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Adhesives — Determination of the mode 1 adhesive fracture energy of structural adhesive joints using double cantilever beam and tapered double cantilever beam specimens

Adhésifs — Détermination de l'énergie de fracture adhésive en mode 1 des adhésifs structurels utilisant des éprouvettes de rayon de cantilever double et des éprouvettes de rayon de cantilever double effilées

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Foreword

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Adhesives — Determination of the mode 1 adhesive fracture energy of structural adhesive joints using double cantilever beam and tapered double cantilever beam specimens

1 Scope

This International Standard specifies a method, based upon linear elastic fracture mechanics (LEFM), for the determination of the fracture resistance of structural adhesive joints under an applied mode I opening load, using double cantilever beam (DCB) and tapered double cantilever beam (TDCB) specimens.

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 10365, *Adhesives — Designation of main failure patterns*

3 Symbols and abbreviated terms

For the purposes of this International Standard, the following symbols and abbreviated terms apply:

A	insert film length (mm), i.e. the distance between the end of the specimen and the tip of the insert film (see Figure 1)
a	crack length (mm), i.e. the distance between the load-line (intersection of plane through pin-hole centres or centres of the hinge axes and plane of crack) and the tip of the precrack or crack on the edge of the specimen (see Figure 1)
a_p	precrack length (mm), measured from the load-line to the tip of the mode I precrack
a_0	insert film length (mm) between the load-line and the tip of the insert film (see Figure 1)
B	width of the specimen (mm)
C	compliance δ/P of the specimen (mm/N)
C_{cs}	compliance of the calibration specimen used to measure the system compliance (mm/N)
C_{max}	compliance of the specimen at maximum load (mm/N)
C_{sy}	compliance of the tensile-loading system (mm/N)
C_{total}	compliance of the tensile-loading system and the calibration specimen used to measure this (mm/N) (see Annex A)

C_0	initial compliance of the specimen, neglecting start-up effects, e.g. due to play in the specimen fixture (mm/N) (see Figure 2)
$C_{0+5\ \%}$	initial compliance of the specimen, C_0, raised by a factor 1,05 (mm/N) (see Figure 2)
E_f	flexural modulus of the arms of the substrate beam, calculated from the DCB mode I crack propagation test (GPa)
E_s	independently measured flexural or tensile modulus of the arms of the substrate beam (GPa); if the substrate is a fibre composite, E_s is the longitudinal modulus of the material in the direction of fibre alignment
F	large-displacement correction
G_{IC}	critical strain energy release rate, or adhesive fracture energy, for the applied mode I opening load (J/m^2)
H	thickness of the load-block (mm)
h	thickness of the substrate beam at a crack length a (mm)
h_a	thickness of the adhesive layer (mm)
l	total length of the specimen (mm)
l_1	distance from the centre of the loading pin or of the piano hinge axis to the mid-plane of the arm of the substrate beam to which the load-block or the piano hinge is attached (mm) (see Figure 1)
l_2	distance between the centre of the pin-hole in the load-block and the edge of the load-block, measured towards the tip of the insert (starter film) or the tip of the mode I precrack (mm) (see Figure 1)
l_3	total length of the load-block (mm) (see Figure 1)
MAX/5 %	either the maximum load on the load-displacement curve or the point of intersection of a straight line with the load-displacement curve with the slope of the straight line corresponding to $C_{0+5\ \%}$ (see Figure 2)
m	specimen geometry factor [see Equation (1)]
N	load-block correction
NL	onset of non-linearity on the load-displacement curve (see Figure 2)
n	slope of a plot of $\log_{10}C$ versus $\log_{10}a$, or $\log_{10}(C/N)$ versus $\log_{10}a$ if load-blocks are being used
P	load measured by the load-cell of the test machine (N)
PROP	increments of the crack length during stable crack growth (propagation) that are marked on the load-displacement curve (see Figure 2)
r^2	correlation coefficient of linear fits
r.h.	relative humidity during the test (%)
VIS	onset of visually recognizable crack growth at the edge of the specimen that is marked on the load-displacement curve (see Figure 2)

Δ crack-length correction for a beam that is not perfectly built-in (mm)

δ displacement of the cross-head of the test machine (mm)

δ_{cor} displacement of the cross-head, corrected for system compliance effects (mm)

4 Principle

A double cantilever beam (DCB) specimen or a tapered double cantilever beam (TDCB) specimen is used to determine the adhesive fracture energy, G_{IC}, of structural adhesive joints.

Resistance to both crack initiation and propagation is determined. The resistance to crack initiation is determined from both a non-adhesive insert placed in the adhesive layer and from a mode I precrack. The resistance to crack propagation is determined from the mode I precrack. The adhesive fracture energy, G_{IC}, (also termed the critical strain energy release rate) for applied mode I loading is calculated and a resistance-curve (R-curve), i.e. a plot of the value of G_{IC} versus crack length, is determined.

5 Apparatus

5.1 Tensile-testing machine, capable of producing a constant cross-head displacement rate between 0,1 mm/min and 5 mm/min in displacement control. The test machine shall be equipped with

a) either a fixture to introduce the load to the pins inserted into the load-blocks or directly into the substrate beams;

b) or grips to hold the piano hinges that allow rotation of the specimen end (see Figure 1).

The test machine shall incorporate a load-cell that shall be calibrated and be accurate to within ± 1 % in the chosen load-range.

NOTE Loads are typically expected to be in the range of 100 N to 5 000 N.

The opening displacement of the test specimen shall be deduced from the position of the cross-head. The test machine shall be equipped with means for recording the complete load versus displacement curves (loading and unloading) during the test.

5.2 Travelling microscope or **video camera**, with suitable magnification, capable of measuring the crack length along the edge of the specimen to an accuracy of at least ± 0,5 mm.

5.3 Micrometer or **vernier calipers**, capable of measuring the thickness of the substrate arms and bonded joints with an accuracy of at least ± 0,02 mm.

5.4 Micrometer or **vernier calipers**, capable of measuring the width of the joints with an accuracy of at least ± 0,05 mm.

5.5 Typewriter correction fluid (“white ink”) or **white spray-paint**.

6 Specimens

6.1 Number of specimens

A minimum of four joints shall be tested.

6.2 Conditioning

Most adhesives will absorb small quantities of water from the atmosphere which may have a significant influence on the measured properties. Following specimen preparation, the adhesive will generally be dry. If testing is carried out within a few days of specimen manufacture, then it is not necessary to condition the specimen under controlled humidity since negligible absorption of water will take place in the thin adhesive layer. However, if the specimen is tested after longer times or if the influence of absorbed water on the properties is of interest, then the humidity shall be controlled by conditioning and the properties will depend on the conditioning time.

In addition, if composite substrates are used, then it may be important to dry these prior to manufacture of the joint. The properties of some adhesives are very sensitive to the presence of small amounts of moisture in a substrate prior to curing. The drying out of the substrates prior to cure will ensure that the integrity of the adhesive joint is not influenced by pre-bond moisture effects.

6.3 Manufacture of adhesive joint specimens

The DCB specimen shall be as shown in Figures 1 a), 1 b) or 1 c). The TDCB specimen shall be as shown in Figure 1 d).

The thickness of the film to be inserted in the adhesive layer during manufacture shall be less than 13 µm. The film shall be non-stick. A PTFE film is recommended. If aluminium foil is used, the foil shall be coated with a release agent prior to use. Appropriate surface treatments for metallic substrates can be found in ISO 17212 [1].

The thickness of the adhesive layer shall be carefully controlled and shall be less than 1 mm (see Notes 1 and 2). The thickness of the layer shall not vary by more than 20 % within a joint, nor shall the average thickness of the layer in one joint differ by more than 20 % from that in another joint in the sample. When fully cured, remove any excess adhesive by mechanical means that do not weaken the bond, so as to leave the joint with smooth sides.

It should be recognized that the value of G_{IC} measured from these tests will depend upon the thickness of the adhesive layer in the joint. The value of the layer thickness shall be determined by the user, based upon the adhesive manufacturer's recommendations or upon consideration of the intended application.

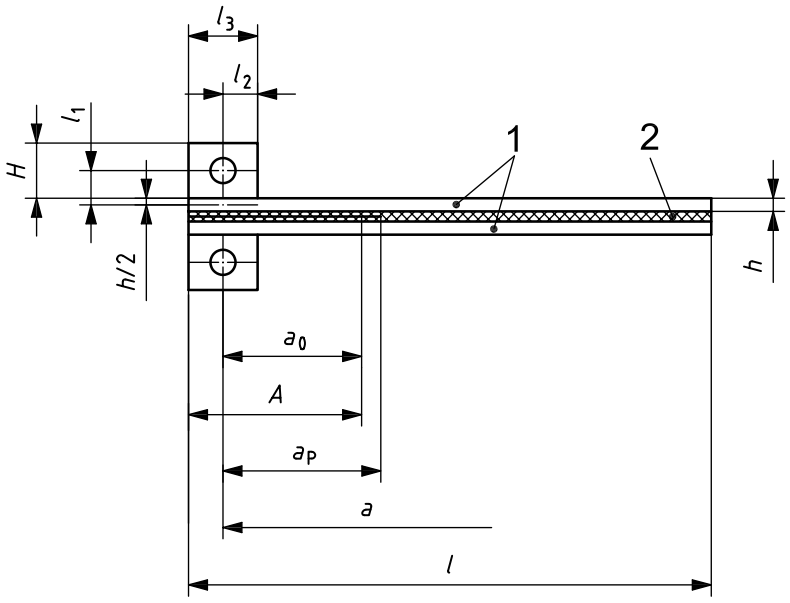
It is not within the scope of this International Standard to specify full manufacturing details of the joints to be tested. Such information should be sought from the adhesive manufacturer and/or the substrate manufacturer.

NOTE 1 The analysis used here assumes that the adhesive layer makes a negligible contribution to the overall compliance of the joint. In round-robin studies and supporting tests, values of h_a from 0,1 mm to 1,0 mm have been used with acceptable results. Values of $h_a > 1{,}0$ mm may be used, but the validity of the analysis has not been demonstrated for these thicker layers.

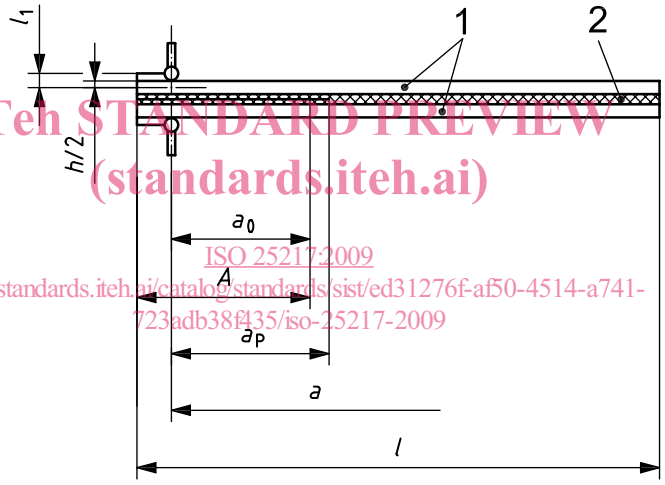
NOTE 2 For some toughened adhesives, the value of G_{IC} determined by this method has been shown to be a strong function of the thickness of the adhesive layer, h_a. As a result, careful consideration is required when selecting the value of h_a. For the selection of h_a, it is instructive to determine the size of the plastic zone ahead of the crack tip. The radius of this zone, r_p, may be approximated for the conditions of plane stress (as exists at the edges of the joint) and plane strain (as exists in the central part of the joint) as follows:

$$r_p = \frac{1}{2\pi}\left(\frac{E_a G_{IC}}{\sigma_y{}^2}\right) \quad \text{(plane stress);} \qquad r_p = \frac{1}{6\pi}\left(\frac{E_a G_{IC}}{\sigma_y{}^2}\right) \quad \text{(plane strain)}$$

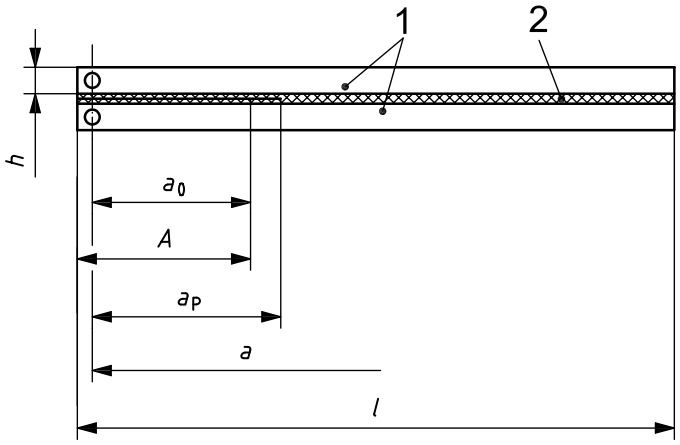
where E_a and σ_y are Young's modulus and the yield strength of the adhesive, respectively. Kinloch and Shaw [4] argued that, as the value of r_p was greater at the edges of the joint, where plane stress conditions exist, then the plane stress value was more applicable to the direct comparison with the adhesive layer thickness, h_a. If $h_a \ll 2r_p$, then the plastic zone may be largely suppressed and a low value of G_{IC} would be anticipated. If $h_a \approx 2r_p$, then the value of G_{IC} may reach a maximum, as shown in Reference [4], as the plastic zone fully develops and potentially distorts due to the presence of the nearby adhesive-substrate interface. If $h_a > 2r_p$, then the value of G_{IC} would be expected to become independent of h_a, which is the most desirable condition, subject to the constraint imposed by Note 1 above.



a) DCB specimen with load-blocks

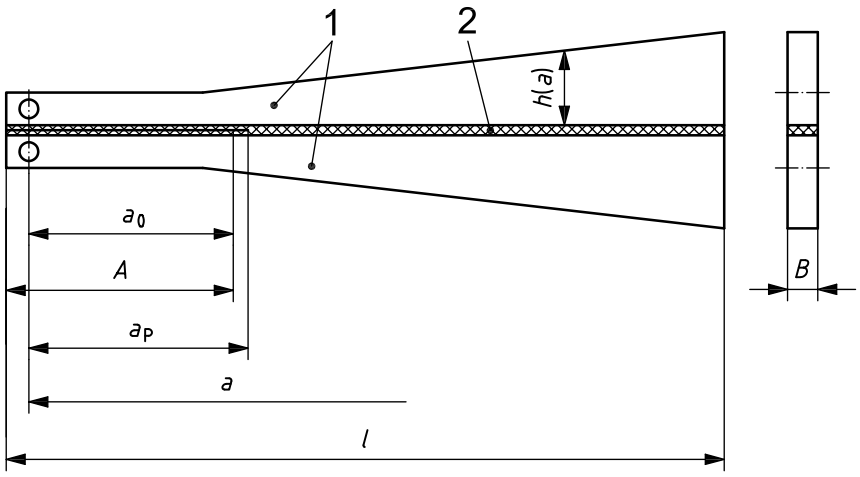


b) DCB specimen with piano hinges (alternative loading arrangement)



c) DCB specimen with metallic substrates where loading holes may be drilled through the arms of the substrate (alternative loading arrangement)

Figure 1 (*continued on next page*)



d) TDCB specimen

Key

1 substrates

2 adhesive

NOTE The crack length, a, is the distance between the load-line (intersection of the plane through the pin-hole centres or the hinge axes and the plane of crack) and the tip of the precrack or crack on the edge of the specimen. The value of h is the thickness of a substrate arm. For the TDCB specimen, the value of h is a function of the crack length, a.

Figure 1 — Geometry of adhesive joint specimens

6.4 Measurement of specimen dimensions

6.4.1 DCB substrates

Measure the thickness of each DCB substrate using a micrometer (5.3) before bonding. Make the measurements at three points along the length of the beam, at 30 mm from either end, and at the mid-length. Calculate the mean value of the thickness of each substrate, h. Determine the thickness of the adhesive layer, h_a, by subtracting the substrate thicknesses, $2h$, from the total thickness of the joint.

6.4.2 TDCB substrates

Measure the thickness of each TDCB substrate using vernier calipers or a micrometer (5.3) before bonding. Make the measurements at three points along the contoured section of the beam, at 30 mm from either end and at the mid-length of the contoured section. Measure the crack length at each of these positions. Calculate m from Equation (1) (see Annex D).

$$\frac{3a^2}{h^3} + \frac{1}{h} = m \tag{1}$$

Repeat the measurements of the total beam thickness after bonding. Determine the adhesive layer thickness, h_a, by subtracting the substrate thicknesses, $2h$ (measured at the locations described), from the total thickness of the joint at each of the three locations.

6.4.3 DCB and TDCB substrates

Remove any excess adhesive from the sides of the beam. After bonding, measure the width of the DCB or TDCB joint with vernier calipers or a micrometer at three points along the length of the beam, at 30 mm from either end and at the mid-length. Calculate the mean value, B.

6.5 Preparation of specimens

Apply a thin layer of typewriter correction fluid ("white ink"), or white spray-paint, on the edges of the specimen after conditioning to facilitate the detection of crack growth.

NOTE Some typewriter correction fluids and paints contain solvents which can harm the adhesive or the laminate matrix material of a composite substrate. An aqueous solvent is usually preferred.

Apply marks every 1 mm from the tip of the insert or the mode I crack for at least the first 10 mm, then apply marks every 5 mm. Apply marks for every 1 mm for the final 5 mm.

For the DCB test specimen, the extent of crack propagation should be approximately 65 mm, and for the TDCB test specimen the extent of crack propagation should be approximately 100 mm.

7 Procedure

7.1 Test set-up and data recording

Perform the test at one of the temperatures specified in ISO 291 or at another temperature agreed between the interested parties. After mounting the specimen in the fixture of the test machine, support the end of the specimen, if necessary, to keep the beam orthogonal to the direction of the applied load. Record the load and the displacement signals of the test machine, electronically or on a paper chart, throughout the test, including the unloading cycle.

If using a tensile-testing machine with a paper chart recorder, the following ratios of cross-head speed to chart speed are recommended:

a) when testing joints with metallic substrates, a ratio of about 1:100;

b) when testing joints with fibre-composite substrates, a ratio of about 1:10.

Measure the crack length along the edge of the specimen to an accuracy of at least $\pm$ 0,5 mm using either a travelling microscope or a video camera with suitable magnification (5.2). If unstable crack growth followed by arrest ("stick-slip") is observed during any stage of the test, follow the procedure in Annex B.

7.2 Initial loading (the precracking stage)

For testing from the insert (starter film), load the specimen at a constant cross-head rate of

a) either 0,1 mm/min to 0,5 mm/min for joints prepared using metallic substrates;

b) or 1,0 mm/min to 5,0 mm/min for joints prepared using fibre-composite (with polymeric matrix) substrates.

NOTE Lower values are more accurate for crack-length measurement. Fibre-composite refers to a material with unidirectional, continuous fibres of carbon or glass in a polymer matrix with the fibre axis along the length of the specimen.

Record the point on the load-displacement curve at which the onset of crack movement from the insert is observed on the edge of the specimen on the load-displacement curve or in the sequence of load-displacement signals [VIS in Figure 2 a)].

Stop the loading as soon as the crack is seen to move on the edge of the specimen. Completely unload the specimen at a constant cross-head rate of up to five times the loading rate. Mark the position of the tip of the precrack on both edges of the specimen. If the crack lengths, a, on the edges of the specimen, i.e. the distance between the load-line and the tip of the precrack, differ by more than 2 mm, consider the results to be suspect and abandon the test.

7.3 Re-loading: Testing from the mode I precrack

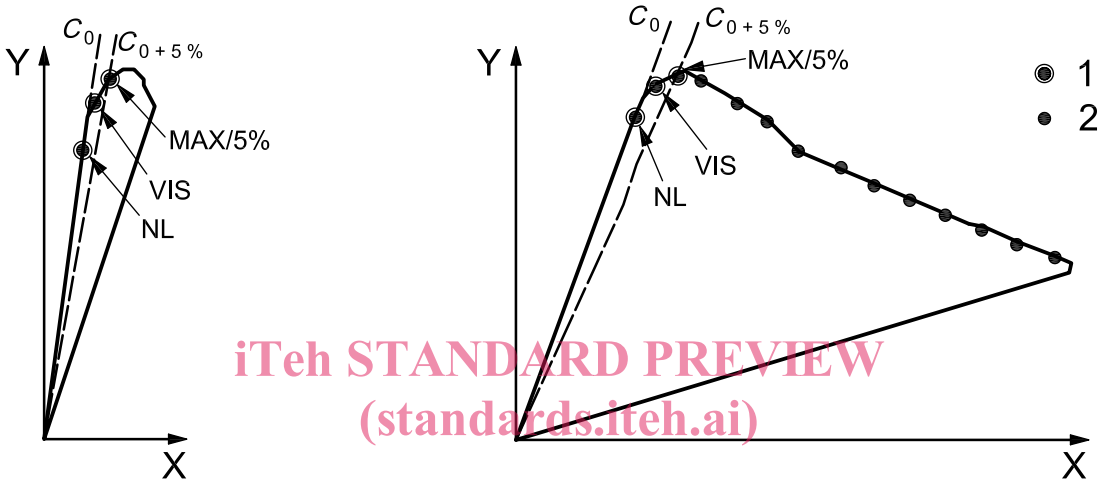
For testing from the mode I precrack which has been formed as a result of the test procedure in 7.2, load the specimen at a constant cross-head rate of

a) either 0,1 mm/min to 0,5 mm/min for joints prepared using metallic substrates;

b) or 1,0 mm/min to 5,0 mm/min for joints prepared using fibre-composite (with polymeric matrix) substrates.

NOTE Lower values are more accurate for crack-length measurement.

Record, on the load-displacement curve or in the sequence of load-displacement signals, the point at which the onset of crack movement from the insert is observed to occur [VIS in Figure 2b)].



a) Testing from the insert with initiation points NL, VIS and MAX/5 %

b) Testing from the mode I precrack with initiation points NL, VIS and MAX/5 % and propagation points (PROP)

Key

X displacement, δ

Y load, P

1 initiation values

2 propagation values

NOTE This figure shows an example where the MAX and the 5 % offset points coincide such that they lie on the same point on the curve. This is not generally the case. Usually, these points will be separated. The MAX/5 % point is assigned to the point $C_{0+5\,\%}$ or MAX, whichever occurs first.

Figure 2 — Schematic load-displacement curve for the DCB test (see Note)

After this, note as many crack-length increments as possible in the first 5 mm on the corresponding load-displacement curves, ideally every 1 mm. Subsequently, note crack lengths at every 5 mm, until the crack has propagated about 60 mm from the tip of the mode I precrack for the DCB test and about 95 mm for the TDCB test. Note every 1 mm for the last 5 mm of crack propagation. Record a minimum number of fifteen propagation points.

Next, unload the specimen at a constant cross-head rate of up to five times the loading rate. Record whether the load-displacement curve returns to its initial point and, if not, follow the procedure in Annex C.

NOTE This might indicate that permanent plastic deformation of the arms of the specimen has occurred.