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} $[M/T^2]$—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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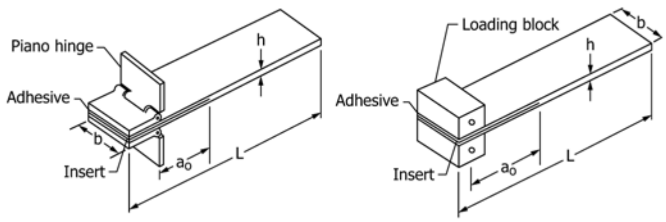
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



(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

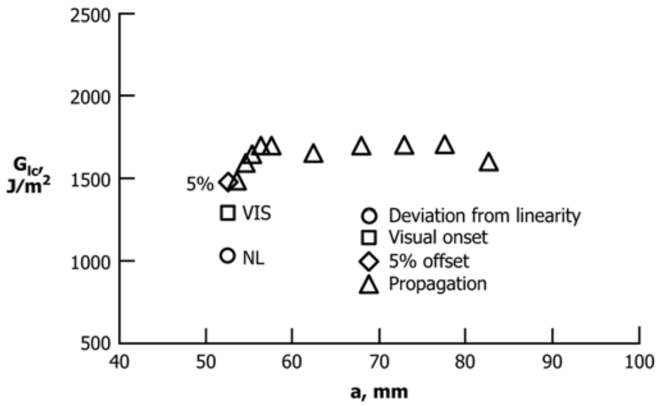


FIG. 2 Delamination Resistance Curve (*R*Curve) from DCB Test

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**).

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

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