



Designation: D5528/D5528M – 21

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

This standard is issued under the fixed designation D5528/D5528M; 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.

1. Scope

1.1 This test method describes the determination of the opening mode-I interlaminar fracture toughness, G_{Ic} , of unidirectional fiber-reinforced polymer matrix composite laminates 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-reinforced laminates with brittle or 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 6.6).

1.3 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.3.1 Within the text, the inch-pound units are shown in brackets.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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. Referenced Documents

2.1 ASTM Standards:²

- D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
- D883 Terminology Relating to Plastics
- D2584 Test Method for Ignition Loss of Cured Reinforced Resins
- D2651 Guide for Preparation of Metal Surfaces for Adhesive Bonding
- D2734 Test Methods for Void Content of Reinforced Plastics
- D3171 Test Methods for Constituent Content of Composite Materials
- D3878 Terminology for Composite Materials
- D5229/D5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials
- D7905/D7905M Test Method for Determination of the Mode II Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites
- E4 Practices for Force Calibration and Verification of Testing Machines
- E6 Terminology Relating to Methods of Mechanical Testing
- E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E456 Terminology Relating to Quality and Statistics

3. Terminology

3.1 Terminology D3878 defines terms relating to high-modulus fibers and their composites. Terminology D883 defines terms relating to plastics. Terminology E6 defines terms relating to mechanical testing. Terminology E456 and Practice E177 define terms relating to statistics. In the event of conflict between terms, Terminology D3878 shall have precedence over the other terminology standards.

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.

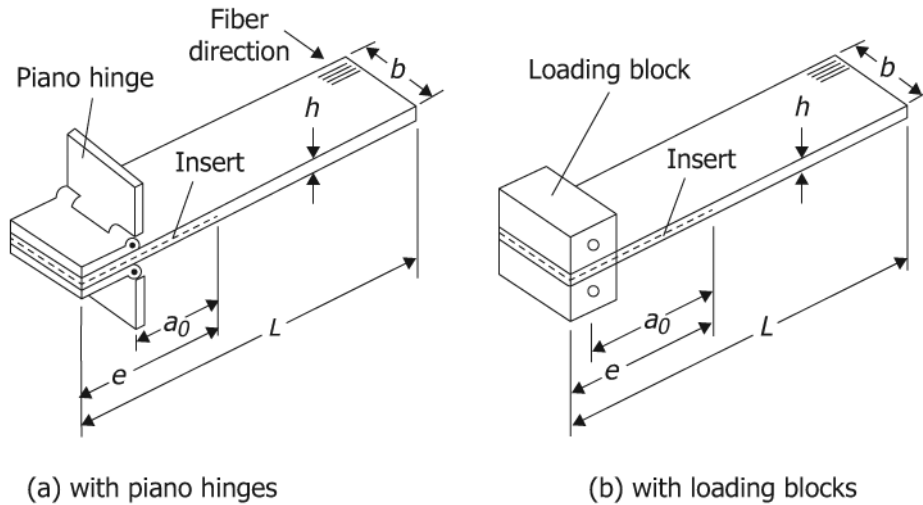


FIG. 1 Double Cantilever Beam Specimen

NOTE 1—If the term represents a physical quantity, its analytical dimensions are stated immediately following the term (or letter symbol) in fundamental dimension form, using the following ASTM standard symbology for fundamental dimensions, shown within square brackets: $[M]$ for mass, $[L]$ for length, $[T]$ for time, $[u]$ for thermodynamic temperature, and $[nd]$ for non-dimensional quantities. Use of these symbols is restricted to analytical dimensions when used with square brackets, as the symbols may have other definitions when used without the brackets.

3.2 Definitions of Terms Specific to This Standard:

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

3.2.2 *mode I interlaminar fracture toughness*, G_{Ic} $[M/T^2]$, n —the critical value of strain energy release rate, G , $[M/T^2]$ for delamination growth $[L]$ as a result of an opening force $[M \cdot L/T^2]$ or opening displacement $[L]$.

3.2.3 *non-precracked (NPC) toughness* $[M/T^2]$, n —an interlaminar fracture toughness value that is determined from the preimplanted insert.

3.2.4 *precracked (PC) toughness* $[M/T^2]$, n —an interlaminar fracture toughness value that is determined after the delamination has been previously advanced from the preimplanted insert.

3.2.5 *strain energy release rate*, G $[M/T^2]$, n —the loss of strain energy, dU $[M \cdot L^2/T^2]$, in the test specimen per unit of specimen width $[L]$ for an infinitesimal increase in delamination length, da $[L]$, for a delamination growing self-similarly under a constant displacement $[L]$; in mathematical form,

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

where:

U = elastic strain energy in the specimen,

b = width of DCB specimen, and

a = delamination length.

3.3 Symbols:

A_I —slope of plot of a/b versus $(C/N)^{1/3}$.

a —delamination length: horizontal distance between load-application point and delamination front (see Fig. 2).

a_0 —initial delamination length: horizontal distance between load-application and end of preimplanted insert (see Fig. 2).

a_i — i^{th} delamination length measured during fracture testing.

b —width of DCB specimen.

C —compliance, δ/P , of DCB specimen.

C_i —compliance of DCB specimen corresponding to the i^{th} delamination length measured during fracture testing.

CV —sample coefficient of variation, in percent.

da —differential increase in delamination length.

dU —differential increase in elastic strain energy.

E_{II} —lamina modulus of elasticity in the fiber direction.

e —total insert length (see Fig. 1).

F —large displacement correction factor.

G —strain energy release rate.

G_I —mode I strain energy release rate.

G_{Ic} —mode I interlaminar fracture toughness.

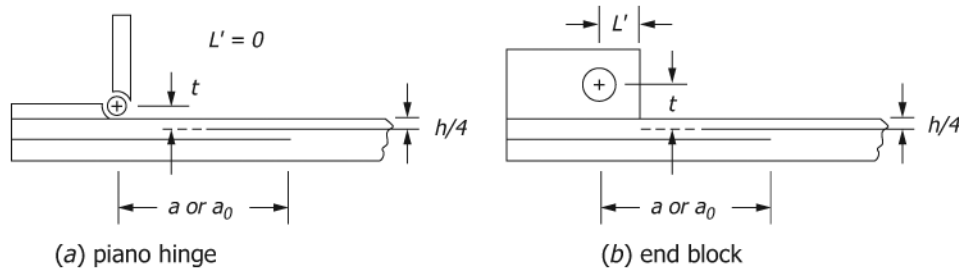


FIG. 2 Methods for Introducing Opening Load to DCB Specimen

G_{Ic}^{est} —estimated value of mode I fracture toughness.
 h —thickness of DCB specimen.
 L —length of DCB specimen.
 L' —horizontal distance from the center of loading-block pin hole to edge of the loading block.
 m —slope of plot of $\log(C/N)$ versus $\log(a)$.
 N —large displacement and loading block correction factor.
 n —number of specimens tested.
 P —applied load.
 P_c —critical force for mode I fracture.
 P_{max} —maximum applied force during DCB test.
 $P_{5\%}$ —applied force at which the specimen compliance has increased by 5%.
 r^2 —correlation coefficient of linear fit of $\log(C/N)$ versus $\log(a)$.
 S_{n-1} —sample standard deviation.
 t —vertical distance from the center of the pin hole to the midplane of the specimen arm.
 U —elastic strain energy in the specimen.
 V_f —fiber volume fraction, in percent.
 $\bar{x}$ —sample mean (average).
 x_i —measured or derived property.
 δ —load point displacement.
 δ_c —critical load point displacement for mode I fracture.
 δ_{NL} —load point displacement containing the initial nonlinearity associated with fixture.
 Δ —effective delamination extension to correct for rotation of DCB arms at delamination front.
 Δ_x —incremental change in $\log(a)$.
 Δ_y —incremental change in $\log(C/N)$.

4. Summary of Test Method

4.1 The DCB specimen shown in Fig. 1 consists of a rectangular, uniform thickness, unidirectional laminated composite specimen containing a preimplanted non-adhesive 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 the delaminated end of the specimen. The arms of the DCB specimen are opened by controlling either the opening displacement or the vertical crosshead movement, while the force and delamination length are recorded.

4.2 A record of the applied force 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, G_{Ic} , is calculated using the compliance calibration (CC) method. The test method provides a non-precracked (NPC) value of G_{Ic} calculated for delamination growth initiating from the preimplanted insert, and a precracked (PC) value of G_{Ic} calculated after the delamination has been previously advanced from the preimplanted insert.

5. Significance and Use

5.1 Susceptibility to delamination is one of the major design concerns for 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 that is independent of specimen geometry or method of force introduction is useful for establishing design allowables used in damage tolerance analyses of composite structures. Knowledge of both the non-precracked and precracked toughness allows the appropriate value to be used for the application of interest.

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 compare quantitatively the values of G_{Ic} obtained from different batches of a specific composite material, for example, to use as a material screening criterion or to develop a design allowable; and

5.2.4 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 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 the thickness for the DCB specimen.

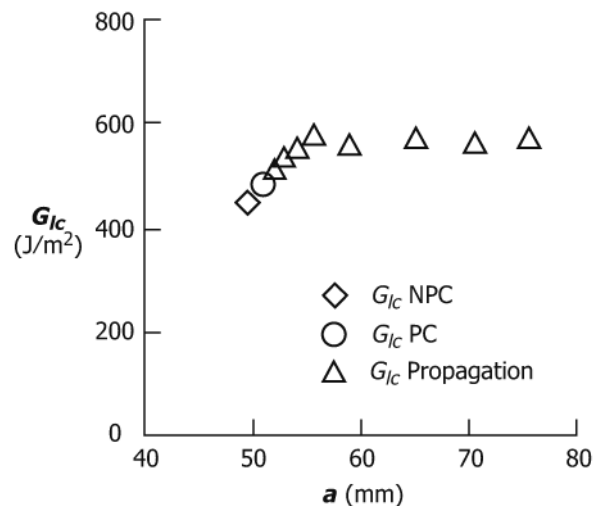


FIG. 3 Schematic of the Delamination Resistance Curve (R-curve) for a Typical DCB Test

6.2 In the DCB test, as the delamination grows from the insert, a resistance-type fracture behavior typically develops where 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 may be generated (Fig. 3). The R-curve may be used to characterize propagation of a delamination in a unidirectional specimen, or it can be used to normalize the maximum cyclic G_I values in mode I fatigue propagation tests.

(1).³ The principal reason for the observed resistance to delamination is the development of fiber bridging (2-4). Fiber bridging is considered to be an artifact of the DCB test. Therefore, the generic significance of G_{Ic} propagation values calculated after growth from 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 (8.3).

6.3 The NPC value of G_{Ic} is determined based on the force-displacement data measured at the point at which the specimen compliance has increased by 5 % or the force has reached a maximum value (see 11.8.1). Physical evidence suggests that the NPC value of G_{Ic} determined based on these force definitions corresponds to the onset of delamination growth having occurred across the entire width of the specimen (5).

6.4 After initiation, 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. A run-arrest extension 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.

6.5 The toughness values obtained by this test method for delamination growth at 0°/0° interfaces may not be representative of the toughness corresponding to delamination growth at interfaces with different relative ply orientations.

6.6 *Application to Other Materials, Layups, and Architectures:*

6.6.1 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 cohesion-type failure within the interleaf and one associated with an adhesion-type failure between the tough polymer film and the more brittle composite matrix.

6.6.2 Non-unidirectional DCB configurations may experience considerable amount of fiber bridging (4, 6) and 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.

6.6.3 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.

6.6.4 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 E4. The testing machine shall be equipped with grips to hold the loading hinges, or clevises to hold the loading blocks, that are bonded to the specimen.

7.2 *Force Indicator*—The testing machine force-sensing device shall be capable of indicating the total force carried by the test specimen. This device shall be essentially free from inertia lag at the specified rate of testing and shall indicate the force with an accuracy over the force 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 at peak load. If not, then the opening displacement shall be obtained from a properly calibrated external gauge or transducer attached to the specimen at the point of force application. The displacement indicator shall indicate the load-point crack opening displacement with an accuracy of within ± 1 % of the indicated value once the delamination occurs.

7.4 *Force Versus Opening Displacement Record*—During the test, force versus opening displacement at the point of force application shall be documented digitally and post-processed.

7.5 *Optical Microscope*—A travelling optical microscope with a magnification no greater than 70 $\times$, or equivalent magnifying devices, shall be positioned on one side of the specimen to observe the delamination front as it extends along one edge of the specimen 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.]. Other methods, such as crack length gauges bonded to a specimen edge, may be used to monitor delamination length, provided their accuracy is as accurate as the optical microscope so that delamination length may be measured to the accuracy specified above.

7.6 *Micrometers and Calipers*—A micrometer with a 4 to 8 mm [0.16 to 0.32 in.] nominal diameter ball interface or a flat anvil interface shall be used to measure the specimen thickness. A ball interface is recommended for thickness measurements when at least one surface is irregular (for example, a coarse peel ply surface, which is neither smooth nor flat). A micrometer or caliper with a flat anvil interface shall be used for measuring length, width, and other machined surface dimensions. The use of alternative measurement devices is permitted if specified (or agreed to) by the test requestor and reported by the testing laboratory. The accuracy of the instrument(s) shall be suitable for reading to the accuracy of the test method.

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

specimen dimensions. For typical specimen geometries, an instrument with an accuracy of ± 0.0025 mm [± 0.0001 in.] is adequate for thickness measurements, while an instrument with an accuracy of ± 0.025 mm [± 0.001 in.] is adequate for measurement of length, width, and other machined surface dimensions.

7.7 Conditioning Chamber—When conditioning materials at non-laboratory environments, a temperature-/vapor-level controlled environmental conditioning chamber is required that shall be capable of maintaining the required temperature to within ± 3 °C [± 5 °F] and the required relative humidity level to within ± 3 %. Chamber conditions shall be monitored either on an automated continuous basis or on a manual basis at regular intervals.

7.8 Environmental Test Chamber—An environmental test chamber is required for test environments other than ambient testing laboratory conditions. This chamber shall be capable of maintaining the test specimen and fixture at the required test environment during the mechanical test. The test temperature shall be maintained within ± 3 °C [± 5 °F] of the required temperature, and the relative humidity level shall be maintained to within ± 3 % of the required humidity level.

8. Sampling and Test Specimens

8.1 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 E122 should be consulted. The method of sampling shall be reported.

8.2 Test laminates must contain an even number of plies, and shall be unidirectional, with delamination growth occurring in the 0° (zero degree) direction (see Fig. 1).

8.3 A non-adhesive insert shall be implanted at the mid-plane of the laminate during layup to form an initiation site for the delamination (see Fig. 1). The insert thickness shall be no greater than 13 μ m [0.0005 in.]. A polymer film is recommended for the insert to avoid problems with folding or crimping at the cut end of the insert. For epoxy matrix composites cured at or below 177 °C [350 °F], an insert made of polytetrafluoroethylene (PTFE) is recommended. For composites with polyimide, bismaleimide, or thermoplastic matrices that are manufactured at relatively high temperatures, that is, greater than 177 °C [350 °F], a thin polyimide insert is recommended. If a polyimide insert is used, the insert shall be painted or sprayed with a mold release agent before it is inserted in the laminate. Caution should be used, as mold release agents containing silicone may contaminate the laminate by migration through the individual layers. It is often helpful to coat the insert at least once and then bake the insert before placing it on the composite. This will help to prevent silicone migration within the composite. For materials outside the scope of this test method, different film materials may be required. Under certain prescribed circumstances (see 13.2), an alternate wedge precracking procedure may be used. Guidelines for generating a wedge precrack are given in Annex A3.

8.4 Specimen Dimensions:

8.4.1 Specimens shall be at least 140 mm [5.5 in.] long and nominally from 20 to 25 mm [0.8 to 1.0 in.] wide, inclusive.

8.4.2 Panels shall be manufactured, and specimens cut from the panels, such that the total insert length, e , is 76 mm [3.0 in.] (see Fig. 1). This distance corresponds to an initial delamination length 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.

8.5 The laminate thickness shall typically be between 3 and 5 mm [0.12 and 0.2 in.]. The initial delamination length, a_0 , measured from the load-application point to the end of the insert, shall typically be 50 mm [2.0 in.]. Alternative laminate thicknesses and initial delamination lengths may be chosen that are consistent with the discussions given as follows; however, very low values of a_0/h are not recommended. For low values of a_0/h (<10), the data reduction procedures given in Section 13 may not be accurate.

8.5.1 For certain composite systems (for example, those with a low-flexural modulus or a high interlaminar fracture toughness), it may be necessary to increase the number of plies (increase the laminate thickness) or decrease the initial delamination length to avoid large displacement of the specimen arms. This displacement is deemed large when the ratio of critical load-point opening displacement at delamination onset, δ_c , to the delamination length, a , is greater than 0.4. To prevent this from occurring, the specimen thickness and initial delamination length, a_0 , shall be designed to satisfy the following criteria (7):

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

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

where:

- a_0 = initial delamination length,
- h = thickness of DCB specimen,
- E_{11} = lamina modulus of elasticity in the fiber direction, and
- G_{Ic}^{est} = estimated value of mode I fracture toughness.

8.6 If specific gravity, density, reinforcement volume, or void volume are to be reported, then obtain these samples from the same panels being tested. Specific gravity and density may be evaluated by means of Test Method D792. Volume percent of the constituents may be evaluated by one of the matrix digestion procedures of Test Method D3171 or, for certain reinforcement materials such as glass and ceramics, by the matrix burn-off technique of Test Method D2584. The void content equations of Test Method D2734 are applicable to both Test Method D2584 and the matrix digestion procedures.

8.7 Force Introduction:

8.7.1 The piano hinges or loading blocks shall be at least as wide as the specimen, between 20 to 25 mm [0.8 to 1.0 in.].

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