TECHNICAL SPECIFICATION

ISO/TS 28660

First edition
2022-04

Plastics — Determination of J-R curves — Fracture toughness

Plastiques — Détermination des courbes J-R — Résistance à la rupture

Reference number
ISO/TS 28660:2022(E)

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Published in Switzerland

Contents

Page

Foreword

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

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Plastics — Determination of J-R curves — Fracture toughness

1 Scope

This document specifies a method for determining the fracture toughness in term of *J* and *R* curves for plastics.

The method is suitable for use with ductile and semi-ductile polymers and polymer blends. It is not intended to be used with materials in which the crack front cannot be distinguished from additional deformation processes in advance of the crack tip. The method is unsuitable for polymers reinforced with fibres.

NOTE *J-R* curves, produced in accordance with this test method, characterizes the crack growth resistance that cannot be characterized by linear elastic fracture mechanics according to ISO 13586.

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

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

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 13586, *Plastics — Determination of fracture toughness (GIC and KIC) — Linear elastic fracture mechanics (LEFM) approach*

ASTM D 6068, *Standard Test Method for Determining J-R Curves of Plastic Materials*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13586, ASTM D6068-96 and the following 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 http://www.electropedia.org/

3.1.1
***J*-integral**
J
line or surface integral over a path that enclosed the crack front from one crack surface to the other, used to characterise the local stress-strain field around the crack front

Note 1 to entry: See Reference [5].

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

3.1.2
***J-R* curve**
***J*-Δa_p**
plot of resistance to stable physical crack extension

3.1.3
net thickness
B_N
distance between the roots of the side grooves in side grooved specimens

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

3.1.4
thickness
B
side to side dimension of the test specimen

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

Note 2 to entry: shown in Figure 2 and Figure 3.

3.1.5
specimen width
W
larger initial dimension of the rectangular cross section of the test specimen

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

Note 2 to entry: shown in Figure 2 and Figure 3.

3.1.6
original crack size
a_0
physical crack size at the start of testing

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

Note 2 to entry: shown in Figure 2 and Figure 3.

3.1.7
original uncracked ligament
b_0
distance from the original crack front to the back edge of the specimen given as follows:

$$b_0 = W - a_0$$

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

3.1.8
crack size
a_p
physical crack size to the observed final crack front

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

Note 2 to entry: The size shall be a calculated average of several measurements along the crack front. See Figure 6 and Figure 7.

3.1.9
crack extension
Δa_p
increase in physical crack size given as:

$$\Delta a_p = a_p - a_0$$

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

3.1.10
specimen span
S
distance between specimen rollers

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

Note 2 to entry: Shown in Figure 1.

3.1.11
corrected energy
U
energy required to extend the crack

Note 1 to entry: It is expressed in Joules (J).

Note 2 to entry: See 8.1.

3.1.12
displacement
f
displacement measured by the transducer or extensometer

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

Note 2 to entry: see Figure 8.

3.1.13
slope
α
slope of the linear portion of the force versus displacement curve

Note 1 to entry: see Figure 8.

3.1.14
geometrical functions
η_{el}, η_{pl}
functions representing the notch depth influence

Note 1 to entry: See 8.2.4.

3.2 Symbols

l total length of the test specimen (see Figures 2 and 3)

U_{Tel} elastic part of total energy *UT,* determined from the area under the force versus displacement (see Figure 8)

U_{Tpl} plastic part of total energy *UT,* determined from the area under the force versus displacement (see Figure 8)

U_T total energy, expressed in joules (J)

U_{el} elastic part of corrected energy *U,* required to extend the crack, in Joules (J)

U_{pl} plastic part of corrected energy *U,* required to extend the crack, in Joules (J)

f_{el} elastic part of displacement, expressed in millimetres (mm)

f_{pl} plastic part of displacement, expressed in millimetres (mm)

4 Principle

This test method describes a multiple specimen technique for determining the *J-R* curves for polymeric materials. The *J-R* curves consist of plot of J versus crack extension in the region of J-controlled growth (see Figure 9). This method uses optical measurements of crack length and crack extension on the fracture surfaces after each test.

There are two options for specimen geometries - three-point bend (SENB) and pin-loaded compact tension (CT) specimens. The *J-R* curves from bend specimens represent lower bound estimation to those obtained from compact tension specimens.

The largest possible specimen with representative microstructure is recommended. The *J-R* curves tend to exhibit lower slope with increasing thickness.

The specimens are notched and tested under slowly increasing displacement.

Test carried out on specimens of different dimensions or with different notches, or specimens prepared under different conditions, may produce results that are not comparable. Other factors, such as test speed or conditioning of the specimens, can also influence the results. Consequently, when comparable data are required, these factors shall be carefully controlled and recorded.

5 Apparatus

5.1 Testing machine

5.1.1 General

The machine shall be in accordance with ISO 7500-1 and ISO 9513, and meet the specification given in 5.1.2 and 5.1.3.

5.1.2 Test speeds

The tensile-testing machine shall be capable of maintaining the test speeds as specified in Table 1.

Table 1 — Recommended test speeds

Test speed (mm/min)	**Tolerance** (%)
0,125	±20
0,25	
0,5	
1	
2	
5	
10	
20	±10
50	
100	
200	
300	
500	

5.1.3 Force indicator

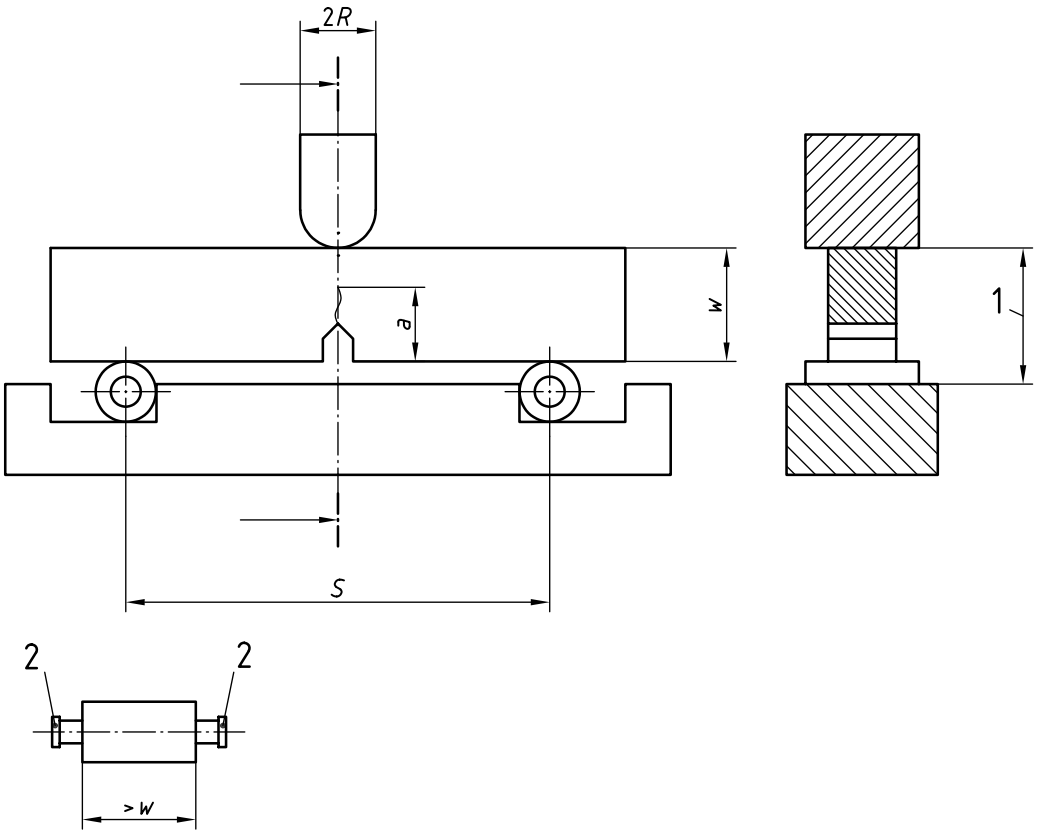
The force measurement system shall be in accordance with class 1 according to ISO 7500-1.

5.2 Displacement transducer

The displacement is recorded during the test. The transducer shall be essentially free from inertia lag at the test speeds being used. It shall measure the displacement with accuracy within the limits of class 2 of ISO 9513 or better. The effects of the transducer on the force measurements shall either be negligible (that is <1 %) or shall be compensated.

5.3 Loading rigs

A rig with either stationary or moving rollers is used for three-point-bending (SENB) tests, as shown in Figure 1. Indentation into the test specimen is minimised by the use of rollers with a larger diameter (> W/4). The measurement of the displacement shall be taken at the centre of the span, *S*.



Key

S	span between rollers	$S = 4W \pm 0{,}1W$
R	radius	$W/8 \le R \le W/2$
1	distance monitored by displacement transducer	
2	bosses for rubber bands - for moving rollers	

NOTE Alternatively, a rig with stationary rollers according to ISO 178 can be used.

Figure 1 — Rig with two rollers and displacement transducer for three-point-bending (SENB) tests

For the compact tensile test, the test specimen is loaded by means of two pins in holes in the specimen. A clip gauge near the pins measures the displacement of the load points during the test.

6 Test specimens

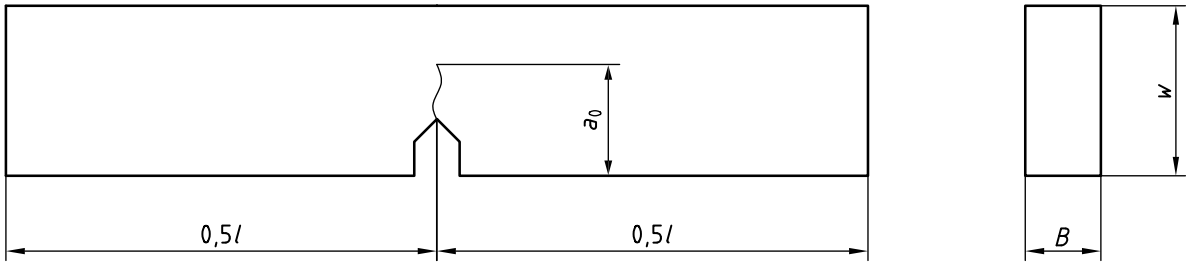
6.1 General

The largest available specimens are recommended for testing in order to obtain a larger portion of *J-R* curve and to obtain the most conservative estimate of crack growth resistance. The *J-R* curve is only appropriate for the thickness that is being evaluated.

6.2 Shape and size

Test specimens for three-point-bending tests (SENB) and for compact tensile (CT) tests shall be prepared in accordance with Figures 2 and 3, respectively. It is usually convenient to make the thickness, *B*, of the test specimens equal to the thickness of a sheet sample. Otherwise, follow the ISO 2818. All in-plane dimensions are proportional to the specimen width, *W* (see Figure 2 and Figure 3). The minimum

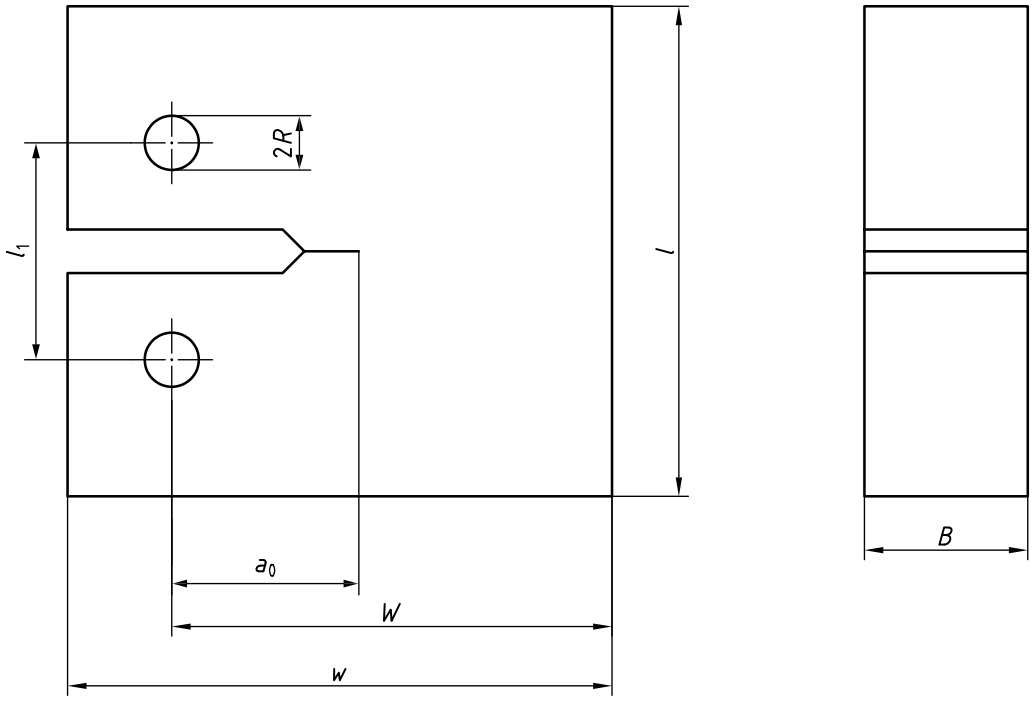
W = 25 mm (fulfil the 9.2). Unnotched test specimens are used for indentation displacement and energy corrections (see Figures 4 and 5, respectively).



Key

W	width	
l	length	$l > 4{,}2\ W$
B	thickness	$W/4 \leq B \leq W/2$ prefer $B = W/2$
a_0	crack length	$0{,}45\ W \leq a_0 \leq 0{,}55\ W$

Figure 2 — Single-edge-notched bend (SENB) test specimen



Key

w	overall width	$w = 1{,}25\ W \pm 0{,}01\ W$
W	width	$l = 1{,}2W \pm 0{,}01\ W$
l	length	$l_1 = 0{,}55\ W \pm 0{,}000\ 5\ W$
$l1$	distance between centres of two holes located symmetrically to the crack plane ± 0,005 W	$R = 0{,}125\ W \pm 0{,}005\ W$
R	radius	$W/4 \leq B \leq W/2$ prefer $B = W/2$
B	thickness	$0{,}45\ W \leq a_0 \leq 0{,}55\ W$
a_0	crack length	

Figure 3 — Compact tensile (CT) test specimen

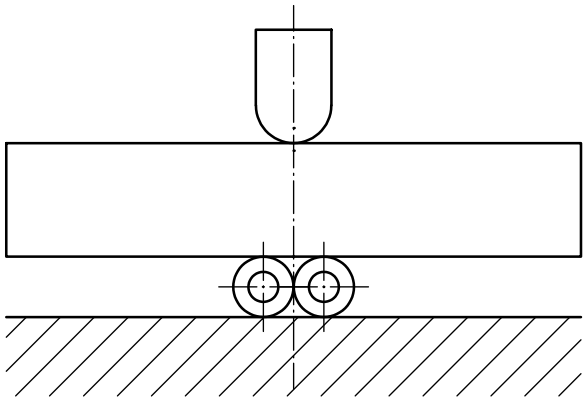


Figure 4 — Arrangement for determining the indentation displacement of a bending-test specimen

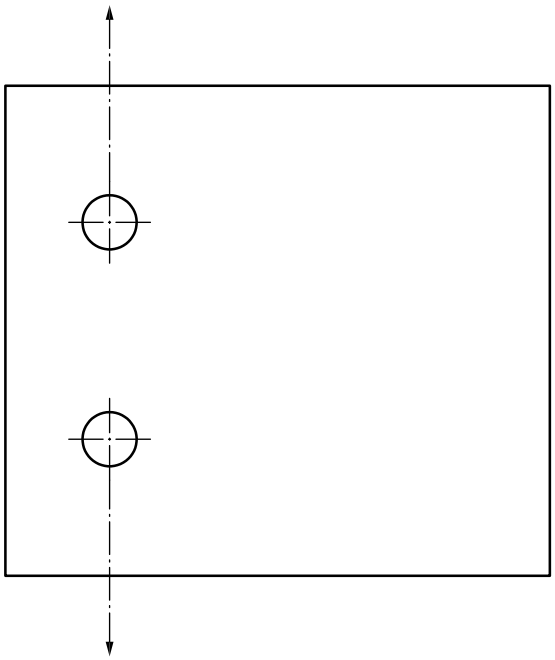


Figure 5 — Arrangement for determining the indentation displacement of a compact tensile specimen

Specimens may need side grooves to promote straighter crack fronts during testing. The side grooves shall be equal in depth and have an inclined angle of 45° ± 5° with a root radius of 0,25 mm ± 0,05 mm. The total thickness reduction may not exceed 0,20 *B*.

6.3 Preparation

Test specimens shall be prepared in accordance with the relevant material International Standard for the material under test with ISO 2818. In the case of anisotropic specimens, take care to indication the reference direction on each test specimen.

6.4 Notching

Method a) or b) can be used for notching

a) Machine a pre-notch into the specimen to a depth of 0,5 *W* using either a saw or a single-point fly cutter. Then generate a natural crack by tapping on a new razor blade placed in the notch and

forcing the crack to grow in advance of the razor blade tip. The length of the razor crack shall not be less than 5 % of the total original crack length a_o and shall be larger than four times the original pre-notch tip radius.

b) Fatigue pre-notching. Suggested notching conditions are given in ASTM E 1152[3]. Because of the possibility of hysteretic heating leading to subsequent damage, frequencies less than 4 Hz are recommended.

7 Procedure

7.1 General

The objective of this procedure is to develop a *J-R* curve consisting of J-values at spaced crack extensions Δa_p (see 9.2). In the multispecimen method, each test specimen is to develop a single point on the *J-R* curve. A series of specimens are loaded to different displacements using crosshead or displacement control. The resulting crack fronts are marked (see Annex A) and crack extensions are measured from the fracture surface. An independent indentation measurement is also conducted to correct for non-fracture related energy dissipation. The *J* value is then calculated from indentation corrected energy for fracture. Each specimen has thus provided a set of J, Δa_p values to describe the *J-R* curve.

7.2 Thickness, width of test specimens

Measure the thickness *B*, B_N and width *W* of each test specimen with an accuracy of ±0,02 mm and in accordance with ISO 16012.

7.3 Conditioning

The test specimen shall be conditioned as specified in the appropriate standard for the material concerned. In absence of this information, the most appropriate condition from ISO 291 shall be selected and conditioning time is at least 16 h, unless otherwise agreed upon by the interested parties, for example for testing at elevated or low temperatures.

The preferred atmosphere is (23 ± 2) °C and (50 ± 10) % R.H., except when the properties of the material are known to be insensitive to moisture, in which case humidity control is unnecessary.

7.4 Test speed

Test speed corresponds to Table 1. A test speed of 1 mm/min is recommended.

7.5 Number of specimens

A minimum of seven specimens are used to generate the power law fit to the data. All shall be machined to the same dimensions. The initial precrack lengths should be as consistent as possible. The objective is to replicate the initial portion of the load versus load-line displacement traces as much as possible.

7.6 Testing procedure

Load each specimen to a selected displacement level that is judged to produce a crack extension in a desired position on the *J-R* curve (see 9.2). Use displacement or clip gage control in order to control the amount of crack growth and minimise crack growth instability.

Unload the specimen, mark the crack front and break the specimen to expose the fracture surface (see Annex A).

7.7 Crack length and crack extension measurement

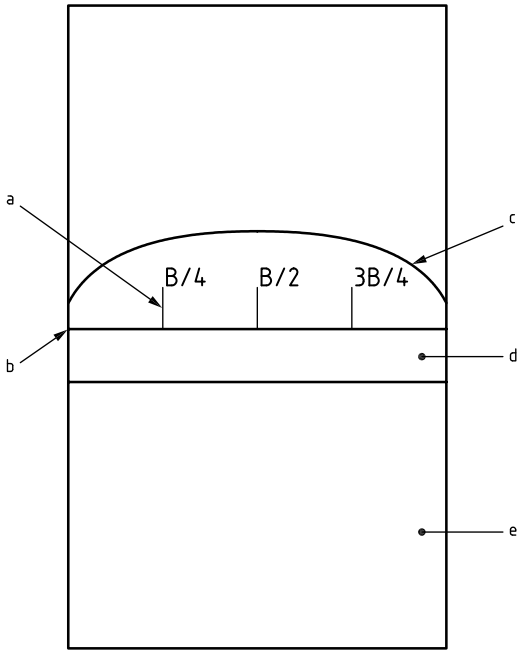
The original crack size a_0 and the individual crack extensions Δa_p are measured from the fracture surface to the nearest 0,01 mm.

The original crack size is calculated from the average of three measurements at distances of B/4, B/2 and 3B/4 (or, for grooved specimens $B_N/4$, $B_N/2$, $3\ B_N/4$) from a side of the specimen along the original crack front on the fracture surface (see Figure 6 and Figure 9).

Along the front of the region if stable crack extension, measure the crack size at five equally spaced points centred about the specimen centreline and extending to 0,005 W from the surfaces of plane sided specimens or from the roots of the side grooves in grooved specimens (see Figure 7 and Figure 9). Calculate the average physical crack size a_p as follows: average the two near-surface measurements, combine the result with remaining three measurements, and determine the average of these four values.

Calculate the crack size using Formula (1):

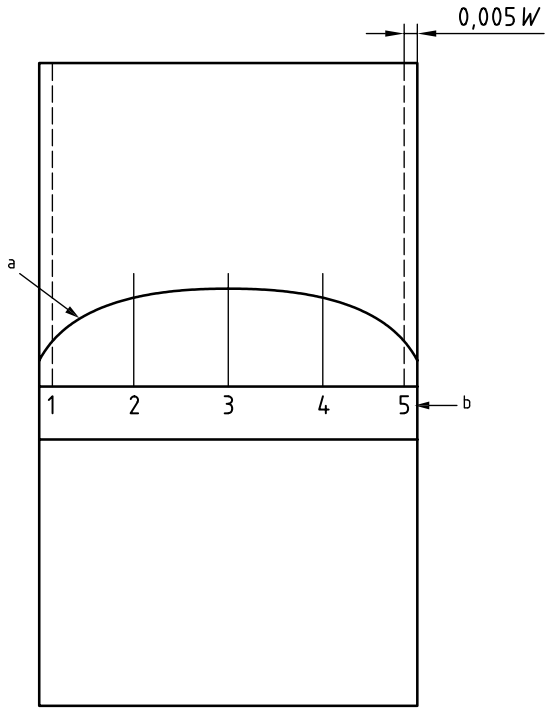
$$\Delta a_p = a_p - a_0 \tag{1}$$



Key

a Measurement points.
b Initial crack length (a_0).
c Final crack front.
d Razor sharpened region.
e Machined notch.

Figure 6 — Measurement of initial crack length (a_0)



Key

a Final crack front.

b Measurement points.

Figure 7 — Measurement of crack extension (Δa)

7.8 Indentation correction

Used an unnotched specimen (shown in Figures 4 and 5, respectively). For the three-point-bending rig, move the rollers together (see Figure 4) in order to minimize flexure. The indentor shall be the same geometry as that used in the *J* tests. For the unnotched CT specimens, clevis grips of the same geometry as those used in the *J* tests shall be used.

Load the specimen to the force that is at least 10 % greater than the maximum force used in the set of individual *J* tests while recording the force versus displacement curve. During all tests, subtract the indentation compliance from the displacement reading. The loading rate and test temperature shall be identical for the indentation correction and *J* tests.

NOTE For SENB specimen, the length can be shorter (min 2*w*).

8 Calculation and interpretation of results

8.1 General

The energy U required to extend the crack is used to calculate J. The total energy U_T determined from the area under the force versus/displacement curved (F versus f) obtained for each specimens is sum of U and U_i, the indentation energy. See Formula (2):

$$U = U_T - U_i \tag{2}$$

The indentation energy U_i is obtained by integrating the force versus displacement curve (F versus f) measured in the indentation test up to the displacement that corresponds to the maximum force for that *J test* specimens.

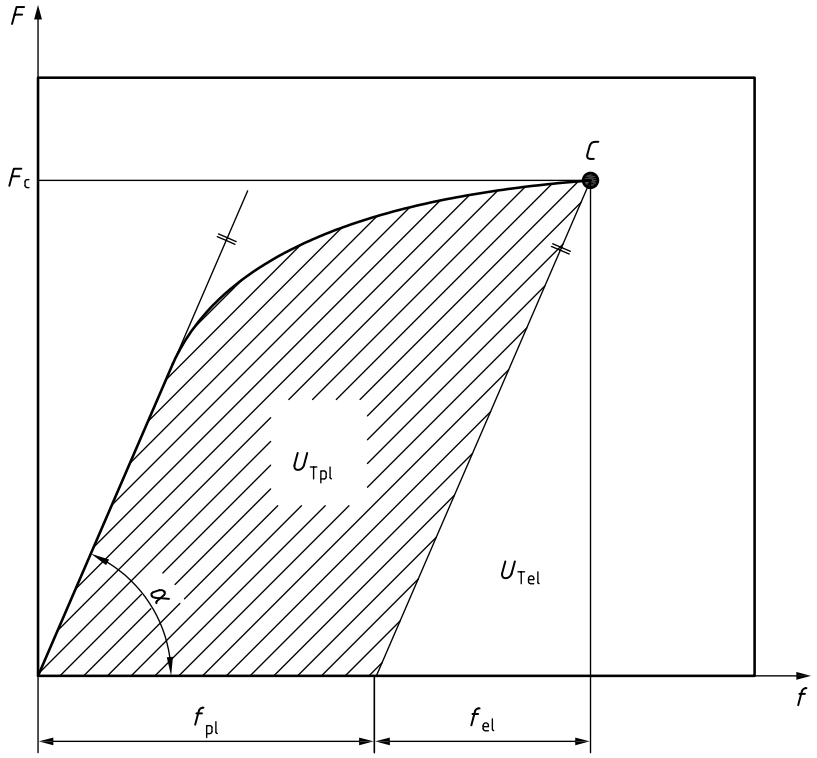
8.2 Calculation of *J* integral

8.2.1 Estimation of the slope

Estimate the slope at the beginning of the load versus load point displacement curve (see Figure 8) using appropriate algorithms.

8.2.2 Splitting of the total energy, U_T

Split the total energy U_T into two parts (see Figure 8).



Key

U_{Tel}	elastic energy
U_{Tpl}	plastic energy
f_{el}	elastic displacement
f_{pl}	plastic displacement
F_c	maximal force
f_c	maximal displacement
C	maximum
α	angle of slope of the curve
F	(kN)
f	(mm)

Figure 8 — Schematic of calculation of *J* integral and the energy

8.2.3 Calculation of corrected energy

Calculate the corrected energy U according to Formula (2). Estimate the elastic and plastic part of this corrected energy according Formulae (3) and (4)

$$U_{\mathrm{el}} = U \times \frac{U_{\mathrm{Tel}}}{U_{\mathrm{T}}} \tag{3}$$

$$U_{\mathrm{pl}} = U \times \frac{U_{\mathrm{Tpl}}}{U_{\mathrm{T}}} \tag{4}$$

NOTE In case the test machine is equipped with software for indentation correction, the energy U can be split directly without use of Formulae (3) and (4). Energy U_T is replaced by U and f is replaced by f. The value of energy U is directly split into U_{el} and U_{pl}.

8.2.4 Calculate *J* integral

Calculate the value of J integral according to Formula (5):

$$J = \eta_{\mathrm{el}} \times \frac{U_{\mathrm{el}}}{B \times (W - a_0)} + \eta_{\mathrm{pl}} \times \frac{U_{\mathrm{pl}}}{B \times (W - a_0)} \times \left[1 - \frac{(0{,}75 \times \eta_{el} - 1) \times \Delta a_0}{(W - a_0)} \right], \tag{5}$$

Values of $\eta_{\mathrm{el,SNEB}}$, $\eta_{\mathrm{el,CT}}$ and η_{pl} are given in Annex A for both types of specimen.

9 Validation of results

9.1 General

None of the original crack size at positions *B/4, B/2, 3B/4* (see 7.7) measurements shall differ by more than 5 % from calculated average (a_0).

None of the measured physical crack extensions shall be less than 50 % of the average Δa_{p}. For subsequent testing, the side groove configuration may be modified within the recommended parameters of 6.2

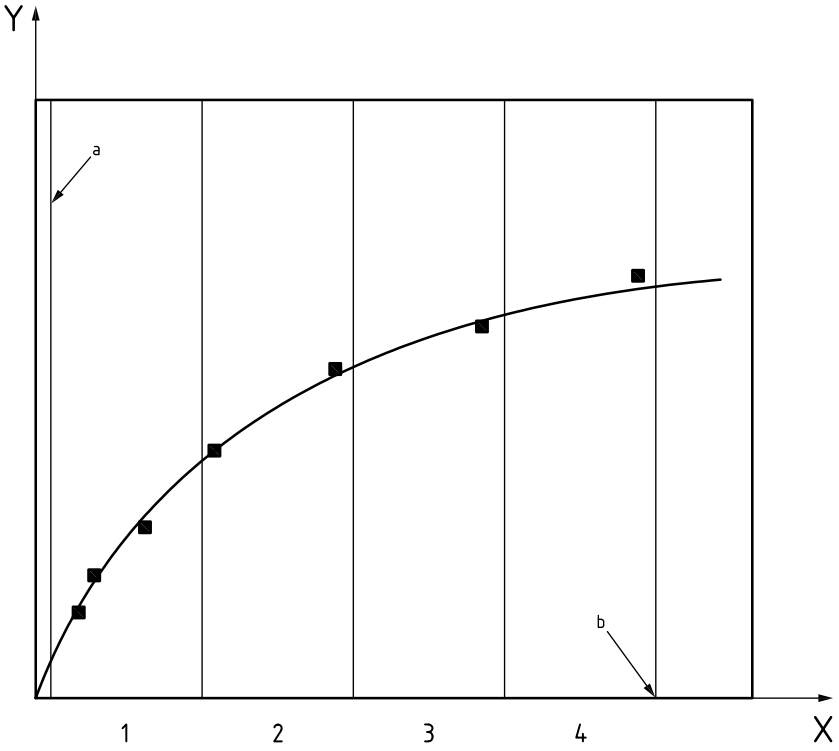
The minimum crack extension shall be >0,05 mm. The maximum crack extension shall be <0,1 b_{o}.

9.2 Construction of *J-R* curves

Plot the curve J vs. Δa_{p} (see Figure 9).

Construct a minimum crack extension line at Δa_{p}=0,05 mm and a maximum crack extension line at Δa_{p}=0,1 b_{o} mm.

Divide the interval between the minimum and maximum crack extension lines into four equally spaced regions (see Figure 9). At least three data points shall be in the first region, two data points in the second quadrant, and at least one data point in each of the two remaining regions.



Key

X physical crack extension, Δa_p (mm)

Y fracture resistance, J (kJ/m^2)

a 0,05 mm exclusion line

b Δa_{max}

Figure 9 — Construction of *J-R* curves

Fit the data to a power law as shown in Formula (6):

$$J = C_1 \Delta a_p^{C_2} \tag{6}$$

The exponent, C_2, shall be less than 1,0.

10 Test reports

The test report shall contain the following information:

a) a reference to this document, i.e. ISO/TS 28660:2022;

b) all details necessary for complete identification of the material tested;

c) the test specimens shape (SENB or CT) and nominal dimensions;

d) the date of testing;

e) notching technique;

f) original crack length, a_0

g) crack extension marking technique;

h) physical crack extension Δa_p;

i) slope at beginning;

j) energies;

k) J integral values;

l) coefficients of power law regression function, C_1 and C_2;

m) temperature and relative humidity during the test;

n) v test speed.

11 Precision and bias

11.1 General

Crack length measurement makes the most significant contribution to the variation in the *J-R* curve. Some control of the crack straightness and crack front shape is required (see 9.1).

A precision statement is given in Annex A.

11.2 Splitting of energy

Splitting the total energy into two parts is important from the physical point of view. The tougher polymers can show different sensitivity to crack initiation (corresponds to U_{el}) and crack growth (dissipated energy corresponds to U_{pl}).

Annex A
(informative)

A.1 Geometrical function

$$\eta_{el} = 0,5 + 5,5\left(\frac{a_0}{W}\right) - 5\left(\frac{a_0}{W}\right)^2 \text{ for SENB specimens}$$

$$\eta_{el} = 11,30 - 68,42\left(\frac{a_0}{W}\right) + 198,75\left(\frac{a_0}{W}\right)^2 - 258,33\left(\frac{a_0}{W}\right)^3 + 125,00\left(\frac{a_0}{W}\right)^4 \text{ for CT specimens}$$

$$\eta_{pl} = 2 \text{ for SENB specimens}$$

$$\eta_{pl} = 2 + 0,522\left(1 - \frac{a_0}{W}\right) \text{for CT specimens}$$

A.2 Crack front marking and identification.

The measurements for stable crack extension are made directly from the fracture surface of each *J* specimen.

The crack front shall be marked in a manner that will differentiate the stable crack extension that occurs during the initial loading in the *J* test from the crack growth process that occurs during the final fracture step that is required to expose the fracture surface. Suggested method is, for example, high-speed impact with or without prior cooling of the specimen (e.g. liquid nitrogen). The minimum cooling time is 30 minutes. In order to better differentiate the fracture surface, a suitable dye can be used before unloading (e.g. alcohol extract from standard pencil used for description the plastic parts).

Optical or scanning electron microscopy must be used to identify the end of the stable crack extension region (optimum magnification for optical microscope is 20 to 40 times). Furthermore, the microscope must allow oblique illumination of the fracture surface.

The end of stable crack growth region is typically determined by the first arrest line after the razor notch or natural crack.

A.3 Precision statement

A.3.1 General

The first interlaboratory tests have been performed in 2011 on PE-HD for extrusion. This interlaboratory test indicated a large scatter on the calculated value of *J* integral and this was attributed to the difficulties involved with the stable crack length measurement. The interlaboratory test continue in year 2013. The aim was unification of methodology of estimation stable crack growth.

The 2013 RRT included one PE-HD extrusion material and one PP-H extruded material and three laboratories from three countries. The specimens were prepared from compression-moulded plaques (130×130×10 mm). Total 12 test specimens per materials were prepared by one laboratory. 3+3 specimens were bended at certain deflection (I. quadrant and IV. quadrant of *R*-curve) by one laboratory and 3+3 at the same deflection by two other laboratories. The stable crack growth was estimated and the fracture surface documented.

A.3.2 Statistical evaluation

CAUTION — Due to limited number of laboratories and materials the following explanation of *r* is only intended to present a meaningful way of considering the approximate precision (repeatability) of this test method.

Concept of *r*

Repeatability: two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the *r* value for that material. *R* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment in the same laboratory.

Table A.1 — Precision data for Δ*a* values

Material	*n*	Quadrant	Δ*a* mm	s_r	*r*	*R*
PE-HD	1	I.	0,222	0,003 2	0,04	0,08
	2		0,182	0,018 0		
	3		0,110	0,033 3		
	1	IV	1,493	0,115 6	0,32	0,52
	2		1,266	0,109 8		
	3		0,870	0,017 8		
PP-H	1	I.	0,145	0,006 7	0,07	0,13
	2		0,147	0,034 6		
	3		0,075	0,020 1		
	1	IV.	1,758	1,004 3	0,53	0,89
	2		1,168	0,127 0		
	3		0,778	0,237 1		

In this table, the statistical properties used are:

Δ*a* average value

s_r within laboratory standard deviation

r 95 % repeatability limit = 2,8 s_r

n number of laboratories

A.4 Recommended test protocol

Recommended test report according to ISO/TS 28660.

Organization:
Name:
Material:
Lot:

Date of testing:
ISO Standard:
Test Temperature [°C]:
Rel. Humidity [%]:

J and *R* curves determination

Notching method: – pre-notch:									
– crack:									
Test speed [mm/min]:									
Specimen No.			1	2	3	4	5	6	7
Specimen type SENB or CT	dimensions	B [mm]							
		B_N [mm]							
		W [mm]							
Displacement level	[mm]								
Max. F	[N]								
Slope									
U_T	[J]								
^{U}Tel									
^{U}Tpl									
U_i									
U									
^{U}el									
^{U}pl									
a_0	[mm]	a_0 (B/4)							
		a_0 (B/2)							
		a_0 (3B/4)							
Average a_0	[mm]								
Valid a_0 ?		Y/N							
b_0	[mm]								
$^{\Delta a}$max	[mm]								
a_p	[mm]	^{a}p1							
		^{a}p2							
		^{a}p3							
		^{a}p4							
		^{a}p5							
Average Δa_p	[mm]								
Check (a_{pi}-a_0)		Y/N							
Check Δa_p		Y/N							
J integral value	[kJm^{-2}]								

Check the data spacing requirement of R curve				
Check No. of points in intervals	1, ≥ 3	Y/N		Spacing requirement fulfilled? Y/N
	2, ≥ 2	Y/N		
	3, ≥ 1	Y/N		
	4, ≥ 1	Y/N		
C_1				
C_2				
Check C_2	< 1,0	Y/N		

Comments:

Organization:
Name:
Material:
Lot:

Date of testing:
ISO Standard:
Test Temperature [°C]:
Rel. Humidity [%]:

Indentation correction

Test speed:

Specimen No.		1	2	3	4	5	6	7
Specimen type SENB or CT								
Max. force	N							
U_i, CT	J							
U_i, SENB	J							

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ICS 83.080.01

Price based on 20 pages