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JAPANESE INDUSTRIAL STANDARD

**Testing methods for interlaminar
fracture toughness of carbon
fibre reinforced plastics**

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In the event of any doubt arising,
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Testing methods for
interlaminar fracture toughness of
carbon fibre reinforced plastics

K 7086-1993

1. Scope This Japanese Industrial Standard specifies testing methods for the interlaminar fracture toughness of carbon fibre reinforced plastics (hereafter referred to as "CFRP").

- Remarks
1. These testing methods cover the determination of the interlaminar fracture toughness of laminated CFRP's reinforced by unidirectional or woven materials.
 2. The deformation mode I as the open mode and II as the edge-sliding mode, respectively, are the subjects of this Standard.
 3. The standards cited in this Standard are as follows:
 - JIS B 0601 Surface roughness — Definitions and designation
 - JIS B 0621 Definitions and designations of geometrical deviations
 - JIS B 7502 Micrometer callipers for external measurement
 - JIS B 7507 Vernier, dial and digital callipers
 - JIS K 6900 Glossary of terms used in plastic industry
 - JIS K 7072 Preparation of carbon fibre reinforced plastic panels for test purpose
 - JIS K 7074 Testing methods for flexural properties of carbon fiber reinforced plastics
 - JIS K 7100 Standard atmospheres for conditioning and testing of plastics
 - JIS Z 8401 Rules for rounding off of numerical values

2. Definitions For the main terms in this Standard the definitions in JIS K 6900 apply, and the rest of the terms shall be as follows:

- (1) interlaminar fracture toughness The critical value of the energy required to create a unit area of a interlaminar delamination crack.
- (2) crack opening displacement (δ) Relative movement between the upper and lower crack surfaces, and hereafter referred to as "COD".
- (3) deformation mode The type of the relative movement between crack surfaces consisting of the three basic modes. In mode I, the opening mode, the relative movement is perpendicular to the crack surfaces. Mode II, the edge sliding mode, refers to the deformation mode where the movement is parallel to the crack surfaces and perpendicular to the crack tip line. In mode III, the tear mode, the movement is parallel to the crack surfaces and the crack tip line.

- (4) G_{IC} , G_{IIC} Initial stage interlaminar fracture toughness for the mode I and mode II deformation, respectively, showing the fracture toughness at the initial stage of fracture.
- (5) G_{IR} , G_{IIR} Propagation of interlaminar fracture toughness for the mode I and mode II deformation, respectively, showing the fracture toughness during fracture propagation.
- (6) load line displacement (u) Deformation of the test specimen measured on the load line. Cross-head displacement is employed usually.
- (7) load point compliance (C) Ratio of load line displacement to the load (u/P).
- (8) COD compliance (λ) Ratio of COD to the load (δ/P).
- (9) initial straight line Line drawn on the initial linear part of record, excluding the non-linear portion of the record just after the start of the loading. This line is obtained for both of the load to the load displacement record and load to COD record.
- (10) crack length (a) The crack length of the double cantilever beam style test specimen is the distance between the load line and the crack tip. In the case of the pin loading type, the line connecting the centers of the pins is the load line. The crack length of the end notched flexure type test specimen is the distance between supporting point in the crack side and the crack tip.
- (11) height of beam (H) Distance between the crack surface and the test specimen surfaces. This is obtained as the half of the thickness of the test specimen.
- (12) corrected compliance method Method to calculate the crack length from relationship between the COD compliance and crack length.
- (13) R-diagram Diagram to show changes in the interlaminar fracture toughness during the crack extension. Horizontal axis of the diagram is the crack length or the amount of crack length increment and the vertical axis is the interlaminar fracture toughness.

3. Type of testing methods Two types of testing methods are specified in this Standard in accordance with the deformation mode.

- (1) Double cantilever beam test [DCB (¹) test] Test to obtain the mode I interlaminar fracture toughness values, G_{IC} and G_{IR} . Double cantilever beam test specimens (DCB test specimens) are employed.
- (2) End notched flexure test [ENF (²) test] Test to obtain the mode II interlaminar fracture toughness values G_{IIC} and G_{IIR} . End notched flexure test specimens (ENF test specimens) are employed.

Notes (¹) DCB is short for Double Cantilever Beam.

(²) ENF is short for End Notched Flexure.

4. Conditioning of test specimens and test temperature and humidity

4.1 Conditioning of test specimens The test specimens shall be conditioned at a standard temperature Grade 2 and a standard humidity Grade 2 [temperature $23 \pm 2^\circ\text{C}$ and relative humidity $(50 \pm 5) \%$] for 48 h or longer before test in accordance with JIS K 7100, as a rule.

4.2 Test temperature and humidity The test shall be conducted in a room at the temperature and humidity specified in 4.1 [temperature $23 \pm 2^\circ\text{C}$ and relative humidity $(50 \pm 5) \%$], as a rule.

5. Testing machine and apparatus

5.1 Testing machine The testing machine shall operate at a constant rate of cross-head or COD movement and comprise the following devices.

5.1.1 Load indicator The load indicator shall be capable of recording the total load applied to the test specimen at any time during the test and shall have a mechanism capable of indicating the load with an accuracy within $\pm 1 \%$ or better at a specified rate of the test.

5.1.2 Extensometer The extensometer shall be capable of recording the total value of the load line displacement applied to the test specimen and shall have a mechanism capable of indicating the load line displacement with an accuracy within $\pm 1 \%$ or better at the specified rate of the test.

5.2 Measuring instruments for dimensions

5.2.1 Micrometer A micrometer to be used for measuring the thickness of the test specimens, having a measuring range of 0 mm to 25 mm specified in JIS B 7502, or equivalent or better in accuracy.

5.2.2 Vernier calliper A vernier calliper to be used for measuring the length, the width and the gauge length of the test specimens, having the maximum measuring length of 300 mm and the minimum read-out value of 0.05 mm specified in JIS B 7507, or equivalent or better in accuracy.

5.2.3 Reading out microscope Reading out microscope to be used for measuring the crack length during the test shall have a measuring range of 0 mm to 200 mm and the minimum read-out value of 0.05 mm, or equivalent or better in accuracy. Microscope magnification of 20 to 70 may be suitable.

6. Test specimens

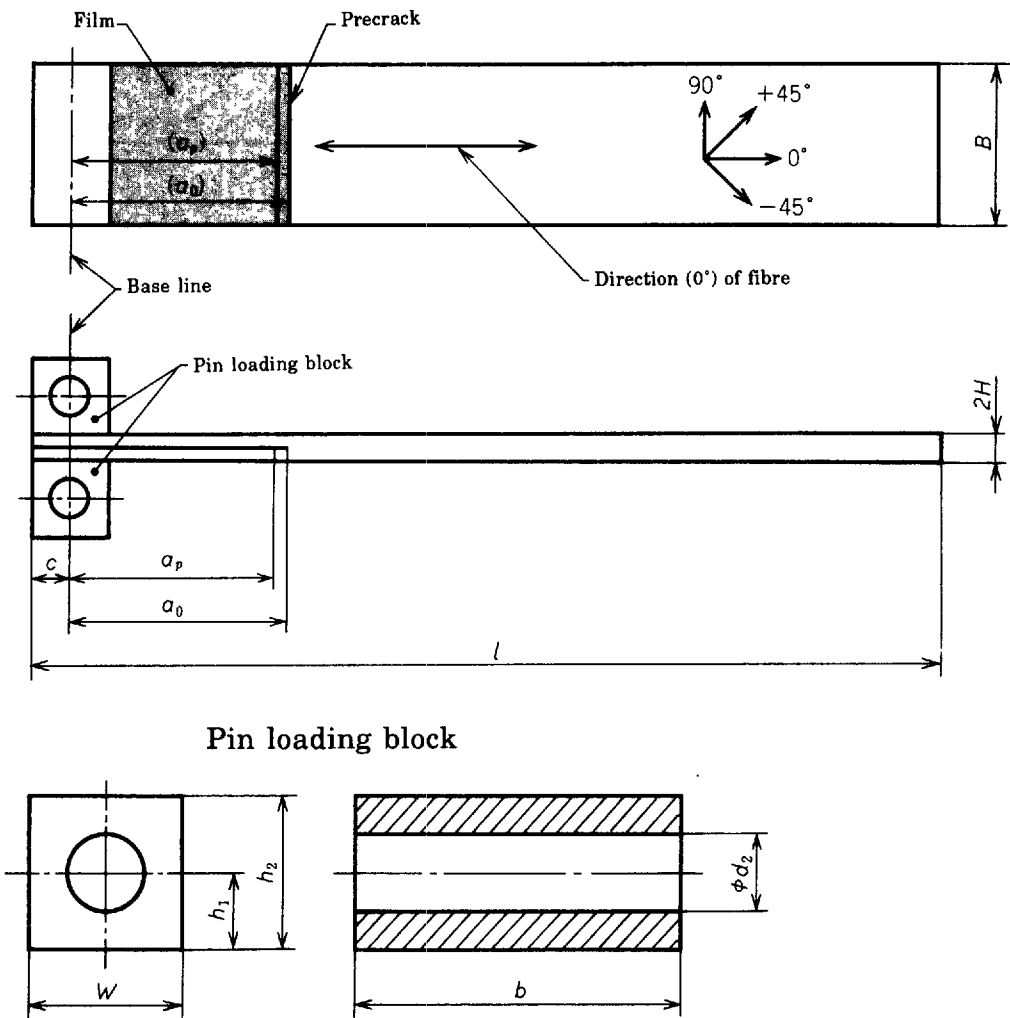
6.1 Type, shape and dimensions of test specimens

6.1.1 DCB test specimen The DCB test specimens shall be used for DCB test in order to measure the mode I interlaminar fracture toughness. The shape and the dimensions of the test specimen are shown in Fig. 1.

6.1.2 ENF test specimen The ENF test specimens shall be used for ENF test in order to measure the mode II interlaminar fracture toughness. The shape and the dimensions of the test specimen are shown in Fig. 2.

Fig. 1. Shape and dimensions of DCB test specimen

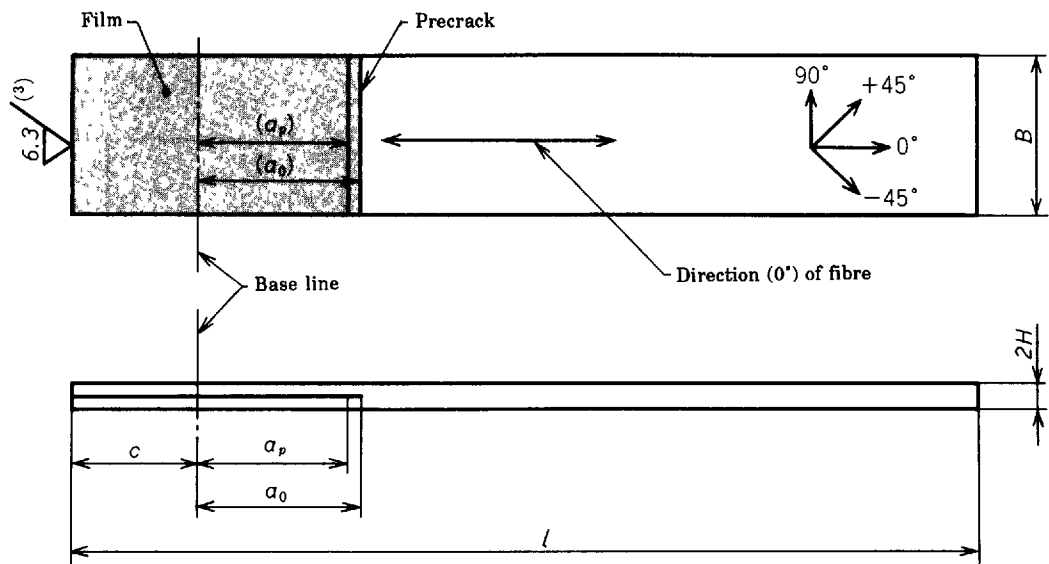
Unit: mm



unit: mm

Item	Mark	Section of test specimen to be measured	Dimension
DCB test specimen	l	Length	$c + a_p + 100$ or over
	$2H$	Thickness	3 ± 0.4
	B	Width	20 to 25
	c	Distance from test specimen edge to load line	6
	a_p	Film crack length	34 to 54
	a_0	Initial crack length	$a_p + (2 \text{ to } 5)$
Pin loading block	W	Width	12
	b	Length	$B \pm 0.2$
	h_1	Half of height	6
	h_2	Height	12
	d_2	Diameter of hole	$5^{+0.1}_{-0.05}$

Fig. 2. Shape and dimensions of ENF test specimen



Note ⁽³⁾ In case COD gauge in Annex 3 Fig. 1 is used.

Unit: mm

Mark	Section of test specimen to be measured	Dimension
l	Length	140 ± 0.5
$2H$	Thickness	3 ± 0.4
B	Width	20 to 25
c	Distance from test specimen edge to load line	20 to 25
a_p	Film crack length	20 to 25
a_0	Initial crack length	22 to 28

Informative reference:

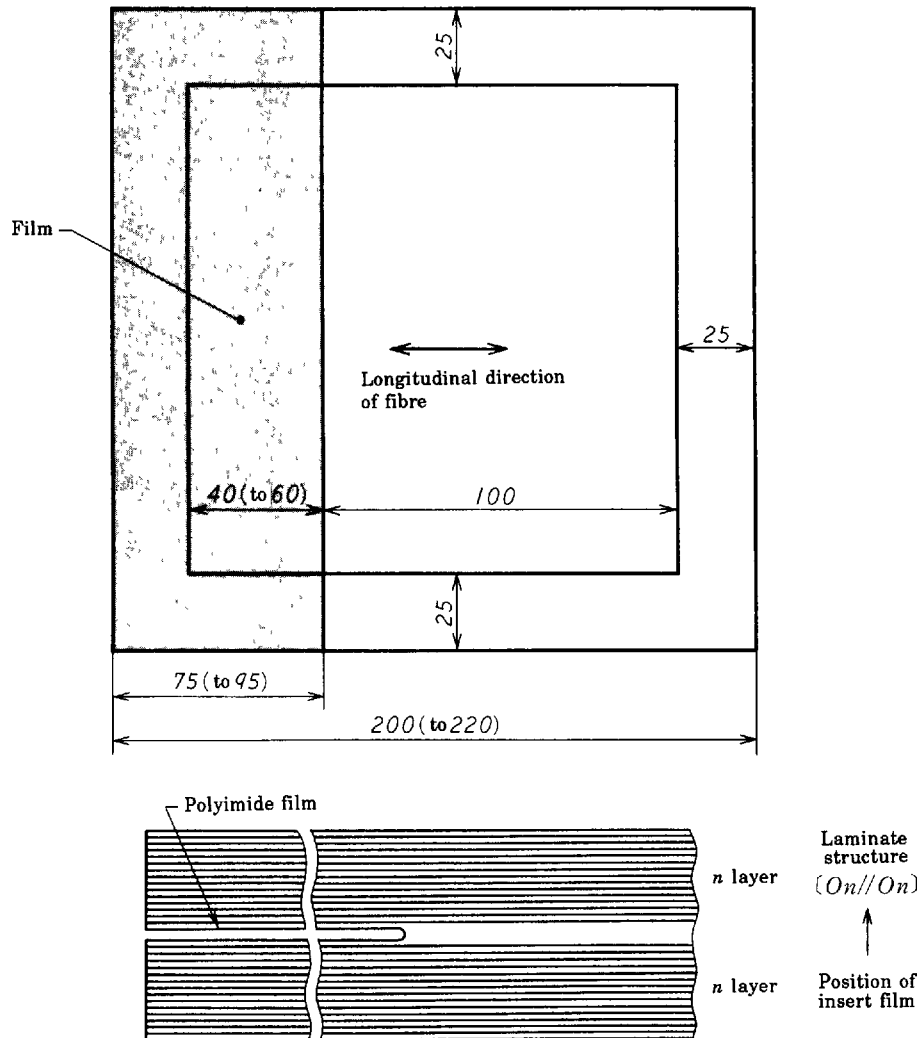
In case of the CFRP which presents a remarkable increase in the interlaminar fracture toughness as the crack propagates, the initial stage of fracture toughness may be evaluated higher than the actual value when a precrack was introduced. In such a case the film defect made of a sheet of the polyimide film or an equivalent material with a thickness of 10 μm (micron meter) or less and inserted in accordance with the procedure described in 6.2 (1) may be used as the starter crack and the test may be conducted without precracking. In order to ensure that the film is not adhered to the crack surfaces, the crack may be opened carefully enough not to extend the crack.

6.2 Preparation of test specimens The test specimens shall be prepared as follows:

- (1) A folded film or a sheet of polyimide or an equivalent material with its surface and edges coated by a release agent shall be placed at the laminate mid-thickness with the film edge perpendicular ($90 \pm 1^\circ$) to the fibre alignment. The total thickness of the film inserted shall not exceed $30 \mu\text{m}$ (micron meter). Here the fibre alignment shall be direction of fibres in the case of the unidirectional reinforced laminates and the warp (or weft) direction in the case of the laminates made of woven fabrics. The number of layers shall be an even number and the misalignment of each layer shall be within $\pm 1^\circ$. Moulding method shall be carried out in accordance with JIS K 7072. An example of the laminate structure, the dimensions and the position of the inserted film of the test specimen are shown in Fig. 3.
- (2) The test panel shall be obtained after trimming the four edges of the laminate.
- (3) The test specimen shall be machined out of the test panel by means of diamond disc cutter, a milling machine or other machining method at least equivalent to those in terms of the accuracy. The test specimens shall be machined so that longitudinal direction agrees to the fibre alignment of the test plate.
- (4) Care shall be taken to ensure that the specimens are not overheated, with preventing by water cooling or other proper method.
- (5) As test result may be affected by angulation of fibre alignment to the cutting direction of the test specimens, ward off such angulation by using a suitable jig and the like in cutting off the test specimens. The longitudinal axis of the test specimen shall be parallel within 0.5 mm in the fibre alignment of the test specimen.
- (6) On the DCB test specimen two blocks for pin loading as shown in Fig. 1 shall be adhered.
- (7) A starter crack shall be made by introducing a 2 mm to 5 mm long precrack at the tip of the inserted film by means of opening the crack with a sharp wedge like a knife. During this procedure the test specimen shall be compressed by a vise or other method to avoid excess amount of propagation of the precrack. In the case of a folded film was used for insert, the film shall be fractured at the fold by the opening procedure.
- (8) In the case of the DCB test specimen, white brittle paint shall be coated on the both sides of the test specimen for the purpose of observation of the crack propagation. For the ENF test specimens, white brittle paint shall be coated at the position as shown in Fig. 2 and the base line shall be drawn by scratching the paint.
- (9) When a COD gauge described in Annex 3 is to be used in the ENF test, the edge surface of the test specimen shall be finished to $50 \mu\text{m}$ of perpendicularity in accordance with JIS B 0621 and to the center line average roughness of $6.3 \mu\text{m}$ in accordance with JIS B 0601.

Fig. 3. An example of moulding of test panel

Unit: mm



6.3 Number of test specimens The number of the test specimens shall be as follows:

- (1) The number of the test specimens shall be five or more.
- (2) The data obtained from abnormal test results shall be discarded, the same number of the tests shall be conducted with new ones.

6.4 Measurement of dimensions of test specimen The thickness of the test specimen shall be measured to the nearest 0.01 mm at three points in the area excluding the zone of the inserted film. The length and the width of the test specimen shall be measured to the nearest 0.05 mm. The difference between the maximum and the minimum values of the three data for thickness shall not exceed 0.2 mm. The thickness of the test specimen is the average value of the three measured thickness values. The difference between the maximum and the minimum values of the measured thickness of the test specimens to be tested shall not exceed 0.4 mm.

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7. Procedure and calculation

7.1 DCB test Follow Annex 1 for the procedure and the calculation of the DCB test.

7.2 ENF test For the procedure and the calculation for convenient measurement of the initial stage interlaminar fracture toughness in mode II deformation, Annex 2 shall be followed. For the procedure and calculation for precise measurement of the interlaminar fracture toughness values for the initiation point of the fracture and the propagation stage, Annex 3 shall be followed.

8. Calculation of average value, standard deviation and coefficient of variation

8.1 Test results Test data shall be obtained from each test result. Calculate average value of five or more test results and round them off to three significant figures in accordance with JIS Z 8401.

8.2 Standard deviation and coefficient of variation When standard deviation and coefficient of variation are required, calculate them from the following equations (1) and (2), and then round them off to two significant figures in accordance with JIS Z 8401.

$$s = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}} \dots\dots\dots (1)$$

$$CV = \frac{s}{\bar{x}} \times 100 \dots\dots\dots (2)$$

where, s : standard deviation
 CV : coefficient of variation (%)
 x : individual measured value
 $\bar{x}$: arithmetic mean of measured values
 n : number of measurements

9. Report The report shall include the following items as required:

- (1) The types and grade of materials tested and manufacturer's name
- (2) Deformation mode of the test, the type of the test specimen, the laminate structure, the carbon fibre content by volume or mass
- (3) Manufacturing and machining method of the test specimen, and the geometry, shape and dimensions of the specimen
- (4) The material and thickness of the inserted film for the starter defect of the test specimen
- (5) Whether precracking was introduced or not to the test specimen and the length of the introduced precracking

- (6) Number of test specimens tested
- (7) Temperature, humidity and time for conditioning of the test specimens
- (8) Test temperature and humidity
- (9) Method of the COD measurement
- (10) Control method of the test
- (11) Test speed
- (12) Test results (Initial crack length. Each value, average values, standard deviations and coefficient of variation of the initial stage interlaminar fracture toughness and the propagation interlaminar fracture toughness. Relationship between the crack length and the COD compliance. Typical load-COD diagram. R-diagrams. Fracture surface appearance and other information if any.)
- (13) Date of testing
- (14) Other necessary items

Annex 1 DCB test

1. Scope This Annex 1 specifies the procedure and the calculation method for obtaining the mode I initial stage interlaminar fracture toughness G_{IC} and the mode I propagating interlaminar fracture toughness G_{IR} by means of the DCB test specimen.

Informative reference:

In this Standard the critical load to calculate the initial stage interlaminar fracture toughness is defined as that observed first in the maximum load P_{max} and the load P_5 at which the compliance in the load to COD diagram increased by 5 % from that for initial straight line. In accordance with this definition, it is possible that the initiation of the crack has taken a place at the critical load and hence G_{IC} is defined as the interlaminar fracture toughness at the initial stage in the fracture propagation rather than that at the initiation of the fracture. It is also considered that the load P_{NL} , at the start point of non-linearity, i.e. the point where the relationship of the load and the COD starts to deviate from the initial straight line, may be used for the calculation of the fracture toughness G_{IC} instead of P_5 . This method reduces to a fracture toughness lower than the value defined by P_5 and results in a safer evaluation. However, the fracture toughness based on P_{NL} shows a considerably big scatter, P_{NL} can be different depending on the person who measures the diagram. Accordingly, the load observed first in P_{max} and P_5 is defined as the critical one in this Standard.

2. Testing machine and apparatus The testing machine and apparatus shall conform to the following requirements as well as those shown in main text of this Standard.

- (1) Testing jig for DCB test The testing jig for DCB test shall be used to apply the mode I load to DCB test specimen by means of a pair of pins during the test and shall consist of universal joint. An example of shape of the testing jig for DCB test is illustrated in Annex 1 Fig. 1.
- (2) Crack opening displacement gauge The crack opening displacement gauge (hereafter referred to as "COD gauge") measures continuously all of the COD applied to the specimen during the test and shall have a mechanism with an error of ± 1 % or better at the test speed specified.