark, origin and date of receipt of the material tested, plus any other pertinent data concerning the material;

c) the date of measurement;

d) the method of sampling and preparation of the test specimens;

e) the average value of the measured thickness d of each test specimen;

f) the test conditions and the conditioning procedure used;

g) details of the way in which the test specimens were clamped;

h) the diameter D_1 of the striker and the nature of the striker surface;

i) the number of test specimens used;

j) the agreed point of failure, if used;

k) the arithmetic mean, standard deviation and coefficient of variation of the

— peak force F_M, in newtons,

— deformation at peak force s_M, in metres,

— energy to peak force W_M, in joules,

— total penetration energy W_T, in joules,

and optionally the

— failure force F_F, in newtons,

— failure deformation s_F, in metres,

— failure energy W_F, in joules;

l) the force-deformation curve $F(s)$ or force-time curve $F(t)$;

m) the natural frequency f_n and total bandwidth B_T of the amplifier train;

n) the appearance of the test specimens after the test (possibly with a representative test specimen as an illustrative example);

o) the test velocity v_0, if not 4,4 m/s;

p) if a lubricant was used, the lubricant type, grade, quality and manufacturer.

Annex A
(informative)

General remarks

In the testing of plastics, tough/brittle transitions are frequently encountered when the test temperature is lowered in stages from high values. At such transitions, the failure energy, for example, rises from a lower to a higher level. These transitions are caused by molecular relaxation processes which become effective only above a certain temperature and which increase the absorption of the impact energy.

Penetration time plays a role similar to that of temperature. If the penetration time is shortened, the transition temperature is shifted to higher temperatures. The relationship between time and temperature is determined by the temperature dependence of molecular relaxation times, which is approximated by the Arrhenius equation:

$$\tau = \tau_0 \exp(E/kT)$$

where

τ is the relaxation time or penetration time;

T is the thermodynamic temperature or position of the tough/brittle transition on the temperature scale;

E is the activation energy.

If the test temperature is in a transition region, a wide scatter of results is frequently observed because the rupture of some specimens is brittle and the rupture of others is tough. In high-density polyethylene, for example, such a transition region lies in the temperature range between – 105 °C and – 140 °C, depending on the relative molecular mass and degree of crystallinity.

The tough/brittle transition can be recognized in the puncture test by the appearance of the damaged specimens, which reveals whether rupture occurred with or without deformation. The two types of rupture behaviour can also be distinguished by means of the force-deformation diagram.

For tough/brittle plastics, the performance and evaluation of impact tests are subject to certain limitations, since the test specimens of a single test series must be assigned to two different parent populations, namely one exhibiting brittle-material behaviour and one exhibiting tough-material behaviour. In such cases, the means and variances are not statistically defined over the entire range of measurements. Nevertheless, it is helpful to employ the mean and standard deviation calculated from the individual measurements to characterize the behaviour of the material.

Where there is a sufficient number of measurements for both parent populations, the characteristic quantities can be calculated separately for the brittle and the tough specimens. If necessary, the choice of assigning measurements to one of the two parent populations should be decided by use of the statistical procedure normally employed for this purpose.

Annex B
(informative)

Bibliography

[1] GRIMMINGER, H. A theory of the interaction typical for the instrumented puncture test by a drop mass. *Z. Werkstofftechnik*, **18** (1987), pp. 218-224.

ICS 83.140.00

Descriptors: plastics, films, sheets, plastic sheets, tests, impact tests, punching tests, determination, shock resistance

Price based on 10 pages