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Composites and reinforcements fibres — Carbon fibre reinforced plastics (CFRPs) and metal assemblies — Characterization of durability of adhesive interfaces by wedge rupture test

Composites et fibres de renfort — Assemblages de plastiques renforcés de fibres de carbone (CFRP) et de métal — Caractérisation de la durabilité des interfaces adhésives à l'aide d'un essai de clivage au coin

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Foreword

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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

This document specifies a method for evaluating the durability of adhesive interfaces in the bonded assemblies of carbon fibre reinforced plastics (CFRPs) and metals by a wedge test with a double cantilever beam (DCB) specimen under specified environmental conditions. The wedge rupture test force the crack to propagate along the CFRP/adhesive or metal/adhesive interfaces, or within the adhesive layer. This test method provides a quantitative value for evaluating the effect of a harsh environmental condition on the durability of adhesive interfaces as an interfacial fracture energy. This method is intended for testing only those bonded plates used in bonding carbon fibre reinforced plastics (CFRPs) to metal assemblies.

The potential benefits to the users of CFRP-metal assemblies of implementing the durability of adhesive interfaces in the bonded plates of carbon fibre reinforced plastics (CFRPs) to metal assemblies based on this document are:

a) expanding CFRP applications to the fields of the combinations 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 of CFRP aircrafts.

It is not the intent of this document to imply the need for:

— omitting relevant field tests for CFRP related engineering;

— generally specifying the dimensions of test specimen to represent CFRPs related bonded or fastened structures;

— superimposing test results for specific applications of the parameters that exceed the range of this document.

Composites and reinforcements fibres — Carbon fibre reinforced plastics (CFRPs) and metal assemblies — Characterization of durability of adhesive interfaces by wedge rupture test

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 and determine any applicable regulatory conditions prior to use. 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 a method for determining the durability of the adhesive joints of carbon fibre reinforced plastics (CFRPs) and metal assemblies by a wedge rupture test using a double cantilever beam (DCB) specimen under specified environmental conditions. This method is intended for evaluating the safety and reliability of adhesives, primers, and surface treatments of the adherends.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes the 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 178, *Plastics — Determination of flexural properties*

ISO 10365, *Adhesives — Designation of main failure patterns*

ISO 9142, *Adhesives — Guide to the selection of standard laboratory ageing conditions for testing bonded joints*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 10365 apply.

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

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

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

3.2 Symbols

a crack length from the wedge/adherend contact point to the crack tip (mm) (see Figure 4)

a_0 initial crack length after insertion of a wedge (mm)

E_1 flexural modulus of the CFRP adherend (GPa); if the substrate is a fibre composite

E_2 flexural modulus of the metal adherend (GPa)

h_1 thickness of the carbon fibre reinforces plastic (CFRP) beam (mm) in a DCB specimen

h_2 thickness of the metal beam (mm) in a DCB specimen

G_C critical strain energy release rate, or adhesive fracture energy, for the applied opening load produced by the insertion of a wedge into the precrack (J/m^2)

Δ wedge thickness (mm)

4 Principle

The durability of the dissimilar adhesive joint interfaces between CFRP and metal is evaluated under harsh environments, such as high humidity, high temperature, high-and-low thermal cycles, salt mist environment, and so on, by the wedge test (see ISO 10354) using a DCB specimen. A wedge is driven into precrack preliminary made in the DCB specimen with an appropriate geometry, and the crack is propagated along the interfaces subjected to tensile opening load. The initial crack made by inserting the wedge is stabilized, but it will be propagated when the specimen is subject to a specified environmental condition. This experiment allows to evaluate the durability of the crack at the interface under a stressed condition quantitatively.

5 Apparatus

5.1 Wedge, with 25 mm width and 30 mm length and with a thickness of Δ, ranging from 1 mm to 3 mm, as shown in Figure 1. The composition of the wedge will not interact chemically with the adherents of the tested specimens. Materials used for the wedge shall be stiff enough not to be deformed during the test. Stainless steel is recommended. The radius of the wedge tip shall be lower than 30° to initiate the insertion into the precrack and the surface of the wedge shall be smooth to make a smooth insertion into the precrack.

Dimensions in millimetres, except indicated otherwise

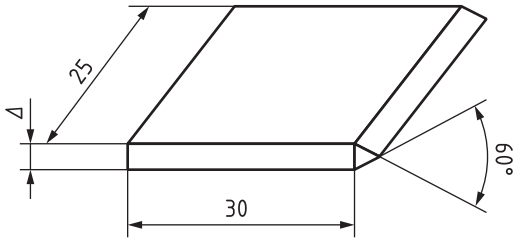


Figure 1 — Dimension of wedge

5.2 Tensile-testing machine, capable of producing a constant cross-head displacement rate between 5 and 15 mm/min in displacement control.

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

5.4 Micrometres or vernier calipers, capable of measuring the thickness of the substrates and bonded plates with an accuracy of at least 0,02 mm.

6 Specimens

6.1 Number of specimens

A minimum of five specimens shall be tested.

6.2 Conditioning

Most adhesives absorbs 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 will take 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, it is important to dry these prior to the manufacture of the specimen. The properties of some adhesives are very sensitive to the presence of small amounts of moisture on 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-bond moisture effects.

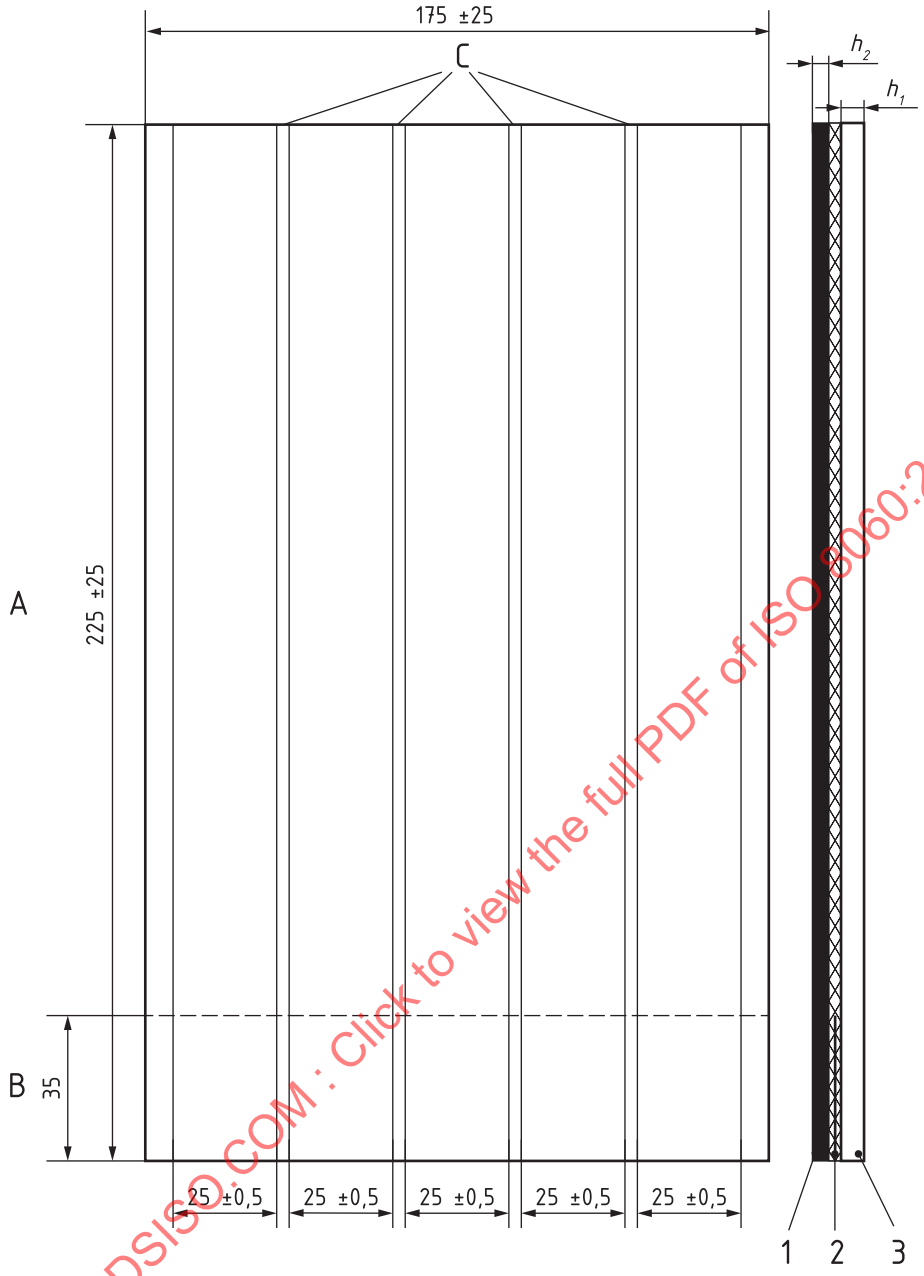
6.3 Preparation of specimens

Specimens shall be prepared by adhesive bonding of CFRP and metal panels as suggested in Figure 2, of which dimensions are 175 ± 25 mm x 200 mm - 250 mm x (h_1 + h_2). Measure the thicknesses of each panel around the corners of the panels and at around the central point to an accuracy of 0,01 mm, and calculate the mean thicknesses. The variation of the thicknesses in the panels shall be less than 0,1 mm. Insert anti-adhesive tape to prepare precrack part as shown in Figure 2. The thickness of the film to be inserted in the adhesive layer 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. Before bonding, both surfaces are degreased and carefully inspected not to be contaminated. Appropriate surface treatments for metallic substrates can be found in ISO 17212.

The two panels are bonded as specified by the manufacturer of the adhesive under test. The thickness of the adhesive layer shall be carefully controlled and shall be less than 1 mm. 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. The thickness of the adhesive shall be controlled by inserting non-stick film with uniform thickness into the edge of the bonded plate, or by adding a small amount of glass beads with a uniform diameter to the adhesive. Cut five strips of which widths are 25 ± 0,5 mm from the bonded panel as shown in Figure 2 by sawing.

Alternatively, specimens may be prepared by adhesive bonding of two plates cut with 225 ± 25 mm length and 25 mm width individually. In this method, remove any excess adhesive from the edge of the specimen by mechanical means, when fully cured, to leave the joint with smooth sides.

Dimensions in millimetres



Key

1	metal plate	A	bonded zone
2	adhesive layer	B	zone inserted by anti-adhesive tape (precrack)
3	CFRP plate	C	sawcut

Figure 2 — Suggested bonded CFRP/metal plate from which specimens are cut

The end of the specimen that contains the anti-adhesive tape is cut as shown in Figure 3 so as to introduce the wedge into the precrack easily.

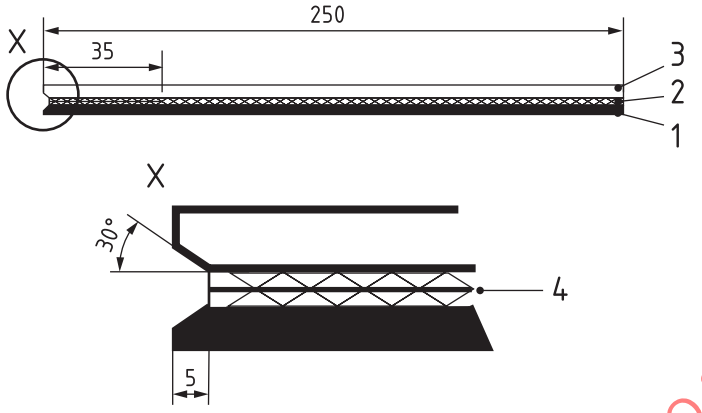
It can be recognized that the value of G_c measured from these tests will depend upon the thickness of the adhesive layer in the joint. The value of the layer thickness shall be determined by the user, based upon the adhesive manufacturer's recommendations or upon consideration of the intended application.

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

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

After bonding, measure the width of the specimen with a vernier caliper or a micrometer at three points along the length of the beam, at 30 mm from either end and at the mid-length. Calculate the mean value.

Dimensions in millimetres



Key

1	CFRP plate	3	metal plate
2	adhesive layer	4	anti-adhesive tape

Figure 3 — Test specimen for wedge test

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

Some typewriter correction fluids and paints contain solvents which can harm the adhesive or the laminate matrix material of a composite substrate. An aqueous solvent is usually preferred.

Apply marks every 1 mm from the tip of the insert for at least the first 10 mm, then apply marks every 5 mm up to the end of the specimen.

7 Testing procedure

Due to the large modulus mismatch of the CFRP/metal dissimilar joint, the crack can escape from the interface and cause the overestimation of the fracture energies with a conventional DCB specimen composed of equivalent thickness beams. The appropriate CFRP/metal thickness combination shall be thus found through the tests with the specimens having different CFRP/metal thickness ratios. An appropriate ratio of the two beams' thickness (h_1/h_2) shall be determined such that the fracture energy is the lowest.

A wedge as shown in Figure 1 is fully driven into the precrack to create an initial crack. Using a universal testing machine, the wedge is pushed slowly into the precrack at the constant speed of 10 mm/min ± 5 mm/min so that the sides and the end flush with the sides of the specimen. To avoid compression overload or buckling of the specimen, an anti-buckling fixture specified in ISO 14126 is recommended for safely inserting the wedge. After the initial crack made by inserting the wedge is stabilized, measure the crack length from the wedge/adherend contact point to the crack tip, on each side of the specimen. The measurement of the initial crack shall be performed one hour or more after no progression of the crack is recognized. Note the mean value as initial crack length a_0. Then, the specimen shall be subjected to an environmental condition as specified in ISO 9142. The crack extends along the interface as shown in Figure 4, and measure the crack length, a, in specified intervals on each side of the specimen, and note the mean values and the residence time in the specified environmental condition.

The interfacial toughness for the initial crack and after the extension of the crack under a specified environmental condition are calculated as the critical strain energy release rate, G_c, using Formula (1)[7]. G_C is given by the Formula (1) in J/m^2, where a is the crack length, Δ is wedge thickness, E is the flexural

modulus, h is the beam thickness, and C is the compensated parameters as described in Formulae (2) and (3). The subscripts 1 and 2 refer to CFRTP beam and metal beam, respectively.

The flexural modulus of the beams shall be measured in accordance with ISO 178.

$$Gc = \frac{3\Delta^2 E_1 h_1^3 E_2 h_2^3}{8a^4} \frac{E_1 h_1^3 C_2^2 + E_2 h_2^3 C_1^2}{\left(E_1 h_1^3 C_2^3 + E_2 h_2^3 C_1^3\right)^2} \quad (1)$$

$$C_1 = 1 + 0{,}64(h_1/a) \quad (2)$$

$$C_2 = 1 + 0{,}64(h_2/a) \quad (3)$$

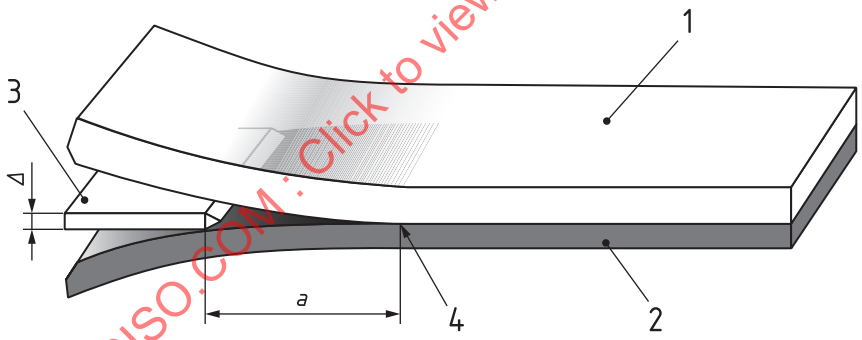
When testing the CFRP/metal combination for the first time, the appropriate CFRP/metal thickness combination shall be determined. The tests are carried out with the specimens with dissimilar thicknesses of CFRP and metal beams, h_1 and h_2, respectively. The thickness ratio (h_1/h_2) are varied ranging from 0,2 to 2 and find the best combination of the thicknesses (h_1 and h_2) to minimize the G_c values obtained after the insertion of the wedge.

NOTE Examples of the determination of the thickness ratio are shown in References [8], [9] and Annex A.

If the CFRP beam fails to break when the wedge is inserted into the precrack, the thicknesses of the beams to be tested shall be extended to 10 mm.

Alternatively, the thicknesses of the beams, h_1 and h_2, may be determined according to ISO 22838.

After the test, the test specimen is separated, and the type of failure pattern shall be assessed by visual inspection of the fracture surface on the two beams in accordance with ISO 10365. If failure behaviour not specified in ISO 10365 is observed, it shall be noted in the test report.



Key

1	metal beam	4	end of crack
2	CFRP beam	a	crack length
3	wedge	Δ	thickness of wedge

Figure 4 — Crack propagation along the adhesive interface in wedge test

8 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. ISO 8060:2024;

b) the bonding process employed;

c) the bonding conditions;

d) the materials and its condition;

e) details of any surface preparation of the adherend plates;

f) the dimensions of the test specimens;

f) environmental condition employed;

g) individual values, mean value and standard deviation of the crack lengths, a_0 and a, in mm;

h) fracture energies, G_c;

i) plot of G_c versus time or cycles of the environmental condition employed;

i) failure description (interfacial failure at metal/adhesive or CFRP/adhesive, cohesive, etc.);

j) special remarks, if any.

Annex A
(informative)

Determination of thickness ratio (h_1/h_2) for CFRTP/Al adhesive joint specimen

Adhesive joint specimens with carbon fibre reinforced thermoplastic (CFRTP) and Al5052 are prepared using a two-component urethane adhesive. The used CFRTP comprises plain woven carbon fibre continuous fabric and polamide6 matrix polymer. The bending modulus of the CFRTP and Al5052 are 37 GPa, and 65 GPa, respectively. A paste commercial adhesive urethane two-component was used for bonding CFRTP and Al, of which tensile modulus is 5,97 GPa.

CFRTP and Al panels with 250 mm length and 150 mm width were bonded with the adhesive cured at 25 °C for 1,5 h and 80 °C for 1 h. The paste adhesives were coated onto both adherents and then a small amount of 200 µm diameter glass spheres were dispersed on the adhesive to control the thickness of the adhesive layer. 80 µm thick poly(tetrafluoroethylene) (PTFE) film was placed onto one end of the substrate before the lamination to incorporate 35 mm length non-bonded region to serve precrack. The Al/CFRTP laminates were fixed with C-type clamps at the four corners and the centre part to apply equal pressure to the laminates during the curing of the adhesives. 25 mm width and 250-length rectangular specimens were cut out from the bonded laminates after curing of the adhesives, incorporating a non-bonded part at one end.

A wedge with 3 mm thick was inserted into the precrack by gently pushing the wedge into the precrack at the constant speed of 10 mm/min by using a testing machine as shown in Figure A.1. The wedge was fully driven into the precrack.

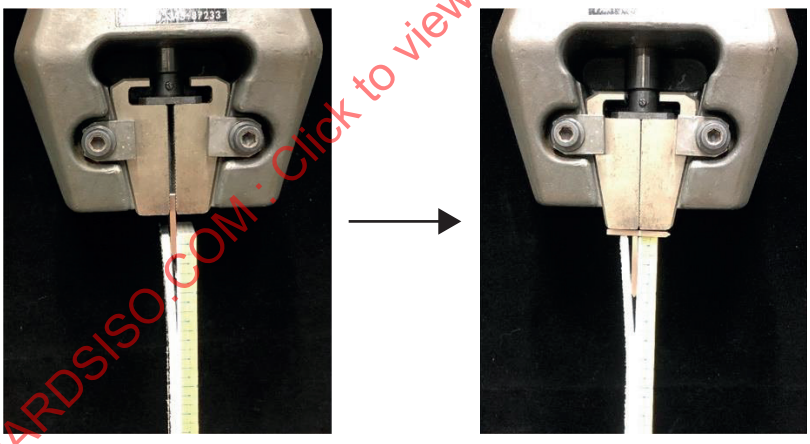


Figure A.1 — Insertion of a wedge into precrack of the specimen using a testing machine

After the initial crack made by the insertion of the wedge was stabilized, the specimens were subject to moisture in a constant climate cabinet kept at 85 °C and 85 % relative humidity (RH) and the crack extension was measured. Figure A.2 shows the *Gc* values versus the CFRTP/Al thickness ratio after the residence times indicated therein. It indicates that the 2/5 thickness ratio gives the minimum *Gc* values.