Designation: D5528 – 13

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; 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 continuous fiber-reinforced composite materials using the double cantilever beam (DCB) specimen (Fig. 1).

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

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

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics
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
E4 Practices for Force 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
E1434 Guide for Recording Mechanical Test Data of Fiber-Reinforced Composite Materials in Databases
E1471 Guide for Identification of Fibers, Fillers, and Core Materials in Computerized Material Property Databases

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)*—fracture mode in which the delamination faces open away from each other.

3.2.2 *Mode I interlaminar fracture toughness, G_{Ic} [M/T²]*—the critical value of *G* for delamination growth as a result of an opening load or displacement.

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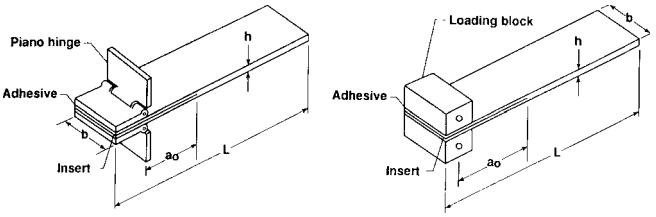
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



(a) with piano hinges (b) with loading blocks

FIG. 1 Double Cantilever Beam Specimen

3.2.3 *strain energy release rate, G [M/T²]*—the loss of energy, dU, in the test specimen per unit of specimen width for an infinitesimal increase in delamination length, *da*, for a delamination growing self-similarly under a constant displacement. In mathematical form,

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

where:

U = total elastic energy in the test specimen,
b = specimen width, and
a = delamination length.

3.3 *Symbols:*

A_1 = slope of plot of a/b versus $C^{1/3}$.
a = delamination length.
a_0 = initial delamination length.
b = width of DCB specimen.
C = compliance, δ/P, of DCB specimen.
CV = coefficient of variation, %.
da = differential increase in delamination length.
dU = differential increase in strain energy.
E_{11} = modulus of elasticity in the fiber direction.
E_{1f} = modulus of elasticity in the fiber direction measured in flexure.
F = large displacement correction factor.
G = strain energy release rate.
G_{Ic} = opening Mode I interlaminar fracture toughness.
h = thickness of DCB specimen.
L = length of DCB specimen.
L' = half width of loading block.
m = number of plies in DCB specimen.
N =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.
P = applied load.
P_{max} = maximum applied load during DCB test.
SD = standard deviation.
t = distance from loading block pin to center line of top specimen arm.
U = strain energy.
VIS = point at which delamination is observed visually on specimen edge.
V_f = fiber volume fraction, %.
δ = load point deflection.
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.

4. Summary of Test Method

4.1 The DCB shown in Fig. 1 consists of a rectangular, uniform thickness, unidirectional laminated composite specimen containing a nonadhesive insert on the midplane that serves as a delamination initiator. Opening forces are applied to the DCB specimen by means of hinges (Fig. 1*a*) or loading blocks (Fig. 1*b*) bonded to one end of the specimen. The ends of the DCB are opened by controlling either the opening displacement or the crosshead movement, while the load and delamination length are recorded.

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

5. Significance and Use

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

5.2 This test method can serve the following purposes:

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

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

5.2.3 To 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.

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.

6.2 In the DCB test, as the delamination grows from the insert, a resistance-type fracture behavior typically develops where the calculated G_{Ic} first increases monotonically, and then stabilizes with further delamination growth. In this test method, a resistance curve (R curve) depicting G_{Ic} as a function of delamination length will be generated to characterize the

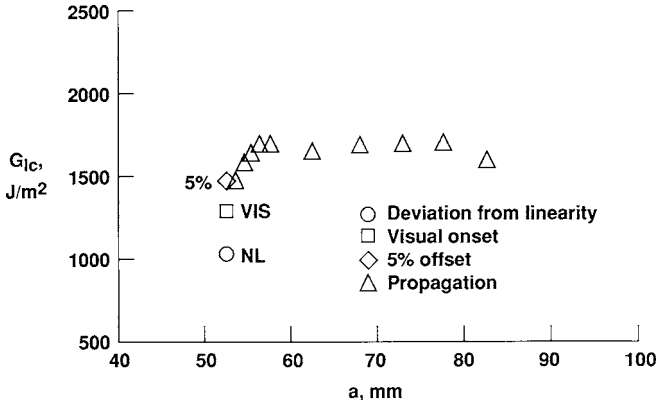


FIG. 2 Delamination Resistance Curve (*R*Curve) from DCB Test

initiation and propagation of a delamination in a unidirectional specimen (Fig. 2). The principal reason for the observed resistance to delamination is the development of fiber bridging (**1-3**).[3] This fiber bridging mechanism results from growing the delamination between two 0° unidirectional plies. Because most delaminations that form in multiply laminated composite structures occur between plies of dissimilar orientation, fiber bridging does not occur. Hence, fiber bridging is considered to be an artifact of the DCB test on unidirectional materials. Therefore, the generic significance of G_{Ic} propagation values calculated beyond the end of the implanted insert is questionable, and an initiation value of G_{Ic} measured from the implanted insert is preferred. Because of the significance of the initiation point, the insert must be properly implanted and inspected (8.3).

6.3 Three definitions for an initiation value of G_{Ic} have been evaluated during round-robin testing (**4**). These include G_{Ic} values determined using the load and deflection measured (1) at the point of deviation from linearity in the load-displacement curve (NL), (2) at the point at which delamination is visually observed on the edge (VIS) measured with a microscope as specified in 7.5, and (3) at the point at which the compliance has increased by 5 % or the load has reached a maximum value (5 %/max) (see Section 11). The NL G_{Ic} value, which is typically the lowest of the three G_{Ic} initiation values, is recommended for generating delamination failure criteria in durability and damage tolerance analyses of laminated composite structures (5.2.4). Recommendations for obtaining the NL point are given in Annex A2. All three initiation values can be used for the other purposes cited in the scope (5.2.1 and 5.2.2). However, physical evidence indicates that the initiation value corresponding to the onset of nonlinearity (NL) in the load versus opening displacement plot corresponds to the physical onset of delamination from the insert in the interior of the specimen width (**5**). In round-robin testing of AS4/PEEK thermoplastic matrix composites, NL G_{Ic} values were 20 % lower than VIS and 5 %/max values (**4**).

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

6.4 Delamination growth may proceed in one of two ways: (1) by a slow stable extension or (2) a run-arrest extension in which the delamination front jumps ahead abruptly. Only the first type of growth is of interest in this test method. An unstable jump from the insert may be an indication of a problem with the insert. For example, the insert may not be completely 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}.

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

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

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

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

7. Apparatus

7.1 *Testing Machine*—A properly calibrated test machine shall be used that can be operated in a displacement control mode with a constant displacement rate in the range from 0.5 to 5.0 mm/min (0.02 to 0.20 in./min). The testing machine shall conform to the requirements of Practices E4. The testing machine shall be equipped with grips to hold the loading hinges, or pins to hold the loading blocks, that are bonded to the specimen.

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

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

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

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

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

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.

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

8.4 *Specimen Dimensions:*

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

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

8.5 The laminate thickness shall normally be between 3 and 5 mm (0.12 and 0.2 in.). The variation in thickness for any given specimen shall not exceed 0.1 mm (0.004 in.). The initial delamination length, measured from the load line to the end of the insert, shall normally be 50 mm (2.0 in.). However, alternative laminate thicknesses and initial delamination lengths may be chosen that are consistent with the discussions given as follows. However, if load blocks are used to introduce the load, very low values of *a/h* are not recommended. For small values of *a/h* (<10), the data reduction procedures given in Section 13 may not be accurate.

8.5.1 For materials with low-flexural modulus or high interlaminar fracture toughness, it may be necessary to increase the number of plies, that is, increase the laminate thickness or decrease the delamination length to avoid large deflections of the specimen arms. The specimen thickness and initial delamination length, a_0, shall be designed to satisfy the following criteria (**6**):

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

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

where:

a_0 = initial delamination length,
h = specimen thickness, and
E_{11} = lamina modulus of elasticity in the fiber direction.

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

8.6 It is recommended that void content and fiber volume be reported. Void content may be determined using the equations of Test Methods D2734. The fiber volume fraction may be determined using a digestion per test in accordance with Test Method D3171.

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.

8.8 *Load Introduction:*

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

8.8.2 *Piano Hinges*—A pair of piano hinge tabs shall be bonded to the end of each specimen as shown in Fig. 1*a*. The hinge tabs shall be made of metal and shall be capable of sustaining the applied load without incurring damage. The maximum load anticipated during a DCB test of a material with a known modulus, E_{11} , and anticipated value of G_{Ic}, may be estimated by **(6)**.

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

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

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

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

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

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

8.8.4.2 *Surface Preparation of the Loading Hinge Tabs or Blocks*—The loading hinge tabs or blocks may be cleaned as in 8.8.4.1. If this procedure results in a bond failure between the specimen and the tabs, it may be necessary to apply a more sophisticated cleaning procedure based on degreasing and chemical etching. Consult Guide D2651 for the surface preparation procedure that is most appropriate for the particular metal used for the hinges.

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

9. Calibration

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

10. Conditioning

10.1 *Standard Conditioning Procedure*—Condition in accordance with Procedure C of Test Method D5229/D5229M unless a different environment is specified as part of the experiment. Store and test specimens at standard laboratory atmosphere of 23 ± 3°C (73 ± 5°F) and 50 ± 10 % relative humidity.

10.2 *Drying*—If interlaminar fracture toughness data are desired for laminates in a dry condition, use Procedure D of Test Method D5229/D5229M.

NOTE 3—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 Measure the width and thickness of each specimen to the nearest 0.05 mm (0.002 in.) at the midpoint and at 25 mm (1 in.) from either end. The variation in thickness along the length of the specimen shall not exceed 0.1 mm (0.004 in.). The average values of the width and thickness measurements shall be recorded.

11.2 Coat both edges of the specimen just ahead of the insert with a thin layer of water-based typewriter correction fluid, or equivalent, to aid in visual detection of delamination onset.

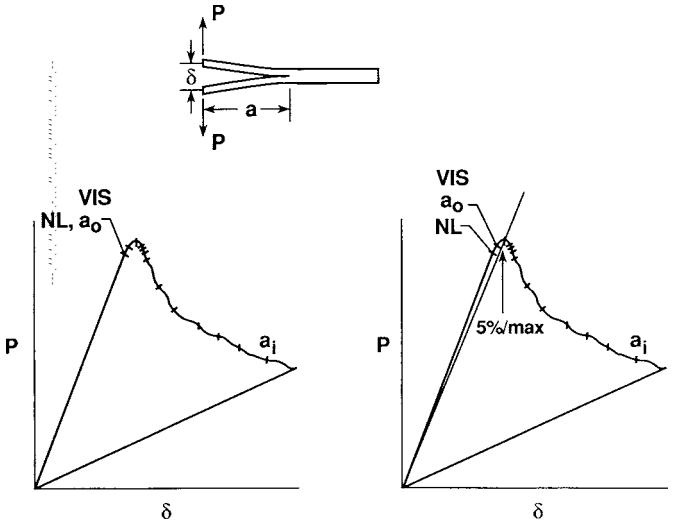
Mark the first 5 mm (0.2 in.) from the insert on either edge with thin vertical lines every 1 mm (0.04 in.). Mark the remaining 20 mm (0.8 in.) with thin vertical lines every 5 mm (0.2 in.). The delamination length is the sum of the distance from the loading line to the end of the insert (measured in the undeformed state) plus the increment of growth determined from the tick marks.

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

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

11.5 The end of the specimen opposite the grips should be supported before loading, as shown schematically in Fig. 3. The supported end may rise off the support as the load is applied. For laminates that are excessively long, the specimen may need to be supported during loading.

11.6 Set an 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. This device shall be capable of pinpointing the delamination front with an accuracy of at least ±0.5 mm (±0.02 in.).



(*a*) brittle matrix (*b*) tough matrix

FIG. 3 Load Displacement Trace from DCB Test

11.7 *Initial Loading:*

11.7.1 Load the specimen at a constant crosshead rate between 1 and 5 mm/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.

11.7.3 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, Fig. 3).

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.

11.7.4 The loading shall be stopped after an increment of delamination crack growth of 3 to 5 mm. If unstable delamination growth from the insert is observed, note in the report and loading shall be continued until the delamination length is increased by 3 to 5 mm beyond the arrest point. Note in the test report if the delamination length increment is outside the range of 3 to 5 mm.

11.7.5 Unload the specimen at a constant crosshead rate of up to 25 mm/min.

11.7.6 After unloading, 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 and if the specimen is removed from the fixture for this procedure.

NOTE 5—Mismatch between the two positions greater than 2 mm may be an indication of asymmetrical loading.

11.7.7 If the insert was properly implanted and inspected (see 8.3), but the R curve shows a decrease in apparent toughness with delamination length, the initial loading process may be replaced by wedge precracking (see Annex A2). Use of wedge precracking is not recommended and must be reported.

11.8 *Reloading:*

11.8.1 The specimen shall be reloaded at the same constant crosshead speed of 1 to 5 mm/min as the initial loading without stopping or unloading until the final delamination length increment has been reached. The load and the displacement values shall be recorded, including the unloading cycle. The position of the delamination shall be pinpointed with an accuracy of at least ±0.5 mm on the edge of the specimen.

11.8.2 Record the load and displacement values at which the onset of delamination movement from the precrack is observed on the edge of the specimen (VIS, Fig. 3).

11.8.3 On continuation of the loading, record the load and displacement values at as many delamination length increments as possible in the first 5 mm, ideally every 1 mm. Subsequently, record these load and displacement data at every 5 mm, until the delamination crack has propagated at least 45 mm from the tip of the precrack, and again at every 1-mm increment of crack growth for the last 5 mm of delamination propagation, up to total delamination length of 50 mm beyond the tip of the precrack (Fig. 3).

11.8.4 Finally, unload the specimen at a constant crosshead rate of up to 25 mm/min.

11.8.5 Mark the positions of the tip of the delamination crack after unloading on both edges of the specimen. Note in the report if these positions differ by more than 2 mm.

NOTE 6—Mismatch between the two positions greater than 2 mm may be an indication of asymmetrical loading.

11.8.6 Any permanent deformation of the specimen after unloading shall be noted in the report. Deviations of the delamination from the midplane of the laminate will invalidate the test results and shall be noted in the report. A replacement specimen shall be tested.

11.9 If an alternative method for monitoring delamination growth is used, such as crack growth gages bonded to the specimen edges, it should collect data in accordance with the principles, accuracy, and magnification as set out in detail above.

11.10 *Interpretation of Test Results*—Several initiation G_{Ic} values may be determined from the load-displacement plots and used along with subsequent propagation values to generate the *R* curve. G_{Ic} values corresponding to the points listed below shall be determined for testing from the starter film and from the Mode I precrack for each specimen. These initiation values are indicated on a typical *R* curve shown in Fig. 2 and are described below. For each of these techniques, the initial delamination length, a_0, should be used to calculate G_{Ic}.

11.10.1 *Deviation from Linearity (NL)*—An initiation (or onset) value for G_{Ic} should be calculated from the load and displacement at the point of deviation from linearity, or onset of nonlinearity (NL). This calculation assumes that the delamination starts to grow from the insert in the interior of the specimen at this point (**5**). The NL value represents a lower bound value for G_{Ic}. For brittle matrix composites, this is typically the same point at which the delamination is observed to grow from the insert at the specimen edges (Fig. 3*a*). For tough matrix composites, however, a region of nonlinear behavior may precede the visual observation of delamination onset at the specimen edges, even if the unloading curve is linear (Fig. 3*b*). Recommendations for obtaining the NL point are given in Annex A2.

11.10.2 *Visual Observation (VIS)*—A visual initiation value for G_{Ic} should be recorded corresponding to the load and displacement for the first point at which the delamination is visually observed to grow from the insert on either edge using the microscope or mirror, or both, specified in 7.5.

11.10.3 *5 % Offset/Maximum Load (5 %/Max)*—A value of G_{Ic} may be calculated by determining the intersection of the load-deflection 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 load-displacement curve (Fig. 3*b*). If the intersection occurs after the maximum load point, the maximum load should be used to calculate this value.

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, unless such flaw constitutes a variable being studied. Retests shall be performed for any specimen on which values are not calculated.

13. Calculation

13.1 *Interlaminar Fracture Toughness Calculations*—Three data reduction methods for calculating G_{Ic} values have been evaluated during round-robin testing (**4**). These consisted of a modified beam theory (MBT), a compliance calibration method (CC) and a modified compliance calibration method (MCC). Because G_{Ic} values determined by the three different data reduction methods differed by no more than 3.1 %, none of the three were clearly superior to the others. However, the MBT method yielded the most conservative values of G_{Ic} for 80 % of the specimens tested (**4**). Hence, the MBT data reduction method is recommended. The area method (**7**) is not recommended because it will not yield an initiation value of G_{Ic} or a delamination resistance curve.

13.1.1 *Modified Beam Theory (MBT) Method*—The beam theory expression for the strain energy release rate of a perfectly built-in (that is, clamped at the delamination front) double cantilever beam is as follows:

$$G_I = \frac{3P\delta}{2ba} \tag{6}$$

where:

P = load,
δ = load point displacement,
b = specimen width, and
a = delamination length.

In practice, this expression will overestimate G_I because the beam is not perfectly built-in (that is, rotation may occur at the delamination front). One way of correcting for this rotation is to treat the DCB as if it contained a slightly longer delamination, $a + |\Delta|$, where Δ may be determined experimentally by generating a leasts squares plot of the cube root of compliance, $C^{1/3}$, as a function of delamination length (Fig. 4). The compliance, C, is the ratio of the load point displacement to the applied load, δ/P. The values used to generate this plot should be the load and displacements corresponding to the visually observed delamination onset on the edge and all the propagation values. Calculate the Mode I interlaminar fracture toughness as follows (**8**):

$$G_I = \frac{3P\delta}{2b(a+|\Delta|)} \tag{7}$$

This approach also allows the flexural modulus, E_{1f}, to be determined as follows:

$$E_{if} = \frac{64(a+|\Delta|)^3\ P}{\delta b h^3} \tag{8}$$

The values of E_{1f} obtained should be independent of delamination length (**8**). However, E_{1f} may increase with delamination length because of fiber bridging.

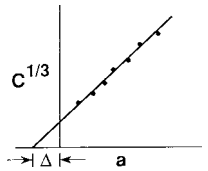


FIG. 4 Modified Beam Theory

13.1.2 *Compliance Calibration (CC) Method*—Generate a least squares plot of log (δ_i/P_i) versus log (a_i) using the visually observed delamination onset values and all the propagation values. Draw a straight line through the data that results in the best least-squares fit. Calculate the exponent n from the slope of this line according to $n = \Delta_y/\Delta_x$, where Δ_y and Δ_x are defined in Fig. 5. Calculate the Mode I interlaminar fracture toughness as follows (**9**):

$$G_I = \frac{nP\delta}{2ba} \tag{9}$$

13.1.3 *Modified Compliance Calibration (MCC) Method*—Generate a least squares plot of the delamination length normalized by specimen thickness, *a/h*, as a function of the cube root of compliance, $C^{1/3}$, as shown in Fig. 6, using the visually observed delamination onset values and all the propagation values. The slope of this line is A_1. Calculate the Mode I interlaminar fracture toughness as follows (**10**):

$$G_I = \frac{3P^2C^{2/3}}{2A_1bh} \tag{10}$$

13.2 *Statistics*—For each series of tests calculate the average value, standard deviation and coefficient of variation (in percent) for each property determined:

$$\bar{x} = \frac{\left(\sum_{i=1}^{n} x_1\right)}{n} \tag{11}$$

$$S_{n-1} = \sqrt{\frac{\left(\sum_{i=1}^{n} x_i^2 - n\,x^{-2}\right)}{(n-1)}} \tag{12}$$

$$CV = \frac{100 \times S_{n-1}}{x} \tag{13}$$

where

$\bar{x}$ = sample mean (average),
S_{n-1} = sample standard deviation,
CV = sample coefficient of variation, in percent,
n = number of specimens, and
x_i = measured or derived property.

14. Report

14.1 Report the following information, or references pointing to other documentation containing this information, to the maximum extent applicable. (Reporting of items beyond the control of a given testing laboratory, such as might occur with material details of panel fabrication parameters, shall be the responsibility of the requester):

NOTE 7—Guides E1309, E1434, and E1471 contain data reporting recommendations for composite materials and composite materials mechanical testing.

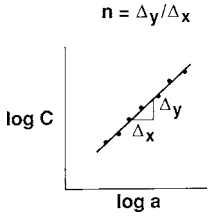


FIG. 5 Compliance Calibration

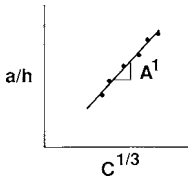


FIG. 6 Modified Compliance Calibration

14.1.1 The revision level or date of issue of this test method,
14.1.2 The date(s) and location(s) of the test,
14.1.3 The name(s) of the test operator(s),
14.1.4 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing,
14.1.5 Description of the fabrication steps used to prepare the laminate including: fabrication start date, fabrication end date, process specification, cure cycle, consolidation method, and a description of the equipment used;
14.1.6 Ply orientation stacking sequence of the laminate,
14.1.7 If requested, report density, reinforcement volume fraction, and void content test methods, specimen sampling method and geometries, test parameters, and test data,
14.1.8 Average ply thickness of the material,
14.1.9 Results of any non-destructive evaluation tests,
14.1.10 Method of preparing the test specimens, including specimen labeling scheme and method, specimen geometry, sampling method, coupon cutting method, identification of tab geometry, tab material, and tab adhesive used;
14.1.11 Calibration dates and methods for all measurement and test equipment,
14.1.12 Type of test machine, alignment data, and data acquisition sampling rate and equipment type,
14.1.13 Measured dimensions for each test specimen,
14.1.14 Conditioning parameters and results, and the procedure used if other than that specified in the test method,
14.1.15 Relative humidity and temperature of the testing laboratory,
14.1.16 Environment of the test machine environmental chamber (if used) and soak time at environment,
14.1.17 Number of specimens tested,
14.1.18 Speed of testing,
14.1.19 Transducer placement on the specimen, transducer type, and calibration data for each transducer used,
14.1.20 Tabulated data of force versus displacement and force-displacement curves for each specimen, and
14.1.21 Tabulated data of stress versus strain and stress versus strain curves for each flexural modulus specimen (if applicable).

14.2 A recommended data reporting sheet is shown in Annex A1. The report shall include the following (reporting of items beyond the control of a given testing laboratory, such as might occur with material details or panel fabrication parameters, shall be the responsibility of the requestor):

14.2.1 *Material*—Complete identification of the material tested; including prepreg manufacturer, material designation,

manufacturing process, fiber volume fraction, and void content. Include the method used to determine fiber volume fraction and void content.

14.2.2 *Coupon Data*—Average nominal thickness and width of each specimen, and maximum thickness variation down the length of the beam, type, and thickness of insert.

14.2.3 *Test Procedure*—Type of load introduction (piano hinges or blocks) and dimensions, drying procedure, relative humidity, test temperature, and loading rate.

14.2.4 *Test Results:*

14.2.4.1 Load-displacement curves indicating load and displacement at first deviation from nonlinearity (NL) and at visual onset of delamination from either edge (VIS). Upon unloading, if the load does not return to zero, damage may have been induced in the beam arms. Note this on the data reduction sheet.

14.2.4.2 Intercept, Δ, for each specimen if modified beam theory (MBT) method is used to reduce the data.

14.2.4.3 Slope, n, of log (δ_i/P_i) versus log (a_i) plot for each specimen if compliance calibration (CC) method is used to reduce the data.

14.2.4.4 Slope, A_1, for each specimen if modified compliance calibration (MCC) method is used to reduce the data.

14.2.4.5 Delamination resistance curve for each specimen, including the NL, VIS, and 5 %/max values of G_{Ic} defined in 13.1, measured from both the insert and the precrack, with the following exceptions:

14.2.4.6 If a postmortem check of the tested specimen reveals any tears, folds, or irregular shape at the end of the insert (that is, the insert is not straight and parallel) where the delamination initiated, then no valid initiation value may be reported.

14.2.4.7 If any propagation value is less than the NL value of G_{Ic}, then no valid initiation value may be reported.

14.2.4.8 Report the number of specimens tested and the mean, standard deviation, and coefficient of variation (standard deviation divided by the mean) of quantities in 14.2.4.2 – 14.2.4.5.

15. Precision and Bias[4]

15.1 Table 1 shows results from round-robin tests conducted in 1987 on AS4/BP907, in 1989 on AS4/3501-6, in 1990 on AS4/PEEK specimens with aluminum inserts, and in 1991 on AS4/PEEK specimens with polyimide film inserts. Table 1 also shows the number of laboratories involved, the number of tests per laboratory, and other pertinent information on the type and thickness of the inserts used. These interlaboratory test programs were designed using Practice E691 as a guide. Further information on the statistical interpretation of the results may be found in Ref **(4)**.

15.2 *Precision*—The following should be used for judging the acceptability of results (see Practice E177):

15.2.1 *Repeatability*—Duplicate test results (obtained by the same operator using the same equipment on the same day) from an individual laboratory for the same material should be considered suspect if they differ by more than the r value for that material, where $r = 2.8\ S_r$, and S_r is the average of the standard deviations for each participating laboratory.

15.2.2 *Reproducibility*—The average result reported by one laboratory for a given material should be considered suspect if it differs from the average measurement of another laboratory, or from measurements in the same laboratory taken by a different operator using different equipment, for the same material by more than the R value for that material, where

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D30-1001.

TABLE 1 Summary of Round-Robin Data

Round	Material	Number of Laboratories	Tests/ Laboratory	Insert	Average Mean, G_{Ic}, kJ/m^2		S_r	$(CV)_r$, %	S_R	$(CV)_R$,%
I	AS4/BP907	9	3	25-μm PTFE	0.400	[A]	0.028	7.0	0.077	19.3
II	AS4/3501-6	3	3	13-μm Kapton	0.085	[A]	0.015	17.6	0.014	16.5
II	AS4/PEEK	3	4	13-μm Kapton	0.983	[B]	0.132	13.4	0.178	18.1
III	AS4/PEEK	16	4	13-μm aluminum foil	1.439	[B]	0.187	13.4	0.261	18.1
III	AS4/PEEK	5	4	7-μm aluminum foil	1.727	[B]	0.226	13.0	0.140	8.1
IV	AS4/PEEK	10	3	13-μm Kapton	1.303	[B]	0.180	13.8	0.207	15.9
V	AS4/PEEK	9	5	7.5-μm Upilex	1.182	[B]	0.126	10.8	0.111	9.4
V	AS4/PEEK	9	5	13-μm Upilex	1.262	[B]	0.132	10.5	0.110	8.7

[A] VIS values using CC method.
[B] NL values using MBT method.
Round I & II—ASTM round robin.
Round III—ASTM and JIS data from international round robin.
Round IV—static tests from ASTM fatigue round robin.
Round V—ASTM/JIS and ESIS data from international round robin.

$R = 2.8\ S_R$, and S_R is the standard deviation from the mean value of G_{Ic} obtained by all participating laboratories.

NOTE 8—These precision data are approximated based on limited data from round-robin test programs (**4**), but they provide a reasonable basis for judging the significance of the results. The ability to measure the delamination front position, as well as the actual variation in material properties from one panel to another, may yield G_{Ic} values with greater variations. No round-robin data were generated for glass epoxy materials, and thus, the applicability of Table 1 to these materials is not known.

15.3 *Bias*—No other test method exists for determining the Mode I interlaminar fracture toughness of composite laminates. Hence, no determination of the bias inherent in the DCB test is available.

16. Keywords

16.1 composite materials; delamination; double cantilever beam; interlaminar fracture toughness; Mode I

ANNEXES

(Mandatory Information)

A1. LARGE DISPLACEMENT AND END BLOCK CORRECTIONS

A1.1 Large displacement effects shall be corrected by the inclusion of a parameter, F, in the calculation of G_I (**11**).

$$F = 1 - \frac{3}{10}\left(\frac{\delta}{a}\right)^2 - \frac{3}{2}\left(\frac{\delta t}{a^2}\right) \quad \text{(A1.1)}$$

where t is shown in Fig. A1.1 for piano hinges. This parameter, F, accounts for both the shortening of the moment arm as well as tilting of the end blocks. For specimens with loading blocks, the distance from the end of the insert to the load line shall be at least 50 mm for the influence of the blocks to be neglected. If not, a second parameter, N, a displacement correction, shall also be included to account for the stiffening of the specimen by the blocks (**11**).

$$N = 1 - \left(\frac{L'}{a}\right)^3 - \frac{9}{8}\left[1 - \left(\frac{L'}{a}\right)^2\right]\left(\frac{\delta t}{a^2}\right) - \frac{9}{35}\left(\frac{\delta}{a}\right)^2 \quad \text{(A1.2)}$$

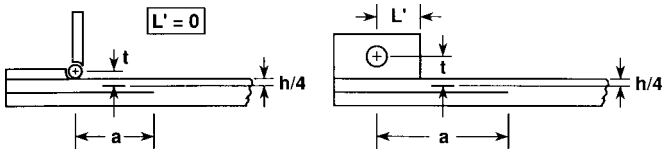
where L' and t are shown in Fig. A1.1 for end blocks.

A1.2 To apply these corrections to either the modified beam theory (MBT) or the compliance calibration (CC or MCC) methods, do the following:

A1.2.1 If piano hinges were used to introduce the opening load, multiply G_I by F to obtain the corrected value of G_I.

A1.2.2 If end blocks were used to introduce the opening load, determine the corrected compliance, C/N, where plotting compliance versus crack length for determining Δ, n, or A_1 (see 13.1.1 – 13.1.3), then multiply G_I by F/N to obtain the corrected value of G_I.

A1.3 These corrections are small for short delamination lengths in 3-mm-thick specimens of 60 % V_f carbon composites, but they may be larger for thin (that is, more flexible) specimens or for long delamination lengths.



(*a*) piano hinge (*b*) end block

FIG. A1.1 Methods for Introducing Opening Load to DCB Specimen

DCB STANDARD DATA REPORTING SHEET	LAB:	DATE:

Material:	Producer:		Panel No.:	Max Cure Temp.:		FAW =	V_f =
Specimen No.	Avg. b (mm)	Avg. h (mm)	Max Δh (mm)	Insert Material		Insert Thickness	
Hinge Type:	Hinge Size:		Block Size:	Surface Prep.:		Adhesive:	
Test Temp.:	Test %RH:		Load Rate:	a_0 =	Δ=	n =	A_1 =

a_0 (mm)	δ (mm)	P (N)	δ/a_0	G_{Ic} (kJ/m^2)	MBT	CC	MCC	Comments
				NL				
				VIS				
				5%				

a (mm)	δ (mm)	P (N)	δ/a	G_{Ic} (kJ/m^2)	MBT	CC	MCC	Comments
				Prop				
				Prop				
				Prop				
				Prop				
				Prop				
				Prop				
				Prop				
				Prop				
				Prop				
				Prop				
				Prop				
				Prop				

FIG. A1.2 DCB Standard Data Reporting Sheet

A2. RECOMMENDATIONS FOR OBTAINING THE NL POINT

A2.1 Physical evidence from X-ray imaging for two types of materials (carbon fiber/epoxy and carton fiber PEEK) shows that the onset of the delamination from the starter film in the interior of the specimen occurs close to the NL point and before the VIS point. The NL point will frequently yield the lowest, most conservative values of the interlaminar fracture toughness. However, it may be difficult to determine the NL point reproducibly on the load-displacement curve.

A2.2 Coefficients of variations of up to 10 % are not uncommon. However, a plot of the analog signals for load versus displacement, typically recorded on a paper chart of an *X-Y* recorder, may yield more consistent results with less variability than those obtained by fitting a curve through the data points recorded electronically during the test with a digital data acquisition device. Performing a linear fit on the load-displacement curve starting at a finite load to avoid nonlinearity as a result of play and using a consistent criterion for deviation from linearity, such as the half thickness of the plotter trace, may yield more consistent results.

A3. GUIDELINES FOR WEDGE PRECRACKING

A3.1 If an alternative to load-induced precracking is necessary (see comments in 11.7.7), the following procedure is recommended for wedge opening. The specimen is clamped at 5 mm beyond the tip of the starter film. The width of the wedge that is driven into the specimen shall be at least the same as that of the specimen and the opening angle shall be as small as possible without the wedge actually touching the tip of the delamination. The wedge may be driven by hand, by tapping on the side, or by using a suitable fixture and a testing machine. The wedge is driven into the specimen until the tip of the wedge is about 2 to 3 mm in front of the clamp. The wedge precrack will usually extend a few mm into the clamp but should be short enough to allow a delamination length increment of at least 50 mm beyond the tip of the precrack. It may be difficult to produce a suitable precrack by wedge opening. The precrack may not always lie in the midplane of the specimen. Deviations of the precrack from the midplane will invalidate the test results and shall be noted in the report.

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