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Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites¹

This standard is issued under the fixed designation D 5528; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Added research report reference to Section 14 editorially in March 2008.

^{ε2} NOTE—Corrected Eq. 3 in July 2008.

Corrected Eq. 3 in July 2008.

^{ε3} NOTE—Eq. 3 was rewritten for clarification in August 2009.

1. Scope

1.1 This test method describes the determination of the opening Mode I interlaminar fracture toughness, G_{IC} , of continuous fiber-reinforced composite materials using the double cantilever beam (DCB) specimen (Fig. 1).

1.2 This test method is limited to use with composites consisting of unidirectional carbon fiber and glass fiber tape laminates with brittle and tough single-phase polymer matrices. This limited scope reflects the experience gained in round-robin testing. This test method may prove useful for other types and classes of composite materials; however, certain interferences have been noted (see 6.5).

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 This standard may involve hazardous materials, operations, and equipment.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D 883 Terminology Relating to Plastics

D 2651 Guide for Preparation of Metal Surfaces for Adhesive Bonding

D 2734 Test Methods for Void Content of Reinforced Plastics

D 3171 Test Methods for Constituent Content of Composite Materials

D 3878 Terminology for Composite Materials

D 5229/D 5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials

E 4 Practices for Force Verification of Testing Machines

E 6 Terminology Relating to Methods of Mechanical Testing

E 122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E 456 Terminology Relating to Quality and Statistics

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 Terminology D 3878 defines terms relating to high-modulus fibers and their composites. Terminology D 883 defines terms relating to plastics. Terminology E 6 defines terms relating to mechanical testing. Terminology E 456 and Practice E 177 define terms relating to statistics. In the event of conflict between terms, Terminology D 3878 shall have precedence over the other terminology standards.

¹ This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.06 on Interlaminar Properties.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

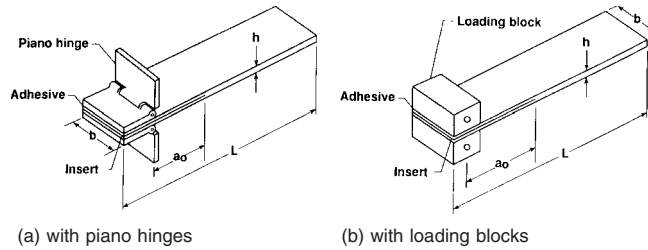


FIG. 1 Double Cantilever Beam Specimen

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *crack opening mode (Mode I)*—fracture mode in which the delamination faces open away from each other.

3.2.2 *Mode I interlaminar fracture toughness, G_{Ic}* —the critical value of G for delamination growth as a result of an opening load or displacement.

3.2.3 *energy release rate, G* —the loss of energy, dU , in the test specimen per unit of specimen width for an infinitesimal increase in delamination length, da , for a delamination growing under a constant displacement. In mathematical form,

$$G = -\frac{1}{b} \frac{dU}{da} \quad (1)$$

where:

U = total elastic energy in the test specimen,

b = specimen width, and

a = delamination length.

3.3 Symbols:

3.3.1 A_1 —slope of plot of a/b versus $C^{1/3}$.

3.3.2 a —delamination length.

3.3.3 a_0 —initial delamination length.

3.3.4 b —width of DCB specimen.

3.3.5 C —compliance, δ/P , of DCB specimen.

3.3.6 CV —coefficient of variation, %.

3.3.7 da —differential increase in delamination length.

3.3.8 dU —differential increase in strain energy.

3.3.9 E_{11} —modulus of elasticity in the fiber direction.

3.3.10 E_{1f} —modulus of elasticity in the fiber direction measured in flexure.

3.3.11 F —large displacement correction factor.

3.3.12 G —strain energy release rate.

3.3.13 G_{Ic} —opening Mode I interlaminar fracture toughness.

3.3.14 h —thickness of DCB specimen.

3.3.15 L —length of DCB specimen.

3.3.16 L' —half width of loading block.

3.3.17 m —number of plies in DCB specimen.

3.3.18 N —loading block correction factor.

3.3.19 NL —point at which the load versus opening displacement curve becomes nonlinear.

3.3.20 n —slope of plot of Log C versus Log a .

3.3.21 P —applied load.

3.3.22 P_{max} —maximum applied load during DCB test.

3.3.23 SD —standard deviation.

3.3.24 t —distance from loading block pin to center line of top specimen arm.

3.3.25 U —strain energy.

3.3.26 VIS —point at which delamination is observed visually on specimen edge.

3.3.27 V_f —fiber volume fraction, %.

3.3.28 δ —load point deflection.

3.3.29 Δ —effective delamination extension to correct for rotation of DCB arms at delamination front.

3.3.30 Δ_x —incremental change in Log a .

3.3.31 Δ_y —incremental change in Log C .

4. Summary of Test Method

4.1 The DCB shown in Fig. 1 consists of a rectangular, uniform thickness, unidirectional laminated composite specimen containing a nonadhesive insert on the midplane that serves as a delamination initiator. Opening forces are applied to the DCB

specimen by means of hinges (Fig. 1a) or loading blocks (Fig. 1b) bonded to one end of the specimen. The ends of the DCB are opened by controlling either the opening displacement or the crosshead movement, while the load and delamination length are recorded.

4.2 A record of the applied load versus opening displacement is recorded on an X-Y recorder, or equivalent real-time plotting device or stored digitally and postprocessed. Instantaneous delamination front locations are marked on the chart at intervals of delamination growth. The Mode I interlaminar fracture toughness is calculated using a modified beam theory or compliance calibration method.

5. Significance and Use

5.1 Susceptibility to delamination is one of the major weaknesses of many advanced laminated composite structures. Knowledge of a laminated composite material's resistance to interlaminar fracture is useful for product development and material selection. Furthermore, a measurement of the Mode I interlaminar fracture toughness, independent of specimen geometry or method of load introduction, is useful for establishing design allowables used in damage tolerance analyses of composite structures made from these materials.

5.2 This test method can serve the following purposes:

5.2.1 To establish quantitatively the effect of fiber surface treatment, local variations in fiber volume fraction, and processing and environmental variables on G_{Ic} of a particular composite material.

5.2.2 To compare quantitatively the relative values of G_{Ic} for composite materials with different constituents.

5.2.3 To develop delamination failure criteria for composite damage tolerance and durability analyses.

6. Interferences

6.1 Linear elastic behavior is assumed in the calculation of G used in this test method. This assumption is valid when the zone of damage or nonlinear deformation at the delamination front, or both, is small relative to the smallest specimen dimension, which is typically the specimen thickness for the DCB test.

6.2 In the DCB test, as the delamination grows from the insert, a resistance-type fracture behavior typically develops where the calculated G_{Ic} first increases monotonically, and then stabilizes with further delamination growth. In this test method, a resistance curve (R curve) depicting G_{Ic} as a function of delamination length will be generated to characterize the initiation and propagation of a delamination in a unidirectional specimen (Fig. 2). The principal reason for the observed resistance to delamination is the development of fiber bridging (1-3).³ This fiber bridging mechanism results from growing the delamination between two 0° unidirectional plies. Because most delaminations that form in multiply laminated composite structures occur between plies of dissimilar orientation, fiber bridging does not occur. Hence, fiber bridging is considered to be an artifact of the DCB test on unidirectional materials. Therefore, the generic significance of G_{Ic} propagation values calculated beyond the end of the implanted insert is questionable, and an initiation value of G_{Ic} measured from the implanted insert is preferred. Because of the significance of the initiation point, the insert must be properly implanted and inspected (8.2).

6.3 Three definitions for an initiation value of G_{Ic} have been evaluated during round-robin testing (4). These include G_{Ic} values determined using the load and deflection measured (1) at the point of deviation from linearity in the load-displacement curve (NL), (2) at the point at which delamination is visually observed on the edge (VIS) measured with a microscope as specified in 7.5, and (3) at the point at which the compliance has increased by 5 % or the load has reached a maximum value (5 %/max) (see Section 11). The NL G_{Ic} value, which is typically the lowest of the three G_{Ic} initiation values, is recommended for generating delamination failure criteria in durability and damage tolerance analyses of laminated composite structures (5.2.3). Recommendations for

³ The boldface numbers in parentheses refer to the list of references at the end of this test method.

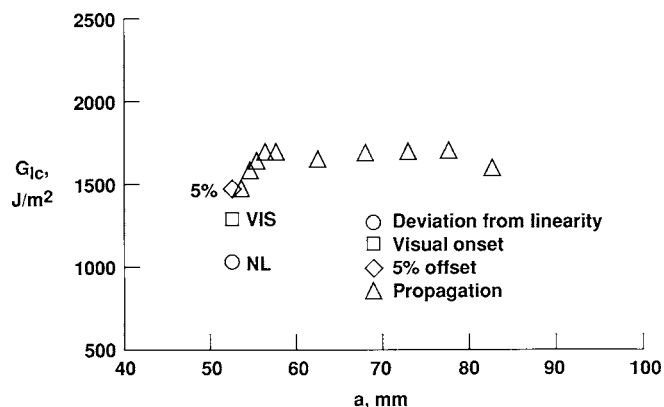


FIG. 2 Delamination Resistance Curve (R Curve) from DCB Test

obtaining the NL point are given in Annex A2. All three initiation values can be used for the other purposes cited in the scope (5.2.1 and 5.2.2). However, physical evidence indicates that the initiation value corresponding to the onset of nonlinearity (NL) in the load versus opening displacement plot corresponds to the physical onset of delamination from the insert in the interior of the specimen width (5). In round-robin testing of AS4/PEEK thermoplastic matrix composites, NL G_{Ic} values were 20 % lower than VIS and 5 %/max values (4).

6.4 Delamination growth may proceed in one of two ways: (1) by a slow stable extension or (2) a run-arrest extension in which the delamination front jumps ahead abruptly. Only the first type of growth is of interest in this test method. An unstable jump from the insert may be an indication of a problem with the insert. For example, the insert may not be completely disbanded from the laminate, or may be too thick, resulting in a large neat resin pocket, or may contain a tear or fold. Furthermore, rapid delamination growth may introduce dynamic effects in both the test specimen and in the fracture morphology. Treatment and interpretation of these effects is beyond the scope of this test method. However, because crack jumping has been observed in at least one material in which the guidelines for inserts (see 8.2) were not violated, the specimens are unloaded after the first increment of delamination growth and reloaded to continue the test. This procedure induces a natural Mode I precrack in the DCB specimen. The first propagation G_{Ic} value is referred to as the Mode I precrack G_{Ic} .

6.5 Application to Other Materials, Layups, and Architectures:

6.5.1 Toughness values measured on unidirectional composites with multiple-phase matrices may vary depending upon the tendency for the delamination to wander between various matrix phases. Brittle matrix composites with tough adhesive interleaves between plies may be particularly sensitive to this phenomenon resulting in two apparent interlaminar fracture toughness values: one associated with a cohesive-type failure within the interleaf and one associated with an adhesive-type failure between the tough polymer film and the more brittle composite matrix.

6.5.2 Nonunidirectional DCB configurations may experience branching of the delamination away from the midplane through matrix cracks in off-axis plies. If the delamination branches away from the midplane, a pure Mode I fracture may not be achieved as a result of the structural coupling that may exist in the asymmetric sublaminates formed as the delamination grows. In addition, nonunidirectional specimens may experience significant anticlastic bending effects that result in nonuniform delamination growth along the specimen width, particularly affecting the observed initiation values.

6.5.3 Woven composites may yield significantly greater scatter and unique R curves associated with varying toughness within and away from interlaminar resin pockets as the delamination grows. Composites with significant strength or toughness through the laminate thickness, such as composites with metal matrices or 3D fiber reinforcement, may experience failures of the beam arms rather than the intended interlaminar failures.

7. Apparatus

7.1 *Testing Machine*—A properly calibrated test machine shall be used that can be operated in a displacement control mode with a constant displacement rate in the range from 0.5 to 5.0 mm/min (0.02 to 0.20 in./min). The testing machine shall conform to the requirements of Practices E 4. The testing machine shall be equipped with grips to hold the loading hinges, or pins to hold the loading blocks, that are bonded to the specimen.

7.2 *Load Indicator*—The testing machine load-sensing device shall be capable of indicating the total load carried by the test specimen. This device shall be essentially free from inertia lag at the specified rate of testing and shall indicate the load with an accuracy over the load range(s) of interest of within ± 1 % of the indicated value.

7.3 *Opening Displacement Indicator*—The opening displacement may be estimated as the crosshead separation, provided the deformation of the testing machine, with the specimen grips attached, is less than 2 % of the opening displacement of the test specimen. If not, then the opening displacement shall be obtained from a properly calibrated external gage or transducer attached to the specimen. The displacement indicator shall indicate the crack opening displacement with an accuracy of within ± 1 % of the indicated value once the delamination occurs.

7.4 *Load Versus Opening Displacement Record*—An X - Y plotter, or similar device, shall be used to make a permanent record during the test of load versus opening displacement at the point of load application. Alternatively, the data may be stored digitally and post-processed.

7.5 *Optical Microscope*—A travelling optical microscope with a magnification no greater than 70 \times , or an equivalent magnifying device, shall be positioned on one side of the specimen to observe the delamination front as it extends along one edge during the test. This device shall be capable of pinpointing the delamination front with an accuracy of at least ± 0.5 mm (± 0.02 in.). A mirror may be used to determine visually any discrepancy in delamination onset from one side of the specimen to the other. Other methods, such as crack length gages bonded to a specimen edge, may be used to monitor delamination length, provided their accuracy is as good as the optical microscope so that delamination length may be measured to the accuracy specified above.

7.6 The micrometer(s) shall use a suitable size diameter ball interface on irregular surfaces such as the bag side of a laminate and a flat anvil interface on machined edges or very smooth tooled surfaces. The accuracy of the instruments shall be suitable for reading to within 1 % of the sample width and thickness. For typical specimen geometries, an instrument with an accuracy of ± 2.5 μ m (0.0001 in.) is desirable for thickness measurement, while an instrument with an accuracy of ± 25 μ m (0.001 in.) is desirable for width measurement.

8. Sampling and Test Specimens

8.1 Test laminates must contain an even number of plies, and shall be unidirectional, with delamination growth occurring in the 0° direction.

8.2 A nonadhesive insert shall be inserted at the midplane of the laminate during layup to form an initiation site for the delamination (see Fig. 1). The film thickness shall be no greater than 13 μm (0.0005 in.). Specimens should not be precracked before testing. By not precracking, an initiation value free of fiber bridging may be obtained and included in the *R* curve. A polymer film is recommended for the insert to avoid problems with folding or crimping at the cut end of the insert, as was observed for aluminum foil inserts during round-robin testing (4). For epoxy matrix composites cured at relatively low temperatures, 177°C (350°F) or less, a thin film made of polytetrafluoroethylene (PTFE) is recommended. For composites with polyimide, bismaleimide, or thermoplastic matrices that are manufactured at relatively high temperatures, greater than 177°C (350°F), a thin polyimide film is recommended. For materials outside the scope of this test method, different film materials may be required. If a polyimide film is used, the film shall be painted or sprayed with a mold release agent before it is inserted in the laminate. (**Warning**—Mold release agents containing silicone may contaminate the laminate by migration through the individual layers. It is often helpful to coat the film at least once and then bake the film before placing the film on the composite. This will help to prevent silicone migration within the composite. Although precracking is not recommended, under certain prescribed circumstances (see 11.7.7) an alternate wedge precracking procedure may be used. Guidelines for generating a wedge precrack are given in Annex A3.)

8.3 Specimen Dimensions:

8.3.1 Specimens shall be at least 125 mm (5.0 in.) long and nominally from 20 to 25 mm (0.8 to 1.0 in.) wide, inclusive.

NOTE 1—Round-robin testing on narrow and wide specimens yielded similar results, indicating that the DCB specimen width is not a critical parameter.

8.3.2 Panels shall be manufactured, and specimens cut from the panels, such that the insert length is approximately 63 mm (2.5 in.) (see Fig. 1). This distance corresponds to an initial delamination length of approximately 50 mm (2.0 in.) plus the extra length required to bond the hinges or load blocks. The end of the insert should be accurately located and marked on the panel before cutting specimens.

8.4 The laminate thickness shall normally be between 3 and 5 mm (0.12 and 0.2 in.). The variation in thickness for any given specimen shall not exceed 0.1 mm. The initial delamination length, measured from the load line to the end of the insert, shall normally be 50 mm (2.0 in.). However, alternative laminate thicknesses and initial delamination lengths may be chosen that are consistent with the discussions given as follows. However, if load blocks are used to introduce the load, very low values of *a/h* are not recommended. For small values of *a/h* (<10), the data reduction procedures given in Section 12 may not be accurate.

8.4.1 For materials with low-flexural modulus or high interlaminar fracture toughness, it may be necessary to increase the number of plies, that is, increase the laminate thickness or decrease the delamination length to avoid large deflections of the specimen arms. The specimen thickness and initial delamination length, *a*₀, shall be designed to satisfy the following criteria (6):

$$a_0 \leq 0.042 \sqrt{\frac{h^3 E_{11}}{G_{1c}}} \quad (2)$$

$$h \geq 8.28 \sqrt[3]{\frac{G_{1c} a_0^2}{E_{11}}} \quad (3)$$

$$h \geq 8.28 \left(\frac{G_{1c} a_0^2}{E_{11}} \right)^{1/3} \quad (3)$$

where:

*a*₀ = initial delamination length,

h = specimen thickness, and

*E*₁₁ = lamina modulus of elasticity in the fiber direction.

However, if the ratio of the opening displacement at delamination onset, *δ*, to the delamination length, *a*, is greater than 0.4, the large deflection corrections in Annex A1 must be incorporated in the data reduction. If these corrections are needed for any delamination length, they should be applied for all delamination lengths.

8.5 It is recommended that void content and fiber volume be reported. Void content may be determined using the equations of Test Methods D 2734. The fiber volume fraction may be determined using a digestion per test in accordance with Test Method D 3171.

8.6 *Sampling*—Test at least five specimens per test condition unless valid results can be gained through the use of fewer specimens, such as the case of a designed experiment. For statistically significant data, the procedures outlined in Practice E 122 should be consulted. The method of sampling shall be reported.

8.7 Load Introduction:

8.7.1 The piano hinges or loading blocks shall be at least as wide as the specimen (20 to 25 mm).

8.7.2 *Piano Hinges*—A pair of piano hinge tabs shall be bonded to the end of each specimen as shown in Fig. 1*a*. The hinge tabs shall be made of metal and shall be capable of sustaining the applied load without incurring damage. The maximum load

anticipated during a DCB test of a material with a known modulus, E_{11} , and anticipated value of G_{Ic} , may be estimated by (6).

$$P_{max} = \frac{b}{a} \sqrt{\frac{h^3 E_{11} G_{Ic}}{96}} \quad (4)$$

8.7.3 *Loading Blocks*—The distance from the loading block pin to the center line of the top specimen arm (distance t in Annex A1) shall be as small as possible to minimize errors as a result of the applied moment arm. These effects will be reduced sufficiently (6) by choosing a distance, t , such that

$$t \leq \frac{h}{4} + 0.01 \sqrt{\frac{0.0434h^3 E_{11}}{G_{Ic}} + a^2} \quad (5)$$

If this criteria cannot be met, then the corrections for loading block effects in Annex A1 should be used to reduce the data.

8.7.4 The bonding surfaces of the loading blocks or hinges and the specimen shall be properly cleaned before bonding to ensure load transfer without debonding of the tabs from the specimen during the test. If debonding occurs, the specimen should not be reused if there is physical evidence that a delamination initiated when the bond failed or if an increased compliance is observed upon reloading.

8.7.4.1 *Surface Preparations of the Specimen*—The bonding surface of the specimen may be lightly grit blasted or scrubbed with sandpaper, then wiped clean with a volatile solvent, such as acetone or methylethylketone (MEK), to remove any contamination.

8.7.4.2 *Surface Preparation of the Loading Hinge Tabs or Blocks*—The loading hinge tabs or blocks may be cleaned as in 8.7.4.1. If this procedure results in a bond failure between the specimen and the tabs, it may be necessary to apply a more sophisticated cleaning procedure based on degreasing and chemical etching. Consult Guide D 2651 for the surface preparation procedure that is most appropriate for the particular metal used for the hinges.

8.7.5 Bonding of the hinges to the specimen shall be performed immediately after surface preparation. The material recommended for bonding is a room temperature cure adhesive. However, in some cases, a superglue, such as cyanoacrylate, has been found to be sufficient. The adhesive may benefit from a postcure if the specimens are dried after the tabs are mounted. Glass beads may need to be added to some adhesives, or other forms of bondline control may be needed to maintain a uniform bond thickness. The loading tabs shall be aligned parallel with the specimen, and with each other, and held in position with clamps while the adhesive cures.

9. Calibration

9.1 The accuracy of all measuring equipment shall have certified calibrations that are current at the time of use of the equipment.

10. Conditioning

10.1 *Standard Conditioning Procedure*—Condition in accordance with Procedure C of Test Method D 5229/D 5229M unless a different environment is specified as part of the experiment. Store and test specimens at standard laboratory atmosphere of $23 \pm 3^\circ\text{C}$ ($73 \pm 5^\circ\text{F}$) and $50 \pm 10\%$ relative humidity.

10.2 *Drying*—If interlaminar fracture toughness data are desired for laminates in a dry condition, use Procedure D of Test Method D 5229/D 5229M.

11. Procedure

11.1 Measure the width and thickness of each specimen to the nearest 0.05 mm (0.002 in.) at the midpoint and at 25 mm (1 in.) from either end. The variation in thickness along the length of the specimen shall not exceed 0.1 mm (0.004 in.). The average values of the width and thickness measurements shall be recorded.

11.2 Coat both edges of the specimen just ahead of the insert with a thin layer of water-based typewriter correction fluid, or equivalent, to aid in visual detection of delamination onset. Mark the first 5 mm (0.2 in.) from the insert on either edge with thin vertical lines every 1 mm (0.04 in.). Mark the remaining 20 mm (0.8 in.) with thin vertical lines every 5 mm (0.2 in.). The delamination length is the sum of the distance from the loading line to the end of the insert (measured in the undeformed state) plus the increment of growth determined from the tick marks.

11.3 Mount the load blocks or hinges on the specimen in the grips of the loading machine, making sure that the specimen is aligned and centered.

11.4 As load is applied, measure the delamination length, a , on one side of the specimen. The initial delamination length, a_0 , is the distance from the load line to the end of the insert. Do not try to locate the end of the insert by opening the specimen. If it is difficult to see the end of the insert on the specimen edge, or to locate the end of the insert from the original mark on the panel, try the following: (1) rub the edge of the specimen in the local area near the insert with a soft lead pencil and (2) polish the edge of the specimen. If none of the above methods are suitable, mark graduations on the specimen edge from the center of the loading pin. When the specimen is loaded, the length of the initial delamination may be determined from these graduation marks. When the delamination grows from the insert, take the first reading at the next whole 1-mm mark. Then, take readings for the next four 1-mm increments of delamination growth and subsequent 5-mm increments as specified above.

11.5 The end of the specimen opposite the grips should be supported before loading, as shown schematically in Fig. 3. The