Protocol for the Determination of the Mode I Adhesive Fracture Energy, G_{IC}, of Structural Adhesives using the Double Cantilever Beam (DCB) and Tapered Double Cantilever Beam (TDCB) Specimens.

(Version 00-08. B.R.K. Blackman and A.J. Kinloch. 22-06-00)

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Reference:

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1. Scope

This 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 the Double Cantilever Beam (DCB) and Tapered Double Cantilever Beam (TDCB) Specimens. The resistance to both crack initiation and propagation are to be determined. The resistance to crack initiation is to be determined from both a non-adhesive insert placed in the adhesive layer and from a mode I precrack. The resistance to crack propagation is to be 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 can be calculated and a resistance-curve (R-curve, i.e. a plot of the value of the adhesive fracture energy G_{IC} versus crack length) can be determined.

2. Normative References

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

ISO 291: l977	*Plastics; standard atmospheres for conditioning and testing.*
ISO 4588: 1991	*Adhesives; preparation of metal surfaces for adhesive bonding.*
ISO 10365: 1992	*Adhesives, designation of main failure patterns.*
ISO 5893: 1993	*Rubber and plastics test equipment; tensile, flexural and compression types (constant rate of traverse); description.*

3 Definitions

For the purpose of this standard the following symbols and conventions apply:

a	crack length, 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 (Figure 1)
a_0	insert film length, distance between the load-line to the tip of the insert film.
a_p	precrack length, measured from the load-line to the tip of the mode I precrack.
A	insert film length, distance between the end of the specimen and the tip of the insert film (Figure 1)
B	width of the specimen
D	crack length correction for beam that is not perfectly built-in
d	displacement of the cross-head of the testing machine
$dcor$	the displacement of the cross-head, corrected for system compliance effects
C	compliance δ/P of the specimen
C_o	initial compliance of the specimen neglecting start-up effects, e.g. due to play in the specimen fixture
C_{max}	compliance of the specimen at maximum load
$C_{5\%}$	initial compliance C_o of the specimen increased by 5%
Csy	the compliance of the tensile loading system
$Ctotal$	the compliance of the tensile loading system and the calibration specimen used to measure this (see annex A.1).
Ccs	the compliance of the calibration specimen used to measure the system compliance.
E_f	flexural modulus of the arms of the substrate beam, calculated from the DCB Mode I crack propagation test

E_s	independently-measured flexural, or tensile, modulus of the arms of the substrate beam
F	large displacement correction
G_{IC}	critical strain energy release rate, or adhesive fracture energy, for applied Mode I opening load
H	thickness of the load-block
h	thickness of each substrate beam at a crack length, a
h_a	thickness of the adhesive layer
l	total length of the specimen
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 (Figure 1)
l_2	distance between the centre of the pin-hole of the load-block and its edge, measured towards the tip of the insert (starter film) or the tip of the Mode I precrack (Figure 1)
l_3	total length of the load-block (Figure 1)
MAX	maximum load of the load-displacement curve (Figure 2)
m	specimen geometry factor (Equation (1))
N	load-block correction
NL	onset of non-linearity on the load-displacement curve
n	slope of a plot of log C versus log a or of log (C/N) versus log a, if load-blocks are being used
P	load measured by the load-cell of the testing machine
PROP	increments of the crack length during stable crack growth (propagation) that are marked on the load-displacement curve (Figure 2)
r^2	correlation coefficient of linear fits
r.h.	relative humidity during test
T	test temperature
T_{mc}	maximum cure temperature of the adhesive

T_d	conditioning (drying) temperature of the joint
t_c	duration of curing of the adhesive
t_d	duration of conditioning (drying) of the joint
VIS	onset of visually recognisable crack growth on the edge of the specimen that is marked on the load-displacement curve (Figure 2)
5%	point of intersection of a straight line with the load-displacement curve, with the slope of the straight line corresponding to $C_{5\%}$

4. Principle

This standard uses the Double Cantilever Beam (DCB) specimen, shown in Figures 1a-c, or the Tapered Double Cantilever Beam (TDCB), shown in Figure 1d, for the determination of the adhesive fracture energy, G_{IC}, of structural adhesive joints.

The Double Cantilever Beam (DCB) specimen shown in Figures 1a-c is well-suited for testing joints consisting of an adhesive which is bonding relatively thin sheets of fibre-composite materials, but may also be used when metallic substrates which possess a relatively high yield stress are being employed (e.g. Figure 1c).

The Tapered Double Cantilever Beam (TDCB) shown in Figure 1d 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 independent of the value of crack length. This is useful since it means that (i) relatively tough adhesives may be tested without plastic deformation of the arms occurring, (ii) the substrates may possess a relatively low yield stress, but again no plastic deformation of the arms may be incurred during the test, and (iii) the measurement of the adhesive fracture energy, G_{IC}, is independent of the crack length, a. 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 quantity:

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

is a constant, where m is the specimen geometry factor.

5. Apparatus

(1) A tensile testing machine in compliance with ISO 5893, capable of producing a constant cross-head displacement-rate between 0.1 and 5 mm/min in displacement control should be used. The testing machine shall be equipped (i) with a fixture to introduce the load to the pins inserted into the load-blocks, or directly into the substrate beams, or (ii) with grips to hold the piano hinges that both allow rotation of the specimen end, see Figure 1.

(2) The testing machine should incorporate a load-cell which should be calibrated and accurate within ±1% for the chosen load-range (loads are typically expected to be in the range of 100-5000 N). The opening displacement of the test specimen should be deduced from the position of the cross-head. The testing machine shall be equipped with means for recording the complete load versus displacement curves (loading and unloading) during the test. (Note that if extensometry is used to measure the opening displacement of the specimen during the test, then the system compliance correction described in Annex A1 can be neglected.)

(3) The crack length should be measured along the edge of the specimen to an accuracy of at least ± 0.5 mm by either using a travelling microscope or a video camera with suitable magnification.

(4) The thickness of DCB specimen arms should be measured with a micrometer or equivalent with an accuracy of 0.02 mm or better. For measuring the width of the joints, a micrometer or vernier calipers with an accuracy of 0.05mm or better shall be used.

6. Specimens

6.1 Manufacture of adhesive joint specimens

It is not the purpose of this document to give full manufacturing details of the joints to be tested. Such information should be sought from the adhesive manufacturer and or the substrate manufacturer. Appropriate surface treatments may also be determined by reference to *ISO 4588* for metallic substrates. The thickness of the film to be inserted in the bondline during manufacture should be less than 13 microns. A PTFE film is recommended although other release films have been successfully used. If aluminium foil is used, the foil should be coated with releasing agent prior to use. The thickness of the adhesive bondline should be carefully controlled. When fully cured, any excess adhesive should be removed by mechanical means to leave the joint with smooth sides.

6.2 Measurement of specimen dimensions

(1) When DCB substrates have been prepared, the thickness of each substrate should be measured using a micrometer *before* bonding. Measurements should be made at three points along the length of the beam (at 30 mm from either end, and at the mid length) and the average obtained. Thus, a value of the thickness of each substrate, h, is obtained.

(2) If the thickness measurements are repeated on the joint after bonding, a value of the bondline thickness, h_a, may be determined by subtracting the substrate thicknesses, nominally 2h, from the total thickness of the joint.

(3) The width, B, of the DCB or TDCB joint should be measured *after* bonding with vernier callipers or a micrometer at three points along the length of the beam (at 30 mm from either end, and at the mid length) and the average obtained. These width measurements should be made *after* any excess adhesive has been removed from the sides of the beam.

6.3 Preparation of Specimens

(1) Adding a thin layer of typewriter correction fluid ("white ink"), or white spray-paint, on the edges of the sample after conditioning will facilitate the detection of the crack growth. It should be noted that some typewriter correction fluids and paints contain solvents which may be harmful to the adhesive or the laminate matrix material of a composite substrate.

(2) For the measurement of the crack growth, marks should indicate every 1 mm from the tip of the insert or of the Mode I crack for at least the first 10 mm, then marks should be applied every 5 mm. Also, such marks should be applied for every 1 mm for the final 5 mm. For the

DCB test specimen the recommended extent of crack propagation is 65 mm, and for the TDCB test specimen the recommended extent of crack propagation is 100 mm.

7. Number of specimens

A minimum of four repeat joints should be tested.

8. Conditioning

The joints should be maintained in normal laboratory conditions for a minimum of twenty four hours prior to testing i.e. they should be maintained at a temperature of 23°C±2°C, and at a relative humidity of 50%±5%.

9. Test Procedure

9.1 Test Set-up and Data Recording

(1) The tests shall be performed under normal conditions in accordance with ISO 291 (23°±2°C, 50%±5% relative humidity) unless prescribed otherwise. After mounting the specimen in the fixture of the testing machine, the end of the specimen may have to be supported in order to keep the beam orthogonal to the direction of the applied load. The load and the displacement signals of the testing machine shall be recorded, either on a paper chart or electronically throughout the test, including the unloading cycle. If using a tensile testing machine with a paper chart recorder, then the following ratios of cross-head speed to chart speed are recommended. When testing joints with metallic substrates, a ratio of cross-head speed to chart speed of about 1:100 is recommended. When testing joints with fibre-composite substrates, a ratio of cross-head speed to chart speed of about 1:10 is recommended.

(2) The crack length should be measured along the edge of the specimen to an accuracy of at least ± 0.5mm by either using a travelling microscope or a video camera with suitable magnification. If unstable crack growth followed by arrest ("stick-slip") is observed during any stage of the test, it should be noted in the report, see Annex B1 for more details.

9.2 Initial loading (the pre-cracking stage).

(1) For testing from the insert (starter film) it is recommended that the specimen should be loaded at a constant cross-head rate of:

(a) 0.1 mm/min for joints prepared using metallic substrates; or

(b) 1.0 mm/min for joints prepared using fibre-composite (with polymeric matrix) substrates.

(2) 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 should be recorded on the load-displacement curve or in the sequence of load-displacement signals (VIS, Figure 2a).

(3) The loading should be stopped as soon as the crack is seen to move on the edge of the specimen, after which the specimen should be completely unloaded at a constant cross-head rate. Unloading may be performed at up to five times the loading rate. The position of the tip of the precrack should be marked 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 the results should be considered suspect and this be noted in the report.

9.3 Re-loading: testing from the mode I precrack

(1) For testing from the mode I precrack, which has been formed as a result of the above test procedure, it is recommended that the specimen be loaded at a constant cross-head rate of:

(a) 0.1 mm/min for joints prepared using metallic substrates; or

(b) 1.0 mm/min for joints prepared using fibre-composite (with polymeric matrix) substrates.

(2) The point on the load-displacement curve at which the onset of crack movement from the Mode I precrack is observed on the edge of the specimen should be recorded on the plot or in the sequence of load-displacement signals (VIS, figure 2b).

(3) After this, as many crack length increments as possible should be noted in the first 5 mm on the corresponding load-displacement curves, ideally every 1 mm. Subsequently, crack lengths are noted 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 tests. Then again every 1 mm for the last 5 mm of crack propagation.

(4) After this, the specimen should be unloaded at a constant cross-head rate. The unloading may be performed at up to five times the loading rate. A note should be made in the report if the load-displacement curve does not return to its initial point, since this indicates that permanent plastic deformation of the arms of the specimen may have occurred, see Annex A2.

(5) The position of the tip of the crack edges of the specimen, i.e. the distance between the load-line and the tip of the crack should be marked 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 crack, differ by more than 2 mm the results should be considered suspect and this be noted in the report.

9.4 Measurement of the Machine Compliance

The tensile testing machine with associated grips and pins will not have an infinite stiffness and hence the compliance associated with the machine set-up should be determined and taken into account in the calculations presented in section 10. This is most conveniently achieved by correcting the displacement measured during the DCB or TDCB test to take account of the deflections in the loading system. The procedure for measuring the system compliance is given in the Normative Annex A1 to this document and this should be followed. It is advisable to conduct the system compliance measurement after the fracture tests have been conducted so that the maximum load obtained from the fracture tests is known. This then determines the load range over which the system compliance is measured. For each test, the corrected values of the displacement are then used in the calculations which follow.

10. Data Analysis

10.1 Determining the raw data from the load-displacement trace

The data required for the analysis are the crack lengths a and the corresponding loads P and displacements δ. The values of δ deduced from the load-displacement record should be corrected for system compliance as described in Annex A1 prior to the determination of G_{IC}. Also, any initial non-linearities in the load-displacement trace should be disregarded by extrapolating the linear region of the loading curve back to zero load, as described in Annexes A1 and A2. The following values should be determined from the load-displacement trace.

Initiation values

The crack length for the initiation values from the insert is the distance between the load-line and the tip insert, a_o. The crack length for the initiation values from the precrack is the distance between the load-line and the tip of the precrack, a_p (Figure 1). If possible, the following initiation values, shown in Figure 2, should be determined for testing from the insert (starter film) and from the Mode I precrack for each specimen:

(1) NL, i.e. deviation from linearity: A region of non-linear behaviour usually precedes the maximum load, even if the unloading curve is linear. The point of deviation from linearity (NL in Figure 2) , is determined by drawing a straight line from the origin but ignoring any initial deviations due to take-up of play in the loading system. Experience has shown that it is difficult to reproducibly determine the position of NL on the load-displacement curve. Performing a linear fit on the load-displacement curve starting at 5% of the maximum load and using a consistent criterion for deviation from linearity (e.g the half-thickness of the plotter trace) is recommended.

(2) VIS, i.e. visual observation: This corresponds to the onset of crack growth, i.e. to 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 (VIS in Figure 2).

(3) 5% or MAX, i.e. 5% increase of compliance or maximum load point: The 5% value corresponds to the point on the load-displacement curve at which the compliance has increased by 5% of its initial value C_O. A best straight line is drawn to determine the initial compliance C_O, ignoring any initial deviation due to take-up of play in the loading system. A new line is then drawn with a compliance equal to C_O + 5%, and the intersection of this new line with the load-displacement trace marked. Which ever point occurs first (i.e. max load or 5%) that is the point to be used.

Propagation values

Besides the initiation points (i.e. NL, VIS, 5% or MAX) obtained both from the insert (starter film) and from the Mode I precrack, propagation values (PROP in Figure 2b) should also be determined. These are determined from the Mode I precrack.

10.2 Determining the values of G_{IC}

As the development of this test protocol is still underway, it is recommended that users employ all the methods of analysis shown below for the given test geometry and these values should be quoted in the report. There are three analysis methods for both the DCB test and the TDCB test. If it is not possible for all methods to be used, then it is recommended that the experimental compliance method be used for both DCB and TDCB tests. Since this method, together with corrected beam theory (CBT) method, are considered to be the more accurate methods for determining the values of G_{IC}. However, if unstable ('stick-slip') crack growth occurs then the simple beam theory (SBT) method should be used for both the DCB and TDCB tests, see the Informative Annex B1 for more details.

10.2.1 Double Cantilever Beam (DCB) Tests

Method (1): Simple Beam Theory (SBT)

The value of the adhesive fracture energy, G_{IC}, may be ascertained from:

$$\mathbf{G_{IC} = \frac{P^2}{2B} \times \frac{dC}{da}} \qquad \textbf{(2)}$$

where C is the compliance and is given by displacement, δ/load, P. For thin adhesive layers, it has been shown (References 1 and 2) from simple beam theory that dC/da may be expressed:

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

where E_S is the independently-measured flexural or tensile modulus of the substrate. This value of the modulus should be measured from an independent modulus test, or quoted if a standard grade of material is used. Hence, combining Equations (1), (2) and (3):

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

where h is the thickness of the arm of one substrate beam.

For the simple beam theory (SBT) method of calculation, the value of G_{IC} should be calculated via Equation (4).

Method (2): Corrected Beam Theory (CBT)

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. A means of correcting for this effect is to treat the beam as containing a slightly longer crack length (a $+|\Delta|$); and $|\Delta|$ may be found experimentally by plotting the cube root of the compliance $C^{1/3}$, or the cube-root of the normalised compliance $(C/N)^{1/3}$, if load-blocks are being used, as a function of crack length a (Figure 3). The load-block correction N is described below. The extrapolation of a linear fit through the data in the plot yields Δ as the negative x-intercept (References 3 and 4). The propagation (PROP) values only are used for the linear fits i.e. all the initiation values are excluded from the linear fits. The adhesive fracture energy G_{IC} is given by:

$$G_{IC} = \frac{3P\delta}{2B(a + |\Delta|)} \quad \text{... (5a)} \quad \text{or} \quad G_{IC} = \frac{3P\delta}{2B(a + |\Delta|)} \cdot \frac{F}{N} \quad \text{... (5b)} \qquad (5)$$

where P is the load, δ the displacement, a the crack length, and B the width of the specimen. All initiation and propagation values of G_{IC}, if applicable, should be calculated. The load-block correction N is applied if load-blocks are being used, for piano hinges and for loading holes drilled directly though the substrate N = 1. The large displacement correction F becomes important if the displacement δ divided by the crack length a, $\delta/a > 0.4$. The large displacement correction F and the load-block correction N are calculated as follows:

$$F = 1 - \frac{3}{10}\left(\frac{\delta}{a}\right)^2 - \frac{3}{2}\left(\frac{\delta l_1}{a^2}\right) \qquad (6)$$

$$N = 1 - \left(\frac{l_2}{a}\right)^3 - \frac{9}{8}\left[1 - \left(\frac{l_2}{a}\right)^2\right]\frac{\delta l_1}{a^2} - \frac{9}{35}\left(\frac{\delta}{a}\right)^2 \qquad (7)$$

where l_1 is the distance from the centre of the loading pin to the mid-plane of the arm of the substrate beam and l_2 the distance from the loading pin centre to the edge of the block (Figure 1). Data with large displacement corrections F < 0.9 should be considered suspect and this be noted in the report.

This approach allows the flexural modulus E_f to be calculated as a function of the crack length a by using:

$$E_f = \frac{8(a + |\Delta|)^3}{CBh^3} \quad \text{(8a)} \qquad \text{or} \qquad E_f = \frac{8(a + |\Delta|)^3}{\frac{C}{N}Bh^3} \quad \text{(8b)} \qquad (8)$$

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 is more than 10% of the average, the values of G_{IC} should be considered suspect and this should be noted in the report. (The value E_f calculated from Equation (8) should not be quoted as the modulus value and this value should not be used in Equation (4), which requires an independently measured or known value of the modulus to be used.)

Method (3): Experimental Compliance Method (ECM) or Berry's Method

An alternative approach is to plot the logarithm of the compliance C, or of the normalised compliance, C/N, if load-blocks are being used, versus the logarithm of the crack length a as shown in Figure 3. Only the propagation (PROP) values are used for the linear fits, i.e. all the initiation values are excluded from the regression analysis. The slope of this plot, n, can then be used to give G_{IC} as follows:

$$\mathbf{G_{IC}} = \frac{\mathbf{nP}\delta}{\mathbf{2Ba}} \quad \textbf{(9a)} \quad \textbf{or} \quad \mathbf{G_{IC}} = \frac{\mathbf{nP}\delta}{\mathbf{2Ba}}\frac{\mathbf{F}}{\mathbf{N}} \quad \textbf{(9b)} \qquad \textbf{(9)}$$

with P the load, δ the displacement, a the crack length, and B the width of the specimen. All initiation and propagation values of G_{IC}, if applicable, should be calculated. The same large-displacement correction F and load-block correction N, if applicable, are used as for the corrected beam theory method (see above).

10.2.2 Tapered Double Cantilever Beam (TDCB) Tests

Method (4): Simple Beam Theory (SBT)

The value of the adhesive fracture energy, G_{IC} , may be ascertained from:

$$\mathbf{G_{IC}} = \frac{\mathbf{P^2}}{\mathbf{2B}} \times \frac{\mathbf{dC}}{\mathbf{da}} \qquad \textbf{(2)}$$

For thin adhesive layers, it has been shown (References 1 and 2) from simple beam theory that dC/da may be expressed by:

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

where E_S is the independently-measured modulus of the substrate beam. Hence, combining Equations (2) and (3) and (1):

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

For the SBT method of calculation, the value of G_{IC} should be determined from Equation (4). If a standard grade of material is used, the quoted modulus may be used in Equation (4). In the report the value of m, and the range of the crack length a for which this value of m is within ±3%, should be quoted. (Values of G_{IC}, calculated where the value of m is outside of the range ±3%, should be considered suspect.)

Method (5): Corrected Beam Theory (CBT)

The simple beam theory expression for G_{IC} described in Method (4) above will incorrectly estimate the compliance of the specimen since (i) the positions of the loading pins, with their surrounding material, are not taken into account in deriving equation (4), and (ii) as for the DCB specimen, the specimen does not behave as a perfectly built-in cantilever beam. These corrections (Reference 6) lead to equation (10):

$$\frac{dC}{da} = \frac{8m}{E_s B} \cdot \left[1 + 0.43\left(\frac{3}{m}\right)^{\frac{1}{3}} \cdot a^{-\frac{1}{3}}\right] \qquad \textbf{(10)}$$

Hence, combining equations (2) and (10):

$$G_{IC} = \frac{4P^2}{E_s B^2} \cdot m \cdot \left[1 + 0.43\left(\frac{3}{m}\right)^{\frac{1}{3}} \cdot a^{-\frac{1}{3}}\right] \qquad (11)$$

(In deriving equation (10), the value of m is approximated to $3a^2/h^3$, i.e. the term 1/h in equation (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 (11) and (2) for tapered beams manufactured with aluminium alloy substrates (Reference 7).)

Method (6): Experimental Compliance Method (ECM)

The value of the adhesive fracture energy, G_{IC}, may again be ascertained from:

$$G_{IC} = \frac{P^2}{2B}\frac{dC}{da} \qquad (2)$$

For the TDCB geometry, when the values of C are plotted against the crack length a, the resulting graph should be linear. The value of dC/da is given by the slope of the straight line and is used to determine G_{IC} in Equation (2). The value of dC/da and the correlation coefficient, r^2, of the regression analysis should be noted on the results sheet. In the calculation of dC/da, only the propagation values should be included in the regression analysis, i.e. all initiation values should be excluded from this linear fit.

11. Test Report

The recommended format of test reports for the DCB and TDCB geometries are shown in Figures 5 (a)- (b). The test report should contain the following information:

11.1 Test report for the DCB test

(1) Equation (4) (i.e. G_{IC} from SBT, Method 1).

(2) Equation (5) (i.e. G_{IC} from CBT, Method 2).

(3) Equation (9) (i.e. G_{IC} from ECM, Method 3).

(4) Equation (8) (i.e. the value of the modulus, E_f).

Using these equations, the parameters listed below should be calculated:

(1) The initiation points of G_{IC} (NL, VIS, 5% or MAX, see Figure 1) obtained from both the insert (starter film) and from the Mode I precrack. (In the calculation of these values of G_{IC} the corresponding measured value of the crack length a should be used in the Equations i.e. a_o or a_p). The values determined from the insert (starter film) and from the Mode I precrack should be entered on the same test results sheet (Figure 5a).

(2) The propagation values of G_{IC} (PROP in Figure 2b) determined from the Mode I precrack as a function of crack length a.

(3) The results from both the insert and the Mode I precrack are then used to draw a resistance-curve (R-curve), i.e. G_{IC} versus crack length a (Figure 4. All initiation and propagation values shall be shown on the R-curve. The minimum number of propagation points recorded should be fifteen, if fewer points are used, this should be noted in the report and the results considered suspect (Reference 5).

(4) The flexural modulus E_f of the substrate should be calculated as a function of the crack length a. The flexural or tensile modulus E_s of a substrate arm should also be independently measured, or quoted if a known Standard Grade of material is employed, and the value obtained should be recorded in the report.

(5) After testing, the joints should be broken open to enable the locus of joint failure to be visually assessed. Record whether it is: (i) cohesive-in-the adhesive, (ii) apparently interfacial along the adhesive/substrate interface or (iii) cohesive-in-the-substrate. If a mixture of such failure paths are seen, estimate and record the percentage of each type. (ISO 10365: 1992).

11.2 Test report for the TDCB test

(1) Equation (4) (i.e. G_{IC} from the SBT, Method 4).

(2) Equation (11) (i.e. G_{IC} from the CBT, Method 5).

(3) Equation (2) (i.e. G_{IC} from the ECM, Method 6).

Using these equations, the parameters listed below should be calculated:

(1) The initiation points of G_{IC} (NL, VIS, 5% or MAX, see Figure 1) obtained from both the insert (starter film) and from the Mode I precrack. (In the calculation of these values of G_{IC} the corresponding measured value of the crack length a should be used.) The values determined from the insert (starter film) and those from the Mode I precrack shall be entered on the same test results sheet (Figure 5b).

(2) The propagation values of G_{IC} (PROP in Figure 2b) determined from the Mode I precrack as a function of crack length a.

(3) The results from both the insert and the Mode I precrack are then used to draw a resistance-curve (R-curve), i.e. G_{IC} versus crack length a (Figure 4). All initiation and propagation values shall be shown on the R-curve. The minimum number of propagation points recorded should be fifteen, if fewer points are used, this should be noted in the report and the results considered suspect (Reference 5).

(4) Also, from the graph of C versus a, the value of the slope, dC/da, and the correlation coefficient, r^2, of the data should be quoted.

(5) After testing, the joints should be broken open to enable the locus of joint failure to be visually assessed. Record whether it is: (i) cohesive-in-the adhesive, (ii) apparently interfacial along the adhesive/substrate interface or (iii) cohesive-in-the-substrate. If a mixture of such failure paths are seen estimate and record the percentage of each type (ISO 10365: 1992).

Annex A: Normative

A.1 Procedure to follow for measuring the compliance of the testing system

Special note: Please ensure that this procedure is carried out by experienced personnel, otherwise damage to equipment may occur when loading the calibration specimen. *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.*

1. Set up the tensile loading system in exactly the manner which was used for the fracture testing. It is recommended that this measurement be performed after the fracture tests, because the maximum load during fracture testing will then be known. This load will now be referred to as the calibration load, P_{cal}.

2. A rigid calibration specimen of known compliance, C_{cs}, is required along with a means of connecting it to the loading system. If pins of circular cross section have been used to load the fracture specimens, these should also be used to load the calibration specimen. (*Note: a calibration specimen made from mild steel with a cross-sectional area of 20mm by 25mm and a distance between loading hole centres of 25mm has been found to work satisfactorily, and will possess a compliance which is usually negligible when compared to the system compliance, C_{sy}.*)

3. With the calibration specimen attached, start to load the specimen at a <u>very</u> slow rate e.g. 0.05mm/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. ***The load will rise rapidly during this procedure, so care should be taken not to overload the load-cell!***

4. From the load-displacement trace obtained, draw the best straight line through the second 50% of the data, thus ignoring the initial non-linearity due to take up of play, as shown in Figure A.1. (This take up of play is also ignored in the fracture tests). From this straight line,

deduce the total compliance, C_{tot}, of the combined system and calibration specimen in (mm/N), as shown in Figure A.1.

5. Calculate the value of the system compliance, C_{sy}, in (mm/N) from:

$$\mathbf{C_{sy} = C_{total} - C_{cs}} \qquad \text{(A.1)}$$

6. All displacement values measured during the fracture tests should then be corrected by:

$$d_{\mathbf{cor}} = d - \mathbf{PC_{sy}} \qquad \text{(A.2)}$$

where δ_{cor} is the corrected value of the displacement in (mm) to be used in equations in section 10, δ is the value of the displacement in (mm) measured in the fracture tests and P is the corresponding load. This correction should be made to all displacement values, i.e. at each value of the crack length that was recorded.

Annex A.2: 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 A2. The complete load, propagation and unload cycle obtained during the test from the Mode I precrack is shown. For both the loading and unloading parts of the trace, the best straight lines should be drawn through the data, ignoring any initial non-linearity due to the take up of play in the system. These lines should be extrapolated back to zero load. The distance between the intercepts of these two lines with the displacement axis is termed δ_{offset}. The maximum value of the displacement attained during the test is termed δ_{max}. The values of δ_{offset} and δ_{max} should be measured from the test trace. It is normal for the term δ_{offset} to be non-zero. The value of (δ_{offset}/ δ_{max}) should be calculated for each test and noted on the results sheet.

The occurrence of plastic deformation in the adherends during a fracture test may be observed visually when the amount of deformation is large. If the joint is carefully broken open after the complete test cycle is finished, then plastic deformation of the substrate arms will have occurred if they remain bent on separation. In the case of the tapered beams, this may be seen if the substrates are held back together as they were before separation. The value of (δ_{offset}/ δ_{max}) and the results of a visual check on the straightness of the beams after breaking open should be noted in the report. Experience has shown that plastic deformation of the substrates can be suspected if $\delta_{offset}/\delta_{max} > 0.05$, where δ_{max} is the displacement required to extend the crack by the distance recommended in section 9.3.

Annex B Informative

B.1 Procedure to follow when unstable or 'stick-slip' crack growth is observed during the fracture test.

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 is shown in Figure B1. The crack grows in shorts bursts separated by periods of crack arrest during this type of propagation. Sometimes the propagation may be partly stable and partly unstable. The reasons for this type of behaviour are not fully understood.

When stick-slip crack propagation is observed during a DCB or TDCB test, it will not be possible to monitor the crack propagation as required by this protocol. The first initiation value of the crack length will be known however, and the crack lengths at subsequent arrest points may be observed using a travelling microscope. Between one arrest point and the next initiation point, the crack will obviously remain stationary. There may then be some stable crack growth before further instability, or the crack may jump directly from the previous arrested value of the crack length. After the crack has propagated sufficiently down the specimen, the joint should be fully unloaded and the unloading trace recorded in the same way as for a stable test. Breaking the joint open may reveal arrest lines on the adhesive that will allow more accurate crack length measurements to be made.

Thus the load, displacement and crack length data will be available at series of initiation and arrest points. As the number of data points will be insufficient to employ the linear regression analysis, needed for the ECM approach and the CBT approach (i.e. for the DCB test), then only the simple beam theory (SBT) method can be used to calculate the values of G_{IC}. Values of G_{IC}(initiation) and G_{IC}(arrest) may be computed using the simple beam theory. However, it should be noted clearly on the results sheet that stick-slip crack propagation was observed, and the type of point, i.e. initiation or arrest, should be clearly stated.

Annex C: Informative

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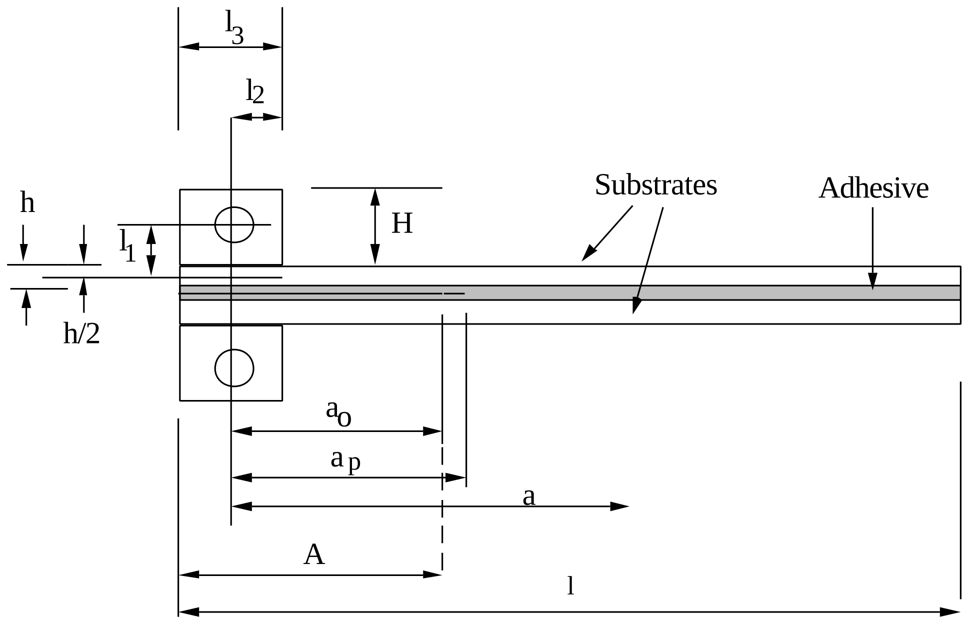
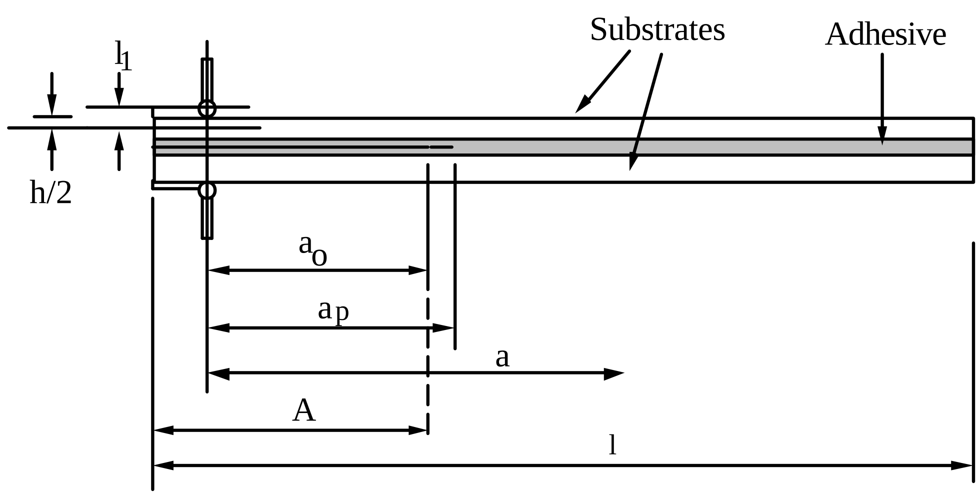
(a)



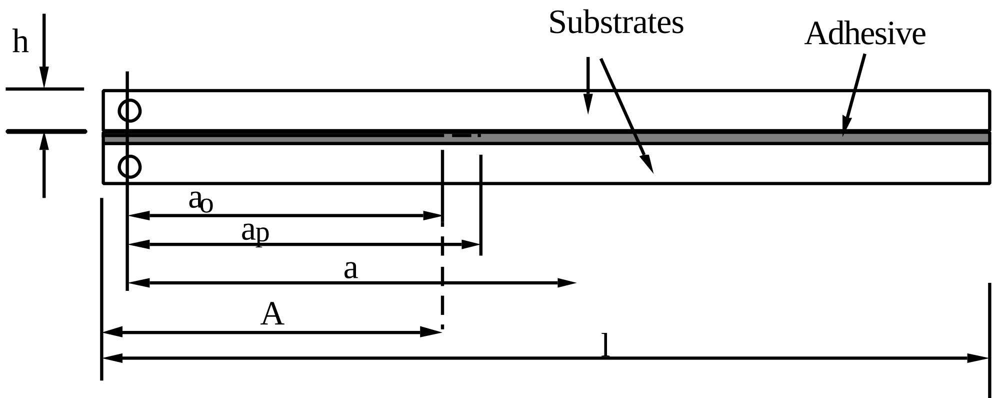
l_3
l_2
Substrates
Adhesive
h
H
l_1
h/2
a_o
a_p
a
A
l

(b)



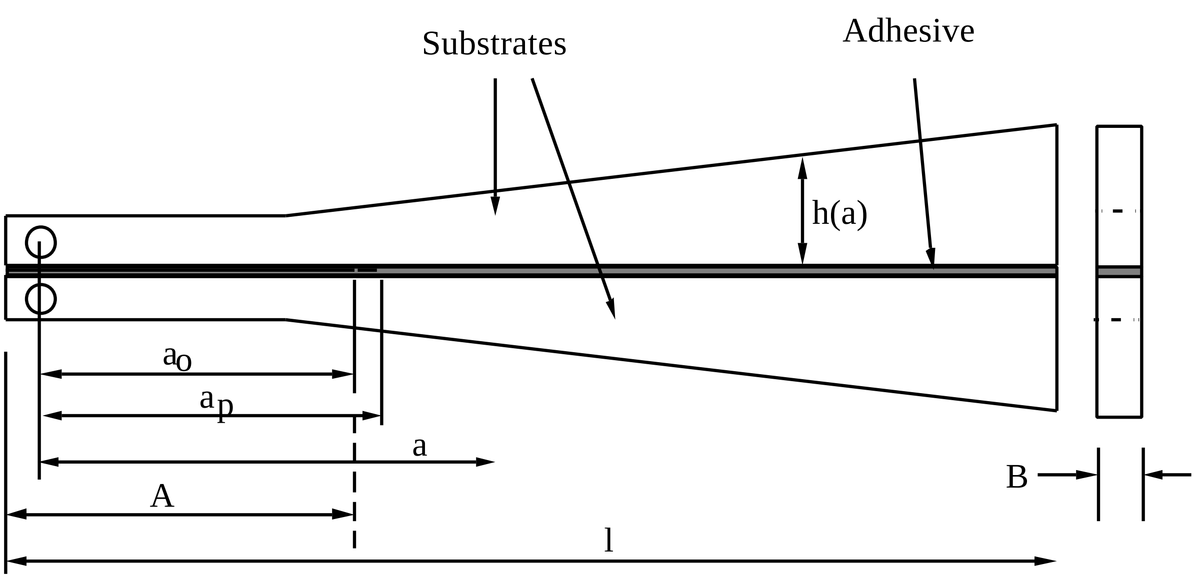
Substrates
Adhesive
l_1
h/2
a_o
a_p
a
A
l

(c)



Substrates
Adhesive
h
a_o
a_p
a
A
l

(d)



Substrates
Adhesive
h(a)
a_o
a_p
a
A
l
B

Figure 1: Geometry for the adhesive joint specimens.

(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).

(d) TDCB specimen.

[The crack length *a* is the distance between the load-line (intersection of the plane through pin-hole centres or the hinge axes and 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. Obviously, for the TDCB specimen, the value of *h* is a function of the crack length *a*.]

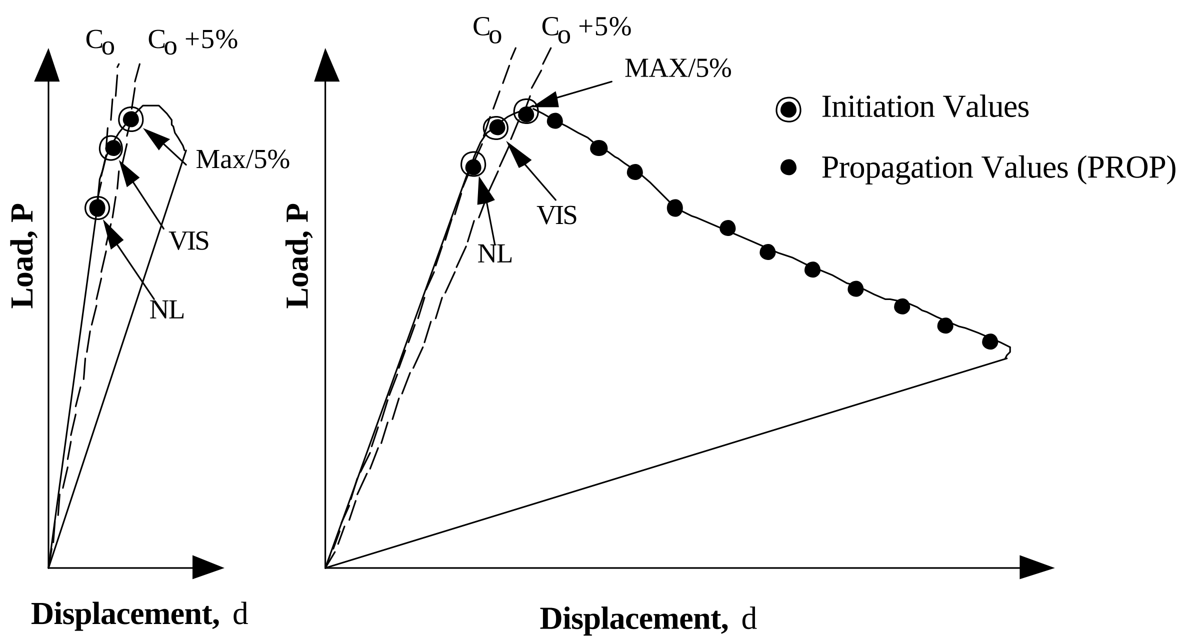


Figure 2: Schematic load-displacement curve for the DCB test.

(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, Max/5%, and propagation points (PROP).

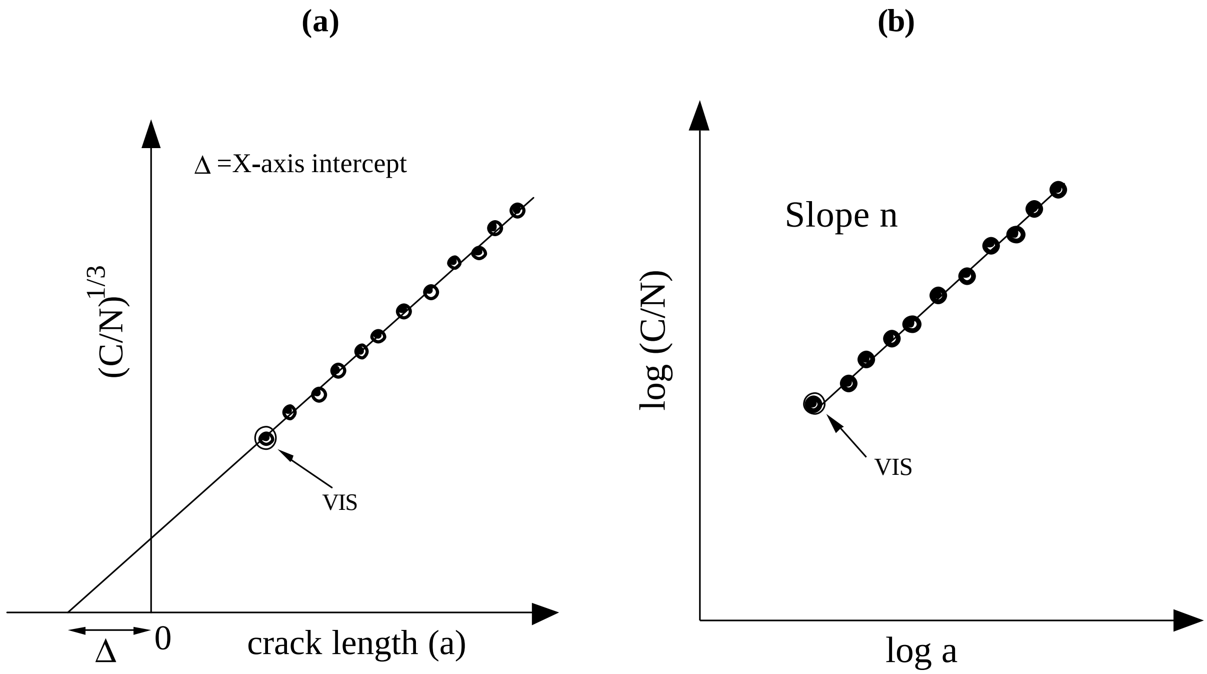


Figure 3: Linear fits used to determine (a) the correction for the Corrected Beam Theory (CBT) Method, and (b) for the slope *n* for the Experimental Compliance Method (ECM) Method. (For the DCB test specimen - and note that the visual point is excluded from the linear regression analysis.)

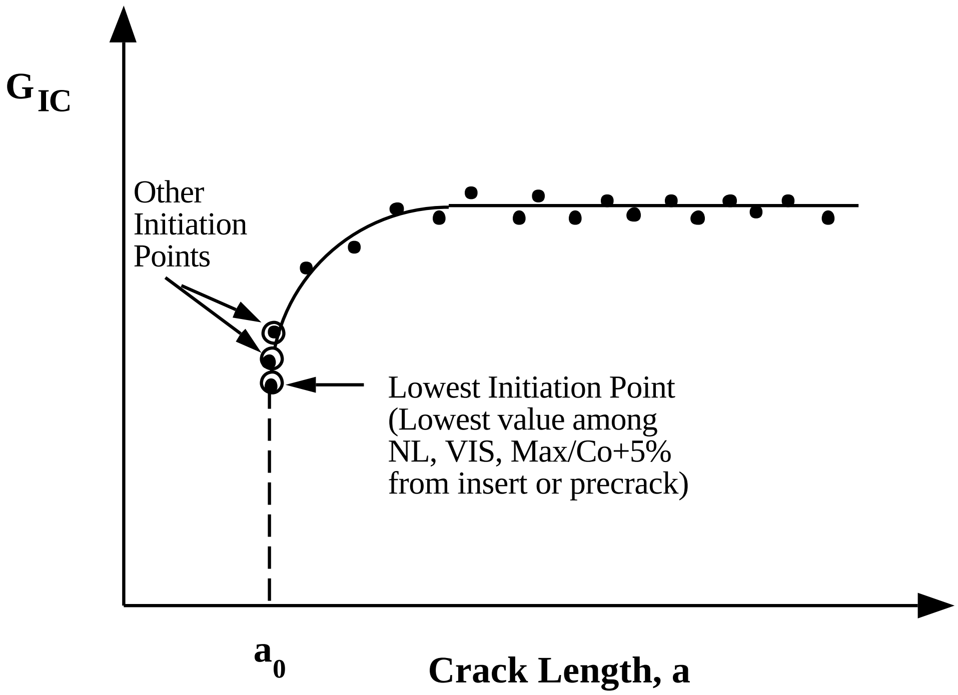


Figure 4. Schematic resistance-curve (R-curve) with G_{IC} value for initiation (i.e. the lowest value among NL, VIS, or MAX/5%) and for propagation (PROP) versus observed crack length a. (For either DCB or TDCB specimens, the specimen type should be stated.)

Figure 5(a) Recommended Test Report Sheet for DCB test

DCB TEST REPORT: PAGE 1 OF 3

Laboratory	
Personnel	
Test date	
Test number / code	

Specimen data

Adhesive	
Substrate	
Surface treatment details	
Specimen length, l (mm)	
Substrate thickness, h, (mm)	
Specimen width, B, (mm)	
Insert film material	
Insert film thickness (μm)	
Insert film total length, A, (mm)	
Insert length from load-line, a_o, (mm)	
Precrack length from load line, a_p, (mm)	
Flexural modulus of substrate, E_S, (GPa)	
Adhesive layer thickness, h_a (mm)	

Joint manufacture and test parameters

Adhesive cure temperature (°C)	
Adhesive cure duration (mins)	
Post cure drying cycle details(°C & hours)	
Fracture test temperature (°C)	
Fracture test relative humidity (%)	
Cross-head loading rate (mm/min)	
Cross-head unloading rate (mm/min)	
End-block dimension, l_1, (mm)	
End block dimension, l_2, (mm)	
Crack growth observations (e.g. stick-slip?)	
Locus of failure (visually assessed)	

Value from unloading line (substrate plasticity check)

δ_{offset} [mm]	δ_{max} [mm]	$\delta_{offset}/\ \delta_{max}$	Substrate bent?*

(*) After breaking open the joint after testing, any permanent deformation seen?)

Measurement of the system compliance (see Annex A.1)

C_{total} [mm/N]	C_{CS} [mm/N]	C_{sy} [mm/N] Eqn [A.1]

DCB TEST REPORT: PAGE 2 OF 3

Experimentally measured values / **Calculated values**

Text	a [mm]	P [N]	δ [mm]	δ_{COR} [mm] Eqn [A.2]	G_{IC}[J/m^2] (*SBT*) Eqn. [4]	G_{IC}[J/m^2] (*CBT*) Eqn. [5]	G_{IC}[J/m^2] (*ECM*) Eqn. [9]	E_f [GPa] Eqn. [8]
NL$^{(insert)}$								
VIS$^{(insert)}$								
MAX/5%$^{(insert)}$								
NL$^{(precrack)}$								
VIS$^{(precrack)}$								
MAX/5%$^{(precrack)}$								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP (*)								
PROP								
PROP								
PROP								
PROP								
PROP								

(*) Minimum number of propagation points required

Mean and standard deviations of propagation values

	G_{IC} [J/m^2] (*SBT*)	G_{IC} [J/m^2] (*CBT*)	G_{IC}[J/m^2] (*ECM*)	E_f [GPa]
Mean value				
Standard deviation				
Coefficient of variation (%)				

DCB TEST REPORT: PAGE 3 OF 3

Linear regression analysis

$(C/N)^{1/3}$ vs a		Log (C/N) vs log (a)	
Δ (mm)	r^2	n	r^2

Intermediate calculated values

a [mm]	F [-] Eqn. [6]	N [-] **Eqn. [7]**	C [mm/N]	(C/N)^1/3 $[mm/N]^{1/3}$	Log (C/N) [mm/N]	Log (a) [mm]	m [1/mm] Eqn. [1]

Figure 5(b). Recommended Test Report Sheet for the TDCB test

TDCB TEST REPORT:PAGE 1 OF 3

Laboratory	
Personnel	
Test date	
Test number / code	

Specimen data

Adhesive	
Substrate	
Surface treatment details	
Specimen length, l, (mm)	
Specimen geometry factor, m, (mm^{-1})	
Specimen width, B, (mm)	
Insert film material	
Insert film thickness (μm)	
Insert film total length, A, (mm)	
Insert length from load-line, a_o, (mm)	
Precrack length from load line, a_p, (mm)	
Flexural modulus of substrate, E_S, (GPa)	
Adhesive layer thickness, h_a, (mm)	

Joint manufacture and test parameters

Adhesive cure temperature (°C)	
Adhesive cure duration (mins)	
Post cure drying cycle details (°C &hours)	
Fracture test temperature (°C)	
Fracture test relative humidity (%)	
Cross-head loading rate (mm/min)	
Cross-head unloading rate (mm/min)	
Is *m* within +/- 3%?	
Crack growth observations (e.g. stick-slip?)	
Locus of failure (visually assessed)	

Value from unloading line (substrate plasticity check)

δ_{offset} [mm]	δ_{max} [mm]	$\delta_{offset}/\ \delta_{max}$	Substrate bent?*

(*)After breaking open the joint after testing, any permanent deformation observed?

Measurement of the system compliance (see Annex A.1)

C_{total} [mm/N]	C_{CS} [mm/N]	C_{sy} [mm/N] Eqn [A.1]

Experimentally measured values

Text	a [mm]	P [N]	δ [mm]
$NL^{(insert)}$			
$VIS^{(insert)}$			
$MAX/5\%^{(insert)}$			
$NL^{(precrack)}$			
$VIS^{(precrack)}$			
$MAX/5\%^{(precrack)}$			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP (*)			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			
PROP			

(*) Minium number of propagation points.

Calculated values

δ_{COR} [mm] Eqn. [A.2]	C [mm/N]	G_{IC} [J/m^2] (*SBT)* Eqn. [4]	G_{IC} [J/m^2] (*CBT)* Eqn. [11]	G_{IC} [J/m^2] (*ECM)* Eqn. [2]

TDCB TEST REPORT:PAGE 3 OF 3

Linear regression analysis:(C versus a)

dC/da [1/N]	r^2 of regression

Mean and standard deviations of propagation values

	G_{IC} [J/m^2] (*SBT)*	G_{IC} [J/m^2] (*CBT)*	G_{IC} [J/m^2] (*ECM*)
Mean value			
Standard deviation			
Coefficient of variation (%)			

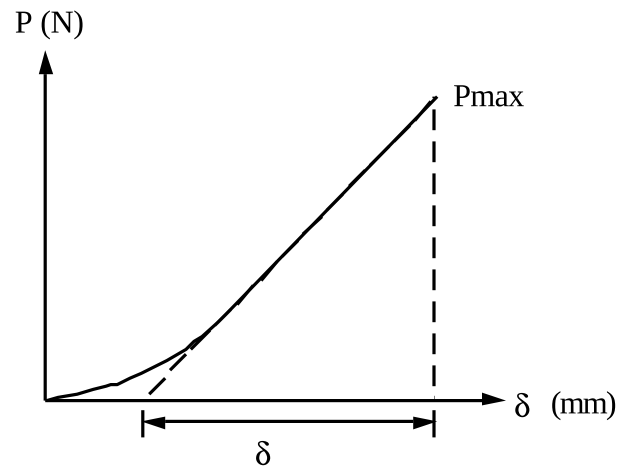


Figure A1. A schematic load-displacement trace obtained during the system compliance measurement. ($C_{total}=\delta/P_{max}$).

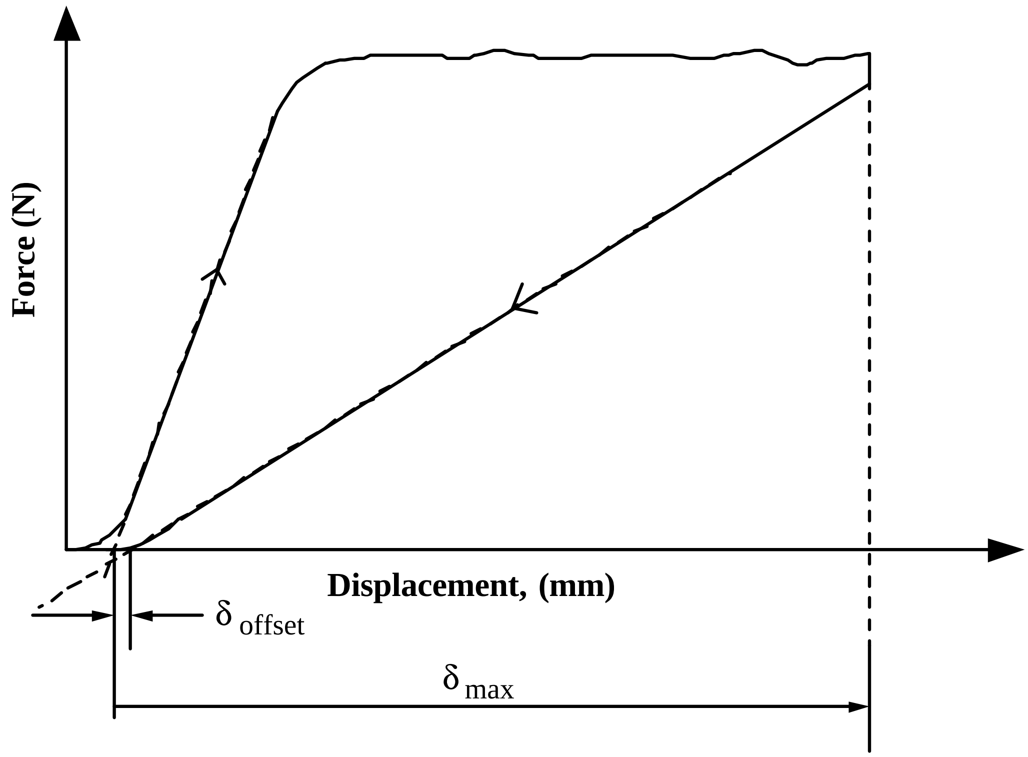


Figure A2. Typical force-displacement trace for a tapered double cantilever beam specimen, showing loading and unloading lines and the displacement offset.

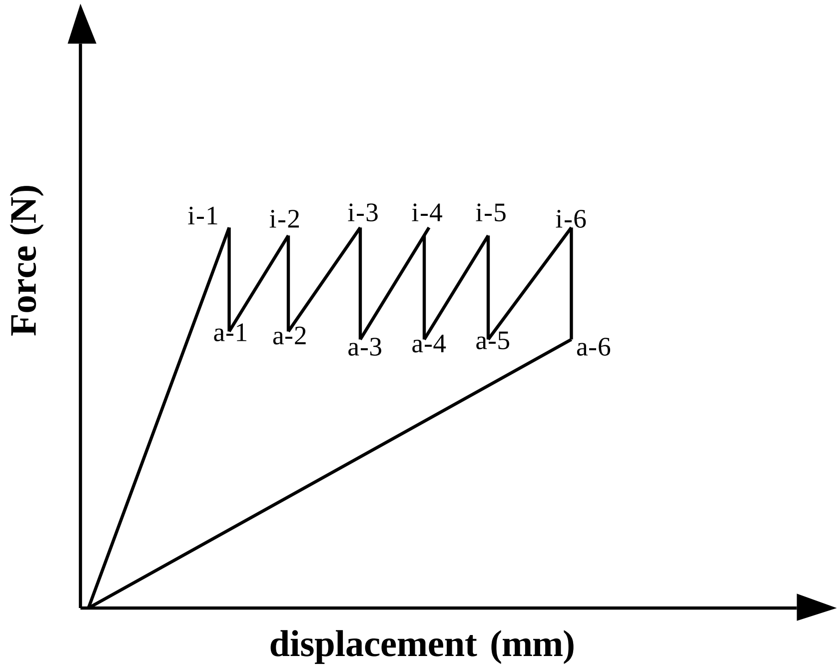


Figure B1. Schematic force-displacement trace for a tapered double cantilever beam specimen, exhibiting unstable 'stick-slip' crack growth behaviour. ('i' indicates initiation points, 'a' indicates arrest points.)