

Designation: D5528 - 13 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 $\frac{D5528}{D5528}$ 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 $\frac{\text{Mode I} \text{mode-I}}{\text{mode-I}}$ interlaminar fracture toughness, G_{Ic} , of $\frac{\text{continuous}}{\text{unidirectional}}$ fiber-reinforced $\frac{\text{polymer matrix}}{\text{polymer}}$ composite $\frac{\text{materials}}{\text{materials}}$ using the double cantilever beam (DCB) specimen (Fig. 1).
- 1.2 This test method is limited to use with composites consisting of unidirectional earbon fiber and glass fiber tape carbon-fiber and glass-fiber-reinforced laminates with brittle andor 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 6.56.6).
- 1.3 <u>Units—The values stated in either SI units or inch-pound units are to be regarded separately as the standard.</u> The values given stated in parentheses are for information only, 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 safety, health, and health 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

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.

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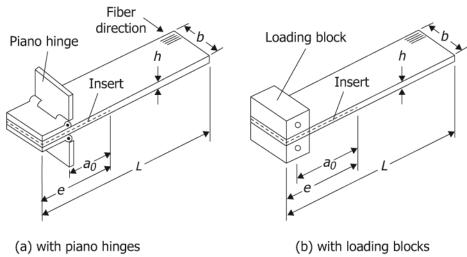


FIG. 1 — Double Double Cantilever Beam Specimen

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

<u>D7905/D7905M</u> 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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1309 Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases (Withdrawn 2015)³

E1434 Guide for Recording Mechanical Test Data of Fiber-Reinforced Composite Materials in Databases (Withdrawn 2015)³

E1471 Guide for Identification of Fibers, Fillers, and Core Materials in Computerized Material Property Databases (Withdrawn 2015)³

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.

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)—(mode I), n—fracture mode in which the delamination faces open away from each other.
 - 3.2.2 <u>Modemode</u> I interlaminar fracture toughness, G_{Ic} [M/T²]—], n—the critical value of <u>strain energy release rate</u>, G_{Ic} [M/T²] for delamination growth [L] as a result of an opening load force [M·L/T² or displacement.] 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/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, $G[M/T^2]$ —[], n—the loss of strain energy, dU, 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, da [L], for a delamination growing self-similarly under a constant displacement. Indisplacement [L]; in mathematical form,

$$G = -\frac{1}{b} \frac{dU}{da} \tag{1}$$

where:

U = total elastic energy in the test specimen,

<u>U</u> = elastic strain energy in the specimen,

b = specimen width, and

= width of DCB specimen, and

a = delamination length.

3.3 Symbols:

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

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

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

a_ith delamination length measured during fracture testing.

b = width —width of DCB specimen.

 $C = \frac{1}{2} \frac{1}{2}$

C,—compliance of DCB specimen corresponding to the ith delamination length measured during fracture testing.

CV = __sample coefficient of variation, %-in percent.

da = differential — differential increase in delamination length. D5528M-21

 $\frac{dU = \text{differential}}{dU = \text{differential}} \text{ increase in elastic strain energy.} \\ 1-4117-8b55-645e7c6faef2/astm-d5528-d5528m-21$

 E_{II} = <u>lamina</u> modulus of elasticity in the fiber direction.

 $\underline{Ee_{TT}}$ = modulus of __total insert length (see Fig. 1elasticity in the fiber direction measured in flexure.).

F = large—large displacement correction factor.

G—strain energy release rate.

(a) piano hina

 G_I —mode I strain energy release rate.

 G_{Ic} = opening Mode _mode I interlaminar fracture toughness.

 G_{Lc}^{est} —estimated value of mode I fracture toughness.

h =thickness of DCB specimen.

L = length—length of DCB specimen.

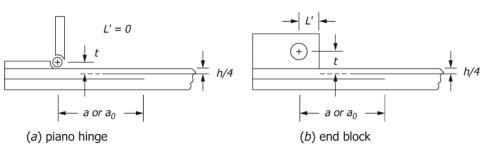


FIG. 4 Modified Beam Theory FIG. 5 Compliance Calibration FIG. 6 Modified Compliance Calibration

(b) end block

FIG. A1.12 — Methods Methods for Introducing Opening Load to DCB Specimen



L' = half width of—horizontal distance from the center of loading-block pin hole to edge of the loading block.

 $m = \text{number of plies in DCB}__\text{slope of plot of } \log(C/N - \text{specimen.}) \text{ versus } \log(a).$

N=loading—large displacement and loading block correction factor.

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

n = slope of plot of Log C versus Log a.—number of specimens tested.

P = applied—applied load.

 P_c —critical force for mode I fracture.

 P_{max} = maximum applied load maximum applied force during DCB test.

 $SDP_{5\%}$ = standard deviation.—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 loading block pin to center line of topthe center of the pin hole to the midplane of the specimen arm.

U = strain energy.—elastic strain energy in the specimen.

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

 $V_f = \text{fiber}$ volume fraction, %: in percent.

 x^{-} —sample mean (average).

 x_i —measured or derived property.

 δ —load point displacement.

 δ_c — critical load point deflection.displacement for mode I fracture.

 δ_{NL} —load point displacement containing the initial nonlinearity associated with fixture.

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

 $\Delta_x = \text{incremental } \underline{\text{--incremental }} \text{ change in } \underline{\text{Log }} \underline{\log(a.a)}.$

 $\Delta_v = \text{incremental}$ —incremental change in $\frac{\text{Log}}{\log(C \cdot C/N)}$.

4. Summary of Test Method

iTeh Standards

- 4.1 The DCB <u>specimen</u> shown in Fig. 1 consists of a rectangular, uniform thickness, unidirectional laminated composite specimen containing a <u>nonadhesive preimplanted non-adhesive</u> insert on the midplane that serves as a delamination initiator. Opening forces are applied to the DCB specimen by means of hinges (Fig. 1aa) or loading blocks (Fig. 1bb) bonded to <u>one-the delaminated</u> end of the specimen. The <u>endsarms</u> of the DCB <u>specimen</u> are opened by controlling either the opening displacement or the <u>vertical</u> crosshead movement, while the <u>loadforce</u> and delamination length are recorded.
- 4.2 A record of the applied <u>loadforce</u> 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 <u>Modemode</u> I interlaminar fracture toughness, G_{Ic} is calculated using a <u>modified beam theory or empliance calibration method</u>: 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 weaknesses of 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 Modemode I interlaminar fracture toughness, toughness that is independent of specimen geometry or method of load introduction, force introduction is useful for establishing design allowables used in damage tolerance analyses of composite structures made from these materials.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. material:
- 5.2.2 To compare quantitatively the relative values of G_{Ic} for composite materials with different eonstituents:
 - 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: 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 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-specimen.

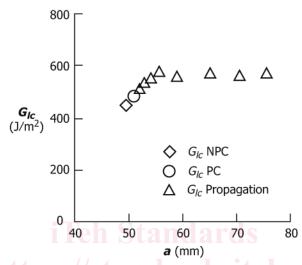


FIG. 23 — Delamination-Schematic of the Delamination Resistance Curve (RGurve) from -curve) for a Typical DCB Test

Document Preview

6.2 In the DCB test, as the delamination grows from the insert, a resistance-type fracture behavior typically develops where the ealculated 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 willmay be generated to (Fig. 3 characterize). The the initiation and -curve may be used to characterize propagation of a delamination in a unidirectional specimen-specimen, or it can be used to normalize the maximum cyclic G_I values in mode I fatigue propagation tests (Fig. 21). The principal reason for the observed resistance to delamination is the development of fiber bridging ((1-2-34).). 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 Fiber bridging is considered to be an artifact of the DCB test on unidirectional materials. test. Therefore, the generic significance of G_{Ic} propagation values calculated beyond the end of the 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 Three definitions for an initiation The NPC 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-is determined based on the force-displacement data measured 7.5, and (3) at the point at which the specimen compliance has increased by 5 % or the loadforce has reached a maximum value (5 %/max) (see Section 1111.8.1). The NLPhysical evidence $G_{SUggests_{Ic}}$ value, which is typically the lowest of the three that the NPC value of G_{Ic} initiation values, is recommended for generating delamination failure criteria in durability and damage tolerance analyses of laminated composite structures (determined based on these force definitions corresponds 5.2.4). Recommendations for obtaining the NL point are given in Annex A2. All three initiation values can be used for the other purposes eited 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

³ The last approved version of this historical standard is referenced on www.astm.org.

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



the physical onset of delamination from the insert in the interior delamination growth having occurred across the entire width 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 <u>Delamination After initiation, delamination growth</u> may proceed in one of two ways: (1)(1) by a slow stable extension or (2)(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 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. 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 8.3) 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} .
- <u>6.5</u> The toughness values obtained by this test method for delamination growth at $0^{\circ}/0^{\circ}$ 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 eohesive-typecohesion-type failure within the interleaf and one associated with an adhesive-typeadhesion-type failure between the tough polymer film and the more brittle composite matrix.
- 6.6.2 Nonunidirectional 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 Modemode 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.
- 6.6.3 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 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[0.02 to 0.20 in./min).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 pinsclevises to hold the loading blocks, that are bonded to the specimen.
- 7.2 <u>LoadForce</u> Indicator—The testing machine <u>load-sensingforce-sensing</u> device shall be capable of indicating the total <u>loadforce</u> carried by the test specimen. This device shall be essentially free from inertia lag at the specified rate of testing and shall indicate the <u>loadforce</u> with an accuracy over the <u>loadforce</u> 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. specimen at peak load. If not, then the opening displacement shall be obtained from a properly calibrated external gagegauge or transducer attached to the specimen. 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 <u>LoadForce</u> Versus Opening Displacement Record—An<u>During</u> X-Y plotter, or similar device, shall be used to make a permanent record during the test of load the test, force versus opening displacement at the point of load application. Alternatively, the data may be storedforce 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 an-equivalent magnifying device; 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.). A mirror may be used to determine visually any discrepancy in delamination onset from one side of the specimen to the other. ± 0.5 mm [± 0.02 in.]. Other methods, such as crack length gagesgauges bonded to a specimen edge, may be used to monitor delamination length, provided their accuracy is as goodaccurate as the optical microscope so that delamination length may be measured to the accuracy specified above.
- 7.6 Micrometers and Calipers—The micrometer(s) shall use a suitable size A micrometer with a 4 to 8 mm [0.16 to 0.32 in.] nominal diameter ball interface on irregular surfaces such as the bag side of a laminate and 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 on machined edges or very smooth tooled surfaces. The 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 instrumentsinstrument(s) shall be suitable for reading to within 1 % of the sample width and thickness. specimen dimensions. For typical specimen geometries, an instrument with an accuracy of ±2.5 µm (0.0001 in.) is desirable ±0.0025 mm [±0.0001 in.] is adequate for thickness measurement, measurements, while an instrument with an accuracy of ±25 mm (0.001 in.) is desirable for width measurement. ±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° direction. (zero degree) direction (see Fig. 1).
- 8.3 A nonadhesive insert shall be inserted implanted at the midplane of the laminate during layup to form an initiation site for the delamination (see Fig. 1). The filminsert 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 [0.0005 in.]. 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-insert. (4). For epoxy matrix composites cured at relatively low temperatures, 177°C (350°F) or less, a thin film 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),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. insert is recommended. If a polyimide filminsert is used, the filminsert shall be painted or sprayed with a mold release agent before it is inserted in the laminate. (Caution Warning Mold 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 filminsert at least once and then bake the filminsert before placing the

film it on the composite. This will help to prevent silicone migration within the composite. Although precracking is not recommended, under For materials outside the scope of this test method, different film materials may be required. Under certain prescribed circumstances (see 11.7.713.2), 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 125 mm (5.0 in.) 140 mm [5.5 in.] long and nominally from 20 to 25 mm (0.8 to 1.0 in.) 25 mm [0.8 to 1.0 in.] wide, inclusive.

Note 2—Round-robin testing on narrow and wide specimens yielded similar results, indicating that the DCB specimen width is not a critical parameter.

8.4.2 Panels shall be manufactured, and specimens cut from the panels, such that the total insert length, e, is approximately 63 mm (2.5 in.) 76 mm [3.0 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.

8.5 The laminate thickness shall normally typically 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 (0.004 in.). The in.]. The initial delamination length, a_0 measured from the load lineload-application point to the end of the insert, shall normallytypically be 50 mm (2.0 in.). However, alternative [2.0 in.]. 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, follows; however, very low values of a/ha₀/h are not recommended. For smallow values of $\frac{dha_0}{h}$ (<10), the data reduction procedures given in Section 13 may not be accurate.

8.5.1 For materials with certain composite systems (for example, those with a low-flexural modulus or a high interlaminar fracture toughness; toughness), it may be necessary to increase the number of plies, that is, increase plies (increase the laminate thicknessthickness) or decrease the initial delamination length to avoid large deflections displacement of the specimen arms. The 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 (67):

$$\frac{\text{ASTM}_{a_0} \ge 0.042 \sqrt{\frac{h^3 E_{11}}{G_{1c}}} \text{M-21}}{G_{1c}}$$
(2)
$$\frac{1}{G_{1c}} = -8b55 - 645e7c6 \text{ fae } [2/\text{astm-d5528-d5528m-21}]}{a_0 \le 0.042 \sqrt{\frac{h^3 E_{11}}{G_{1c}^{est}}}}$$
(2)
$$\frac{h \ge 8.28 \left(\frac{G_{1c} a_0^2}{E_{11}}\right)^{1/3}}{E_{11}}$$
(3)
$$h \ge 8.28 \left(\frac{G_{cs} a_0^2}{E_{11}}\right)^{1/3}$$
(3)

where:

 a_0 = initial delamination length, = specimen thickness, and = thickness of DCB specimen,

lamina modulus of elasticity in the fiber direction. = lamina modulus of elasticity in the fiber direction, and

estimated value of mode I fracture toughness.

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.

8.6 It is recommended that void content and fiber volume be reported. Void content may be determined using the equations of Test Methods 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 D2734D792. The fiber volume fraction may be determined using a digestion per test in accordance with 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 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.

- 8.7 *Load*Force Introduction:
- 8.7.1 The piano hinges or loading blocks shall be at least as wide as the specimen (20 to 25 mm). 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 loadforce without incurring damage. damage or excessive deformation. The maximum loadforce anticipated during a DCB test of a material with a known modulus, E_{II} , and anticipated value of G_{Ic}^{est} , may be estimated determined by ((67).).

$$P_{max} = \frac{b}{a} \sqrt{\frac{h^{3} E_{11} G_{1c}}{96}}$$
 (4)

$$P_{max} = \frac{b}{a_0} \sqrt{\frac{h^3 E_{11} G_{Ic}^{est}}{96}}.$$
 (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 A1Fig. 2)b) shall be as small as possible to minimize errors as a result of the applied moment arm. These effects will be reduced sufficiently (67) by choosing a distance, t, such that

$$t \le \frac{h}{4} + 0.01 \sqrt{\frac{0.0434h^3 E_{11}}{G_{1c}} + a^2}$$
 (5)

$$t \le \frac{h}{4} + 0.01 \sqrt{\frac{0.0434h^3 E_{11}}{G_{Ic}^{est}} + a_0^2}$$
 (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 <u>cleanedprepared</u> before bonding to ensure <u>loadforce</u> 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), 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 8.8.4.18.7.4.1. If this procedure results in a bond failure between the specimen and the tabs, it may be necessary to apply a more sophisticated eleaning—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. 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 maintainproduce 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 Standard Conditioning Procedure—Condition in accordance with Procedure C of The recommended pre-test condition is effective moisture equilibrium at a specific relative humidity as established by Test Method D5229/D5229M-unless a different environment is specified as part of the experiment. Store and test specimens at standard laboratory atmosphere of $23 \pm 3^{\circ}$ C (73 $\pm 5^{\circ}$ F) and $50 \pm 10^{\circ}$ % relative humidity.; 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 Drying—If interlaminar fracture toughness data are desired for laminates in a dry condition, use Procedure D of Test Method The pre-test specimen conditioning process, to include specified environmental exposure levels and resulting moisture content, shall be reported with D5229/D5229M. 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."

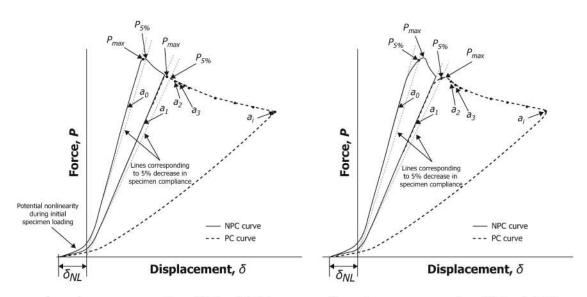
11. Procedure

iTeh Standards

- 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.
- 11.1 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. 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.
- 11.3 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.

- 11.2 As load is applied, measure the delamination length, $\underline{\text{Mount the}}$ 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:specimen in the loading machine, making sure that the specimen's width is centered relative to the load line.
- 11.3 The Prior to loading, the end of the specimen opposite the grips should be supported before loading, as shown schematically in hinges/loading-blocks may be supported to keep the specimen horizontal. Fig. 3. The supported end may will rise off the support as the load is applied. For laminates that are excessively long, the specimen may need to be supported during loading force is applied.
- 11.4 Set anthe 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. 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.). ± 0.5 mm [± 0.02 in.].
- 11.5 Initial Loading:
- 11.5.1 Load the specimen at a constant crosshead rate between 1 and 55 mm -mm/min/min [0.04 and 0.20 in./min].
- 11.7.2 Record the load and the displacement values, continuously if possible. Record the position of the delamination with an accuracy of at least ± 0.5 mm.
- 11.5.2 During loading, record the point on the load-displacement curve, or the load-displacement data values, at which the visual onset of delamination movement was observed on the edge of the specimen (VIS,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 Fig. 3).per loading cycle are recommended.

Note 4—If the start of delamination growth is difficult to observe, a change of illumination conditions or a crosshead speed from the lower end of the range is recommended.



a) specimen response where NPC and PC G_{IC} values are calculated based on P_{max}

b) specimen response where NPC and PC G_{IC} values are calculated based on $P_{5\%}$

FIG. 34 —Load Displacement TraceExample Force-Displacement Curves from DCB TestTests