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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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ISO 25217 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

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.

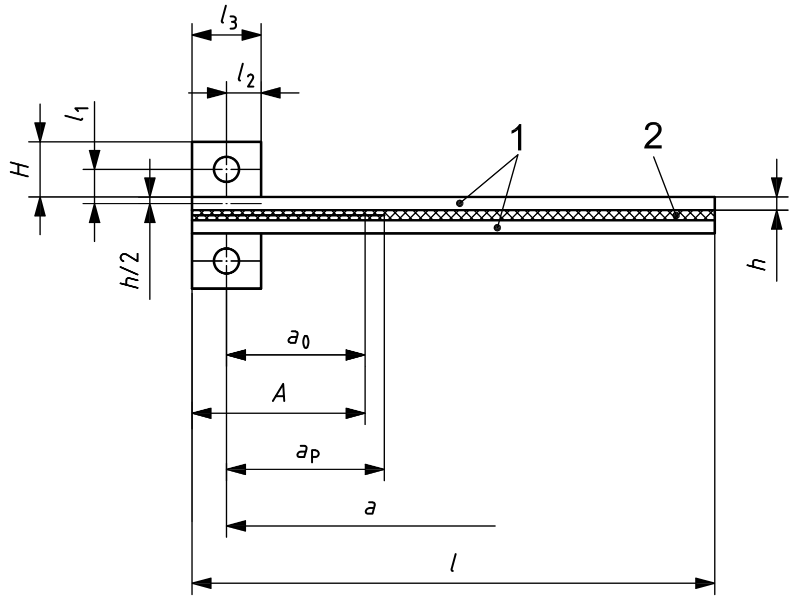
It is not within the scope of this International Standard to specify full manufacturing details of the joints to be tested. Such information should be sought from the adhesive manufacturer and/or the substrate manufacturer.

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 $h_{\mathrm{a}} > 1{,}0$ mm may be used, but the validity of the analysis has not been demonstrated for these thicker layers.

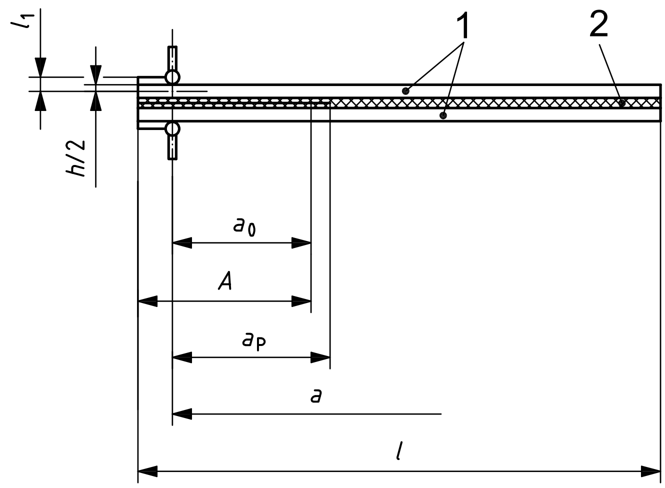
NOTE 2 For some toughened adhesives, the value of G_{IC} determined by this method has been shown to be a strong function of the thickness of the adhesive layer, h_{a}. As a result, careful consideration is required when selecting the value of h_{a}. For the selection of h_{a}, it is instructive to determine 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:

$$r_{\mathrm{p}} = \frac{1}{2\pi}\left(\frac{E_{\mathrm{a}} G_{\mathrm{IC}}}{\sigma_{\mathrm{y}}^{2}}\right) \quad \text{(plane stress);} \qquad r_{\mathrm{p}} = \frac{1}{6\pi}\left(\frac{E_{\mathrm{a}} G_{\mathrm{IC}}}{\sigma_{\mathrm{y}}^{2}}\right) \quad \text{(plane strain)}$$

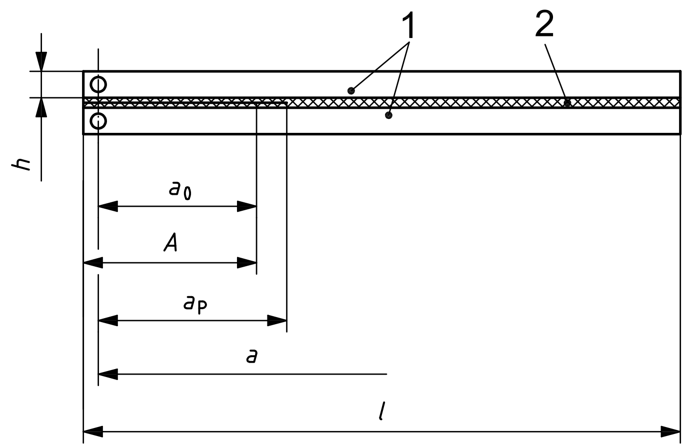
where E_{a} and σ_{y} are Young's modulus and the yield strength of the adhesive, respectively. Kinloch and Shaw [4] argued that, as the value of r_{p} was greater at the edges of the joint, where plane stress conditions exist, then the plane stress value was more applicable to the direct comparison with the adhesive layer thickness, h_{a}. If $h_{\mathrm{a}} \ll 2r_{\mathrm{p}}$, then the plastic zone may be largely suppressed and a low value of G_{IC} would be anticipated. If $h_{\mathrm{a}} \approx 2r_{\mathrm{p}}$, 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 $h_{\mathrm{a}} > 2r_{\mathrm{p}}$, 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

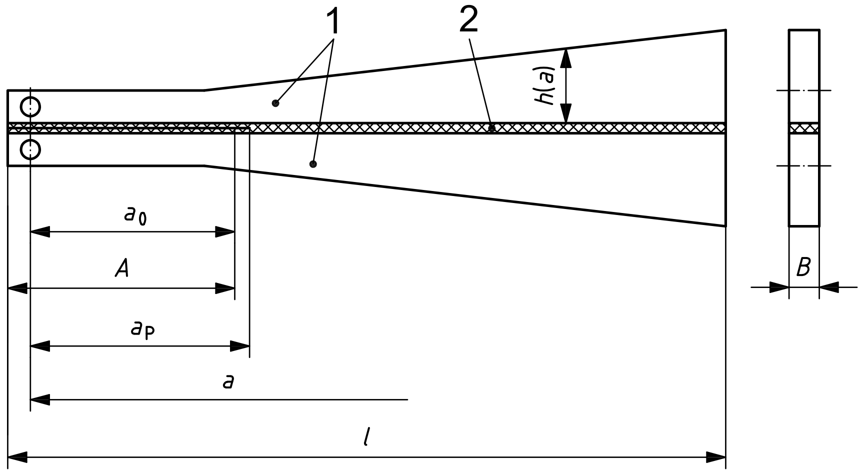


b) DCB specimen with piano hinges (alternative loading arrangement)



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

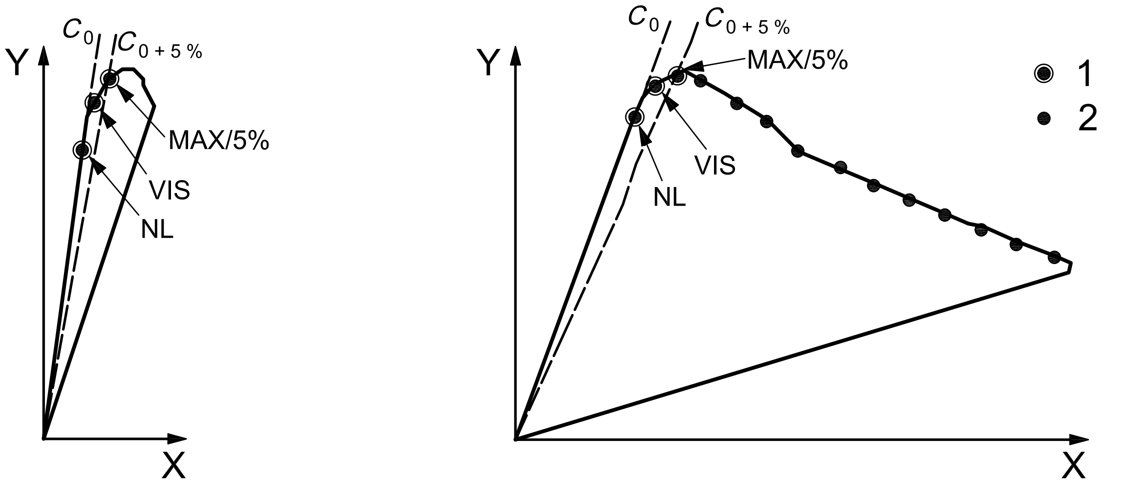
For testing from the mode I precrack which has been formed as a result of the test procedure in 7.2, load the specimen at a constant cross-head rate of

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.

Mark the position of the tip of the crack, i.e. mark the distance between the load-line and the tip of the crack on both edges of the specimen. If the crack lengths, a, on each of the two edges of the specimen, i.e. the distance between the load-line and the tip of the crack, differ by more than 2 mm, abandon the test.

NOTE This might indicate asymmetric loading or other problems with the test.

Break the joints open to enable the locus of joint failure to be visually assessed. Visually assess the locus of joint failure in accordance with ISO 10365.

7.4 Measurement of machine compliance

If the stiffness of the tensile-testing machine together with its associated grips and pins is not known, determine the compliance associated with the machine set-up as specified in Annex A. Take the compliance into account in the calculations presented in Clause 8, unless extensometry is used to measure the opening displacement of the specimen during the test, when the system compliance correction described in Annex A shall be neglected. Conduct the system compliance measurement after the fracture tests have been conducted so that the maximum load obtained from the fracture tests, and hence the load range over which the system compliance is to be measured, is known. Correct the displacement measured during the DCB or TDCB test to take account of the deflections in the loading system. For each test, use the corrected values of the displacement in the calculations.

If an extensometer attached to the specimen or loading pins is used to measure the displacement, then the system compliance correction is not made.

8 Data analysis

8.1 Determination of the raw data from the load-displacement trace

8.1.1 General

Disregard any initial non-linearity in the load-displacement trace by extrapolating the linear region of the loading curve back to zero load, as described in Annexes A and C. Determine initiation values (see 8.1.2) and propagation values (see 8.1.3) from the load-displacement trace.

8.1.2 Initiation values

Determine the crack length for the initiation values from the insert as the distance between the load-line and the tip of the insert, a_0. Determine the crack length for the initiation values from the precrack as the distance between the load-line and the tip of the precrack, a_p (see Figure 1). Determine the following initiation values, shown in Figure 2, for testing from the insert (starter film) and from the mode I precrack for each specimen:

a) NL: Determine the point of deviation from linearity by drawing a straight line from the origin, ignoring any initial deviations due to take-up of play in the loading system. Perform a linear fit on the load-displacement curve, starting at 5 % of the maximum load, using a consistent criterion for deviation from linearity (e.g. the half-thickness of the plotter trace).

   NOTE A region of non-linear behaviour usually precedes the maximum load, even if the unloading curve is linear. Experience has shown that it is difficult to determine reproducibly the position of NL on the load-displacement curve.

b) VIS: Determine the first point at which the crack is observed to move from the tip of the insert or of the mode I precrack on the edge of the specimen.

c) MAX/5 %: Determine the 5 % value as that point on the load-displacement curve at which the compliance has increased by 5 % of its initial value, C_0, as follows. Draw a best straight line to determine the initial compliance, C_0, ignoring any initial deviation due to take-up of play in the loading system, and draw a new line with a compliance equal to $C_{0+5\,\%}$. Mark the intersection of this new line with the load-displacement trace. Use whichever point occurs first, i.e. the maximum load (MAX) or the load at 5 % increase in the initial compliance.

8.1.3 Propagation values

Determine propagation values [PROP in Figure 2 b)] from the mode I precrack.

8.2 Determination of G_{IC}

8.2.1 General

Employ all three analysis methods shown below for the given test geometry, unless unstable (“stick-slip”) crack growth occurs, when only simple beam theory (SBT) is used (see Annex B).

8.2.2 Double cantilever beam (DCB)

8.2.2.1 Analysis method 1: Simple beam theory (SBT) (see Annex D)

Measure E_s from an independent modulus test or quote it if a standard grade of material is used.

Calculate G_{IC} from Equation (2) for the SBT method.

$$G_{IC} = \frac{4P^2}{E_s B^2}\left(\frac{3a^2}{h^3}+\frac{1}{h}\right) = \frac{4P^2}{E_s B^2}\cdot m \tag{2}$$

8.2.2.2 Analysis method 2: Corrected beam theory (CBT) (see Annex D)

NOTE 1 The simple beam theory expression for the compliance of a perfectly built-in DCB specimen will underestimate the compliance as the beam is not perfectly built-in.

Treat the beam as containing a slightly longer crack length, $(a + |\varDelta|)$; find $|\varDelta|$ experimentally by plotting the cube root of the compliance, $C^{1/3}$, or the cube root of the normalized compliance, $(C/N)^{1/3}$, if load-blocks are being used, as a function of crack length, a [see Figure 3 a)]. The load-block correction, N, is described in Equation (6).

Extrapolate a linear fit through the data in the plot to yield $\varDelta$ as the negative X-intercept. Use only the propagation (PROP) values for the linear fits, i.e. exclude all the initiation values from the linear fits.

Calculate G_{IC} using Equation (3) if piano hinges or drill holes are used to introduce the load, or Equation (4) if load-blocks are used:

$$G_{IC} = \frac{3P\delta}{2B\left(a+|\varDelta|\right)} \,.\, F \tag{3}$$

$$G_{IC} = \frac{3P\delta}{2B\left(a+|\varDelta|\right)} \cdot \frac{F}{N} \tag{4}$$

Calculate all applicable initiation and propagation values of G_{IC}. The load-block correction, N, is only applied if the specimen in Figure 1 a) is used.

NOTE 2 For piano hinges and for loading holes drilled directly though the substrate, $N = 1$. For loading holes drilled directly though the substrate, $l_1 = 0$. The large-displacement correction, F, becomes important if $\delta/a > 0{,}4$. In Equations (3) and (4), (7) and (8) and (9) and (10), Equation (3) or (7) or (9) is used if piano hinges or drill holes are used to introduce the load [see Figures 1 b) and 1 c)] and Equation (4) or (8) or (10) is used if load-blocks are used (see Figure 1 a).

Calculate the large-displacement correction, F, and the load-block correction, N, as shown in Equations (5) and (6), respectively:

$$F = 1 - \frac{3}{10}\left(\frac{\delta}{a}\right)^2 - \frac{3}{2}\left(\frac{l_1 \delta}{a^2}\right) \tag{5}$$

$$N = 1 - \left(\frac{l_2}{a}\right)^3 - \frac{9}{8}\left[1 - \left(\frac{l_2}{a}\right)^2\right]\frac{l_1 \delta}{a^2} - \frac{9}{35}\left(\frac{\delta}{a}\right)^2 \tag{6}$$

where

l_1 is the distance from the centre of the loading pin to the mid-plane of the arm of the substrate beam to which the load-block is attached;

l_2 is the distance from the loading-pin centre to the edge of the block (see Figure 1).

Consider data with large-displacement corrections, F, $<$ 0,9 as suspect.

NOTE 3 If the displacement correction, F, is $<$ 0,9 then, ideally, the test specimen should be redesigned, e.g. with thicker substrates, to reduce the required displacements and thus reduce the large-displacement correction factor.

Calculate the flexural modulus, E_f, as a function of the crack length, a, by using Equation (7) if piano hinges or drill holes are used to introduce the load, or Equation (8) if load-blocks are used:

$$E_f = \frac{8\left(a + |\Delta|\right)^3}{CBh^3} \tag{7}$$

$$E_f = \frac{8\left(a + |\Delta|\right)^3}{\frac{C}{N}Bh^3} \tag{8}$$

NOTE 4 This calculation is a useful check on the procedure as a value of the flexural modulus, E_f, independent of crack length should be obtained.

If the maximum variation of E_f is more than 10 % of the average, consider the values of G_{IC} as suspect.

The value E_f calculated from Equation (7) or (8) shall not be quoted as the modulus value and this value shall not be used in Equation (2), which requires an independently measured or known value of the modulus to be used.

8.2.2.3 Analysis method 3: Experimental compliance method (ECM) or Berry's method (see Annex D)

Plot the logarithm of the compliance, or of the normalized compliance, C/N, if load-blocks are being used, versus the logarithm of the crack length, a, as shown in Figure 3 b). Use only the propagation (PROP) values for the linear fits, i.e. exclude all the initiation values from the regression analysis.

Use the slope, n, of this plot to calculate G_{IC}. Use Equation (9) if piano hinges or drill holes are used to introduce the load, or use Equation (10) if load-blocks are used.

$$G_{IC} = \frac{nP\delta}{2Ba} \cdot F \tag{9}$$

$$G_{IC} = \frac{nP\delta}{2Ba} \cdot \frac{F}{N} \tag{10}$$

Calculate all applicable initiation and propagation values of G_{IC}. The same large-displacement correction, F, and load-block correction, N, if applicable, are used as for the corrected beam theory method (see 8.2.2.2).

8.2.3 Tapered double cantilever beam (TDCB)

8.2.3.1 Analysis method 4: Simple beam theory (SBT) (see Annex D)

Measure E_s from an independent modulus test, or quote it if a standard grade of material is used.

Calculate G_{IC} from Equation (2) for the simple beam theory (SBT) method given in 8.2.2.1.

In the test report, quote the value of m and the range of the crack lengths, a, for which this value of m is within ± 3 %.

Values of G_{IC} calculated where the value of m is outside the range ± 3 % shall be considered suspect.

8.2.3.2 Analysis method 5: Corrected beam theory (CBT) (see Annex D)

Calculate G_{IC} from Equation (11):

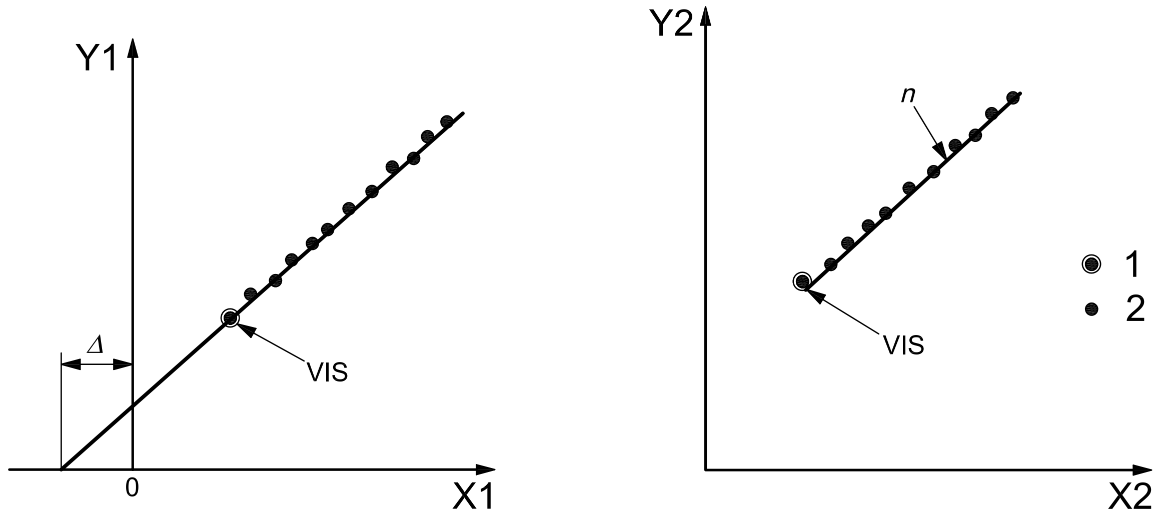
$$G_{IC} = \frac{4P^2 m}{E_s B^2}\left[1 + 0{,}43\left(\frac{3}{ma}\right)^{\frac{1}{3}}\right] \tag{11}$$

8.2.3.3 Analysis method 6: Experimental compliance method (ECM) (see Annex D)

Calculate the value of the adhesive fracture energy, G_{IC}, using Equation (12). Obtain the value of $\mathrm{d}C/\mathrm{d}a$ from the slope of the straight line of C plotted against a, using only the propagation (PROP) values in the regression analysis, i.e. excluding all initiation values from the linear fit.

NOTE For the TDCB geometry, when the values of C are plotted against the crack length, a, the resulting graph should be linear since

$$G_{IC} = \frac{P^2}{2B} \cdot \frac{\mathrm{d}C}{\mathrm{d}a} \tag{12}$$



a) **The correction for the corrected beam theory (CBT) method for the DCB test specimen**

b) **The slope n for the experimental compliance method (ECM) for the DCB test specimen**

Key

X_1 crack length, a

Y_1 $(C/N)^{1/3}$

X_2 $\log a$

Y_2 $\log(C/N)$

Δ X-axis intercept

n slope

1 initiation values

2 propagation values

NOTE The visual point is excluded from the linear regression analysis.

Figure 3 — Linear fits

9 Test report

9.1 Test report for the DCB test

The test report shall contain G_{IC} from SBT (method 1), G_{IC} from CBT (method 2) and G_{IC} from ECM (method 3) from Equations (2), (3) or (4) and (9) or (10), respectively, except when Annex B applies (i.e. unstable or "stick-slip" crack propagation is observed), when only G_{IC} from SBT (method 1) is obtained.

In addition, the following shall be included in the report:

a) the initiation points of G_{IC} (NL, VIS or MAX/5 % — see Figure 2), obtained from both the insert (starter film) and from the mode I precrack using the corresponding measured value of the crack length, a, i.e. a_0 or a_p;

b) the propagation values of G_{IC} [PROP in Figure 2 b)], determined from the mode I precrack as a function of crack length, a;

c) a resistance-curve (R-curve), i.e. G_{IC} versus crack length, a (see Figure 4), showing all initiation and propagation values (see Blackman, *et al.* [10]) from both the insert and the mode I precrack;

d) the flexural modulus, E_f, of the substrate [from Equation (7) or (8)] (the flexural or tensile modulus, E_s, of a substrate arm shall also be independently measured, or quoted if a known standard grade of material is employed, and the value recorded in the test report);

e) whether the locus of joint failure (see 7.3) is

1) cohesive, in the adhesive,

2) apparently interfacial along the adhesive/substrate interface,

or

3) cohesive, in the substrate.

If a mixture of such failure paths is seen, estimate and record the percentage of each type.

9.2 Test report for the TDCB test

The test report shall contain G_{IC} from SBT (method 4), G_{IC} from CBT (method 5) and G_{IC} from ECM (method 6) from Equations (2), (11) and (12), respectively, except when Annex B applies (i.e. unstable or "stick-slip" crack propagation is observed), when only G_{IC} from SBT (method 4) and G_{IC} from CBT (method 5) are obtained.

In addition, the following shall be included in the report:

a) the initiation points of G_{IC} (NL, VIS or MAX/5 % — see Figure 2), obtained from both the insert (starter film) and from the mode I precrack using the corresponding measured value of the crack length, a, i.e. a_0 or a_p;

b) the propagation values of G_{IC} [PROP in Figure 2 b)], determined from the mode I precrack as a function of crack length, a;

c) a resistance-curve (R-curve), i.e. G_{IC} versus crack length, a, (see Figure 4), showing all initiation and propagation values (see Blackman, *et al.* [10]) from both the insert and the mode I precrack;

d) the value of the slope, $\mathrm{d}C/\mathrm{d}a$, of the graph of C versus a and the correlation coefficient, r^2, for the graph;

e) whether the locus of joint failure (see 7.3) is

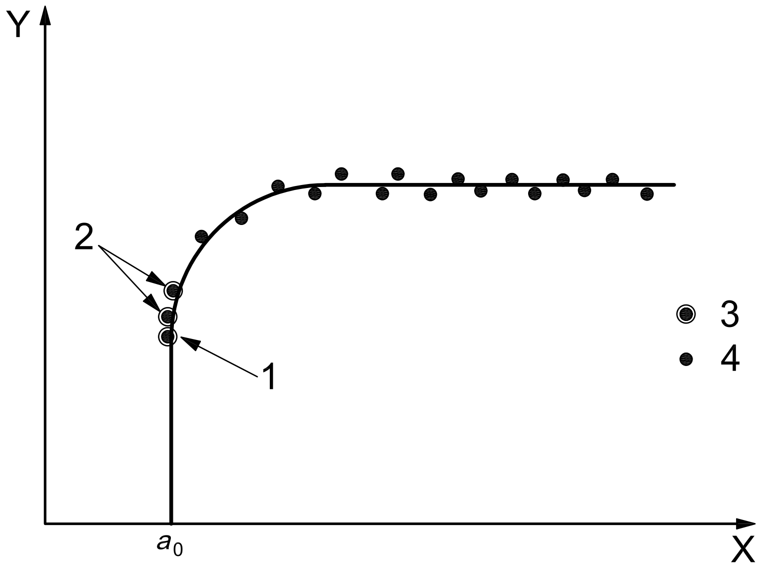
1) cohesive, in the adhesive,

2) apparently interfacial along the adhesive/substrate interface,

or

3) cohesive, in the substrate.

If a mixture of such failure paths is seen, estimate and record the percentage of each type.



Key

X crack length, a

Y G_{IC}

1 lowest initiation point (lowest value among NL, VIS and MAX/5 % from insert or precrack)

2 other initiation points

3 initiation values

4 propagation values

For both DCB and TDCB specimens, the specimen type shall be stated.

Figure 4 — Schematic resistance-curve (R-curve) with G_{IC} values for initiation (i.e. the lowest value among NL, VIS and MAX/5 %) and for propagation (PROP) versus observed crack length, a

Annex A
(informative)

Measurement of test system compliance

CAUTION — Ensure that this procedure is carried out by experienced personnel, otherwise damage to equipment can occur when the calibration specimen is loaded.

NOTE 1 It has been observed from round-robin testing that this correction procedure can have a significant effect on the shape of the R-curves and on the values of the back-calculated modulus in the DCB tests.

The apparatus specified in 5.1 shall be used. Set up the tensile-loading system in exactly the manner that was used for the fracture testing. Define the calibration load, P_{cal}, as the maximum load during fracture testing.

Connect a rigid calibration specimen of known compliance, C_{cs}, to the loading system. If pins of circular cross-section were used to load the fracture specimens, use these to load the calibration specimen.

NOTE 2 A calibration specimen made from mild steel with cross-sectional dimensions of 20 mm by 25 mm and a distance between loading-hole centres of 25 mm has been found to work satisfactorily and will possess a compliance which is usually negligible when compared to the system compliance, C_{sy}.

Start to load the specimen at a very slow rate, e.g. 0,05 mm/min, up to the calibration load value, P_{cal}. If using a chart recorder to monitor displacement, run this at 100 times the rate of the cross-head. When the load reaches the value of P_{cal}, stop the cross-head and unload the sample.

NOTE 3 The load will rise rapidly during this procedure, so care should be taken not to overload the load-cell.

Draw the best straight line through the linear part of the load-displacement trace obtained, thus ignoring the initial non-linearity due to take up of play, as shown in Figure A.1.

NOTE 4 This take up of play is also ignored in the fracture tests.

Deduce the total compliance, C_{total}, of the combined system and calibration specimen (in mm/N) from this straight line, as shown in Figure A.1.

NOTE 5 $C_{\text{total}} = \delta_{\text{cal}}/P_{\text{cal}}$

Calculate the value C_{sy} (in mm/N) from Equation (A.1):

$$C_{\text{sy}} = C_{\text{total}} - C_{\text{cs}} \tag{A.1}$$

Correct all the displacement values measured during the fracture tests, i.e. at each value of the crack length that was recorded, using Equation (A.2):

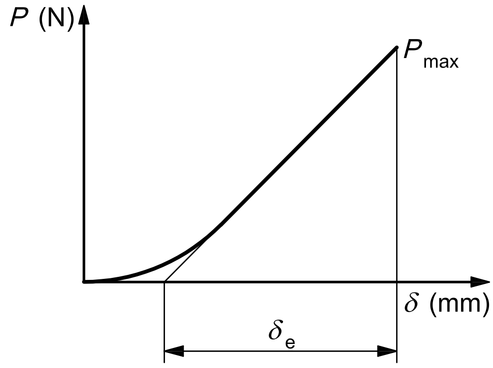
$$\delta_{\text{cor}} = \delta - PC_{\text{sy}} \tag{A.2}$$

where

δ_{cor} is the corrected value of the displacement (in mm) to be used in the equations in Clause 8;

δ is the value of the displacement (in mm) measured in the fracture tests;

P is the corresponding load.



Key

δ_e effective cross-head displacement (ignoring initial non-linearity due to take-up of play)

Figure A.1 — Schematic load-displacement trace obtained during the system compliance measurement

Annex B
(normative)

Procedure to follow when unstable or “stick-slip” crack growth is observed during the fracture test

NOTE 1 It is not uncommon for adhesive joints to exhibit unstable or “stick-slip” crack propagation during a DCB or TDCB fracture test. A schematic example of a load-displacement trace obtained from a TDCB joint exhibiting “stick-slip” crack growth behaviour is shown in Figure B.1. During this type of propagation, the crack grows in short bursts separated by periods of crack arrest. Sometimes the propagation is partly stable and partly unstable. The reasons for this type of behaviour are not fully understood.

NOTE 2 When “stick-slip” crack propagation is observed during a DCB or TDCB test, it will not be possible to monitor the crack propagation.

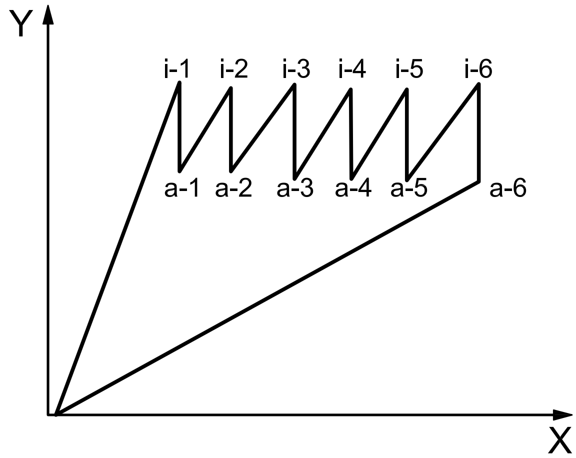
Observe the first initiation value of the crack length and the crack lengths at subsequent arrest points, using a travelling microscope.

After the crack has propagated sufficiently down the specimen, fully unload the joint and record the unloading trace recorded in the same way as for a stable test. Break the joint open to reveal arrest lines on the adhesive so that more accurate crack-length measurements can be made.

NOTE 3 Between one arrest point and the next initiation point, the crack will remain stationary. There might be some stable crack growth before further instability, or the crack might jump directly from the previous arrested value of the crack length.

Record that “stick-slip” crack propagation was observed, and clearly indicate alongside each data point the type of point, i.e. initiation or arrest, in the test report.

Do not average initiation and arrest values of G_{IC} together. However, average values of G_{IC} from the initiation points and values of G_{IC} from the arrest points can be deduced separately.



Key

X displacement (mm)

Y force (N)

i indicates initiation points

a indicates arrest points

Figure B.1 — Schematic force-displacement trace for a tapered double cantilever beam specimen exhibiting unstable “stick-slip” crack growth behaviour

Annex C
(normative)

Procedure to detect the occurrence of plastic deformation during a DCB or TDCB adhesive joint test

A schematic load-displacement trace obtained from testing a bonded tapered double cantilever beam specimen is shown in Figure C.1. The complete load, propagation and unload cycle obtained during the test from the mode I precrack is shown.

Draw the best straight lines through the data, for both the loading and unloading parts of the trace, ignoring any initial non-linearity due to the take up of play in the system. Extrapolate the lines back to zero load. Measure the values of the distance between the intercepts of these two lines with the displacement axis, δ_{offset}, and the maximum value of the displacement attained during the test, δ_{max}.

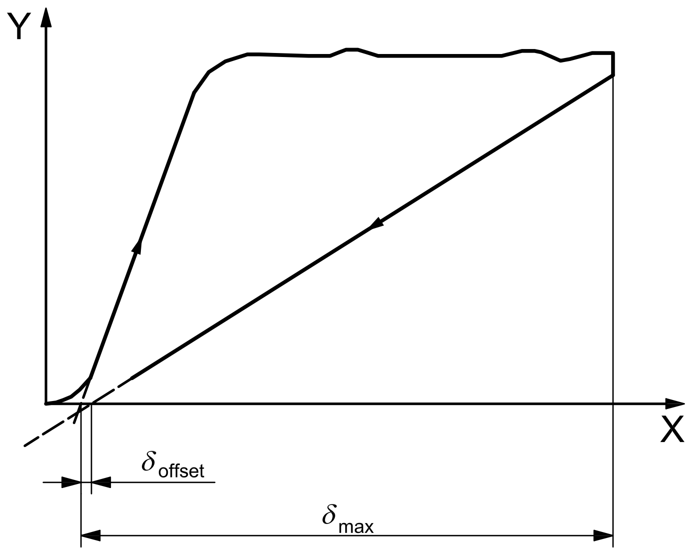
NOTE 1 It is normal for the term δ_{offset} to be non-zero.

Calculate the value of $\delta_{\text{offset}}/\delta_{\text{max}}$ for each test and note this value on the results sheet.

Carefully break the joint open after the complete test cycle is finished. Visually determine whether plastic deformation has occurred: plastic deformation of the substrate arms will have occurred if they remain permanently deformed on separation. In the case of tapered-beam specimens, hold the substrates back together as they were before separation.

Note the straightness of the beams after breaking open in the test report.

NOTE 2 Experience has shown that plastic deformation of the substrates can be suspected if $\delta_{\text{offset}}/\delta_{\text{max}} > 0{,}05$, where δ_{max} is the displacement required to extend the crack by the distance given in 7.3.



Key

X displacement (mm)

Y force (N)

Figure C.1 — Typical force-displacement trace for a tapered double cantilever beam specimen, showing loading and unloading lines and the displacement offset

Annex D
(informative)

Summary of background theory

D.1 General

The test methods specified in this International Standard enable the fracture resistance of structural adhesive joints to be determined under mode I tensile-loading conditions. The use of either the double cantilever beam (DCB) specimen or the tapered double cantilever beam (TDCB) specimen is accommodated.

The methods describe the measurement of the resistance both to crack initiation and to crack propagation through the adhesive layer. Points of crack initiation are determined directly from an insert film, moulded into the centre of the adhesive layer during joint manufacture, and from a mode I precrack created during the test. The property measured by these methods is the mode I adhesive fracture energy, G_{IC}. G_{IC} may also be described as the critical strain energy release rate, the energy release rate or the fracture toughness of the joint.

The double cantilever beam (DCB) specimen is well suited for testing joints consisting of an adhesive bonding relatively thin sheets of fibre-composite materials, but may also be used when metallic substrates, which possess a relatively high yield stress, are employed [see e.g. Figure 1 c)].

The tapered double cantilever beam (TDCB) specimen is designed so that, over a large range of values of crack length, the rate of change of compliance with crack length is constant and so is independent of the value of the crack length. This is useful since

a) relatively tough adhesives can be tested without plastic deformation of the arms occurring;

b) the substrates can possess a relatively low yield stress, but again no plastic deformation of the arms is encountered during the test;

c) the measurement of the adhesive fracture energy, G_{IC}, is independent of the crack length, a.

There are three analysis methods for both the DCB test and the TDCB test. The corrected beam theory (CBT) method and the experimental compliance method (ECM) are considered to be the more accurate methods for determining the value of G_{IC}.

NOTE DCB specimens are cheaper to manufacture, so are the test specimen of first choice provided plastic deformation of the substrates is avoided. For tougher adhesives and substrates with lower yield stresses, i.e. in joints more likely to violate LEFM test conditions, the TDCB is favoured.

To develop a linear change of compliance with crack length, the height of the specimen is varied by contouring the substrate beam so that the specimen geometry factor, m, is a constant [see Equation (D.1)].

$$\frac{3a^2}{h^3} + \frac{1}{h} = m \qquad \text{(D.1)}$$

where

a is the crack length;

h is the thickness of the substrate beam at crack length a.

The equations used to calculate the adhesive fracture energy, G_{IC}, in the double cantilever beam (DCB) test method (analysis methods 1 to 3) and in the tapered double cantilever beam (TDCB) test method (analysis methods 4 to 6) are described here.

D.2 Analysis method 1: Simple beam theory (SBT) — Double cantilever beam (DCB)

The value of the adhesive fracture energy, G_{IC}, is calculated using Equation (D.2):

$$G_{IC} = \frac{P^2}{2B} \cdot \frac{dC}{da} \tag{D.2}$$

where

C is the compliance, given by δ/P;

B is the width of the specimen;

P is the load measured by the load-cell of the test machine.

For thin adhesive layers, it has been shown by Mostovoy, *et al.* [5] and Kinloch [6] that, from simple beam theory, dC/da may be expressed as Equation (D.3):

$$\frac{dC}{da} = \frac{8}{E_s B}\left(\frac{3a^2}{h^3} + \frac{1}{h}\right) \tag{D.3}$$

where E_s is the independently measured flexural or tensile modulus of the substrate.

By combining Equations (D.1), (D.2) and (D.3), Equation (D.4) is obtained and G_{IC} can be calculated:

$$G_{IC} = \frac{4P^2}{E_s B^2}\left(\frac{3a^2}{h^3} + \frac{1}{h}\right) = \frac{4P^2}{E_s B^2} \cdot m \tag{D.4}$$

D.3 Analysis method 2: Corrected beam theory (CBT) — Double cantilever beam (DCB)

The simple beam theory expression for the compliance of a perfectly built-in DCB specimen will underestimate the compliance as, in practice, the beam is not perfectly built-in (see Hashemi, *et al.* [7] and Blackman, *et al.* [8]).

The adhesive fracture energy, G_{IC}, is calculated using Equation (D.5) if piano hinges or drill holes are used to introduce the load, or Equation (D.6) if load-blocks are used:

$$G_{IC} = \frac{3P\delta}{2B\left(a + |\Delta|\right)} \cdot F \tag{D.5}$$

$$G_{IC} = \frac{3P\delta}{2B\left(a + |\Delta|\right)} \cdot \frac{F}{N} \tag{D.6}$$

where

N is a load-block correction;

F is a large-displacement correction;

δ is the displacement;

Δ is a crack-length correction for a beam that is not perfectly built-in.

The large-displacement correction, F, and the load-block correction, N, are calculated as shown in Equations (D.7) and (D.8), respectively (see Hashemi, *et al.* [7]):

$$F = 1 - \frac{3}{10}\left(\frac{\delta}{a}\right)^2 - \frac{3}{2}\left(\frac{l_1 \delta}{a^2}\right) \tag{D.7}$$

$$N = 1 - \left(\frac{l_2}{a}\right)^3 - \frac{9}{8}\left[1 - \left(\frac{l_2}{a}\right)^2\right]\frac{l_1 \delta}{a^2} - \frac{9}{35}\left(\frac{\delta}{a}\right)^2 \tag{D.8}$$

where

l_1 is the distance from the centre of the loading pin to the mid-plane of the arm of the substrate beam to which the load-block is attached;

l_2 is the distance from the loading pin centre to the edge of the block (see Figure 1).

The flexural modulus, E_f, is calculated as a function of the crack length, a, by using Equation (D.9) if piano hinges or drill holes are used to introduce the load, or Equation (D.10) if load-blocks are used:

$$E_\text{f} = \frac{8\left(a + |\Delta|\right)^3}{CBh^3} \tag{D.9}$$

$$E_\text{f} = \frac{8\left(a + |\Delta|\right)^3}{\frac{C}{N} Bh^3} \tag{D.10}$$

This calculation is a useful check on the procedure, as a value of the flexural modulus E_f independent of crack length should be obtained.

D.4 Analysis method 3: Experimental compliance method (ECM) or Berry's method — Double cantilever beam (DCB)

The logarithm of the compliance, or of the normalized compliance, C/N, if load-blocks are being used, is plotted against the logarithm of the crack length, a. The slope, n, of this plot gives G_IC as follows:

$$G_\text{IC} = \frac{nP\delta}{2Ba} \cdot F \tag{D.11}$$

$$G_\text{IC} = \frac{nP\delta}{2Ba} \cdot \frac{F}{N} \tag{D.12}$$

The same large-displacement correction, F, and load-block correction, N, if applicable, are used as for the corrected beam theory method.

D.5 Analysis method 4: Simple beam theory (SBT) — Tapered double cantilever beam (TDCB)

This is the same as in analysis method 1, except the value of m is a constant for this particular geometry.

D.6 Analysis method 5: Corrected beam theory (CBT) — Tapered double cantilever beam (TDCB)

The simple beam theory expression for G_{IC} described in analysis method 4 will incorrectly estimate the compliance of the specimen since:

a) the positions of the loading pins, with their surrounding material, are not taken into account in deriving Equation (D.4);

b) as for the DCB specimen, the specimen does not behave as a perfectly built-in cantilever beam.

Corrections (see Blackman, *et al.* [9]) lead to Equation (D.13):

$$\frac{\mathrm{d}C}{\mathrm{d}a} = \frac{8m}{E_{\mathrm{s}}B}\left[1+0{,}43\left(\frac{3}{ma}\right)^{\frac{1}{3}}\right] \tag{D.13}$$

Combining Equations (D.2) and (D.13), Equation (D.14) is obtained:

$$G_{\mathrm{IC}} = \frac{4P^2m}{E_{\mathrm{s}}B^2}\left[1+0{,}43\left(\frac{3}{ma}\right)^{\frac{1}{3}}\right] \tag{D.14}$$

In deriving Equation (D.13), the value of m is approximated to $3a^2/h^3$, i.e. the term $1/h$ in Equation (D.1) is neglected. The error in the value of G_{IC} that is introduced by this approximation is insignificant, and round-robin testing has demonstrated good agreement between the values of G_{IC} deduced via Equations (D.14) and (D.2) for tapered beams manufactured with aluminium alloy substrates (see Blackman, *et al.* [10]).

D.7 Analysis method 6: Experimental compliance method (ECM) — Tapered double cantilever beam (TDCB)

The adhesive fracture energy, G_{IC}, is calculated using Equation (D.2), where the values of C plotted against the crack length, a, produce a linear graph.

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