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Fibre-reinforced plastic composites — Determination of mode I interlaminar fracture toughness, G_{IC}, for unidirectionally reinforced materials

Composites plastiques renforcés de fibres — Détermination de la ténacité à la rupture interlaminaire en mode I, G_{IC}, de matériaux composites à matrice polymère renforcés de fibres unidirectionnelles

Reference number
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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

This second edition cancels and replaces the first edition (ISO 15024:2001), which has been technically revised.

The main changes are as follows:

— a new double cantilever beam (DCB) has been added [Figure 1 c)].

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Fibre-reinforced plastic composites — Determination of mode I interlaminar fracture toughness, G_{IC}, for unidirectionally reinforced materials

1 Scope

This document specifies a method for the determination of mode I interlaminar fracture toughness (critical energy release rate), G_{IC}, of unidirectional fibre-reinforced plastic composites using a double cantilever beam (DCB) specimen.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 527-1, *Plastics — Determination of tensile properties — Part 1: General principles*

ISO 1268 (all parts), *Fibre-reinforced plastics — Methods of producing test plates*

ISO 7500-1, *Metallic materials — Calibration and verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Calibration and verification of the force-measuring system*

ISO 9513, *Metallic materials — Calibration of extensometer systems used in uniaxial testing*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
mode I interlaminar fracture toughness
critical energy release rate
G_{IC}
resistance to the initiation and propagation of a delamination crack in unidirectional fibre-reinforced polymer matrix composite laminates under mode I opening load

Note 1 to entry: It is measured in joules per square metre.

3.2
mode I crack opening
crack-opening mode due to a load applied perpendicular to the plane of delamination using the double cantilever beam specimen

Note 1 to entry: The double cantilever beam specimen shown in Figure 1 is shown in Figure 1.

3.3
NL point
point of deviation from linearity on the load versus displacement trace

Note 1 to entry: As shown in Figure 2.

3.4
VIS point
point of the onset of delamination, as determined by visual observation, at the edge of the specimen, marked on the load-displacement trace

Note 1 to entry: As shown in Figure 2.

3.5
5 % / MAX point
point which occurs first on loading the specimen between:

a) the point of 5 % increase in compliance ($C_{5\ \%}$) from its initial value (C_0); and

b) the maximum load point.

Note 1 to entry: See Figure 2.

3.6
PROP points
points of discrete delamination length increments beyond the tip of the insert or starter crack tip marked on the load-displacement trace, points where the crack has been arrested being excluded

Note 1 to entry: See Figure 2.

3.7
delamination-resistance curve
R curve
cross-plot of G_{IC} for initiation and subsequent propagation values for *mode I crack opening* (3.2) as a function of delamination length

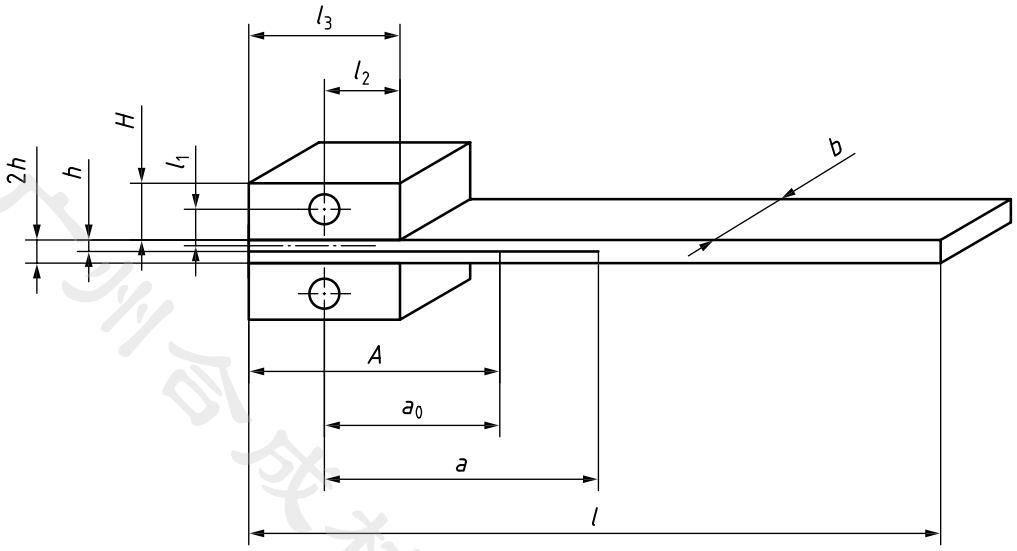
Note 1 to entry: See Clause 10.

4 Principle

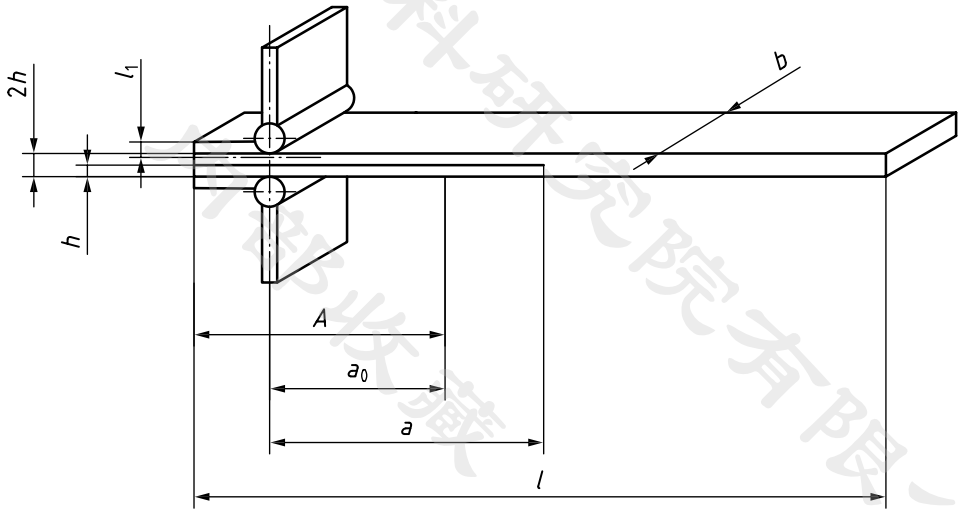
A mode I double cantilever beam (DCB) specimen, as shown in Figure 1, is used to determine G_{IC}, the critical energy release rate, or interlaminar fracture toughness, of fibre-reinforced plastic composites. Figure 1 represents three different loading arrangement for the specimen as following, a) Specimen loading using load blocks, b) Specimen loading using piano hinges, c) Specimen loading using insert hinges (see Annex D). The test method is limited to zero-degree unidirectional lay-ups only (see B.1). Data reduction yields initiation and subsequent propagation values of G_{IC} for mode I opening fracture toughness. A delamination-resistance curve, or R curve, is generated by plotting G_{IC} on the ordinate as a function of delamination length plotted on the abscissa.

The aim of the test method is to determine initiation values for the composite material tested. Fibre bridging is observed in a DCB test and it might not be representative of the composite material tested. Fibre bridging is considered to be the main cause for the observed shape of the R curve, which typically rises before reaching a roughly constant value of G_{IC} for long delamination lengths. A crack-opening load is applied to the DCB specimen, perpendicular to the plane of delamination, through load blocks or piano hinges under displacement control at a constant rate. The DCB specimen contains a thin, non-adhesive starter film embedded at the midplane as shown in Figure 3, which is used to simulate an initial delamination. The specimen is precracked by unloading the DCB specimen immediately after the first increment of delamination growth from the insert, followed by re-loading. The onset of stable delamination growth is monitored, and the delamination initiation and propagation readings are recorded. The R curve is plotted with the initiation values from both the insert and the mode I precrack,

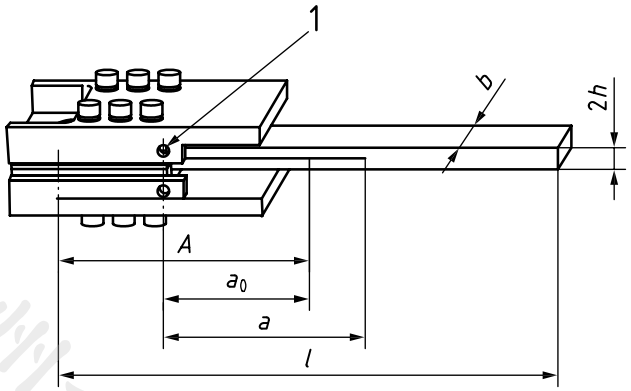
and with the propagation from the precrack. Under certain prescribed circumstances (see 9.2.7), an alternative wedge precracking procedure can be used but is not recommended.



a) Specimen loading using load blocks



b) Specimen loading using piano hinges



c) Specimen loading using insert hinges

Key

b specimen width

$2h$ specimen thickness

a_0 initial delamination length

a total delamination length

A insert length

l specimen length

l_1 distance from the loading pin (or piano hinge axis) to the midplane of the half-beam to which the load block (or piano hinge) is attached

l_2 distance from centre of loading pin (or piano hinge axis) to edge of load block (or piano hinge)

l_3 block length

H block thickness

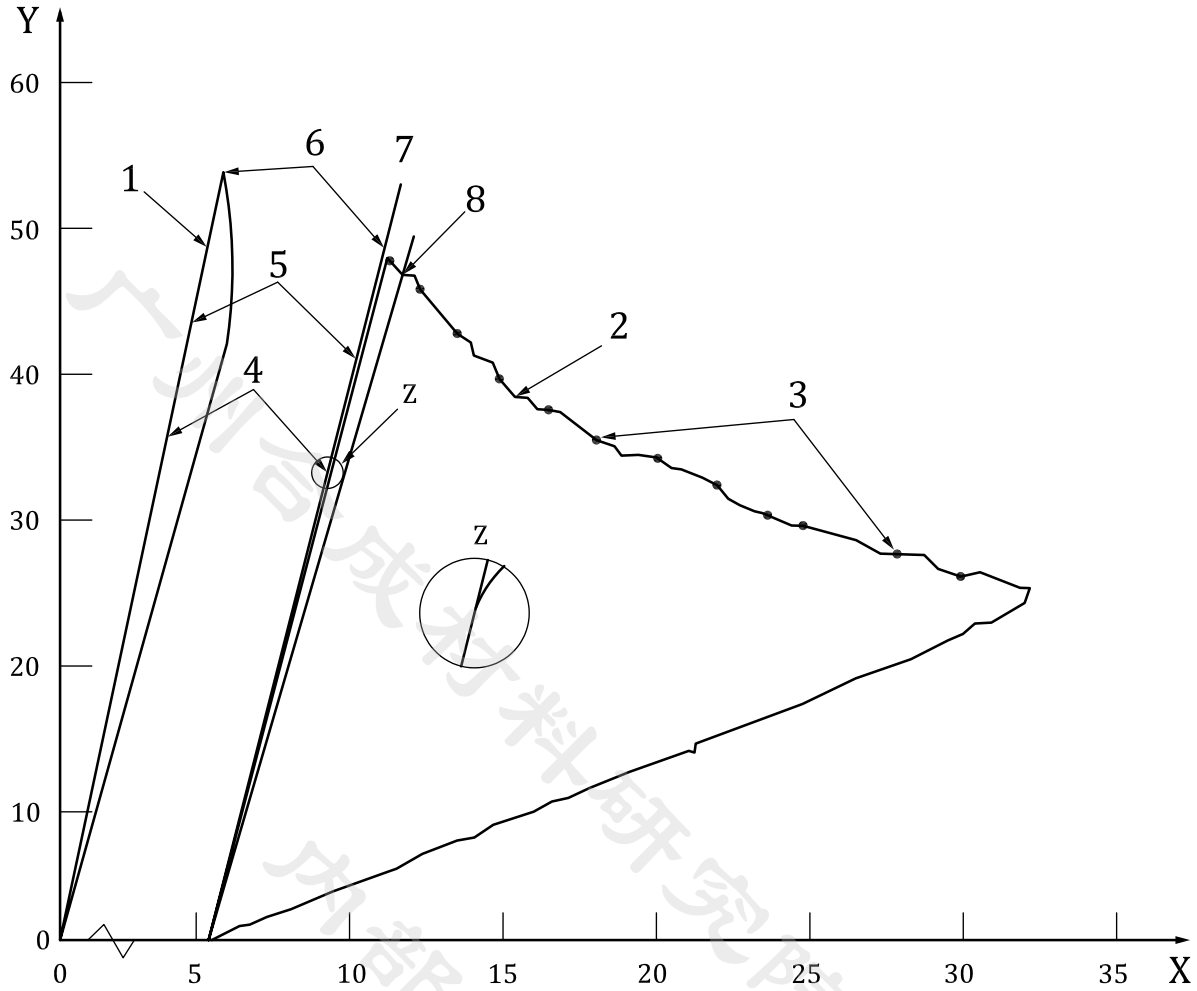
1 centres of hinge axis

NOTE 1 Alternative loading arrangements are (a) load blocks and (b) piano hinges.

NOTE 2 The fibre orientation is parallel to the length l.

NOTE 3 Details of DCB test with insert hinges are described in Annex D.

Figure 1 — Geometry for the double cantilever beam (DCB) specimen with a starter delamination



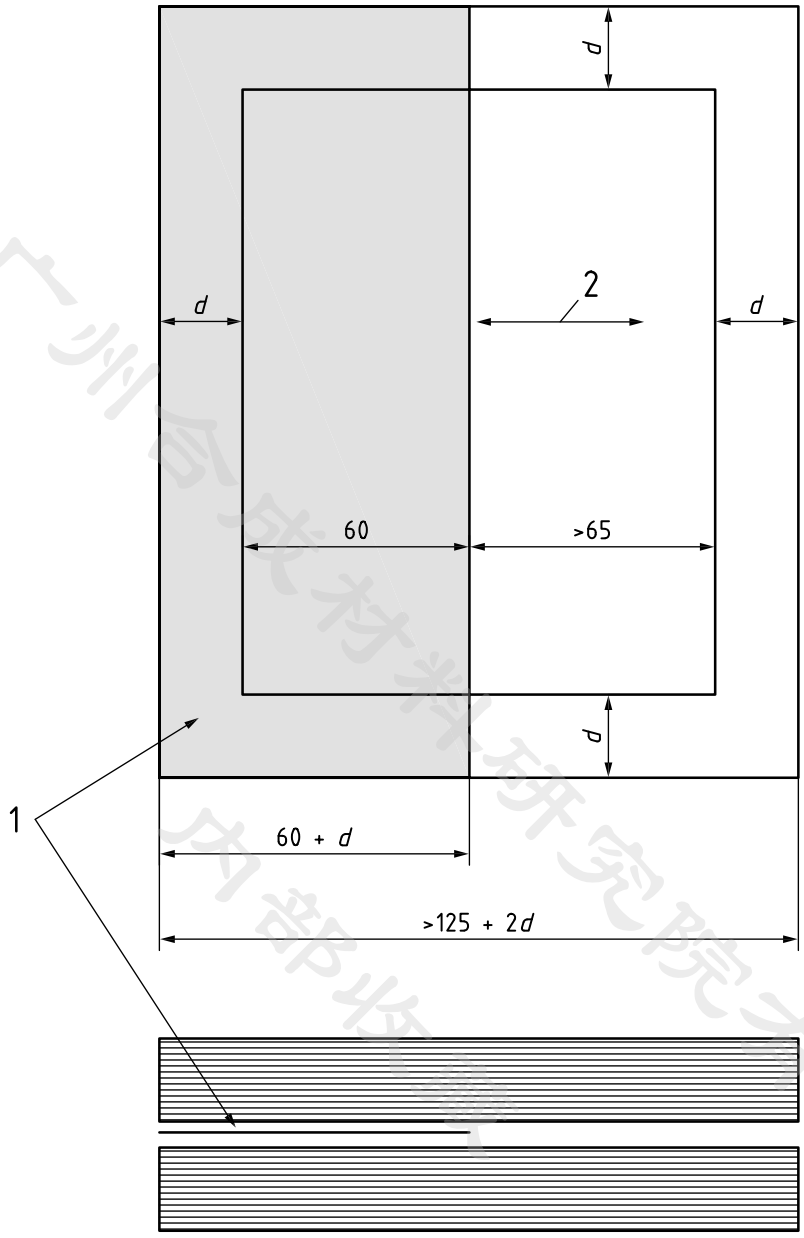
Key

X displacement, in millimetres
Y load, in newtons
1 crack initiation followed by unloading
2 crack propagation by re-initiation from the resulting mode I precrack
3 crack propagation markers
4 NL point
5 VIS point
6 MAX point
7 C_0
8 $C_{5\,\%}$

NOTE Figure shows case where 5 % values follow maximum load, and reload curve has been offset 5 mm for clarity

Figure 2 — Load-displacement curve for a DCB test

Dimensions in millimetres



Key

1 film insert

2 fibre direction

d margin to allow for initial trimming

Figure 3 — Example of test plate preparation showing the laminate structure, the dimensions and the position of the film insert

5 Apparatus

5.1 Test machine

5.1.1 General

The testing machine shall be in accordance with the following requirements.

5.1.2 Speed of testing

The testing machine shall be capable of maintaining the constant displacement rate required in 9.2.1 and 9.3.1 within a tolerance of ±20 %, as specified in ISO 527-1.

5.1.3 Fixture

The test machine shall be equipped with a fixture to introduce the load to the pins inserted into the load blocks or with grips to hold the piano hinges. In each case, rotation of the specimen end shall be allowed. The axis of the load-introduction fixtures shall be aligned with the loading axis of the test machine.

5.1.4 Load and displacement measurements

The force measurement system shall be in accordance with class 1 as defined in ISO 7500-1. The displacement measurement shall be in accordance with class 2 of ISO 9513 within the relevant range used for results determination. Apply machine compliance compensation if the crosshead monitor is used, to ensure that the required accuracy level is as well achieved under loading conditions.

5.1.5 Recorder

The test machine shall allow the displacement and corresponding load to be measured and recorded, preferably on a continuous basis.

5.2 Load blocks or piano hinges

Load blocks or piano hinges, as shown in Figure 1, may be used for introducing the load into the specimen. They shall be at least as wide as the specimen. For the load blocks in Figure 1 a), the maximum value of l_3 shall be 15 mm. The hole to inset the loading pin shall be at the centre of l_3.

5.3 Measuring apparatus

5.3.1 Micrometer, or equivalent, capable of reading to 0,02 mm or less, suitable for measuring the thickness of the specimen. The micrometer shall have contact faces appropriate to the surface being measured (i.e. flat faces for flat, polished surfaces and hemispherical faces for irregular surfaces).

5.3.2 Vernier calipers, or equivalent, capable of reading to 0,05 mm or less, for measuring the width of the specimen.

5.3.3 Linear scale (ruler), with 1 mm divisions, for measuring the specimen length and marking the edges of the specimen to monitor the delamination crack growth.

5.4 Travelling microscope (optional)

A travelling microscope may be used to measure the delamination length. If used, it shall have a travel range of 0 mm to 200 mm, have a magnification no greater than × 70 and be readable to 0,05 mm.

5.5 Non-adhesive insert film

A polymer film of thickness not exceeding 13 µm shall be used as a non-adhesive insert. For epoxy resin matrix composites cured at temperatures below 180 °C, a film of polytetrafluoroethylene (PTFE) is recommended. For composites cured at temperatures above 180 °C (for example those including polyimide or bismaleimide thermoplastics), a film of polyimide is recommended (see B.2).

5.6 Ancillary equipment

5.6.1 Desiccator, for storing the test specimens after conditioning, including a suitable desiccant such as silica gel or anhydrous calcium chloride.

5.6.2 Mould release agent. When a polyimide film is used as the non-adhesive insert film, a mould release agent of the polytetrafluoroethylene (PTFE) type is recommended (see B.2).

5.6.3 Adhesive. A cyanoacrylate adhesive or epoxy adhesive of the two-component room-temperature-cure type to bond the load blocks or piano hinges to the test specimen (see Annex A).

5.6.4 Solvent. Organic solvent such as acetone or ethanol (see Annex A).

5.6.5 Sandpaper (abrasive paper), with 500 grade grit or finer (see Annex A).

5.6.6 White ink. Water-soluble typewriter correction fluid.

6 Test specimen

6.1 Test plate preparation

A test plate shall first be prepared in accordance with the part of ISO 1268 appropriate to the production process used. The recommended plate thickness is 3 mm for 60 % by volume carbon-fibre-reinforced composites and 5 mm for 60 % by volume glass-fibre-reinforced composites.

An even number of unidirectionally aligned layers shall be used (seeB.1). The non-adhesive film insert shall be placed at laminate mid-thickness during lay-up. The insert shall not exceed 13 µm, in order to simulate a sharp crack and cause minimum disturbance of the individual plies of the laminate. Guidelines for the insert material and its preparation are given in B.2.

If a polyimide film is used, the film shall be painted or sprayed with a mould release agent before insertion into the laminate. The film shall be cut to the proper size for insertion into the laminate before applying the mould release agent. Mould release agents containing silicone may contaminate the laminate by migration through the individual layers. Baking of the film will help to prevent silicone migration within the composite. The film shall be coated and baked twice for 30 min at 130 °C. Care shall be exercised in handling the film so that the coated layer of release agent is not damaged or removed from the film.

Figure 3 shows an example of how the test plate can be configured. The positioning of the insert shall allow for the initial trimming of the test plate.

6.2 Specimen preparation

6.2.1 Preferred specimens

Machine the test specimens from the trimmed test plate, with their longitudinal axes parallel to the fibre direction in the test plate. Specimens shall be identified to indicate their original position in the test plate. The specimen configuration and dimensions are illustrated in Figure 1. The dimensions and tolerances for the preferred specimens are shown in Table 1. Specimen surfaces shall not be machined to meet the thickness requirement.

The thickness and width of individual specimens shall not vary by more than ±1 % of the mean value for that type of specimen.

Table 1 — Recommended specimen dimensions and tolerances

	Unit	Carbon fibre	Glass fibre	Tolerance
Width, b	mm	20	20	±0,5
Minimum length, l	mm	125	125	—
Thickness, $2h$	mm	3	5	±0,1

6.2.2 Alternative specimens

Other specimen thicknesses may be used, depending on the tensile modulus of elasticity and the anticipated interlaminar fracture toughness of the specimen. Guidelines for choosing a specimen thickness that will yield negligible displacement corrections based on the anticipated interlaminar fracture toughness are given in B.3.

Other specimen widths between 15 mm and 30 mm may be used. Increasing the length of the specimen is not critical. However, shortening is not recommended because it will reduce the maximum delamination length that can be investigated and thus yield too few data points for the analysis.

6.3 Checking and measurement of the test specimens

After machining the specimens, check that they are free from twist and warpage, and free from machining damage. Check that the cut edges are suitably smooth to allow preparation for monitoring the crack length in accordance with B.4 and B.5.

Measure and record the length, l, of each specimen to the nearest millimetre. Measure the width, b, to the nearest 0,02 mm at three evenly spaced points along the length. Measure the thickness, $2h$, to the nearest 0,02 mm at these three points along the centreline of the specimen, and at two additional points near the edge at the middle measurement point, to check for tapering of the specimen.

Record the mean thickness and width of each specimen and check that the values are within the range given in Table 1. Check also that the variations along the specimen are within the range given in Table 1. Discard specimens not meeting these requirements.

Measure the length of the insert at both side edges of the specimen. Record the average value, but if the insert length measurements differ by more than 1 mm on the two edges this shall be noted in the report. The minimum distance of the tip of each insert edge from the near ends of the load blocks or piano hinges shall be 45 mm.

6.4 Attachment of loading points

Bond the load blocks or piano hinges for load introduction on the surfaces at the end of the specimen where the insert has been placed, as shown in Figure 1. The load-introduction fixtures shall be well aligned with the specimen, and with each other, and held in position with clamps while the adhesive sets. The requirements for bonding the load blocks or piano hinges given in Annex A shall be followed.

6.5 Measurement of delamination length

For the measurement of the delamination lengths, marks shall be drawn at 5 mm intervals along the edge of the specimen, extending at least 55 mm beyond of the tip of the insert. Additionally, the first 10 mm and last 5 mm shall be marked at 1 mm intervals.

7 Number of specimens

A minimum number of five specimens shall be tested. Specimens found to be invalid (see 9.3.6) shall be discarded and new specimens tested in their place.

8 Conditioning

The specimens shall be dried using the drying temperature and duration recommended by the resin supplier. This conditioning shall be performed after bonding of the load blocks or piano hinges. After conditioning, the specimens may be stored in a desiccator for not more than 24 h before testing.

Conditioning is required to obtain baseline data on test specimens with a uniform moisture content, because the interlaminar fracture toughness of polymer-matrix composites is sensitive to the amount of moisture present in the resin. Hence, a dry condition is recommended for this document. Guidelines for conditioning are given in B.6.

9 Test procedure

9.1 Test set-up

9.1.1 The test shall be performed under standard conditions in accordance with ISO 291 [i.e. 23 °C ± 2 °C, (50 ± 5) % relative humidity].

9.1.2 Mount the specimen in the fixture of the test machine. Support the end of the specimen, if required, in order to keep the beam perpendicular to the direction of the applied load.

9.1.3 For recommendations on further aspects of test preparation, see B.4.

9.2 Initial loading

9.2.1 Load the specimen at a constant cross-head rate between 1 mm/min and 5 mm/min.

9.2.2 Record the load and the displacement values, continuously if possible. Record the position of the delamination with an accuracy of at least ±0,5 mm (seeB.5).

9.2.3 During loading, record the point on the load-displacement curve, or record the load-displacement data values, at which the onset of delamination movement is visually observed on the edge of the specimen (VIS, see Figure 2).

If the start of delamination growth is difficult to observe, a change of illumination conditions or the use of a cross-head speed from the lower end of the range is recommended.

9.2.4 Stop the loading after 3 mm to 5 mm of delamination crack growth. If unstable delamination growth from the insert is observed (see B.7), this shall be noted in the report and loading shall be continued until the delamination length has increased by 3 mm to 5 mm beyond the arrest point. Note in the test report if the delamination length is outside the range of 3 mm to 5 mm.

9.2.5 Unload the specimen at a constant cross-head rate of up to 25 mm/min.

9.2.6 After unloading, mark the position of the tip of the precrack on both edges of the specimen. Note in the test report if the position on the two edges differs by more than 2 mm and if the specimen is removed from the fixture for this procedure.

NOTE Mismatch of greater than 2 mm between the two positions can be an indication of asymmetrical loading.

9.2.7 In the atypical case that the R-curve shows a decrease in apparent toughness with delamination length (as indicated in Figure 8), the initial-loading process may be replaced by wedge precracking. Use of wedge precracking (see B.8) is not recommended, since it may be difficult to produce a suitable

precrack by wedge opening. Its use shall be reported. In addition, the precrack may not always lie in the midplane of the specimen. Deviations of the precrack from the midplane will invalidate the test results and shall also be reported.

9.3 Re-loading

9.3.1 The specimen shall be re-loaded at the same constant cross-head speed of 1 mm/min to 5 mm/min as the initial loading, without stopping or unloading, until the final delamination length increment has been reached (see 9.3.3). The load and displacement values shall be recorded, including those for the unloading cycle. The position of the delamination shall be pinpointed with an accuracy of at least ±0,5 mm on the edge of the specimen.

9.3.2 Record the load and displacement values at which the onset of delamination movement from the precrack is observed on the edge of the specimen (VIS, Figure 2).

9.3.3 On continuation of the loading, record the load and displacement values at as many delamination length increments as possible in the first 5 mm, ideally every 1 mm. Subsequently, record the load and displacement data at every 5 mm, until the delamination crack has propagated at least 45 mm from the tip of the precrack, and again at every 1 mm increment of crack growth for the last 5 mm of delamination propagation, up to a total delamination length of 50 mm beyond the tip of the precrack (see Figure 2).

9.3.4 Finally, unload the specimen at a constant cross-head rate of up to 25 mm/min.

9.3.5 Mark the positions of the tip of the delamination crack after unloading on both edges of the specimen. Note in the report if these positions differ by more than 2 mm.

NOTE Mismatch of greater than 2 mm between the two positions can be an indication of asymmetrical loading.

9.3.6 Any permanent deformation of the specimen after unloading shall be noted in the report. Deviations of the delamination from the midplane of the laminate will invalidate the test results and shall be noted in the report. In such cases, a replacement specimen shall be tested.

10 Calculation of G_{IC}

10.1 Interpretation of test results

Several initiation fracture toughness G_{IC} values are determined from the corresponding points on the load-displacement curve. G_{IC} values corresponding to the points listed below shall be determined for testing from the starter film and from the mode I precrack for each specimen. These initiation values are indicated on the typical R-curve shown in Figure 7. These values are determined as follows.

— **The NL point** is determined by drawing a straight line through the linear portion of the load versus displacement trace to obtain the point of deviation from linearity, or onset of non-linearity (NL in Figure 2). Recommendations for obtaining this point are given in B.9.

— **The VIS point** is determined from the first visual observation that the delamination has moved from the tip of the insert, or from the mode I precrack, on the edge of the specimen (VIS in Figure 2). The corresponding load and displacement data at this point are used for the calculation. A travelling microscope (5.4) may be used to determine the VIS point.

— **The 5 % / MAX point** corresponds to the values of the load and displacement which occur first on loading the specimen. For the **5 %** value, a straight line is drawn to determine the initial compliance C_0, 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 1,05 C_0. The intersection of the new line with the load-displacement curve yields the load and displacement to be used for the calculation of G_{IC}, unless the intersection

is at a larger displacement than the maximum load point. In the latter case, the **maximum load** and the corresponding displacement shall be used for the calculation of G_{IC}.

— **PROP points** are determined for each delamination length measured during propagation (PROP in Figures 7 and 8). Data taken at points where the crack has been arrested are excluded. The minimum number of PROP points shall be 15. If fewer points are used, this shall be noted in the report because the values determined from the linear fit may be influenced by statistical effects.

10.2 Data reduction

10.2.1 General

Either Method A (see 10.2.2) or Method B (see 10.2.3) shall be used for the data reduction. Both methods will give equivalent results. The data required for the analysis are:

— a_0 initial delamination length;

— a total delamination length ($a = a_0$ + measured delamination length increments);

— P load;

— δ load line displacement;

— C load line compliance δ / P;

— b width of specimen;

— $2h$ thickness of specimen.

10.2.2 Method A: Corrected beam theory (CBT)

Establish the relation between the delamination length and the compliance by plotting the cube-root of the compliance, $C^{1/3}$ [or $(C/N)^{1/3}$ if load blocks are being used, where N is the load block correction described below], as a function of delamination length a for the reloading data (see Figure 4). The extrapolation of a linear fit through the data in the plot yields Δ as the x-intercept.

If the Δ-value from the fit is positive, a value of $\Delta = 0$ shall be used and this shall be noted in the report. The VIS and the PROP points are used for the linear fit, but not for the NL or 5 % / MAX points. If the VIS point has not been determined or clearly lies outside the range defined by the PROP points when plotted as in Figure 4, it shall be excluded from the linear fit and this shall be noted in the report.

The critical energy release rate G_{IC} is given by Formula (1):

$$G_{IC} = \frac{3P\delta}{2b(a+|\Delta|)} \times F \quad \text{or} \quad G_{IC} = \frac{3P\delta}{2b(a+|\Delta|)} \times \frac{F}{N} \tag{1}$$

where

F is the large-displacement correction (described in this subclause);

N is the load block correction.

The G_{IC} values corresponding to all initiation and propagation points shall be calculated. The delamination length for the initiation value from the insert shall be taken as the distance between the load line and the tip of the insert (a_0 in Figure 1) whereas the delamination length for the initiation value from the precrack shall be taken as the distance between the load line and the tip of the precrack (a in Figure 1).

The large-displacement correction F shall be applied for all specimens (see Reference [14]). This correction factor will contribute significantly if $\delta / a > 0{,}4$. The large-displacement correction F and the load block correction N are calculated as follows (for piano hinges, $N = 1$):

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

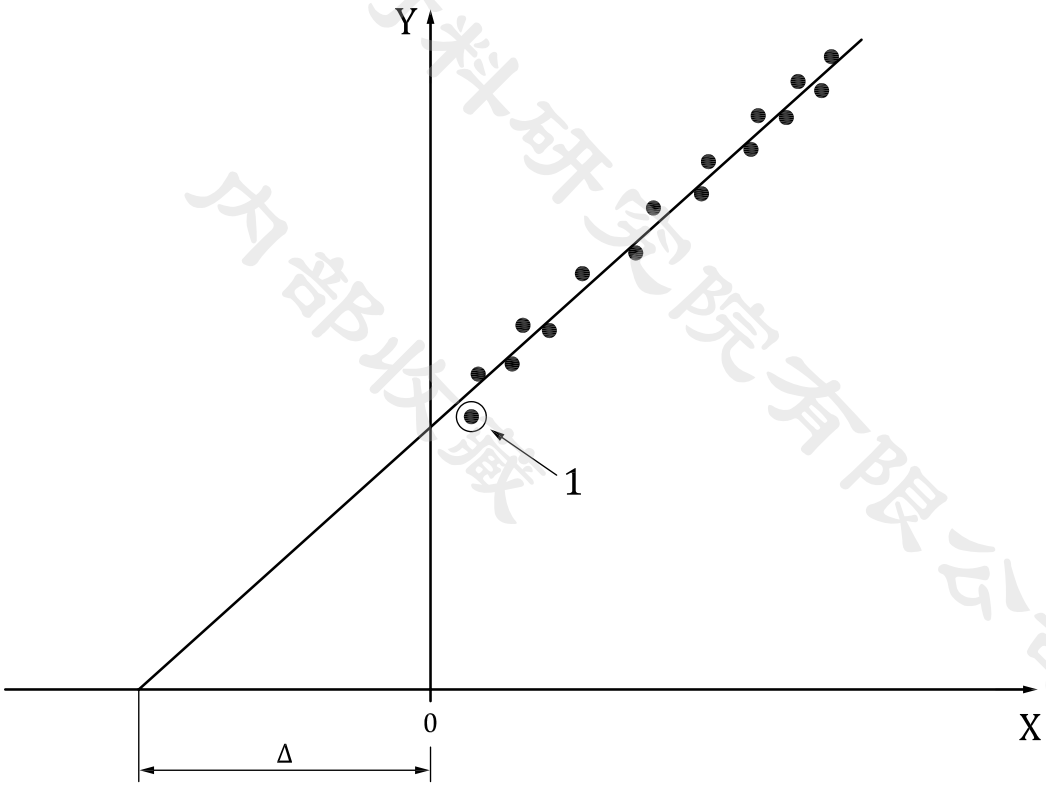
$$N = 1 - \left(\frac{l_2}{a}\right)^3 - \frac{9}{8}\left[1 - \left(\frac{l_2}{a}\right)^2\right]\frac{\delta l_1}{a^2} - \frac{9}{35}\left(\frac{\delta}{a}\right)^2 \tag{3}$$

where

l_1 is the distance from the loading pin (or piano hinge axis) to the midplane of the half-beam to which the load block (or piano hinge) is attached;

l_2 is the distance from the loading-pin centre to its edge (see Figure 1);

If large-displacement corrections which are less than 0,9 are found, this shall be noted in the report.



Key

X delamination length, a

Y cube-root of the normalized compliance, $(CIN)^{1/3}$

1 VIS

Δ x-axis intercept Δ of the linear fit of $(C/N)^{1/3}$, versus a

NOTE The VIS point can be excluded from the linear fit (see 10.2.2).

Figure 4 — Linear fit used to determine the correction *Δ* in the corrected beam theory method

10.2.3 Method B: Modified compliance calibration (MCC)

Establish the relation between the delamination length and the compliance by plotting the width-normalized cube root of the compliance $(bC)^{1/3}$ [or $(bC/N)^{1/3}$ if load blocks are used], as a function of the thickness-normalized delamination length $a/2h$ for the reloading data (see Figure 5). The slope of this relation is defined as m.

The critical energy release rate G_{IC} is given by Formula (4):

$$G_{IC} = \frac{3\,m}{2(2h)} \times \left(\frac{P}{B}\right)^2 \times \left(\frac{bC}{N}\right)^{2/3} \times F \tag{4}$$

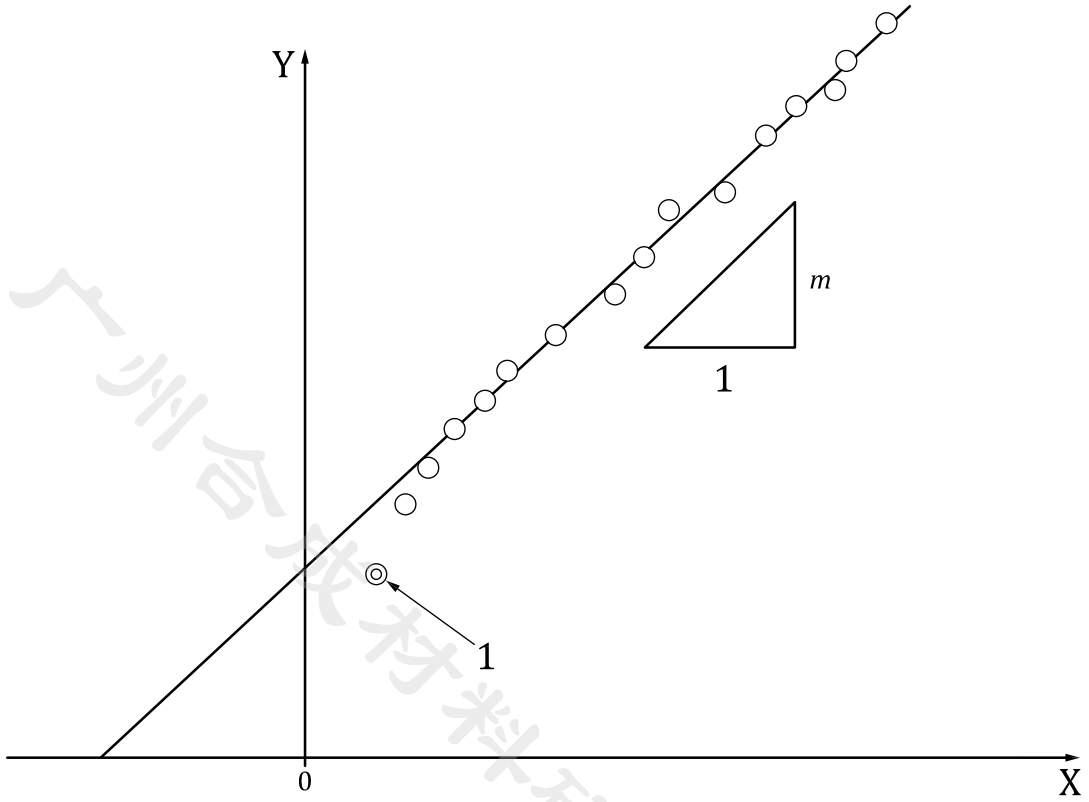
where factors F and N are given by Formulae (2) and (3), respectively. The G_{IC} values corresponding to all initiation and propagation values shall be calculated.

For the case where the delamination length is measured in the horizontal direction (x in Figure 6) using a travelling microscope (as described in B.5), the delamination length x may be used for plotting Figure 4 or 5 and to calculate G_{IC}. In this case, the large-displacement correction factor F is equal to 1. However, if end blocks are used instead of piano hinges, correction factors N in accordance with Formula (3) are required for G_{IC}.

10.3 Data sheets, data plots and statistical calculation

All results corresponding to NL, VIS and 5 % / MAX points from the starter film, the mode I precrack, and PROP values are used to draw a delamination-resistance curve (R-curve), consisting of a plot of G_{IC} versus delamination length a for each specimen (see Figures 7 and 8). When quoting characteristic material values from testing, the five replicates required (see Clause 7), the arithmetic mean, the standard deviation σ, and the coefficient of variation CV of G_{IC} (CV = σ/mean in %) corresponding to each VIS, NL and 5 % / MAX point shall be calculated.

A single test result sheet shall be used to report the test data obtained using the insert (values corresponding to NL, VIS and 5 % / MAX points) and from the mode I precrack (values corresponding to NL, VIS, 5 % / MAX and PROP points) for each specimen. Recommended test result sheets are included in Annex C.



Key

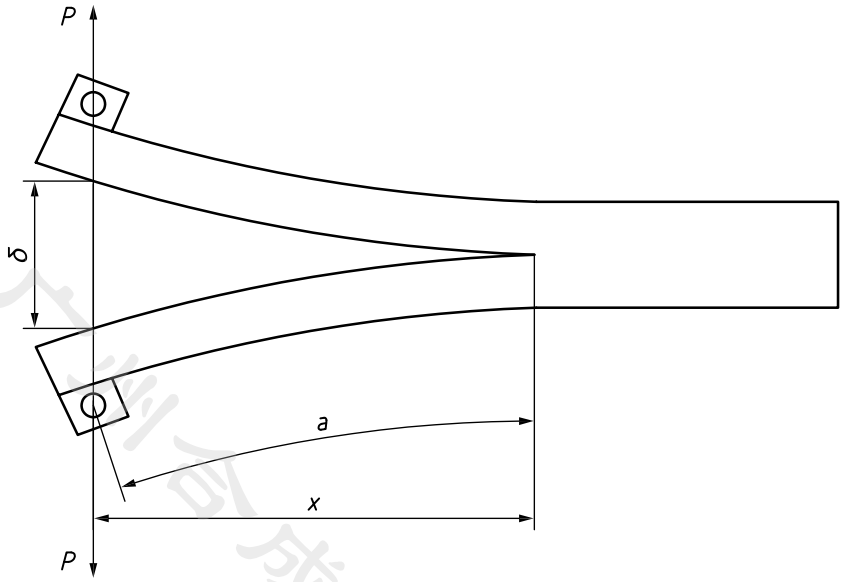
X thickness-normalized delamination length, $a/2h$

Y $(bCIN)^{1/3}$

1 VIS

NOTE The VIS point can be excluded from the linear fit (see 10.2.3).

Figure 5 — Linear fit used to determine the slope *m* in the modified compliance calibration (MCC) method



Key

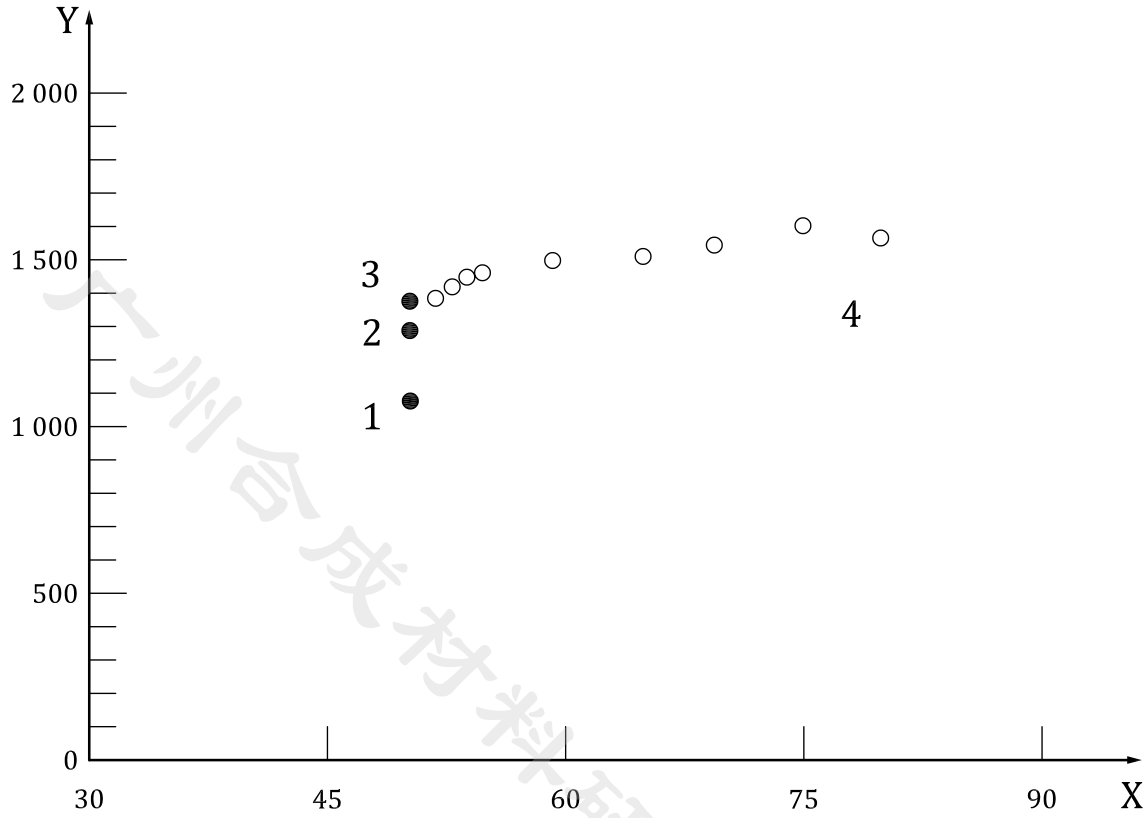
P load

δ load line displacement

a delamination length

x delamination length in the horizontal direction

Figure 6 — DCB specimen under load showing the delamination length measured along the horizontal direction, *x*, and along the curved coordinate scale fixed to the specimen, *a*



Key

X delamination length (mm)

Y G_{IC} (J/m^2)

1 NL

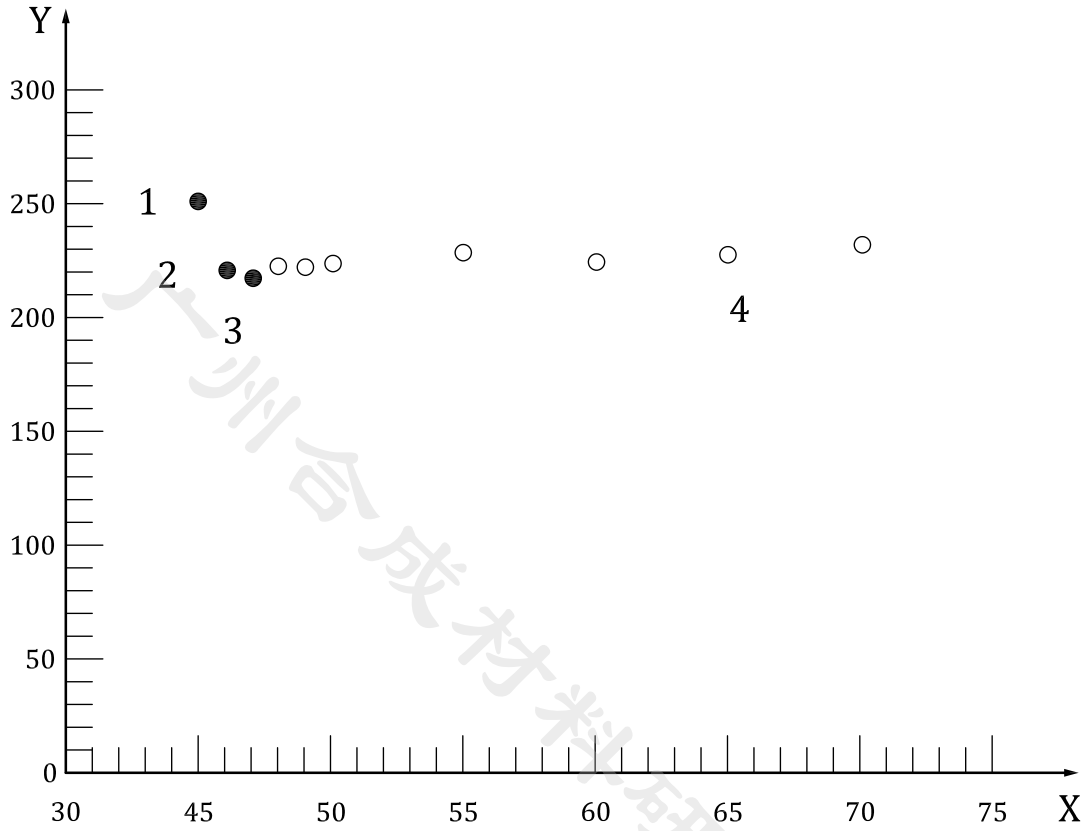
2 VIS

3 5 %

4 PROP

NOTE Initiation G_{IC} values labelled NL, VIS and 5 %, and propagation values, PROP, are defined in Clause 3. Initial-loading values only are shown.

Figure 7 — Typical delamination-resistance curve (R-curve) showing decreasing delamination resistance with delamination length



Key

X delamination length (mm)
Y G_{IC} (J/m^2)
1 NL
2 VIS
3 5 %
4 PROP

NOTE Initiation G_{IC} values labelled NL, VIS and 5 %, and propagation values, PROP, are defined in Clause 3. Initial loading values only are shown.

Figure 8 — Atypical delamination-resistance curve (R-curve) showing decreasing delamination resistance with delamination length

11 Precision

Table 2 shows the materials used and the precision data obtained in two interlaboratory evaluations of this test method. Initiation values quoted are NL values, which for the carbon-fibre-reinforced epoxy (CFRE) material, were identical to VIS values. For the carbon-fibre-reinforced thermoplastic (CFRT) material, the NL point preceded the VIS point. The test procedure was a modification of that in this standard as these tests were conducted continuously with no unloading step following the initial precrack. In all cases, the typical R-curve was obtained (see Figure 7).

Table 2 — Precision data abstracted from ASTM D 5528-94a[a]

Material	No. of labs	Tests per lab	Insert	Average G_{IC} kJ/m^2	s_r	$(CV)_r$ %	s_R	$(CV)_R$ %
CFRE	3	3	13 µm poly-imide	0,085	0,015	17,6	0,014	16,5
CFRT	9	5	7,5 µm poly-imide	1,182	0,126	10,8	0,111	9,4
CFRT	9	5	13 µm poly-imide	1,262	0,132	10,5	0,110	8,7

[a] These results are selected from data first published in ASTM D 5528-94a,[10] to which reference should be made for further information. It is noted in this ASTM standard that the data are limited to selected carbon-fibre-reinforced materials, and variations may be greater for other materials. Further information is also given in Reference [5]. The precision measures of "repeatability" and "reproducibility" are defined in ASTM D 5528-94a as:

Repeatability: Duplicate test results (obtained by the same operator using the same equipment on the same day) from an individual laboratory for the same material should be considered suspect if they differ by more than the "r" value for that material, where $r = 2{,}8s_r$ and s_r is the average of the standard deviation for each participating laboratory.

Reproducibility: The average result reported by one laboratory for a given material should be considered suspect if it differs from the average measurement of another laboratory, or from measurements in the same laboratory taken by a different operator using different equipment, for the same material by more than the "R" value for that material, where $R = 2{,}8s_R$ and s_R is the standard deviation from the mean values of all participating laboratories.

The latest update of ASTM D5528-21 published in 2021[12] does not contain this precision data.

12 Test report

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 15024:2023, indicating the method of analysis;

b) all details necessary to identify the material tested (e.g. laminate manufacturer, fibre material, polymer material, maximum cure temperature T_{mc}, duration of curing t_c);

c) the number of specimens tested, the test date and the test laboratory;

d) the location of each specimen on the test plate;

e) the average thickness, average width, maximum thickness variation along the length, and length of each specimen, the insert material used and the thickness and length of the insert, noting if the insert length measurements differ by more than 1 mm on the two edges;

f) the type of starter film insert, and mould release agent used;

g) the test and conditioning conditions used;

h) the type of load-introduction device used (blocks or hinges), their dimensions, details of their surface preparation, if applicable, and the adhesive used for bonding them to the specimen;

i) the type of precracking used (e.g. mode I or wedge opening), noting if the specimen was removed from the fixture after precracking from the insert;

j) the displacement rates for loading and unloading for testing from the insert and from the mode I precrack;

k) the delamination length measured from the load line on both edges of the specimen after testing (unloading) from the insert, noting if the delamination length measurements differ by more than 2 mm on the two edges;

l) if the CBT method was used, report the x-axis intercept Δ of the linear fit of the cube-root of the normalized compliance, $(C/N)^{1/3}$, versus the delamination length a, as well as the correlation coefficient r^2 of the linear fit, noting if the VIS value was excluded from the fit;

m) if the MCC method was used, report the slope m of the linear fit of the normalized cube-root of the corrected compliance, $(bC/N)^{1/3}$, versus the thickness-normalized delamination length $a/2h$, as well as the correlation coefficient r^2 of the linear fit, noting if the VIS value was excluded from the fit;

n) a copy of the load-displacement curve for each specimen;

o) a table of G_{IC} values and a plot of G_{IC} (with all values corresponding to the points as defined in 10.1) versus delamination length a (R-curve) for each specimen, including large-displacement corrections and load block corrections, if applicable, and noting if the large-displacement correction F is lower than 0,9;

p) the average value and the coefficient of variation for each set of values corresponding to the VIS, NL and 5 % / MAX points;

q) The value of $(C_{5\ \%\ /\mathrm{MAX}} - C_0) \times 100/C_0$, i.e. the percent change in compliance between the initial compliance C_0 and the compliance at the 5 % or MAX point, whichever is applicable;

r) any deviation from the prescriptions of this document (e.g. concerning the dimensions of the specimens or the fibre orientation);

s) any observations on the test (e.g. deviation of the precrack or the delamination from the midplane or occurrence of stick-slip, fibre-bridging, permanent deformation after unloading or sticking of the insert) that may have affected the test procedure or the results;

t) the results of material characterization (e.g. fibre and void-volume fraction), if available.

Annex A
(normative)

Preparation and bonding of the load blocks or piano hinges

The load blocks or piano hinges and the specimen shall first be lightly abraded. Use only sandpaper or grit blasting because the loads required to delaminate the specimens used in this test are quite low. The blocks or hinges and the specimens shall then be cleaned with a solvent. If bond failure occurs, it may be necessary to consult ISO 17212 for a more sophisticated procedure. Bonding of the blocks or hinges shall be done immediately after the surface preparation. In previous tests on similar specimens, a cyanoacrylate adhesive has been found adequate in most cases. Alternatively, a tough, room-temperature-cure adhesive may be used. The surface preparation and the type of adhesive used shall be noted in the report.

Annex B
(informative)

Recommendations for testing

B.1 Guidelines for specimen lay-up

This document has been developed for zero-degree unidirectional laminates only. These lay-ups exhibit very little anti-clastic bending. Hence, the plate bending stiffness components satisfy the condition that the ratio $(D_{12})^2/(D_{11}D_{22})$ is much less than 1, D_{11}, D_{12} and D_{22} being components of the bending-stiffness matrix for the upper and lower arm of the double cantilever beam[9]. Multidirectional lay-ups may not satisfy this condition, and hence may exhibit significant anti-clastic bending. Furthermore, multidirectional lay-ups typically exhibit crack branching away from the specimen midplane, and hence are not recommended.

B.2 Guidelines for starter film insert material and preparation

A polymer film is recommended as the starter film to avoid problems with folding or crimping that have been observed with aluminium films. For epoxy-matrix composites cured at temperatures below 180 °C, a thin film of polytetrafluorethylene (PTFE) is recommended. For composites that are manufactured at temperatures above 180 °C (for example polyimide or bismaleimide thermoplastics), a thin polyimide film is recommended.

B.3 Guidelines for specimen thickness

If possible, the specimen thickness is chosen as shown in Formula (B.1):

$$2h > 8{,}28\left(\frac{G_{IC}\,a_0^2}{E_{11}}\right)^{1/3} \tag{B.1}$$

where

$2h$ is the thickness of the specimen;

G_{IC} is the anticipated maximum fracture toughness;

a_0 is the initial delamination length ($a_0 = A - l_3 + l_2$ for load blocks);

E_{11} is the tensile modulus of elasticity of the specimen in the direction of the fibres.

Application of this criterion requires prior knowledge of values of G_{IC} for the material to be tested and, if available, test results or data from published literature should be used. Specimen thicknesses determined using this criterion should result in a large-displacement correction factor F that is close to 1.

B.4 Test preparation

Preparation of at least one extra specimen is recommended (i.e. six instead of the minimum five) when

a) testing a material for the first time or

b) the operator has no experience in visually detecting delamination onset for the material being tested.

The load cell and load-cell range can be chosen by assuming typical loads of less than 500 N.

Applying a thin layer of water-soluble typewriter correction fluid ("white ink") along the edges of the specimens after conditioning will facilitate the observation of delamination growth. Some correction fluids use solvents that are harmful to some types of matrix material, however, and it is recommended that the composition of the fluid be checked before application.

Delamination growth may be followed by eye, or by using a travelling microscope. See B.5 for more details.

In transparent laminates, the delamination length may be followed inside the specimen by marking the specimen across its width rather than at the edge.

B.5 Automation of delamination length measurement

Delamination length measurement may be automated by installing a position sensor on the travelling microscope. To measure x, marks are scribed on the specimen as specified in 6.5. The travelling microscope is then positioned at the first mark and the position of the microscope noted. When the delamination front passes the grid line of the microscope, the delamination length will be the same as the position of the microscope. The microscope is then moved to the next mark and this process is repeated until the test is terminated.

B.6 Guidelines for conditioning

ISO 291 gives guidelines for conditioning before testing, and for use during testing, that are typical of test laboratory climates. The only temperatures allowed in ISO 291 are +23 °C and +27 °C, with a corresponding selection of humidities. If agreed, however, +20 °C may be used. All three temperatures may yield considerable humidity content in the specimens, depending on the matrix material, even after the recommended duration of 88 h. The results from drying at elevated temperatures versus ISO 291 may vary, and therefore may not be compatible. Hence, it is recommended that specimens be tested with a minimum humidity content after drying at elevated temperatures (e.g. +70 °C for epoxies, and one-day maximum storage in a desiccator).

B.7 Unstable delamination growth

Delamination growth in most fibre-reinforced laminates is not steady but progresses at a somewhat irregular speed, even when followed visually on a macroscopic scale. Unstable delamination growth is characterized by phases of no, or very slow, delamination growth followed by rapid, almost instantaneous, delamination growth. This typically produces sharp drops in the load-displacement curve, i.e. zones of virtually infinite slope. Often it is impossible to record delamination length readings during unstable delamination growth. Unstable delamination growth is usually followed by arrest (no delamination growth), and then a reload phase during which the load increases and produces a (local) maximum in the load when delamination growth continues.

B.8 Guidelines for wedge precracking

If an alternative to load-induced precracking is necessary (see Clause 4 and 9.2.7), the following procedure is recommended for wedge opening. The specimen is clamped at 5 mm beyond the tip of the starter film. The width of the wedge that is driven into the specimen shall be at least the same as that of the specimen and the opening angle shall be as small as possible without the wedge actually touching the tip of the delamination. The wedge may be driven in by hand, by tapping on the end, or by using a suitable fixture with the test machine. The wedge is driven into the specimen until the tip of the wedge is about 2 mm to 3 mm in front of the clamp. The wedge precrack will usually extend a few millimetres

into the clamp but should be short enough to allow a delamination length increment of at least 50 mm beyond the tip of the precrack.

B.9 Recommendations for obtaining the NL point

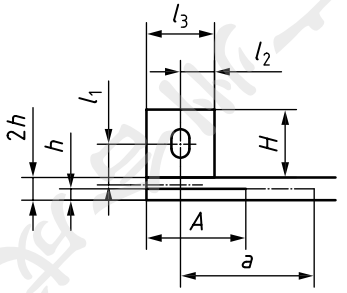
Physical evidence from X-ray imaging for two types of material (carbon-fibre/epoxy and carbon-fibre/PEEK) shows that the onset of delamination from the starter film in the interior of the specimen occurs close to the NL point and before the VIS point. The NL point will frequently yield the lowest, most conservative, values of the interlaminar fracture toughness. However, it may be difficult to determine the NL point reproducibly on the load-displacement curve. Coefficients of variation of up to 10 % are not uncommon. However, a plot of the analogue signals for load versus displacement, typically recorded on the paper chart of an X-Y recorder, may yield more consistent results with less variability than those obtained by fitting a curve through the data points recorded electronically during the test with a digital data acquisition device. Performing a linear fit on the load-displacement curve starting at a finite load to avoid nonlinearity due to play and using a consistent criterion for deviation from linearity, such as that of the midpoint of the plotter trace, may yield more consistent results.

Annex C
(informative)

Recommended test result sheet

Table D.1

Test result sheet for DCB mode I, page 1 of 2			**Fibre:**	**Resin:**	
Specimen identification	.....	No. of specimens	.....		
Test laboratory	.....	Test personnel	.....	Test date	.....
Laminate manufacturer	.....	Resin manufacturer	.....	Fibre manufacturer	.....

Specimen length	l (mm)	Load introduction	.....		**Average NL ± σ**
Average thickness	$2h$ (mm)	Block thickness	H (mm)		**CV NL**
Max. thickness variation	$\Delta(2h)$ (mm)	Block length	l_3 (mm)		**Average VIS ± σ**
Specimen width	b (mm)	Block/hinge width	(mm)		**CV VIS**
Insert material	.....	Surface preparation	.....		**Av. 5 %/MAX ± σ**
Insert thickness	(µm)	Adhesive	.....		**CV 5 %/MAX**
Insert length	A (mm)	Conditioning temperature	T_d (°C)		
Mould release agent	.....	Conditioning duration	t_d (h)		
Precrack length	a_0 (mm)	Test temperature	T (°C)		
Max. cure temperature	T_{mc} (°C)	Relative humidity	rh (%).....		
Cure duration	t_c (h)	Loading/unloading rate	(mm/min)		
Max. delamination length	a_{max} (mm)	Distances l_1 and l_2	(mm)		
% change in compliance	(%).....	Correction	.....	**Minimum initiation value:**	

Point	a (mm)	Load (N)	δ (mm)	Location of specimen and insert in test plate	Comments	G_{IC} CBT (J/m^2)	G_{IC} MCC (J/m^2)	Flexural modulus E_f (GPa)
NL (insert)				Length.....mm				
5 %/MAX (insert)								
VIS (insert)								
NL (precrack)								
5 %/MAX (precrack)								
VIS (precrack)								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP								
PROP				Width.....mm				

Table D.1 *(continued)*

Test result sheet for DCB mode I, page 2 of 2				**Fibre:**		**Resin:**	
Specimen identification	.....	No. of specimens	.....				
Test laboratory	.....	Test personnel	.....			Date of test	.....
Laminate manufacturer	.....	Resin manufacturer	.....			Fibre manufacturer	.....

Method A (CBT)		Difference in insert length on each side ≤ 1 mm?	yes/no	**Observations and comments**
Slope of fit		Difference in precrack length on each side ≤ 2 mm?	yes/no	
y-intercept of fit		Precrack tip 3 mm to 5 mm from insert?	yes/no	
Δ (mm)		Difference in total delamination on each side ≤ 2 mm?	yes/no	
Correlation coefficient		Specimen removed after precracking?	yes/no	
		VIS included in linear fit?	yes/no	
Method B (MCC)		Stable delamination growth throughout test?	yes/no	
Slope of fit		All correction factors $F \geq 0{,}9$?	yes/no	
y-intercept of fit		Load line displacement/$a < 0{,}4$?	yes/no	
m		Reference to ISO 17212?	yes/no	
Correlation coefficient				

Point	F	N	F/N	C/N	$(C/N)^{1/3}$	$(bC/N)^{1/3}$	$a/2h$	δ/a	Checks OK?
NL (insert)									
5 %/MAX (insert)									
VIS (insert)									
NL (precrack)									
5 %/MAX (precrack)									
VIS (precrack)									
PROP									
PROP									
PROP									
PROP									
PROP									
PROP									
PROP									
PROP									

Annex D
(informative)

DCB test with flat insert hinge

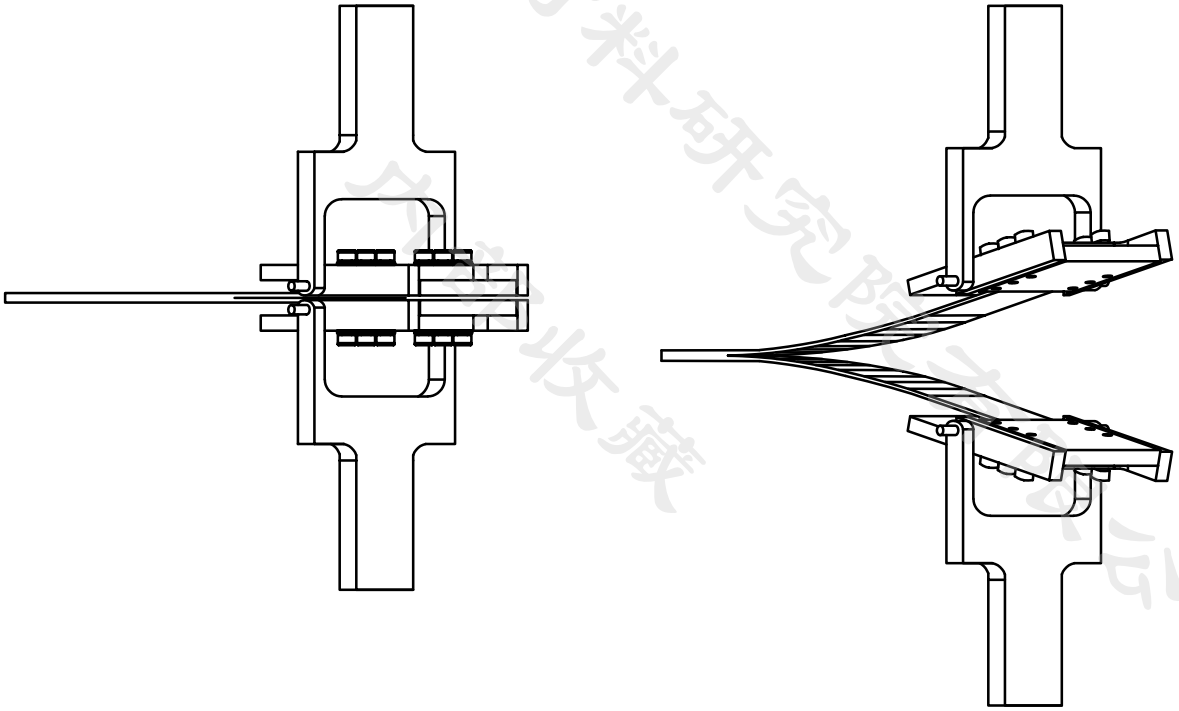
D.1 DCB test concept with insert hinge

Figure D.1 shows the

a) set up of a DCB test with flat insert hinges and

b) DCB specimen and insert hinge under the conditions of opening displacement.

This DCB test with flat insert hinges does not require adhesive between the specimen and flat insert hinges. The test procedure is same as the test methods using a DCB specimen bonded with loaded blocks or hinges.

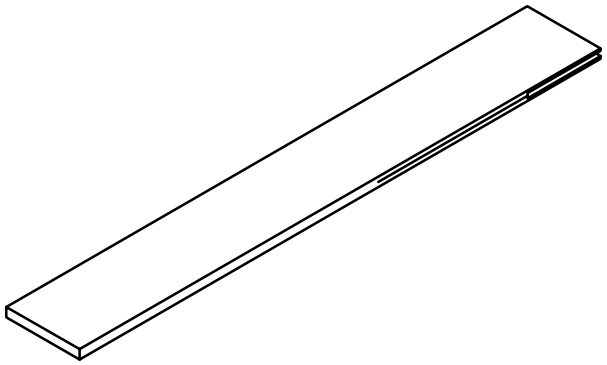


a) Before starting **b) Under conditions of opening displacement**

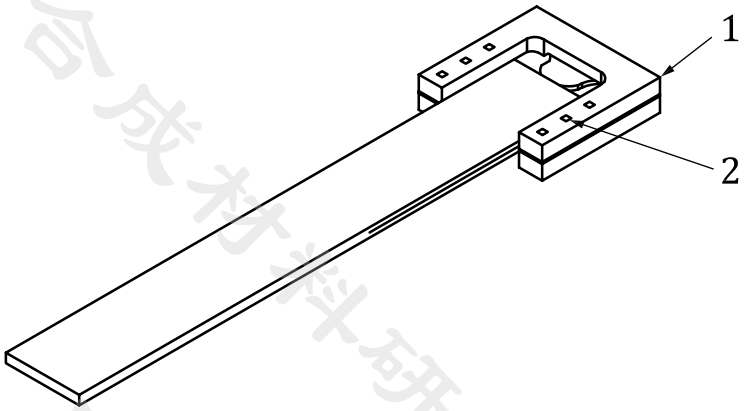
Figure D.1 — DCB specimen and flat insert hinge

D.2 DCB test fixture and specimen

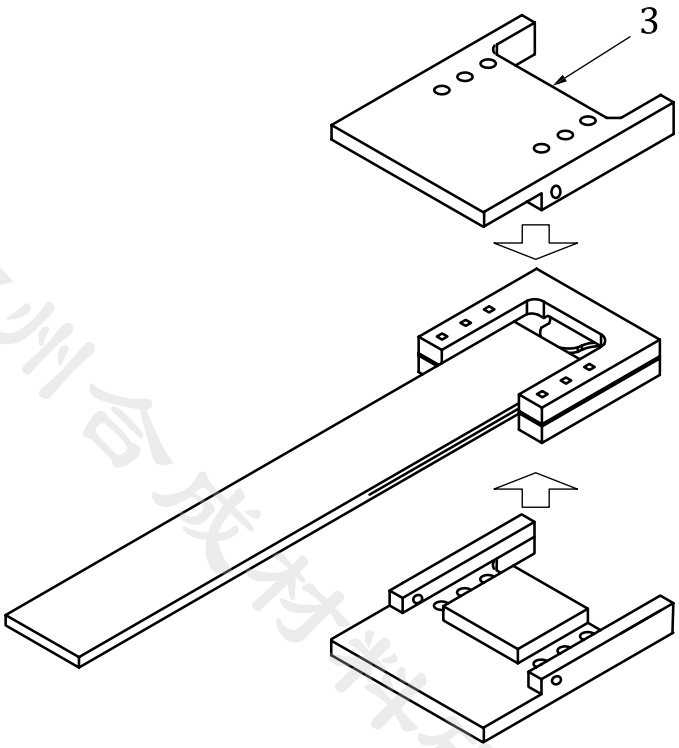
Figures D.2 a) to-c) show the procedure of setting up a DCB specimen with flat insert hinges. As shown in Figure D.2 c), each cantilever beam is fixed to the hinge parts with the bolt. In this test fixture, insert parts have female threads on the outside, and the hinge part is thick for gripping the cantilever beam with the insert parts.



a) DCB specimen



b) DCB specimen set with insert parts



c) DCB specimen with insert and hinge parts tightened with bolts

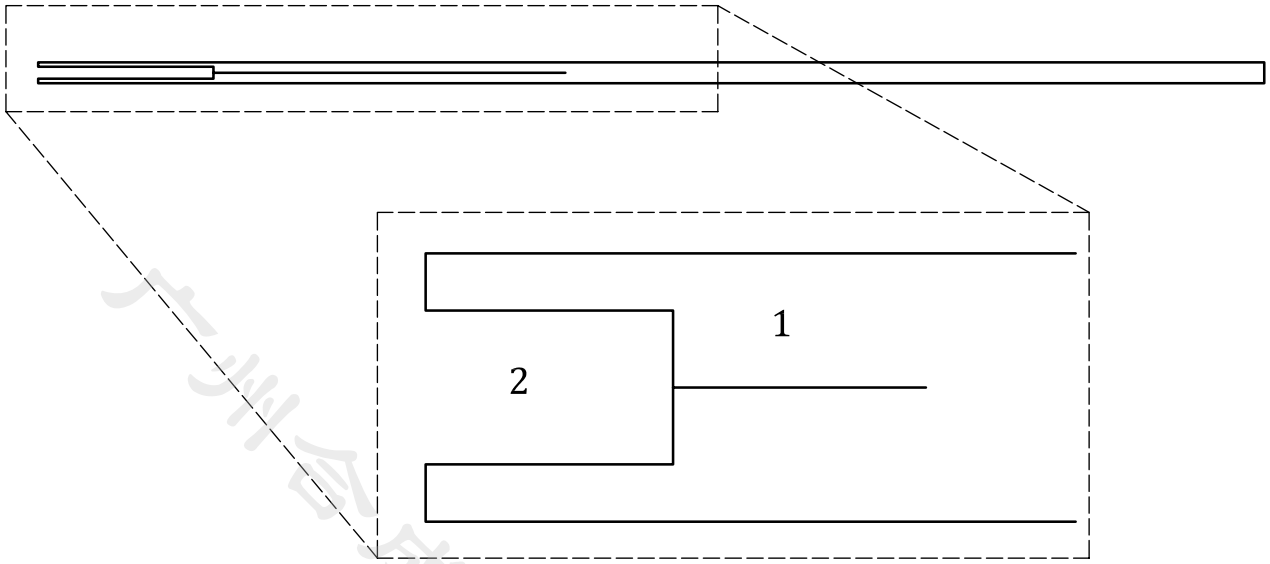
Key

1 insert part
2 female threads
3 hinge part

Figure D.2 — DCB specimen and flat insert hinge

D.3 DCB test specimen

Figure D.3 shows the side view of the DCB specimen for insert hinges. A space for the insert parts is prepared at the tip of the DCB specimen to set the insert parts (Figure D.2(b)). This space can be additionally machined after cut as the coupon specimen from the test plate in accordance with the procedure in Annex B and Figure D.3. Alternatively, this space can be manufactured by laminating a spacer-applied release agent in the laminate process. DCB specimens with space for insert parts were carried out for academic papers[11],[12].



Key

1 insert film

2 space for insert parts

Figure D.3 — Side view of DCB specimen for flat insert hinge

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