INTERNATIONAL STANDARD

ISO 15024

First edition
2001-12-01

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
ISO 15024:2001(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15024 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Annex A forms a normative part of this International Standard. Annexes B and C are for information only.

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

1 Scope

1.1 This International Standard 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.

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing*

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

ISO 4588:1995, *Adhesives — Guidelines for the surface preparation of metals*

ISO 5893:—[1)], *Rubber and plastics test equipment — Tensile, flexural and compression types (constant rate of traverse) — Description*

3 Terms and definitions

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

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

NOTE It is measured in joules per square metre.

1) To be published. (Revision of ISO 5893:1993)

3.2
mode I crack opening
the crack-opening mode due to a load applied perpendicular to the plane of delamination using the double cantilever beam specimen shown in Figure 1

3.3
NL point
the point of deviation from linearity on the load versus displacement trace as shown in Figure 2

3.4
VIS point
the point of the onset of delamination, as determined by visual observation, at the edge of the specimen, marked on the load-displacement trace as shown in Figure 2

3.5
5 % / MAX point
the 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) as shown in Figure 2;

b) the maximum load point as shown in 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 in Figure 2, points where the crack has been arrested being excluded

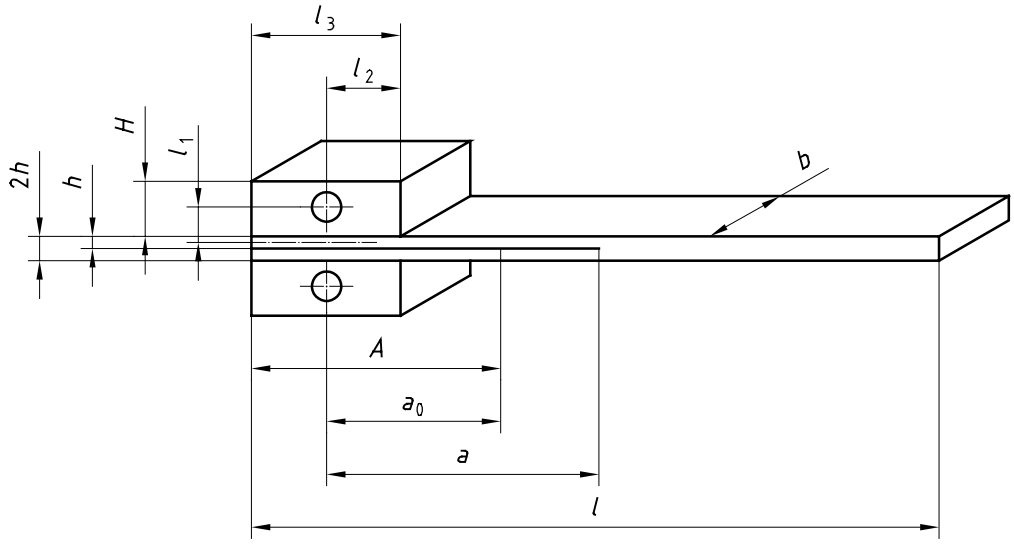
3.7
delamination-resistance curve
R-curve
a cross-plot of G_{IC} for initiation and subsequent propagation values for mode I crack opening as a function of delamination length (see clause 10)

4 Principle

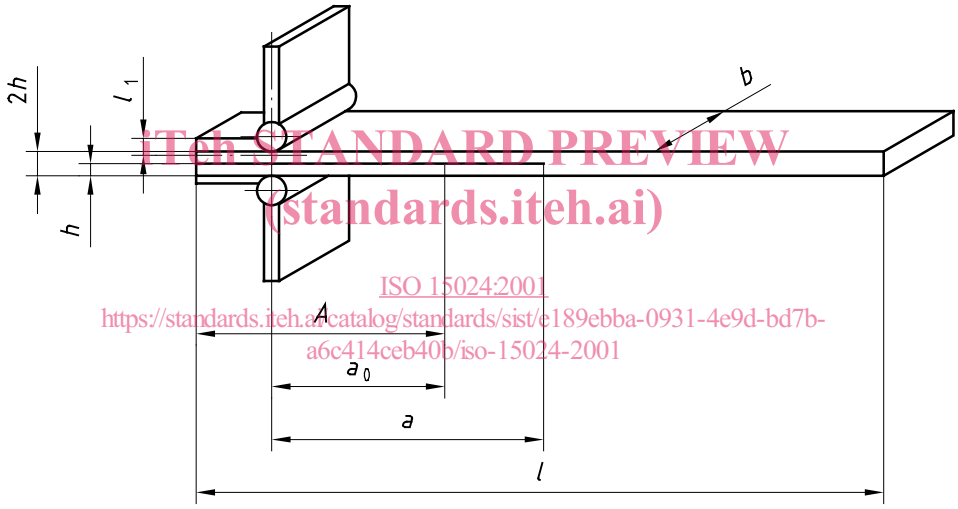
A mode I double cantilever beam (DCB) specimen, as shown in Figure 1, is used to determine G_{IC}, the critical energy release rate, or interlaminar fracture toughness, of fibre-reinforced plastic composites. The test method is limited to zero-degree unidirectional lay-ups only (see clause B.1). Data 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. Delamination typically occurs between plies of dissimilar orientation in composite structures. However, in the DCB test the delamination cracks are grown between similar zero-degree unidirectional plies, resulting in fibre bridging after the delamination crack initiates. This fibre bridging is an artifact of the DCB test and is not 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) Starter delamination using load blocks



b) Starter delamination using piano hinges

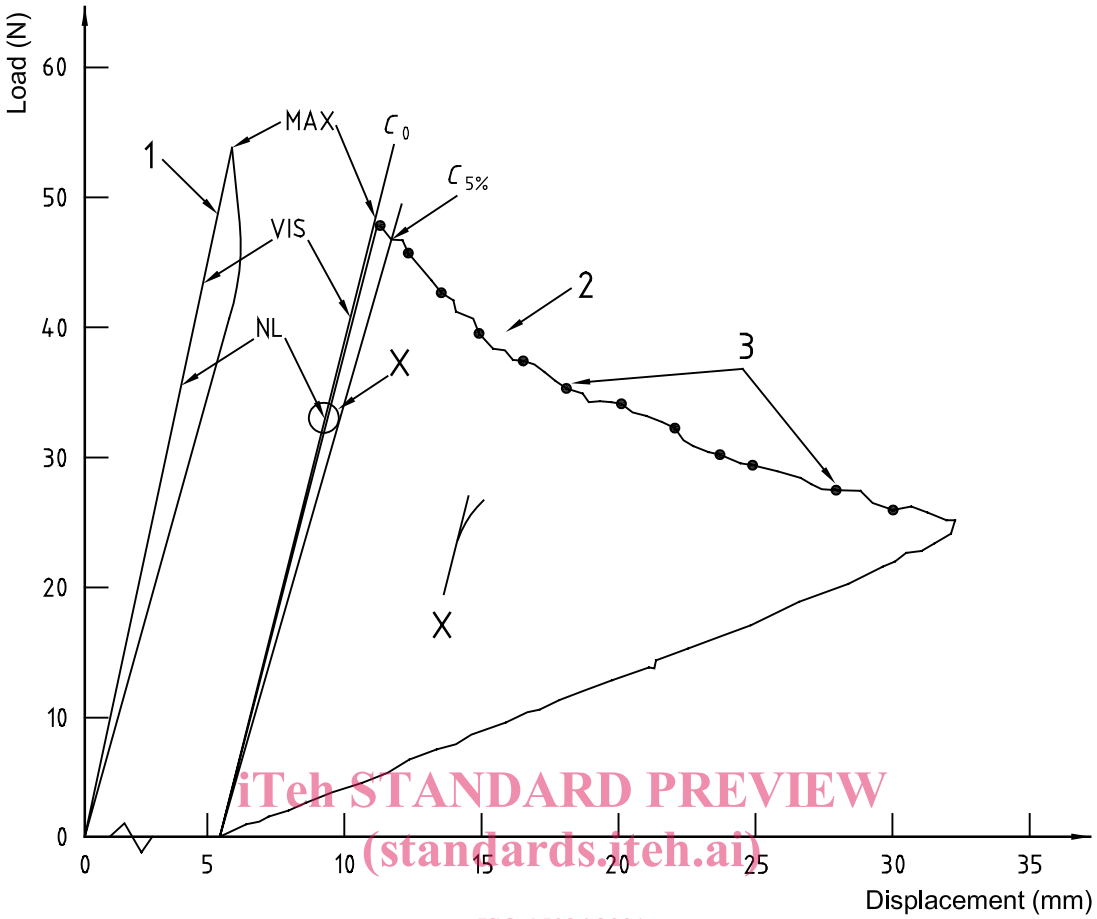
Key

b	Specimen width	l_1	Distance from centre of loading pin (or piano hinge axis) to midplane of specimen
$2h$	Specimen thickness	l_2	Distance from centre of loading pin (or piano hinge axis) to edge of load block (or piano hinge)
a_0	Initial delamination length	l_3	Block length
a	Total delamination length	H	Block thickness
A	Insert length		
l	Specimen length		

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

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

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

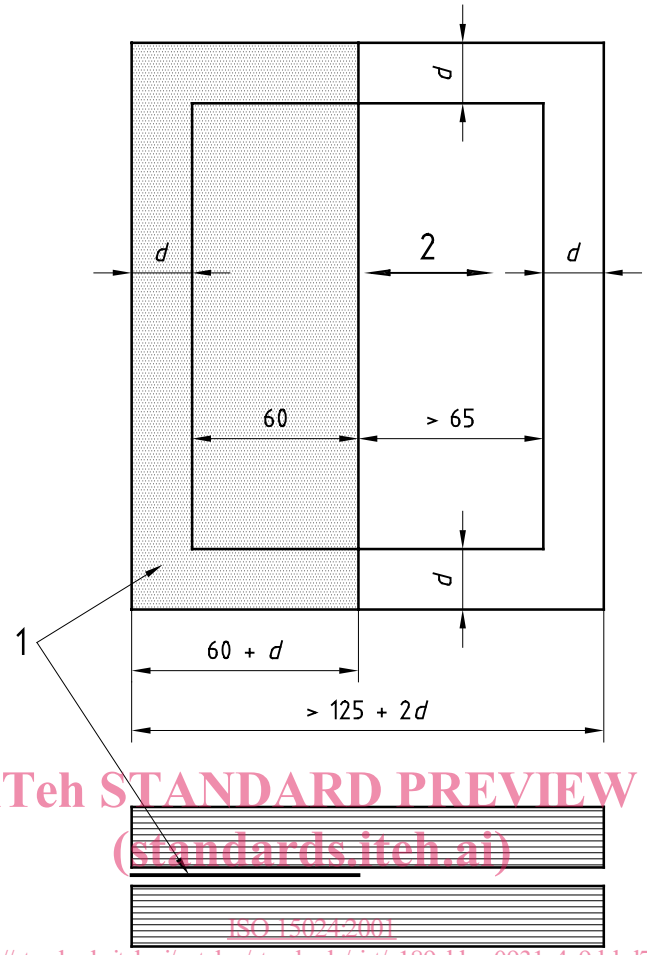


Key

1 Crack initiation followed by unloading
2 Crack propagation
3 Crack propagation markers

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 showing (1) initiation from the insert followed by unloading and (2) re-initiation from the resulting mode I precrack followed by crack propagation and unloading



Key

1 Film insert
2 Fibre direction
d Margin to allow for initial trimming

Figure 3 — An 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 tensile-testing machine shall comply with ISO 5893 and the requirements given in 5.1.2 to 5.1.5.

5.1.2 Speed of testing

The test machine shall be capable of maintaining the constant displacement rate required in 9.2.1 and 9.3.1, as specified in ISO 5893.

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 load cell shall be calibrated and shall have a maximum permissible error of ±1 % of the indicated value. The error in the displacement measurement, normally taken from cross-head movements corrected for any significant loading-train deflection, shall be no greater than ±1 % of the indicated value.

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 clause 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 clause 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 clause A.1).

5.6.4 Solvent: Organic solvent such as acetone or ethanol (see clause A.1).

5.6.5 Sandpaper (abrasive paper), with 500 grade grit or finer (see clause A.1).

5.6.6 White ink: Water-soluble typewriter correction fluid.

6 Test specimens

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 (see clause B.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 clause 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 clause 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 clauses 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. Requirements for bonding the load blocks or piano hinges are given in clause A.1.

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.

NOTE 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 International Standard. Guidelines for conditioning are given in clause B.6.