Designation: D5528/D5528M – 21

Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites[1]

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.*

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.

[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.

Current edition approved Nov. 15, 2021. Published January 2022. Originally approved in 1994. Last previous edition approved in 2013 as D5528 – 13. DOI: 10.1520/D5528_D5528M-21.

[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.

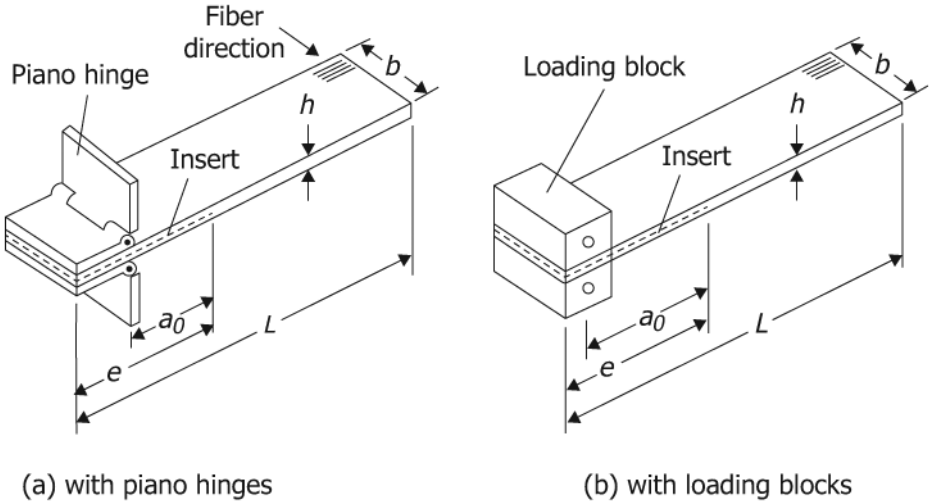

FIG. 1 Double Cantilever Beam Specimen

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/T²], n*—the critical value of strain energy release rate, *G*, *[M/T²]* for delamination growth [*L*] as a result of an opening force [*M·L/T²*] or opening displacement [*L*].

3.2.3 *non-precracked (NPC) toughness [M/T²], n*—an interlaminar fracture toughness value that is determined from the preimplanted insert.

3.2.4 *precracked (PC) toughness [M/T²], 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/T²], n*—the loss of strain energy, *dU* [*M·L²/T²*], 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_1—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_{11}—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.

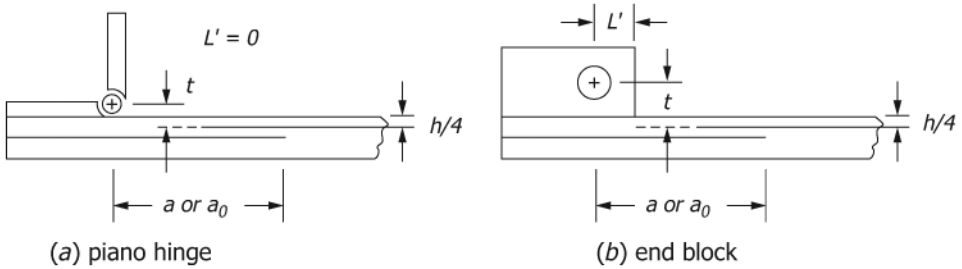

FIG. 2 Methods for Introducing Opening Load to DCB Specimen

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 nonprecracked (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.

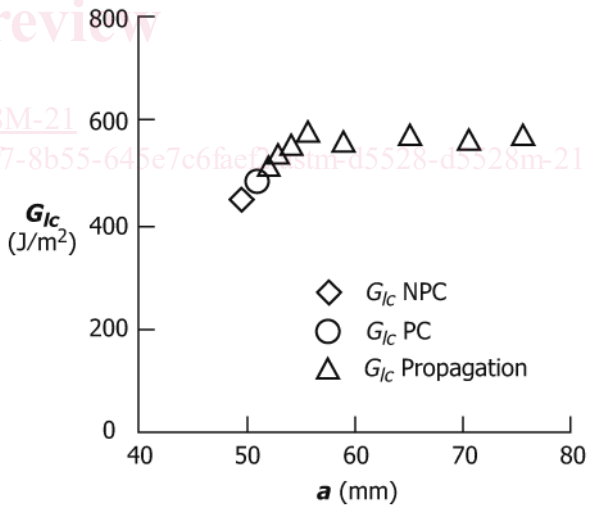

FIG. 3 Schematic of the Delamination Resistance Curve (*R*-curve) for a Typical DCB Test

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 disbonded 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×, 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 within 1 % of the

[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 midplane 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 µm [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):

$$a_0 \le 0.042 \sqrt{\frac{h^3 E_{11}}{G_{Ic}^{est}}} \tag{2}$$

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

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 anticipated during

a DCB test of a material with a known modulus, E_{11}, and estimated value of G_{Ic}^{est}, may be determined by (7).

$$P_{max} = \frac{b}{a_0}\sqrt{\frac{h^3 E_{11} G_{Ic}^{est}}{96}}. \quad (4)$$

8.7.3 *Loading Blocks*—The distance from the loading block pin to the center line of the top specimen arm (distance t in Fig. 2b) shall be as small as possible to minimize errors as a result of the applied moment arm. These effects will be reduced sufficiently (7) by choosing a distance, t, such that

$$t \le \frac{h}{4} + 0.01\sqrt{\frac{0.0434 h^3 E_{11}}{G_{Ic}^{est}} + a_0^2} \quad (5)$$

8.7.4 The bonding surfaces of the loading blocks or hinges and the specimen shall be properly prepared before bonding to ensure force 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 isopropyl alcohol, 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 surface preparation procedure based on degreasing and chemical etching. Consult Guide D2651 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. The adhesive may benefit from a postcure if the specimens are dried after the tabs are mounted. Glass beads or other forms of bondline control may be needed to produce 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.

8.8 *Labeling*—Label the plate specimens so that they will be distinct from each other and traceable back to the raw material, and will neither influence the test nor be affected by it.

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 The recommended pre-test condition is effective moisture equilibrium at a specific relative humidity as established by Test Method D5229/D5229M; however, if the test requestor does not explicitly specify a pre-test conditioning environment, no conditioning is required and the test specimens may be tested as prepared.

10.2 The pre-test specimen conditioning process, to include specified environmental exposure levels and resulting moisture content, shall be reported with the test data.

NOTE 2—The term "moisture," as used in Test Method D5229/D5229M, includes not only the vapor of a liquid and its condensate, but the liquid itself in large quantities, as for immersion.

10.3 If no explicit conditioning process is performed, the specimen conditioning process shall be reported as "unconditioned" and the moisture content as "unknown."

11. Procedure

11.1 *Specimen Preparation:*

11.1.1 Following final specimen machining, but before conditioning and testing, measure the width and thickness of each specimen to the nearest 0.05 mm [0.002 in.] at the midpoint and at 50 mm [2 in.] from either end. The individual and average values of the three width measurements and three thickness measurements shall be recorded. The variation in specimen width among all measurements shall not exceed 0.5 mm [0.02 in.], and the variation in specimen thickness shall not exceed 5 % of the mean value. Measure and record the vertical distance from the center of the pin hole to the midplane of the specimen arm, t, as defined in Fig. 2. If loading blocks are used, measure and record the horizontal distance from the center of loading-block pin hole to edge of the loading block, L', as defined in Fig. 2.

NOTE 3—The test requester may request that additional measurements be performed after the machined specimens have gone through any conditioning or environmental exposure.

11.1.2 Coat both long edges of the specimen with a thin layer of water-based typewriter correction fluid, or equivalent, to aid in visual detection of delamination growth. Once the coating is dry, mark the location of the insert tip with thin vertical lines on both edges. The vertical lines shall be made with a mechanical pencil containing a 0.5 mm [0.002 in.] diameter lead or smaller. Measure and record the initial delamination length, a_0, with an accuracy of at least ±0.5 mm [0.02 in.]. The initial delamination length is the distance from the load-application point to the end of the insert. Mark the first 10 mm [0.4 in.] from the insert tip with thin vertical lines every 1 mm [0.04 in.] on both edges. Mark the additional 20 mm [0.8 in.] length with thin vertical lines every 2 mm [0.2 in.] on both edges.

11.2 Mount the specimen in the loading machine, making sure that the specimen's width is centered relative to the load line.

11.3 Prior to loading, the end of the specimen opposite the hinges/loading-blocks may be supported to keep the specimen horizontal. The supported end will rise off the support as the force is applied.

11.4 Set the optical microscope (see 7.5), or an equivalent magnifying device, in a position to observe the motion of the delamination front as it grows along one edge of the specimen. This device shall be capable of pinpointing the delamination front with an accuracy of at least ±0.5 mm [±0.02 in.].

11.5 *Initial Loading:*

11.5.1 Load the specimen at a constant crosshead rate between 1 and 5 mm/min [0.04 and 0.20 in./min].

11.5.2 The force and displacement data are to be recorded continuously or at frequent and regular intervals during the initial and reloading cycles; a sampling rate of 5 Hz or greater and a target minimum of 500 data points per loading cycle are recommended.

11.5.3 The loading shall be stopped after initial delamination growth is between 3 to 5 mm [0.12 to 0.20 in.]. If unstable delamination growth from the insert is observed, it shall be noted in the test report. If the unstable delamination growth is less than 3 mm [0.12 in], loading shall be continued until delamination length growth is between 3 to 5 mm [0.12 to 0.20 in.] beyond the insert. If the unstable delamination growth is greater than 5 mm [0.20 in], the loading shall be stopped. Note in the test report if the initial delamination length increment is outside the range of 3 to 5 mm [0.12 to 0.20 in.].

11.5.4 Unload the specimen at a constant crosshead rate of up to 25 mm/min [1 in./min], while continuously recording the force and displacement. Pause the unloading at approximately 50 % of the maximum force reached during initial loading. Using the optical microscope, or an equivalent device, measure the delamination length and record it as the first propagation delamination length, a_1. Mark the position of the tip of the precrack on both edges of the specimen. Note in the test report if the position on the two edges differs by more than 2 mm. Continue to unload the specimen until the applied opening force returns to zero (ensuring displacement reading is not zeroed at any stage of the test).

NOTE 4—Mismatch between the two positions greater than 2 mm [0.08 in.] may be an indication of fixture misalignment resulting in asymmetrical loading. If the resulting PC G_{Ic} value determined based on a_1 (see 11.8) is an outlier relative to all other PC G_{Ic} values from the same batch, a replacement specimen shall be tested.

11.6 *Reloading:*

11.6.1 The specimen shall be reloaded at the same constant crosshead speed of 1 to 5 mm/min [0.04 to 0.20 in./min]. Record the force and the displacement values, continuously using the sample rate chosen for the initial loading cycle

11.6.2 On reloading, record the force and displacement values at as many delamination length increments as possible in the first 10 mm [0.4 in.] of delamination growth beyond the insert, ideally every 1 mm [0.04 in.]. Subsequently, record these force and displacement data at delamination lengths in 2-4 mm increments of delamination growth, until the delamination has propagated at least 30 mm [1.2 in.] from the insert.

11.6.3 Finally, unload the specimen at a constant crosshead rate of up to 25 mm/min [1 in./min]. Pause the unloading at approximately 50 % of the force reached at the end of the test. Mark the positions of the tip of the delamination on both edges of the specimen. Note in the report if these positions differ by more than 2 mm [0.08 in.]. Unload the specimen to the original displacement position (corresponding to the zero-load point prior to the beginning of the first load cycle) and note the indicated applied force. A negative value of this indicated applied force may be due to permanent deformation of the specimen arms. Any permanent deformation of the specimen after unloading shall be noted in the report. Specimen arms will be noted as being permanently deformed if removal of curvature of the arms resting on a rigid, flat surface (with fracture surface facing up) requires more than manual loading with an index finger. The specimen arms will be machined away from the intact portion of the specimen in order to perform this assessment of permanent deformation. Data from specimens exhibiting permanent deformation are invalid.

NOTE 5—Mismatch between the two positions greater than 2 mm [0.08 in.] may be an indication of fixture misalignment resulting in asymmetrical loading. Any instances of delamination extension that exhibit a mismatch between delamination front position greater than 2 mm [0.08 in.] shall be excluded from compliance calibration (11.8.1).

11.7 If an alternative method for monitoring delamination growth is used (for example, crack growth gauges bonded to the specimen edges, digital video camera), it should collect data in accordance with the principles, accuracy, and magnification as set out in detail above.

11.8 *Interpretation of Test Results*—The first value of toughness (that is, NPC G_{Ic}) corresponds to delamination growth from the insert and is obtained from the initial force-displacement plot. The second value of toughness (that is, PC G_{Ic}) corresponds to delamination growth from a precracked delamination front and is obtained from the force-displacement plot after subsequent reloading. The NPC and PC values of G_{Ic}, and propagation values of G_{Ic} may be used to generate the *R*-curve, as shown in Fig. 3. The NPC G_{Ic} shall be determined using the initial delamination length, a_0, while PC G_{Ic} shall be determined using the subsequent delamination length, a_1, defined in 11.5.4. Both NPC and PC values of G_{Ic} shall be determined based on the critical force and displacement values defined in 11.8.1. All propagation values shall be determined based on the critical force and displacement values corresponding to visually observed delamination lengths, a_i, described in 11.6.2.

11.8.1 *Definition of Critical Force Used to Calculate NPC and PC Value of G_{Ic}*—A non-precracked (NPC) and precracked (PC) value of G_{Ic} shall be calculated by determining the intersection of the force-displacement curve, once it has become nonlinear, with a line drawn from the origin and offset by a 5 % increase in compliance from the original linear region of the force-displacement curve (see Fig. 4). The original linear region of the force-displacement curve is defined as that ranging between 25 % and 75 % of peak force. The 25 % limit is chosen to be sufficient to exclude any data affected by initial nonlinearity in the load-displacement response (see Fig. 4a). Compliance is determined using a linear least squares regression analysis of the displacement versus force (δ versus P) data over the previously defined load range as described in Annex A4. If the intersection occurs after the maximum force (P_{max}) point, the critical force, $P_c = P_{max}$, should be used to calculate G_{Ic} (see Fig. 4a). If the intersection occurs before P_{max}, the force corresponding to that intersection, $P_c = P_{5\%}$, should be used to calculate G_{Ic} (see Fig. 4b).

12. Validation

12.1 Values for toughness shall not be calculated for any specimen that fails by breaking in some manner other than delamination advance, such as breaking at some obvious flaw,