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Composites and reinforcements fibres — Determination of the fracture energy of bonded plates of carbon fibre reinforced plastics (CFRPs) and metal using double cantilever beam specimens

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

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.

Introduction

The potential benefits to the users of CFRP/metal assemblies of implementing the adhesive fracture energy of DCB specimen based on this document are:

a) expanding CFRP applications to the fields where it could be used in combination with metallic components;

b) the detection or the prevention of physical properties loss — such as ion migration and time-related degradation in sealant film, injected calking layer and glass fibre reinforced plastics (GFRPs) layer;

c) demonstrating the conformity to specified conditions for type certification requirements in the engineering such as aircraft developments;

d) evaluating the procedures for maintenance, repair and overhaul (MRO) in the engineering operations such as CFRP in aerospace, or in constructions such as steel bridges and industrial applications (e.g. pipework repair, etc.)

Composites and reinforcements fibres — Determination of the fracture energy of bonded plates of carbon fibre reinforced plastics (CFRPs) and metal using double cantilever beam specimens

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices. It is recognized that some of the materials permitted in this document might have a negative environmental impact. As technological advances lead to more acceptable alternatives for such materials, they will be eliminated to the greatest extent possible. At the end of the test, care should be taken to dispose of all waste in an appropriate manner.

1 Scope

This document specifies the test method for the determination of adhesive fracture energy of adhesively bonded plates of carbon fibre reinforced plastic (CFRP) and metal using a double cantilever beam (DCB) specimen. The test method is also applicable to bonded joints between metals and other composite materials, such as glass fibre reinforced plastics.

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 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 10365, *Adhesives — Designation of main failure patterns*

ISO 25217, *Adhesives — Determination of the mode 1 adhesive fracture energy of structural adhesive joints using double cantilever beam and tapered double cantilever beam specimens*

3 Terms, definitions and symbols

3.1 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

3.2 Symbols

A	insert film length (mm), i.e. the distance between the end of the specimen and the tip of the insert film (see Figure 1)
a	crack length (mm), i.e. the distance between the load-line (intersection of plane through pin-hole centres and plane of crack) and the tip of the precrack or crack on the edge of the specimen (see Figure 1)
a_p	pre-crack length (mm), measured from the load-line to the tip of the precrack (see Figure 1)
a_0	insert film length (mm) between the load-line and the tip of the insert film (see Figure 1)
Δa_i	crack growth at i-th load step in the stick-slip behaviour of the crack propagation (see Figure 1)
b	width of the specimen (mm) (see Figure 1)
C	compliance δ/P of the specimen (mm/N)
C_0	initial compliance of the specimen, neglecting start-up effects, e.g. due to play in the specimen fixture (mm/N) (see Figure 2)
$C_{0+5\,\%}$	initial compliance of the specimen, C_0, raised by a factor 1,05 (mm/N) (see Figure 2)
E_f	flexural modulus of the arms of the substrate beam, calculated from the crack propagation using DCB test (GPa)
E_1	flexural modulus of the carbon fibre reinforces plastic (CFRP) beam in DCB specimen (GPa)
E_2	flexural modulus of the metal beam in DCB specimen (GPa)
$(EI)_{eq}$	equivalent stiffness (N·m^2) (see Figure 7)
F	large-displacement correction
G_C	adhesive fracture energy for the applied opening mode (J/m^2)
H	height of the load-block (mm) (see Figure 1)
h_1	thickness of the carbon fibre reinforces plastic (CFRP) beam (mm) in DCB specimen (see Figure 1)
h_2	thickness of the metal beam (mm) in DCB specimen (see Figure 1)
h_a	thickness of the adhesive layer (mm) (see Figure 1)
I_1	moment of inertia of area in CFRP (m^4)
I_2	moment of inertia of area in metal (m^4)
l	total length of the specimen (mm) (see Figure 1)
l_1	distance from the centre of the loading pin to the mid-plane of the arm of the substrate beam to which the load-block is attached (mm) (see Figure 1)
l_2	distance between the centre of the pin-hole in the load-block and the edge of the load-block, measured towards the tip of the insert (starter film) or the tip of the precrack (mm) (see Figure 1)
l_3	total length of the load-block (mm) (see Figure 1)

MAX/5 % either the maximum load on the load-displacement curve or the point of intersection of a straight line with the load-displacement curve with the slope of the straight line corresponding to $C_{0+5\,\%}$ (see Figure 2)

N load-block correction

NL onset of nonlinearity on the load-displacement curve (see Figure 2)

P load measured by the load-cell of the test machine (N)

$P(\delta)$ is the experimentally obtained load value indicating the maximum extent of the portion of load-displacement curve, from the origin O to the point A in Figure 3"

$P_{\mathrm{l}}(\delta)$ is a linearly increasing load value indicated by the dashed line a in Figure 3, and it is calculated by Formula (8):

PROP increments of the crack length during stable crack growth (propagation) that are marked on the load-displacement curve (see Figure 2)

VIS onset of visually recognizable crack growth at the edge of the specimen that is marked on the load-displacement curve (see Figure 2)

Δ crack-length correction for a beam that is not perfectly built-in (mm) (see Figure 7)

δ displacement of the cross-head of the test machine (mm)

ρ radius of curvature of the bonded plate specimen (m) (see Figure 6)

4 Principle

A double cantilever beam (DCB) specimen is used to determine the adhesive fracture energy of structural bonded joint between a metal and CFRP components

Resistance to both crack initiation and propagation is determined. The resistance to crack initiation is determined from both a non-adhesive insert placed in the adhesive layer and from a precrack. The resistance to crack propagation is determined from the precrack. The adhesive fracture energy versus applied opening load is estimated and a resistance-curve (R-curve), i.e. a plot of the value of the adhesive fracture energy versus crack length, is determined.

In the case where the CFRP/metal interlaminar toughness and interface are significantly tough, the plastic deformation of the metal beam preferentially occurs during the crack propagation. This causes the overestimation of fracture energy values. In such cases, double cantilever beam specimens with dissimilar thicknesses shall be used. An appropriate ratio of the two beams thickness (h_1/h_2) shall be determined such that the plastic deformation of the metal beams during the crack propagation is avoided.

5 Apparatus

5.1 Tensile-testing machine, capable of maintaining a crosshead displacement speed between 0,125 mm/min and 10 mm/min accurate to ±20 % and higher speeds accurate to ±10 %. The test tensile testing machine shall be equipped with a fixture to introduce the load to the pins inserted into the loading-blocks. Measurement of test system compliance is described in Annex C and Figure C.1.

The tensile testing machine shall comply with ISO 7500-1 and the force measurement system shall comply with ISO 7500-1:2018, class 1.

The opening displacement of the test specimen shall be deduced from the position of the test machine cross-head. The test machine shall be equipped with means for recording the complete load versus displacement curves (loading and unloading) during the test.

5.2 Travelling microscope or **video camera**, with suitable magnification, capable of measuring the crack length along the edge of the specimen to an accuracy of at least ±0,5 mm.

5.3 Micrometer or **vernier callipers**, capable of measuring the thickness of the substrate arms and bonded plates with an accuracy of at least ±0,05 mm.

5.4 Micrometer or **vernier callipers**, capable of measuring the width of the specimens with an accuracy of at least ±0,05 mm.

5.5 White spray-paint or **typewriter correction fluid** ("white ink").

6 Specimens

6.1 Number of specimens

A minimum of five specimens shall be tested.

6.2 Conditioning

Most adhesives absorb small quantities of water from the atmosphere which can have a significant influence on the measured properties. Following specimen preparation, the adhesive will generally be dry. If testing is carried out within a few days of specimen manufacture, then it is not necessary to condition the specimen under controlled humidity since negligible absorption of water takes place in the thin adhesive layer. However, if the specimen is tested after longer times or if the influence of absorbed water on the properties is of interest, then the humidity shall be controlled by conditioning and the properties will depend on the conditioning time (see ISO 291).

In addition, if composite substrates are used, it can be important to dry these prior to manufacture of the specimen. The properties of some adhesives are very sensitive to the presence of small amounts of moisture in a substrate prior to curing. The drying out of the substrates prior to cure will ensure that the integrity of the adhesive joint is not influenced by pre-bonding moisture effects.

6.3 Manufacture of adhesive joint specimens

6.3.1 General

The DCB specimen shall be as shown in Figure 1. The thickness of the film to be inserted in the adhesive layer during manufacture shall be less than 13 µm. The film shall be non-stick. For joint specimens bonded at temperatures below 180 °C, a thin polytetrafluoroethylene (PTFE) film is recommended. For specimens bonded at temperatures above 180 °C, a thin polyimide film is recommended. Appropriate surface treatments for metallic substrates can be found in ISO 17212[5].

The thickness of the adhesive layer shall be carefully controlled and shall be less than 1 mm in accordance with ISO 25217. The thickness of the layer shall not vary by more than 20 % within a plate, nor shall the average thickness of the layer in one joint differ by more than 20 % from that in another joint. When fully cured, remove any excess adhesive by mechanical means that do not weaken the bond, to leave the joint with smooth sides.

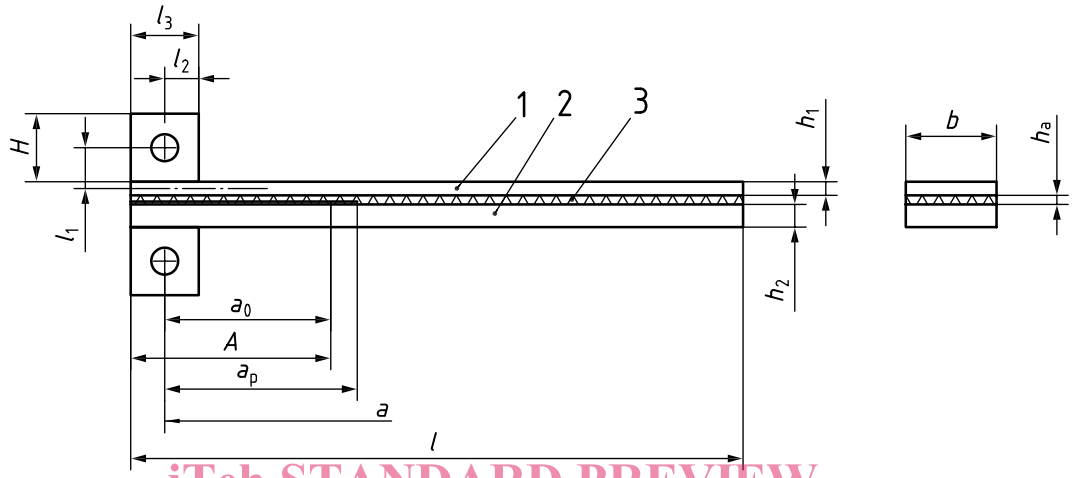
It should be recognized that the value of G measured from these tests depends upon the thickness of the adhesive layer in the joint. The value of the layer thickness shall be determined by the user, based upon the adhesive manufacturer's recommendations or upon consideration of the intended application.

It is not within the scope of this document to specify full manufacturing details of the specimens to be tested. Such information should be sought from the adhesive manufacturer and/or the substrate manufacturers.

Repeat the measurements of the total beam thickness after bonding. Determine the adhesive layer thickness, h_a, by subtracting the substrate thicknesses from the total thickness of the joint at each of the three locations.

6.3.2 DCB specimen measurements

Remove any excess adhesive from the sides of the beam. After bonding, measure the width of the DCB specimen with vernier calipers or a micrometer at three points along the length of the beam, at 30 mm from either end and at the mid-length. Measurement tolerance to be ±0,5 mm. Calculate the mean value, *b*.



Key

1 composite (CFRP) beam
2 metal beam
3 adhesive layer

Figure 1 — Geometry of DCB bonded plate specimen with load-blocks

6.4 Preparation of specimens

Apply a thin layer of white spray-paint, or typewriter correction fluid ("white ink"), on the edges of the specimen after conditioning to facilitate the detection of crack growth.

NOTE Some typewriter correction fluids and paints contain solvents which can harm the adhesive or the laminate matrix material of a composite substrate. A material with an aqueous solvent is usually safe to use.

Apply marks every 1 mm from the tip of the insert or the precrack for at least the first 10 mm, then apply marks every 5 mm. Apply marks for every 1 mm for the final 5 mm.

Measure the radius of curvature of the warped specimen following the method illustrated in 7.6.

If the specimen radius of the curvature, ρ, is smaller than 1,5 m, such specimens shall not be tested in order to avoid the effect of thermally induced residual stress. The value of ρ shall be calculated using Formula (2) and as shown in Figure 6.

For the DCB test specimen, the extent of crack propagation should be approximately 50 mm. If early breakage happens at shorter propagation, the data should be recorded with information of failure mode as defined by ISO 10365.

7 Procedure

7.1 Test set-up and data recording

The test shall be performed at one of the temperatures specified in ISO 291 or at another temperature agreed between the interested parties. After mounting the specimen in the fixture of the test machine, support the end of the specimen, if necessary, to keep the test beam orthogonal [i.e. at 90 degrees (90°)] to the direction of the applied load. Record the load and the displacement signals of the test machine electronically throughout the test, including the unloading cycle.

If using a tensile-testing machine with a paper chart recorder, the ratios of cross-head speed to chart speed are recommended to be about 1:10.

Measure the crack lengths, with the respective load and displacement along both edges of the specimen to an accuracy of at least ±0,5 mm using a travelling microscope, a scale, a vernier calliper or a video camera with suitable magnification (5.2). Calculate an average of both lengths.

7.2 Initial loading (precracking stage)

For testing from the insert (starter film), load the specimen at a constant cross-head rate of 1,0 mm/min to 5,0 mm/min.

NOTE Lower values are more accurate for crack-length measurement.

Record the point on the load-displacement curve at which the onset of crack movement from the insert is observed on the edge of the specimen, on the load-displacement curve or in the sequence of load-displacement signals [see VIS in Figure 2 a)].

Stop the loading as soon as the crack is seen to move on the edge of the specimen. Completely unload the specimen at a constant cross-head rate of up to five times the loading rate. Mark the position of the tip of the precrack on both edges of the specimen.

7.3 Re-loading: Testing from the precrack

For testing from the precrack which has been formed as a result of the test procedure in 7.2, load the specimen at a constant cross-head rate of 1,0 mm/min to 5,0 mm/min.

NOTE 1 Lower values are more accurate for crack-length measurement.

Record, on the load-displacement curve or in the sequence of load-displacement signals, the point at which the onset of crack movement from the insert is observed to occur [see VIS in Figure 2 b)].