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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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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.

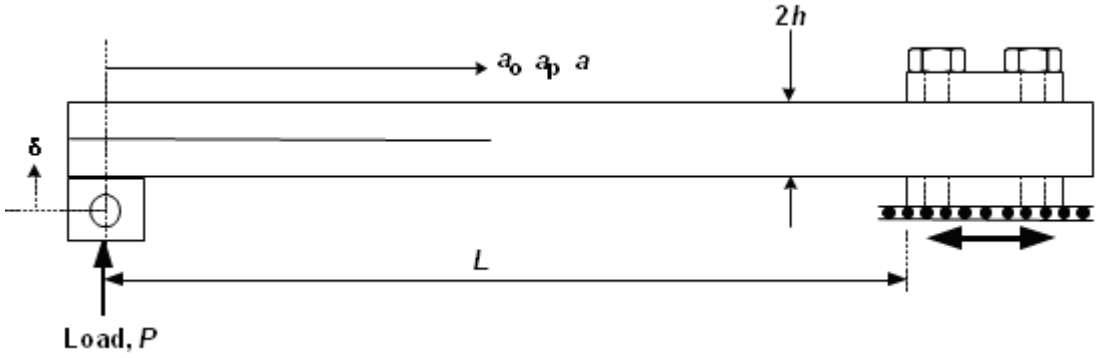


Figure 1 — ELS test specimen showing the clamping fixture and loading

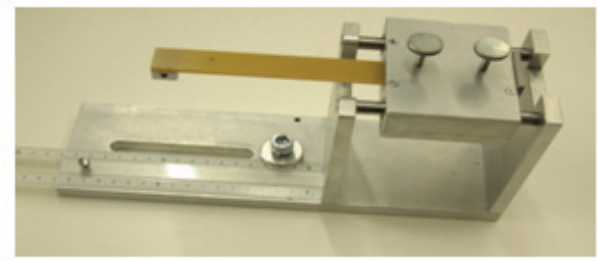


Figure 2 — Two alternative ELS test fixtures

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).

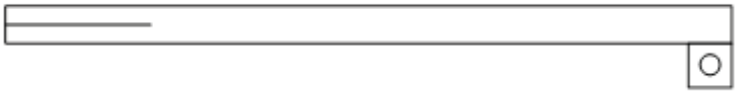
6.3 Attaching the load-block to the specimen

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.

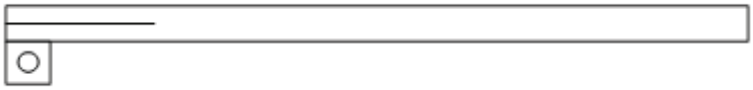
A tough, room-temperature cure adhesive (e.g. two part epoxy) is recommended. If bond failure occurs it might be necessary to consult ISO 4588 for a more sophisticated surface treatment procedure. Bonding of the load-block should be done immediately after the surface preparation.

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.