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Fibre-reinforced plastic composites — Determination of the mode II fracture resistance for unidirectionally reinforced materials using the calibrated end-loaded split (C-ELS) test and an effective crack length approach

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National foreword

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Fibre-reinforced plastic composites — Determination of the mode II fracture resistance for unidirectionally reinforced materials using the calibrated end-loaded split (C-ELS) test and an effective crack length approach

Composites plastiques renforcés de fibres — Détermination de la résistance à la rupture en mode II de matériaux renforcés de fibres unidirectionelles en utilisant l'essai de délaminage (C-ELS) et une approche de la longueur de fissure réelle

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Introduction

Previous attempts to determine mode II delamination resistance curves (R-curves) for composites have been hampered by the experimental difficulty of determining crack length in the absence of any applied beam opening displacement and when a complex damage zone develops ahead of the crack front. The effects of friction in the different mode II test specimens have also been widely debated and have typically been determined to introduce errors of between 1 % and 3 % in G_{IIC} determination for ELS specimens (n.b. friction effects would appear to be more significant in 3 point loaded end notch flexure (3ENF) (to be standardized by ASTM) and, particularly, in the 4 point loaded (4ENF) test specimen. Stabilized ENF was not popular in round-robin trials).

The procedure presented here uses the end-loaded split test apparatus and specifies an experimental procedure to calibrate the clamping fixture and simultaneously determine the flexural modulus of the specimen. This serves two purposes. Firstly, the clamp calibration has been found to significantly reduce scatter in the results between different test laboratories and secondly, it provides an accurate means by which crack lengths can be calculated and thus their measurement can be avoided. Although this procedure still includes an experimental determination of crack length, the use of calculated (or effective crack lengths) means that values of G_{IIC} can be determined without experimentally measured crack length values. The procedure is a development of that published by ESIS (the European Structural Integrity Society), Technical Committee 4, Polymers and Composites[1], who carried out the preliminary enabling research through a series of round-robin exercises conducted in 2004 and 2007.

Fibre-reinforced plastic composites — Determination of the mode II fracture resistance for unidirectionally reinforced materials using the calibrated end-loaded split (C-ELS) test and an effective crack length approach

1 Scope

This International Standard specifies a method for the determination of mode II shear load delamination resistance. G_{IIC}, (critical energy release rate), of unidirectional fibre-reinforced plastic composites using the calibrated end-loaded split (C-ELS) test.

It is applicable to carbon-fibre and glass-fibre reinforced thermosets and thermoplastics.

The scope is not necessarily limited to these fibres and lay-ups, but for laminates with other types of fibres or lay-ups, no recommendations for specimen dimensions and fibre volume content are currently available.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 5893, *Rubber and plastics test equipment — Tensile, flexural and compression types (constant rate of traverse) — Specification*

ISO 15024, *Fibre-reinforced plastic composites — Determination of mode I interlaminar fracture toughness, GIC, for unidirectionally reinforced materials*

3 Symbols and abbreviated terms

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

a	measured delamination length, distance between the load-line (intersection of the plane through the pin-hole centre of the load-block normal to the specimen width and the plane of delamination) and the tip of the delamination on the edge of the specimen (see Figure 1)
a_o	insert film length, measured from the load-line to the tip of the insert film (seeFigure 1)
a_p	precrack length, the length between the load-line and the tip of the precrack formed in during the precracking step
b	width of the specimen
C	compliance δ/P of the specimen
C_{max}	compliance of the specimen at maximum load
C_0	initial compliance of the specimen neglecting start-up effects, e.g. due to play in the specimen fixture

$C_{5\,\%}$	initial compliance, C_0, of the specimen increased by 5 %
δ	displacement of the cross-head of the testing machine
E_1	elastic modulus determined from “three-point bending” flexural test or from the clamp calibration test
G_{IIC}	critical energy release rate for mode II shear loading
H	height of the load-block
l	total length of the specimen
l_1	distance between the centre of the loading pin and the mid-plane of the specimen beam to which the load-block is attached (see Figure 5), i.e. equal to $(H + h)/2$ if the pin hole is through the centre of the block
l_2	distance between the centre of the loading pin and the edge of the load block, measured towards the tip of the insert (starter film) or the tip of the mode I or mode II precrack (see Figure 5), i.e. equal to $l_3/2$ if the pin hole is through the centre of the block
l_3	length of the load-block (see Figure 5)
L	free length of the specimen between load-line and clamp (see Figure 1)
MAX	maximum load on the load-displacement trace (see Figure 7)
m	slope of C versus a^3 plot
NL	onset of nonlinearity on the load-displacement trace (see Figure 7)
P	load measured by the load-cell of the testing machine
PROP	increments of the delamination length during stable delamination growth (propagation) that are marked on the load-displacement curve (see Figure 7)
r^2	correlation coefficient of linear fit
VIS	onset of visually recognizable delamination growth on the edge of the specimen that is marked on the load-displacement trace (see Figure 7)
$2h$	total thickness of the specimen (thickness of each specimen arm is h)
5 %	point of intersection of a straight line with the load-displacement trace, with the slope of the straight line corresponding to $C_{5\,\%}$

4 Principle

This procedure specifies a method for the determination of the delamination resistance of unidirectional fibre-reinforced polymer laminates under mode II shear load using the calibrated end-loaded split (C-ELS) test. The resistance to the initiation and propagation of a delamination is determined from a non-adhesive insert and from a mode I (opening) or a mode II (shear) precrack. The critical energy release rate for mode II loading can be calculated and a resistance-curve (R-curve, i.e. a plot of the critical energy release rate versus delamination length) determined.

5 Apparatus

A tensile testing machine in compliance with ISO 5893, capable of producing a constant load-rate between 1 mm/min and 5 mm/min in displacement control should be used. The load-cell should be

calibrated and accurate within ±1 % for the chosen load-range (loads are typically expected to be in the range of 100 N to 1 000 N). The testing machine shall be equipped with a fixture to introduce the load to the pin inserted into the load-block that allows rotation of the specimen end.

The recommended loading jig requires a clamping arrangement to freely slide in bearings in the horizontal direction (side-ways) with a fixed load point. This is shown schematically in Figure 1. Two test fixtures used in the round-robin programmes (see Clause 9) are shown in Figure 2.

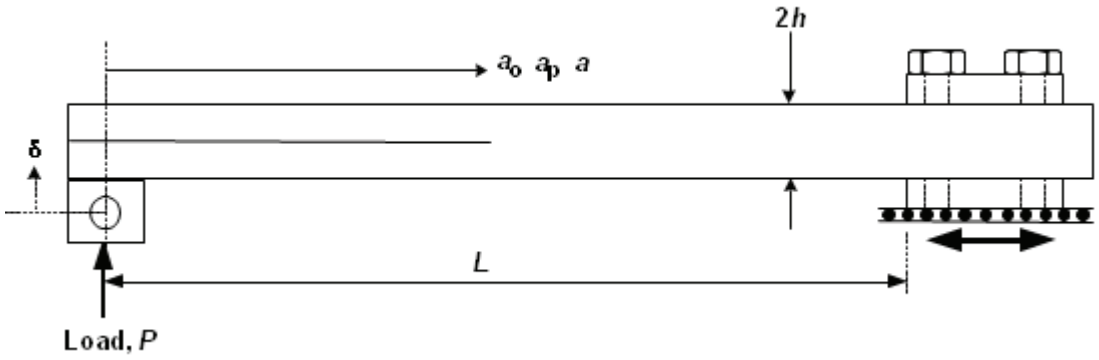
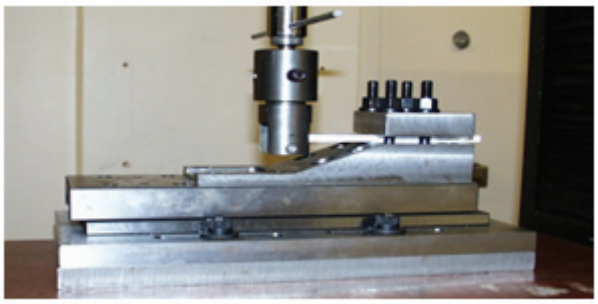


Figure 1 — ELS test specimen showing the clamping fixture and loading



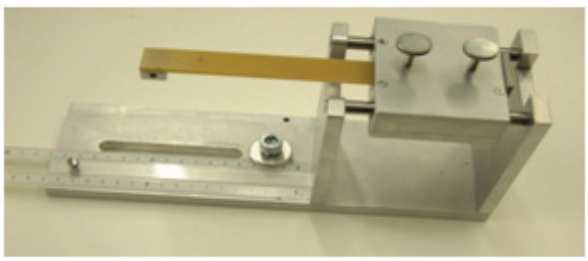


Figure 2 — Two alternative ELS test fixtures

A calibrated lever arm (torque wrench) is required to apply a consistent pressure while fixing the specimens into the sliding fixture. It is recommended that this device can apply closing torsion in the range of 0 Nm to 30 Nm to a precision of ±1 Nm. During the test, the load shall be applied vertically on the load-block by pulling upward provided the clamp is symmetrical with respect to the specimen. The testing machine shall be equipped with means for recording the complete load-displacement traces (loading and unloading) that allow a determination of the loads and the corresponding displacements with an accuracy of ±1 %. Vernier callipers or a micrometer should be used to measure the specimen thickness ($2h$) to an accuracy of ±0.02 mm and the specimen width, b, to an accuracy of ±0.02 mm. A travelling microscope (or video camera) shall be used to monitor the length of the delamination along one edge of the specimen with a magnification of between ×10 and ×25.

6 Specimens

6.1 Preparation of specimens

The recommended specimen width, *b*, and length, *l*, are 20 mm and 190 mm, respectively. The specimen length shall not be less than the active length of the insert, a_0, plus 110 mm; thus, $l \geq a_0 + 110$ mm. For recommendations on the length of a_0, see 6.2. The free length, *L*, is typically 100 mm. The recommended specimen thickness ($2h$) is 3 mm for 60 % by volume carbon fibre-reinforced and 5 mm for 60 % by volume glass fibre-reinforced composites.

Other specimen dimensions can be used, but the specimen width should be between 15 mm and 30 mm. Increasing the length of the specimen is not critical, shortening will reduce the maximum delamination length that can be investigated, and thus, yield too few data points for the analysis (see 8.1). If specimens are too thin or not sufficiently stiff, delamination growth might not be induced or occur at large displacements only, or permanent deformation of the specimen might occur, invalidating the assumptions of linear elastic fracture mechanics.

6.2 The initial defect

A crack starter film should be placed at the laminate mid-thickness during the lay-up of the composite panel prior to moulding. The film should be PTFE or another fluoro-polymer with excellent non-stick properties. The film should be thin (between 10 microns and 13 microns) to minimize the disturbance of the laminate. The upper service temperature of the film should be greater than the cure temperature of the laminate. When the composite panel is trimmed, the active starter film length should satisfy the requirement that $a_0 > 50$ mm, so that the influence of the load-block can be neglected. This initial defect will be extended in mode I or mode II loading prior to testing (see 7.2).

6.3 Attaching the load-block to the specimen

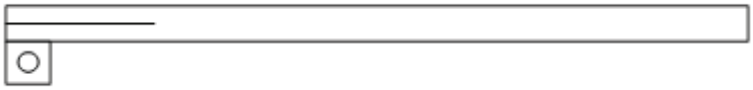
One load-block should be bonded to each specimen for the purposes of load-introduction [see Figure 3 b)]. The block should be of the same width as the specimen. Prior to bonding, the load-block and the specimen (in the position where the block will adhere) should firstly be lightly abraded using an abrasive paper or grit blasting. Both the load-block and the specimen should then be cleaned with a solvent.

A tough, room-temperature cure adhesive (e.g. two part epoxy) is recommended. If bond failure occurs it might be necessary to consult ISO 4588 for a more sophisticated surface treatment procedure. Bonding of the load-block should be done immediately after the surface preparation.

The load-block should be well aligned with the specimen and held in position with a clamp while the adhesive sets. Specimen edges should be smoothed prior to determining the dimensions. For the clamp calibration procedure (as described in 7.1), one specimen should be prepared with the load-block bonded to the end not containing the insert film as shown in Figure 3 a). After the clamp calibration, this load-block can be removed and one should be bonded at the insert end [see Figure 3 b)] to allow the specimen to be tested in mode II.

NOTE If the specimen is sufficiently long, that the end-block attached for the clamp calibration measurement does not interfere with the clamping of the specimen in the subsequent mode II test, then this load-block can be left in place on the beam.

a) (1 specimen per sample)



b) (5 specimens per sample)

Figure 3 — Position of the load-blocks for a) inverse ELS specimen for clamp calibration and b) for the ELS fracture specimen

6.4 Moisture conditioning

Moisture conditioning is required for obtaining baseline data in order to test specimens with uniform moisture content. The drying conditions (temperature and duration) shall be chosen according to the recommendations of the resin supplier. Conditioning should be performed after bonding of the load-block. Before testing, the specimens can be stored in a desiccator for at most three days after conditioning.

NOTE Other conditioning procedures can be applied for the investigation of specific conditioning effects.

6.5 Final specimen preparation and measuring dimensions

In preparation for the visual measurement of crack length, applying a thin layer of typewriter correction fluid ("white ink") on the edges of the specimen after conditioning will facilitate the measurement. The following procedure is recommended:

a) Apply a thin coat of white type-writer correction fluid to the edge of one side of each specimen. (Ensure the use of a new bottle and try to apply the coating in a single brush stroke, avoiding re-brushing if possible. Practicing on another specimen or the reverse side of the test specimen is recommended).

b) When the layer is dry, locate the position of the end of the insert film and mark this with a black pen. (A nib of 0,1 mm is recommended).

c) Mark the specimen edge at regular increments, starting one division before the end of the film insert and extending to a = 100 mm. (Drawing straight vertical lines across the beam edge at the crack length increments is helpful). The mark increment should be chosen to be either 2,0 mm or 2,5 mm, depending upon the specimen length available for crack propagation. For shorter lengths, the narrower increment should be selected.

d) With the specimen to be used for the clamp calibration, draw lines at 50 mm, 60 mm, 70 mm, 80 mm, 90 mm, 100 mm, and 110mm from the load-block pin hole centre (load-line). (These will be the positions at which the specimen will be clamped in the clamp calibration test; see 7.1.)

Some typewriter correction fluids contain solvents that might be harmful to the laminate matrix material. A water-based paint is thus recommended.

NOTE The vertical lines will shear when the crack front passes and might kink due to the shear strain ahead of the crack tip. The crack position is determined from the lines shearing.

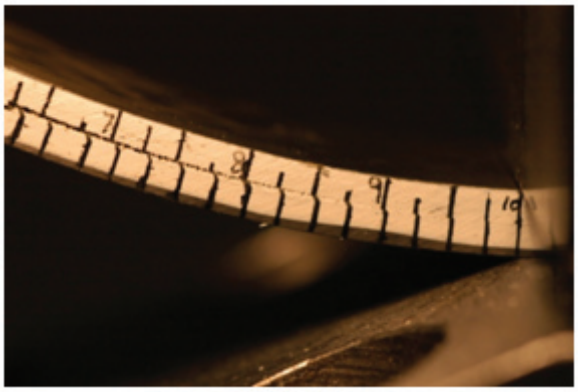


Figure 4 — A specimen during loading in an ELS test, showing the crack length markers at 2,5 mm increments along the specimen edge (L = 100 mm for this test)

Measure and record the length, l, of each specimen to the nearest mm. 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 three points along the centre-line of the specimen. The variation in thickness and average values of the width and the thickness should be recorded for each specimen. The variation in thickness, i.e. the maximum difference between the thickness measurements, should not exceed 0,1 mm for each specimen. The length, l_3, and height, H, of the end block should be measured to the nearest 0,1 mm and be recorded. The load-block details are shown in Figure 5.

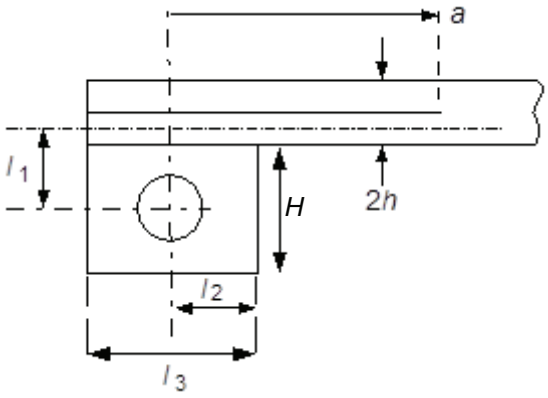


Figure 5 — Load-block dimensions

6.6 Number of specimens

A minimum number of five specimens shall be tested. A minimum of one calibration specimen per sample of nominally identical specimens should be used.

7 Procedure

7.1 Performing the calibration of the ELS fixture

The following procedure should be followed:

a) Position the specimen in the clamping fixture as shown in Figure 6, loosely tighten the clamp, attach the load-block to the test machine, e.g. via the loading pin, then clamp the specimen with a free length, L = 110 mm and record the clamping pressure applied.

b) Apply load to the load-block at a cross-head displacement rate of 1 mm/min such that the beam deforms elastically and record the load-displacement trace.

Loading to a maximum load of 250 N is recommended for CFRP specimens and 150 N for GFRP specimens.

NOTE It is important that the insert film is held fully within the clamp, so that it does not influence the measured beam compliance. Adjust the length, L, if necessary to ensure this.

c) Stop the loading, and then, fully unload the specimen and repeat the procedure with the beam clamped at free lengths of 100 mm, 90 mm, 80 mm, 70 mm, 60 mm, and 50 mm. The unloading can be performed at up to 10 mm/min.

NOTE It is important that the clamping of the specimen in the ELS fixture can be performed in a reproducible manner. This is conveniently achieved using a torque wrench, such that the retaining nuts can be tightened to the same level of torque in each test. (A torque of 8 Nm has been found to be suitable for the clamping of carbon-fibre epoxy specimens, see Figure 2, upper test fixture.)

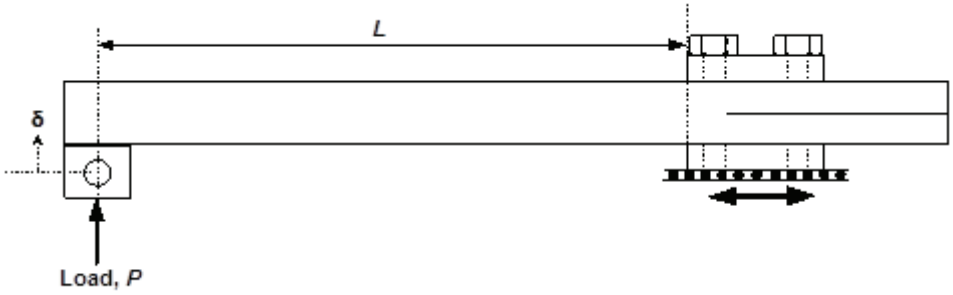


Figure 6 — The clamp calibration set up with the delamination fully within the clamp

When the clamp calibration has been performed, the specimen used can then have the load-block removed and another should be bonded to the end containing the crack (see 6.3) so that a fracture test can be performed on this specimen [see Figure 3 b)].

7.2 Pre-cracking the specimens

Experience has shown that initiation values of G_{IIC} will depend on the precracking method used. Specimens should always be precracked and either mode I or mode II precracking can be used. The procedure used should be clearly stated in the report.

7.2.1 Mode I precracking

The specimens should be loaded in mode I (tensile opening) until the crack has grown at least 2 mm (but no greater than 5 mm) ahead of the insert. The procedures of ISO 15024 should be consulted for mode I loading using the double cantilever beam (DCB) test specimen.

NOTE If mode I rather than mode II precracking is chosen (see 7.2), two load blocks shall be bonded to each specimen rather than one. After precracking, one of the two load blocks shall be removed before mode II testing.

7.2.2 Mode II precracking

The procedures for precracking in mode II are somewhat more complex, as it is intended that unstable crack initiation from the insert be avoided. Thus, the following procedure is recommended. A load-scale of up to 500 N is usually sufficient for composite laminates.

a) Determine the initial length of the crack, a_0.

b) Determine an initial free length, L, via $L = a_0 \times (4/3)$ and clamp the specimen at this value of L.

c) Load the specimen at a displacement rate of 1 mm/min until the delamination has grown at least 2 mm (but no more than 5 mm) ahead of the insert. The load-displacement trace should be recorded during this procedure.

d) Immediately stop loading and fully unload the specimen (displacement rates of up to 5 mm/min can be used for unloading).

e) Record the length of the newly formed precrack, a_p.

f) Release the clamp, remove the specimen, and repeat for the remaining specimens in the sample.

NOTE The initial free length is chosen such that the initial ratio of (a/L) = 0,75. This is chosen to promote stability. An energy analysis predicts that the ELS test is stable provided approximately that (a/L) > 0,55. Thus, the factor of 0,75 provides a margin for safety, promoting the conditions for stability.

7.3 Testing the samples in mode II from the precrack formed in 7.2

A travelling microscope and specimen illumination should be used to observe delamination growth. A magnification of between ×10 to ×25 should be employed. The following procedure should be used for each specimen in the sample.

a) Determine the free length, L, such that (a_p/L) > 0,55 and clamp the specimen.

NOTE Stability can be improved by using larger values of a_p/L, but using larger values will reduce the length available for crack propagation.

b) Load the specimen at a displacement rate of 0,5 mm/min, until the crack has grown to within 10 mm from the clamp, recording the observed crack lengths on the load-displacement trace. Crack lengths should be recorded at 2,0 mm (or 2,5 mm) intervals. The first point should correspond to the instant when the crack is seen to grow ahead of the precrack.

NOTE The slow loading rate will improve the accuracy of the crack length measurements.

c) Immediately stop the loading and fully unload the specimen (displacement rates of up to 5 mm/min can be used for unloading).

d) Continue to record the load and displacement during the unloading.

e) Remove the specimen and inspect for any damage.

NOTE Some materials have been observed to microcrack severely ahead of the main crack tip. For materials exhibiting this effect, it is helpful to observe the black vertical lines drawn on the edge of the specimen. Microcracking might not result in the shearing of the lines. Photographs of the crack appearance can be helpful.

8 Data analysis

8.1 The points for data analysis

The data required for the analysis are delamination lengths, a, and the corresponding loads, P, and displacements, δ. Several points for the determination of G_{IIC} can be determined from the test. If mode II precracking was undertaken, initiation values of G_{IIC} should be determined from both the insert (starter film) and from the mode II precrack. If mode I precracking was undertaken, initiation values of G_{IIC} should only be determined from the precrack. In addition, a number of propagation points are to be determined, as outlined below.

8.1.1 Initiation points

For the purpose of this International Standard, crack initiation is defined via the 5 % or max criteria, i.e. by a 5 % increase in the initial compliance or by the maximum load point: The 5 % value corresponds to

the point on the load-displacement trace at which the compliance has increased by 5 % of its initial value, C_0. A best 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 C_0 + 5 % whose intersection with the load-displacement curve yields the load and displacement to be used for the calculation, unless the intersection is at a larger displacement than the maximum load in which case the maximum load and the corresponding displacement have to be used.

NOTE Round-robin testing has shown that the definition of crack initiation based on the nonlinear (NL) point is prone to significant scatter. In addition, the visually determined definition of crack initiation, the (VIS) point, is not consistent with the effective crack length approach recommended here (see 8.3.3).

8.1.2 Propagation points

In addition to the initiation points, propagation points (see Figure 7) can be determined for each delamination length measured during the crack propagation stage.

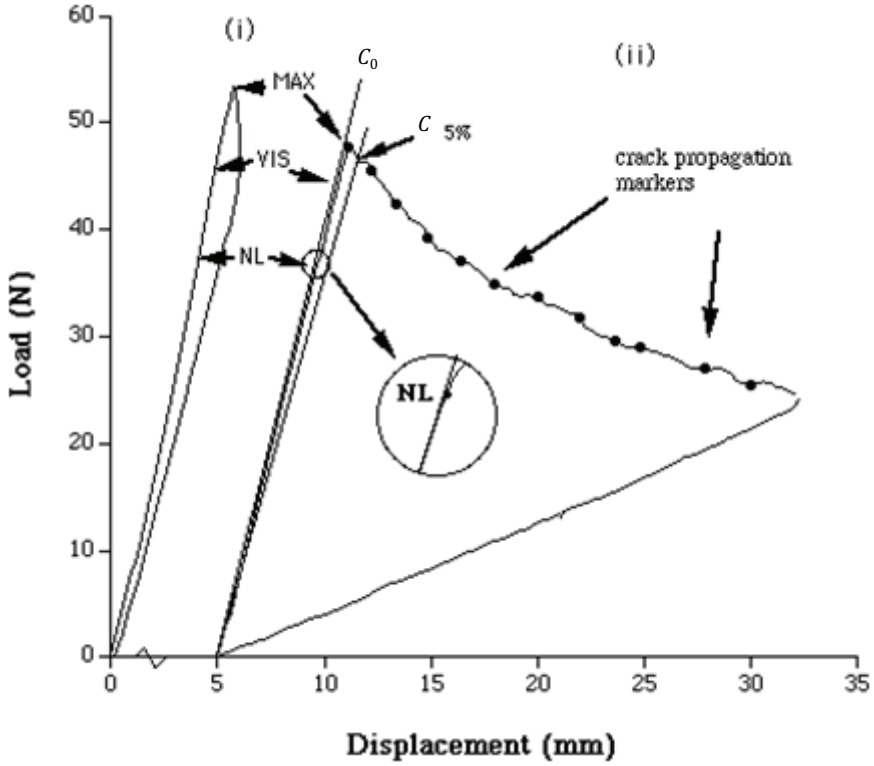


Figure 7 — Load versus displacement trace for (i) precracking from the insert and (ii) testing from the precrack. The (NL), (VIS), (5 %), and (Max) initiation points and subsequent propagation (Prop) points are shown

8.2 Determination of the ELS clamp correction

The analysis of the clamp calibration data (as recorded in 7.1) is performed assuming that the data take the linear form as expressed in Formula (1).

$$C^{\frac{1}{3}} = slope \cdot L + slope \cdot \left|\Delta_{\text{clamp}}\right| \tag{1}$$

where

$$slope = \left(\frac{1}{2bh^3 E_1} \right)^{\frac{1}{3}}$$

Thus, the procedure is as follows:

a) Perform a linear regression of the data for each loading (excluding any initial nonlinear take up effects) and hence deduce the compliance, C, for each value of free length, L.

b) Plot these values on a graph of $C^{1/3}$ versus L, and perform a linear regression of these data, extending the regression back to $C^{1/3} = 0$.

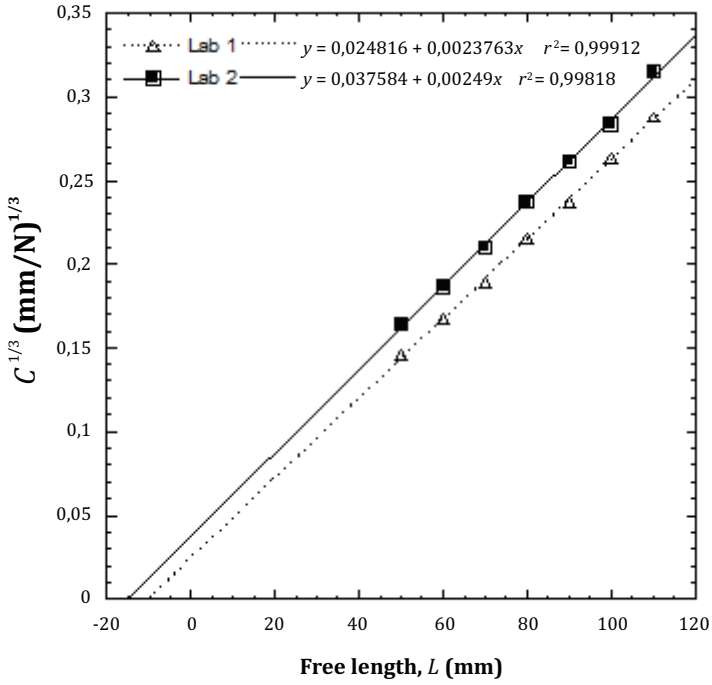
c) From the graph, determine the slope of the regression line and its intercept with the L-axis.

d) The positive value of the intercept with the L-axis is referred to as the clamp correction, Δ_{clamp}.

e) The slope of the regression line should be recorded and can be used to determine the flexural modulus of the specimen.

The intercept of the regression line with the $C^{1/3} = 0$ line should occur at a negative value on the L-axis (see Figure 8). This negative intercept corresponds to a positive value of Δclamp. A zero intercept would imply an infinitely rigid clamp. If a positive intercept is obtained, the data should be checked for errors and, if necessary, the calibration should be repeated.

A typical representation of the data are shown in Figure 8, as determined in two test laboratories using different test fixtures.



NOTE Δ_{clamp} = C1/3 – axis intercept/slope for the linear regressions, i.e. 10,4 mm for Lab 1 and 15,1 mm for Lab 2)

Figure 8 — Typical clamp calibration data (values of $C^{1/3}$ versus L) determined in two laboratories

8.3 Determination of G_{IIC} values

The values of G_{IIC} can be determined for values of crack initiation and propagation to generate the resistance curve (R-curve), i.e. values of G_{IIC} as a function of crack length. Three data reduction schemes are described. Method 1, the experimental compliance method (ECM) and method 2, the simple beam theory (SBT) method; both require experimental values of crack length to be determined however, method 3, the corrected beam theory with effective crack length (CBTE) is independent of measured crack length. Methods 2 and 3 require a value for the flexural modulus of the specimen and this should be determined via three point bending or from the clamp calibration procedure. Method 3 has been found to return the best reproducibility (see Clause 9) and is the recommended method to follow.

The values G_{IIC} are determined using the Irwin-Kies formula:

$$G_{IIC} = \frac{P^2}{2b} \cdot \frac{dC}{da} \tag{2}$$

where

P is the load;

b is the specimen width;

C is the compliance;

a is the crack length.

8.3.1 Method 1: experimental compliance method (ECM)

An experimental compliance method based upon the cubic relationship between the compliance, C and the measured crack length, a, can be written as:

$$C = C_o + ma^3 \tag{3}$$

where C_o and m are constants. Thus, if values of C (for crack propagation points) are plotted versus the cube of the measured crack length, a^3, values then a linear regression of these data will yield a slope, m and an intercept with the C axis of C_o. Formula (3) can be differentiated with respect to crack length and substituted into Formula (2) to give:

$$G_{IIC} = \frac{3P^2a^2m}{2b} \tag{4}$$

The values of G_{IIC} should be determined for each point using the ECM method and the R-curve should be drawn. The values of r^2 determined from the regression analysis of C versus a^3 should be noted in the test report.

8.3.2 Method 2: simple beam theory (SBT)

The compliance, C, of the ELS specimen shown in Figure 1 can be expressed as:

$$C = \frac{\delta}{P} = \frac{3a^3 + L^3}{2bh^3E_1} \tag{5}$$

where

δ is the load-line displacement;

L is the free length;

h is the half specimen thickness (see Figure 1);

E_1 is the flexural modulus of the specimen.

Formula (5) can be differentiated with respect to the crack length and substituted into Formula (2) to give:

$$G_{IIC} = \frac{9P^2a^2}{4b^2h^3E_1} \tag{6}$$

The values of G_{IIC} should be determined for each point using the SBT method and the R-curve should be drawn.

8.3.3 Method 3: corrected neam theory using effective crack length (CBTE)

Using the corrected beam theory with effective crack length approach, the crack length is calculated using the measured compliance and the known flexural modulus of the specimen, E_1. This also requires the value of the clamp correction, determined in 8.2.

In Formula (5), no correction was made for beam root deflections and rotations at either the crack tip or clamping point and no correction was included for the transverse shear effects in the composite

arms. These can be taken into account using corrected beam theory as expressed in Formula (7). The compliance of the ELS specimen can be expressed as:[2]

$$C = \frac{\delta}{P} = \frac{3\left(a_e\right)^3 + \left(L + \Delta_{\text{clamp}}\right)^3}{2bh^3E_1} \tag{7}$$

where

a_e is the effective (calculated) crack length;

Δ_{clamp} is the clamp correction.

Formula (7) can be re-arranged:

$$a_e = \left[\frac{1}{3}\left\{2bCh^3E_1 - \left(L + \Delta_{\text{clamp}}\right)^3\right\}\right]^{\frac{1}{3}} \tag{8}$$

and substituting for crack length in Formula (6):

$$G_{\text{IIC}} = \frac{9P^2a_e^2}{4b^2h^3E_1} \tag{9}$$

The values of G_{IIC} should be determined using the CBTE method [Formula (9)] for each point and the R-curve should be drawn. The value of E_1 can be deduced from slope of the clamp calibration data, i.e. the slope of the linear regression to the $C^{1/3}$ versus L data, as:

$$E_1 = \frac{1}{2b\left(h \times slope\right)^3} \tag{10}$$

It should be noted that in the analysis described above, corrections have not been included for large displacement or load-block effects. In an earlier protocol,[1] these were corrected via the use of correction factors F and N. Round-robin studies have shown that these represent only relatively small corrections and thus, for simplicity, they have been excluded in this procedure. The effect of such corrections can be determined, and this is discussed in Annex A.

9 Precision

Data from a round-robin completed on a carbon-fibre epoxy composite material,[3] which was tested in four laboratories are shown in Table 1. Each laboratory tested five specimens.

Table 1 — Test results from an inter-laboratory ESIS TC4 round-robin on a carbon-fibre epoxy laminate

	G_{IIC} (ECM) (J/m²) Method 1			G_{IIC} (SBT) (J/m²) Method 2			G_{IIC} (CBTE) (J/m²) Method 3		
	Init vis[a]	Init 5 %	Mean prop	Init vis	Init 5 %	Mean prop	Init vis	Init 5 %	Mean prop
Mean	981	1 064	1 052	870	920	921	1 098	1 172	1 102
SD	253	236	206	162	105	101	193	99	51
CoV (%)	25,8	22,2	19,5	18,6	11,5	11,0	17,6	8,5	4,7

NOTE SBT: simple beam theory; ECM: experimental compliance method; CBTE: corrected beam theory with effective crack length. Analysis points were: Init vis = visual initiation, Init 5 % = 5 % compliance offset initiation, and Mean prop = the mean of the propagation values recorded. Mean and SD are the reproducibility of the results obtained across the four test laboratories.

[a] Incomplete data.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard (i.e. ISO 15114) and to the referring standards;

b) a complete identification of the material (e.g. laminate manufacturer, fibre-material, polymer material, maximum cure temperature, T_{mc}, duration of curing, t_c, location of specimen on plate);

c) test date, test laboratory, test personnel identification;

d) number and label of specimens tested and type(s) of method used for the analysis;

e) average thickness, average width, maximum thickness variation along the length, and length of each specimen, insert (starter film) material, and thickness, length of the insert; note if insert lengths measurements differ by more than 1 mm on both edges;

f) conditioning temperature, T_d, and conditioning duration, t_d, and temperature, T, and relative humidity, r.h., during the test;

g) dimensions of the load-block, surface preparation, if applicable, and adhesive.

h) type of precracking used (i.e. mode I or mode II);

i) load-rate for loading and unloading, for testing from the insert and from the mode I or mode II precrack;

j) E-modulus value, E_1, from "three-point bending" test or from the clamp calibration test;

k) slope, m, of plot of the compliance, C, versus the cube of the delamination length, a^3, if method 1 is used for the data analysis (see 8.3) and the correlation coefficient, r^2, of the linear fit;

NOTE Propagation points only are used for this linear regression.

l) copy of the load-displacement trace for each specimen;

m) table of G_{IIC} ($C_{5\,\%}$ initiation and propagation values) and plot of G_{IIC} ($C_{5\,\%}$ initiation and propagation values) versus delamination length, i.e. an (R-curve) for each specimen;

n) average values and standard deviation for the initiation value as defined as the 5 % offset or maximum load (stating whether this is from the insert or precrack, and if a precrack, stating the precracking mode used) and average value and standard deviation of the last 10 propagation values (PROP) or of the last 50 % of the propagation values, whichever contains the larger number of data points, from all specimens tested;

NOTE If a specimen is excluded from averaging, the reason for this should be noted in the report.

o) observations from testing (e.g. deviation of the precrack or the delamination from the midplane, stick-slip, occurrence of fibre-bridging, permanent deformation after unloading, sticking of insert film, no plateau in the R-curve) that might have affected the test procedure or the results;

p) any deviation from the prescriptions of this protocol (e.g. dimensions of specimens, fibre orientation).

q) plot of $C^{1/3}$ vs L for the clamp calibration and the value of Δ_{clamp} determined;

r) a photograph of the ELS fixture used.

Annex A
(informative)

Large displacement and load-block correction factors

Large displacements of the specimen during the test give rise to a reduction in the moment arm. This effect has been analysed by Williams[4] and it has been shown that it can be corrected via the correction factor, F, which is given by:

$$F = 1 - \theta_1 \left(\frac{\delta}{L} \right)^2 - \theta_2 \left(\frac{\delta l_1}{L^2} \right) \tag{A.1}$$

Where δ, L, and l_1 have the meaning as defined in Clause 3 and where θ_1 and θ_2 are defined below.

The effects of bonding a load-block to the test specimen will be to stiffen the arm and additionally, the offset to the load-point will incur additional rotational effects. This effect was also analysed by Williams and it was shown that it can be corrected via the factor, N, which is given by:[4]

$$N = 1 - \theta_3 \left(\frac{l_2}{L} \right)^3 - \theta_4 \left(\frac{\delta l_1}{L^2} \right) - \theta_5 \left(\frac{\delta}{L} \right)^2 \tag{A.2}$$

and where θ_3, θ_4, and θ_5 are defined below. The correction factor, F, is a multiplying factor applied to $G_c \rightarrow (G_c.F)$, i.e. is applied to Formulae (4), (6), and (9). The correction factor, N, is a dividing factor applied to $C \rightarrow (C/N)$, i.e. is applied to Formulae (1), (3), (4), (5), and (7).

In situations when the crack length is not measured, i.e. if only the CBTE (method 3) is employed, then initial values of F and N can be determined using the initial crack length value, $a = a_p$ in the formulae for θ given below.

The values of θ are given by:

$$\theta_1 = \frac{3}{20} \left\{ \frac{15 + 50\left(\frac{a}{L} \right)^2 + 63\left(\frac{a}{L} \right)^4}{\left[1 + 3\left(\frac{a}{L} \right)^3 \right]^2} \right\} \tag{A.3}$$

$$\theta_2 = \frac{-3\left(\frac{L}{a} \right)\left[1 + 3\left(\frac{a}{L} \right)^2 \right]}{1 + 3\left(\frac{a}{L} \right)^3} \tag{A.4}$$

$$\theta_3 = \frac{4}{1 + 3\left(\frac{a}{L} \right)^3} \tag{A.5}$$

$$\theta_4 = \frac{-9}{4} \frac{\left\{ \left[1 - \left(\frac{a}{L} \right) \right]\left[1 + 3\left(\frac{a}{L} \right)^3 \right] + 4\left(\frac{a}{L} \right)^2 \left[1 - \left(\frac{l_2}{a} \right)^2 \right]\left[1 + 3\left(\frac{a}{L} \right)^2 \right] \right\}}{\left[1 + 3\left(\frac{a}{L} \right)^3 \right]^2} \tag{A.6}$$

$$\theta_5 = \frac{36}{35} \cdot \frac{1+\frac{3}{8}\left(\frac{a}{L}\right)^3\left[35+70\left(\frac{a}{L}\right)^2+63\left(\frac{a}{L}\right)^4\right]}{\left[1+3\left(\frac{a}{L}\right)^3\right]^3} \tag{A.7}$$

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[5] ISO 17212, *Structural adhesives — Guidelines for the surface preparation of metals and plastics prior to adhesive bonding*

[6] ISO 14125, *Fibre-reinforced plastic composites — Determination of flexural properties*

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