

# Standard Test Method for Determination of the Mode II Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites<sup>1</sup>

This standard is issued under the fixed designation D7905/D7905M; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

- 1.1 This test method describes the determination of the mode II interlaminar fracture toughness,  $G_{IIc}$ , of unidirectional fiber-reinforced polymer matrix composite laminates under mode II shear loading using the end-notched flexure (ENF) test (Fig. 1).
- 1.2 This method is limited to use with composites consisting of unidirectional carbon-fiber- and glass-fiber-reinforced laminates. 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 Section 6).
- 1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.
- 1.3.1 Within the text the inch-pound units are shown in brackets.
- 1.4 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:<sup>2</sup>

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D883 Terminology Polyting to Plastics

D883 Terminology Relating to Plastics

D2584 Test Method for Ignition Loss of Cured Reinforced Resins

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

D5687/D5687M Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation

D7264/D7264M Test Method for Flexural Properties of Polymer Matrix Composite Materials

**E4** Practices for Force Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing E18 Test Methods for Rockwell Hardness of Metallic Materials

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-modulous 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 precendence over the other terminology standards.

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

FIG. 1 ENF Test Fixture and Specimen Nomenclature

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 Compliance Calibration (CC) Method—the method of data reduction where the relationship between specimen compliance  $[T^2/M]$  and delamination length [L] is determined prior to testing by measuring specimen compliance  $[T^2/M]$  at multiple simulated delamination lengths.
- 3.2.2 Mode II Interlaminar Fracture Toughness,  $G_{IIc}$  [M/ $T^2$ ]—the critical value of strain energy release rate, G, [M/ $T^2$ ] for delamination growth [L] due to an in-plane shear force [M/ $T^2$ ] or displacement [L] oriented perpendicular to the delamination front.
- 3.2.3 Non-precracked (NPC) toughness [M/T²]—an interlaminar fracture toughness value that is determined from the preimplanted insert.
- 3.2.4 Precracked (PC) Toughness [M/T<sup>2</sup>]—an interlaminar fracture toughness value that is determined after the delamination has been advanced from the preimplanted insert.
- 3.2.5 Strain Energy Release Rate,  $G [M/T^2]$ —the loss of strain energy,  $dU [ML^2/T^2]$ , in the test specimen per unit of specimen width [L] for an infinitesimal increase in delamination length, da [L], for a delamination growing self-similarly under constant displacement [L]. In mathematical form,

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

where:

U = total elastic strain energy in the specimen;

a = delamination length; and

B = specimen width.

- 3.3 Symbols:
- 3.3.1~A—intercept of the linear fit of compliance versus crack length cubed data
  - 3.3.2 a—delamination length
  - 3.3.3  $a_i$ —insert length in the trimmed specimen
- 3.3.4  $a_j$ —the j<sup>th</sup> crack length used during compliance calibration (j = 1,2)

- 3.3.5  $a_o$ —delamination length used in fracture test
- $3.3.6~a_{calc}$ —crack length calculated from an unloading curve after the NPC test
  - 3.3.7  $a_{PC}$ —actual crack length used during the PC test
- 3.3.8  $a_{vis}$ —visually determined crack length after the NPC test
  - 3.3.9 B—specimen width
  - 3.3.10 *C*—specimen compliance
- 3.3.11  $C_0$ —specimen compliance during load-up of the fracture test (See Figure 6 in 13.1)
- 3.3.12  $C_u$ —specimen compliance from unloading after the non-precracked test
- 3.3.13  $\delta$ —displacement of loading roller during testing perpendicular to the plane of the specimen (Fig. 1)
  - 3.3.14  $E_{1f}$ —flexural modulus of the specimen
  - 3.3.15 *G*—total strain energy release rate
  - 3.3.16  $G_{IIC}$ —mode II interlaminar fracture toughness
- 3.3.17  $G_Q$ —candidate mode II interlaminar fracture toughness
- 3.3.18  $\%G_Q$ —peak percentage of  $G_Q$  achieved during compliance calibration
  - 3.3.19 *h*—specimen half-thickness (Fig. 2)
  - 3.3.20 *L*—specimen half-span (Fig. 2)
- 3.3.21  $L_c$ —distance from the center of the support roller at the cracked end of the specimen to the cracked end of the specimen (Fig. 2)
- 3.3.22  $L_u$ —distance from the center of the support roller at the uncracked end of the specimen to the uncracked end of the specimen (Fig. 2)
- $3.3.23 \, m$ —slope of the linear fit of compliance versus crack length cubed data
- 3.3.24 *P*—force applied to center loading roller and perpendicular to the plane of the specimen (Fig. 1)
  - 3.3.25  $P_c$ —critical force for mode II fracture
- 3.3.26  $P_j$ —the compliance calibration force used at crack length  $a_i$
- $3.3.27\ P_{Max}$ —maximum value of force on the force-displacement curve
  - 3.3.28  $r_1$ —radius of the loading roller (Fig. 2)

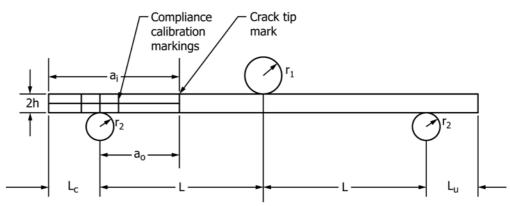


FIG. 2 ENF Specimen, Fixture, and Dimensions

- 3.3.29  $r_2$ —radius of the support rollers (Fig. 2)
- 3.3.30  $r^2$ —correlation coefficient of linear fit of compliance versus crack length cubed
- $3.3.31 \Delta s$ —Maximum measured difference in crack length along the delamination front of the precrack
  - 3.3.32 *U*—total elastic strain energy in the specimen

# 4. Summary of Test Method

- 4.1 The ENF specimen shown in Fig. 1 consists of a rectangular, uniform thickness, unidirectional laminated composite specimen containing a non-adhesive insert at the midplane that serves as a delamination initiator. Forces are applied to the specimen through an ENF fixture under displacement controlled loading.
- 4.2 Delamination growth is not stable in the ENF test. A method is presented so that the initiation values of the mode II interlaminar fracture toughness are obtained from the preimplanted insert as well as from a precrack.
- 4.3 A record of the applied force versus center roller displacement is to be obtained using an x-y recorder or equivalent real-time plotting device, or else it may be obtained and stored digitally. The mode II interlaminar fracture toughness,  $G_{IIc}$ , is obtained using the compliance calibration (CC) method. This is the only acceptable method of data reduction for this test (1).
- 4.4 This standard recommends that static mode II precracking is performed and a recommended method is described. Other precracking methods may be used provided that a record of the shape of the precracked delamination front is obtained prior to the PC test. Precracking methods that typically leave crack front markings for post-test evaluation of these values include mode I and fatigue mode II.

# 5. Significance and Use

5.1 Susceptibility to delamination is one of the major design concerns for many advanced laminated composite structures. Knowledge of a laminated composite material's resistance to interlaminar fracture is useful for product development and

material selection. Furthermore, a measurement of the mode II interlaminar fracture toughness that is independent of specimen geometry or method of force introduction is useful for establishing design allowables used in damage tolerance analyses of composite structures. Knowledge of both the non-precracked and precracked toughnesses 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  $G_{Hc}$  of a particular composite material;
- 5.2.2 To compare quantitatively the relative values of  $G_{Hc}$  for composite materials with different constituents;
- 5.2.3 To compare quantitatively the values of  $G_{Hc}$  obtained from different batches of a specific composite material, for example, to use as a material screening criterion or to develop a design allowable; and
- 5.2.4 To develop delamination failure criteria for composite damage tolerance and durability analyses.

### 6. Interferences

- 6.1 Linear elastic behavior is assumed in the calculation of G used in this method. This assumption is valid when the zone of damage or nonlinear deformation at the delamination front, or both, is small relative to the smallest specimen dimension, which is typically the specimen's thickness for the ENF test.
- $6.2~G_{Hc}$  is obtained for both non-precracked and precracked specimens based on the maximum load point.  $G_{Hc}$  based on the nonlinear load point or other measures, such as a compliance offset, may also be obtained if desired. However, definitions of this type have not been related to any specific physical occurrences in the ENF test.
- 6.3 The three loading noses in the ENF test fixture may be fixed, rotatable, or rolling. Fixed loading noses or pins supported in a v-groove are recommended, and loading noses of this type were used in the interlaboratory test program that was conducted in support of this standard. The type of supports that are used is to be reported as described in Section 14. The loading noses should uniformly contact the specimen across its width. Lack of uniform contact can affect results, most commonly due to non-uniform loading across the width of the

<sup>&</sup>lt;sup>3</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

specimen. Formulas used in this standard assume a uniform line loading across the entire specimen width at the loading nose and at the specimen supports; deviations from this type of loading are beyond the scope of this standard.

- 6.4 There is an inherent error associated with the use of Eq 7 to obtain the calculated crack length, and it is not expected that the calculated crack length will exactly correspond to the true length of the precrack. However, since toughness is computed by CC, it has been shown (2) that this error in crack length will not affect the accuracy of the computed toughness provided that the recommended approach is followed.
- 6.5 For very tough composites, large deformations at the onset of delamination growth could affect the accuracy of the ENF test. For typical unidirectional glass and carbon reinforced unidirectional composites, it has been shown (1) that the combined effects of friction and geometric nonlinearities will affect the accuracy of the recommended approach by approximately 2.5% or less for glass-reinforced polymer matrix composites with toughnesses up to 1.45 kJ/m<sup>2</sup> [8.28 in.-lbf/ in.<sup>2</sup>] and by 3% or less for polymer matrix composites with carbon reinforcement with toughnesses up to 2.10 kJ/m<sup>2</sup> [12.0 in.-lbf/in.<sup>2</sup>]. Testing of composites that exhibit greater toughness may produce somewhat larger errors. One means of checking for nonlinearities is to examine the difference between the nonlinear point and the maximum load point. If this is found to be greater than approximately 5% of  $P_{Max}$ , further investigations may be in order to determine the reason for the discrepancy, for example, material nonlinearity, geometric nonlinearity, or subcritical crack advance. The results of this investigation may be used to choose a new test geometry, for example to eliminate geometric nonlinearities, or to choose a definition of critical load that is different from  $P_{Max}$ , for example in the case of subcritical crack advance.
- 6.6 A precracking method that only produces a short crack "jump," e.g., by positioning a specimen with a crack tip close to the center loading roller, may produce precracked toughness values that are significantly higher than those that will be produced for a long crack jump following the recommended procedure (2,3).
- 6.7 The toughness measured using this method is sensitive to reinforcement volume and void content. Consequently, the test results may reflect manufacturing quality as much as material properties.
- 6.8 Number of Points for CC—The use of a three-point CC was studied extensively in References (2,4) and resulted in the recommended approach (subsection 11.9). However, equivalent results will be obtained with a five-point CC, and one may use this approach following Note 4 (11.9).
- 6.9 The toughness values obtained by this test method for delamination growth at 0°/0° interfaces may not be representative of the toughness required for delamination growth at interfaces with different relative ply orientations.

# 7. Apparatus

7.1 Testing Machine—A properly calibrated test machine shall be used which can be operated in a displacement control

mode with a constant displacement rate in the range from 0.025 to 1.6 mm/min [0.001 to 0.063 in./min]. The testing machine will conform to the requirements of Practices E4.

- 7.2 The testing machine shall be equipped with a loading fixture as shown in Fig. 1 and Fig. 2.
- 7.2.1 A fixture geometry with a nominal specimen span length (2L) of 100 mm [4 in.] and a nominal half-span length (L) of 50 mm [2 in.] is required.
- 7.2.2 The loading roller shall have a radius,  $r_I$ , in the range of 4.7 to 9.6 mm [0.185 to 0.378 in.]. The support rollers shall have the same radius,  $r_2$ , which shall be in the range of 3.0 to 6.4 mm [0.118 to 0.250 in.]. The loading roller shall be centered between the two support rollers (Fig. 2). All rollers shall have finely ground surfaces free of indentation and burrs with all sharp edges relieved, with a hardness of 60 to 62 HRC, as specified in Test Methods E18. Loading and support rollers may be arranged in a fixed, rotatable, or rolling arrangement, where rotation may occur only about the roller center points as viewed in the orientation of Fig. 1 and Fig. 2. All other movement of the support rollers shall be restrained, and loading rollers shall only be free to move vertically when viewed in the orientation of Fig. 1 and Fig. 2 (i.e., perpendicular to the plane of the specimen).
- 7.2.3 The system compliance, defined as the compliance of the load frame with the test fixture installed, shall be less than 3% of the measured compliance of the specimens that are tested. The system compliance shall be determined by using an essentially rigid calibration bar with the ENF test fixture and a span length (2L) of 100 mm [4.0 in.]. It is recommended that the calibration bar is at least as stiff as a steel bar with a moment of inertia, I, equal to 6 cm<sup>4</sup> [0.144 in.<sup>4</sup>]. When this is the case, the system compliance can be determined as the slope of the deflection versus force data from the test of the calibration bar in the ENF fixture. For calibration bars with a lower moment of inertia, the bar's compliance should be accounted for. Here, the system compliance may be computed as the slope of the deflection versus force data from the test of the calibration bar minus the compliance of the calibration bar, defined as  $L^3/(6EI)$ , where L is the half-span length and E and I are the Young's modulus and moment of inertia, respectively, of the calibration bar. The system compliance shall then be compared to the minimum compliance from all specimens tested to ensure that the 3% requirement is met. It is recommended that the system compliance tests be performed with a nominal loading rate of 0.05 mm/min [0.002 in./min], but rates in the range of 0.02 to 0.08 mm/min [0.0008 to 0.003 in./min]
- 7.2.4 The fixture cannot have rotational bearings that allow rotation about an axis parallel to the length direction of the specimen.
- 7.2.5 It is recommended that the test fixture be equipped with alignment features to ensure that (1) the loading and support rollers are parallel, and (2) the longitudinal direction of the specimen is perpendicular to the roller direction (3).
- 7.3 Force Indicator—The testing machine's force-sensing device shall be capable of indicating the total force carried by the test specimen. This device shall be essentially free from inertia-lag at the specified rate of testing and shall indicate the

force with an accuracy over the force range(s) of interest of within  $\pm$  1% of the indicated value. Forces are dependent on the specimen geometry and toughness. A method to calculate the expected forces can be found in Annex A1.

- 7.4 Load Point Displacement Indicator—The load point displacement may be obtained from the crosshead separation of the load frame provided that the compliance requirement of subsection 7.2.3 is satisfied. Otherwise, the load point displacement shall be taken from a properly calibrated external gage or transducer and/or a stiffer test fixture and/or load frame should be used. The load point displacement indicator shall indicate the load point displacement with an accuracy of  $\pm$  1% at the displacement at which delamination growth occurs.
- 7.5 Force versus Load Point Displacement Record—A digital record of force versus load point displacement shall be stored for subsequent post-processing.
- 7.6 The micrometer(s) or caliper(s) used to measure specimens prior to testing shall have a flat anvil interface for the measurement of smooth surfaces or a suitably sized ball interface for the measurement of rough surfaces, such as the bag side of a laminate. The accuracy of the instruments shall be suitable for reading to within  $\pm$  1% of the sample width and thickness. For typical specimen geometries, an instrument with an accuracy of  $\pm$  2.5 µm [0.0001 in.] is desirable for thickness measurements, while an instrument with an accuracy of  $\pm$  25 µm [0.001 in.] is desirable for width measurements.

# 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 Specimen and Test Configuration—Test laminates must contain an even number of plies and must be unidirectional, with delamination growth occurring in the  $0^{\circ}$  (zero degree) direction. Specimen dimensions shall conform to those presented in Fig. 3 and Fig. 4, which are chosen such that placement of the specimen within the fixture will be as defined in Table 1 and Fig. 2.

# 8.3 Manufacturing:

- 8.3.1 A flat composite plate shall be manufactured with a preimplanted non-adhesive film insert. Specimens are to be cut from these plates as shown in Fig. 3 and Fig. 4. Fabrication and machining are to be performed following Guide D5687/D5687M.
- 8.3.2 A non-adhesive film insert shall be implanted at the midplane of the laminate during layup to form an initiation site for the delamination (Fig. 3 and Fig. 4). The film thickness shall be no greater than  $13 \mu m$  [0.0005 in.]. A polymer film is recommended for the insert to avoid problems with folding or crimping at the cut end of the insert. For epoxy matrix

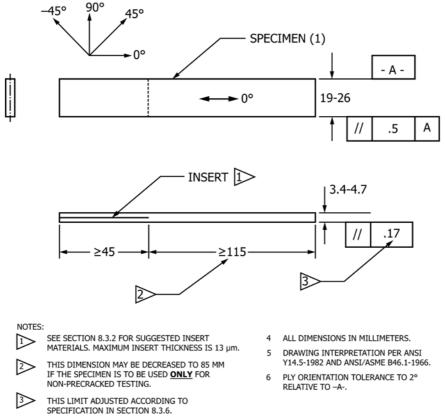


FIG. 3 Specimen—ENF Test (SI units)

# D7905/D7905M - 14 90° 45° SPECIMEN (1) - A -. 0° 0.75-1.00 .02 INSERT 1> 0.133-0.185 .007 ≥4.6 NOTES: SEE SECTION 8.3.2 FOR SUGGESTED INSERT ALL DIMENSIONS IN INCHES MATERIALS. MAXIMUM INSERT THICKNESS IS DRAWING INTERPRETATION PER ANSI 0.0005 INCHES

FIG. 4 Specimen—ENF Test (inch-pound units)

THIS DIMENSION MAY BE DECREASED TO 3.35

FOR NON-PRECRACKED TESTING.

THIS LIMIT ADJUSTED ACCORDING TO SPECIFICATION IN SECTION 8.3.6.

INCHES IF THE SPECIMEN IS TO BE USED ONLY

TABLE 1

Parameter	Value or Range
L	50 mm [2.0 in.]
$L_c$	≥ 15 mm [≥ 0.6 in.]
	≥ 45 mm [≥ 1.8 in.] when the same specimen is to
$L_u$	be used for non-precracked and precracked testing. Otherwise:
	≥ 15 mm [≥ 0.6 in.]
$a_o$	30 mm [1.2 in.]

composites cured at or below 177°C [350°F], a thin film made of polytetrafluoroethylene (PTFE) is recommended. For composites with polyimide, bismaleimide, or thermoplastic matrices that are manufactured at relatively high temperatures, i.e., greater than 177°C [350°F], a thin polyimide film is recommended. 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. Caution should be used, as mold release agents containing silicone may contaminate the laminate by migration through the individual layers. It is often helpful to coat the 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. It also is often necessary to decohere the light bond that might form between the insert and the composite (2). For materials outside the scope of this standard, different film materials and procedures may be required.

8.3.3 The plate shall be made in such a way that the specimen dimensions presented in Fig. 3 and Fig. 4 may be

achieved. Manufacturing large panels with a full-width insert in the center of the length direction is recommended to prevent thickness variations in the test specimens. After manufacture, these panels are cut width-wise along the centerline of the insert to create two plates, each with an edge view as shown in Fig. 3 and Fig. 4. A typical panel would be 400 mm [16 in.] long in the  $0^{\circ}$  direction with a 100 mm [4 in.] insert. Depending on the saw blade and amount trimmed at the edges, this will yield two plates that are approximately 200 mm [8 in.] long with an initial insert length ( $a_i$ ) of approximately 50 mm [2 in.].

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PLY ORIENTATION TOLERANCE TO 2°

RELATIVE TO -A-

8.3.4 Prior to cutting the plate into specimens, the end of the insert should be accurately located and marked, and markings should be placed on the plate such that location of each specimen relative to the original plate geometry will be identifiable subsequent to cutting.

8.3.5 Individual specimens are to be cut such that they fall within the range of allowable lengths and widths specified in Fig. 3 and Fig. 4.

8.3.6 Subsequent to cutting, measure the width, B, at the three points of each specimen that will correspond to the contact locations of the three rollers when the specimen is tested in the non-precracked configuration. Measure the thickness, 2h, of each specimen at six points, with two thickness measurements at each of the points where the width was measured; one on the left side and one on the right side. The individual and average values of the three width measurements and the six 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.

- 8.4 Labeling—Label the specimens so that they will be distinct from each other and traceable back to the raw material, and in a manner that will both be unaffected by the test and not influence the test.
- 8.5 *Void Content*—It is recommended that void content and fiber volume be reported. Void content may be determined using Test Method D2734 and fiber volume fraction may be determined using Test Method D3171.

## 9. Calibration

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

# 10. Conditioning

- 10.1 The recommended pre-test condition is effective moisture equilibrium at a specific relative humidity as established by Test Method D5229/D5229M, however, if the test requestor does not explicitly specify a pre-test conditioning environment, no conditioning is required and the test specimens may be tested as prepared.
- 10.2 The pre-test specimen conditioning process, to include specified environmental exposure levels and resulting moisture content, shall be reported with the test data.

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

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

### 11. Procedure

- 11.1 Parameters to be Specified Prior to Test:
- 11.1.1 The specimen sampling method, specimen geometry, and conditioning travelers (if required),
  - 11.1.2 The properties and data reporting format desired,
  - 11.1.3 The environmental conditioning test parameters, and
- 11.1.4 If performed, the sampling method, specimen geometry, and test parameters used to determine density and constituent volumes.
- 11.2 Condition the specimens as required. Store the specimens in the conditioned environment until test time, if the test environment is different than the conditioning environment.
  - 11.3 Specimen Preparation:
- 11.3.1 Measure and record the width and thickness of each specimen as specified in subsection 8.3.6.
- 11.3.2 A light coating of white or silver spray paint, or equivalent, shall be applied to the specimen edges. This is to assist in the visual detection of the delamination tip and in making compliance calibration (CC) markings (Fig. 2). Once the paint is dry, the tip of the insert shall be marked with a thin vertical pencil line. The edges shall then be marked with three vertical compliance calibration markings, within the cracked

region, at distances of 20, 30, and 40 mm [0.8, 1.2, and 1.6 in.] from the tip of the insert.

- 11.3.3 If specific gravity, density, reinforcement volume, or void volume, or combinations thereof, are to be reported, then obtain these samples from the same panels being tested. Specific gravity and density may be evaluated by means of Test Method D792. Volume percent of the constituents may be evaluated by one of the matrix digestion procedures of Test Method D3171, or, for certain reinforcement materials such as glass and ceramics, by the matrix burn-off technique of Test Method D2584. The void content equations of Test Method D2734 are applicable to both Test Method D2584 and the matrix digestion procedures.
- 11.4 Test Environment—If possible, test the specimen under the same fluid exposure level used for conditioning. However, cases such as elevated temperature testing of a moist specimen place unrealistic requirements on the capabilities of common testing machine environmental chambers. In such cases, the mechanical test environment may need to be modified, for example, by testing at elevated temperature with no fluid exposure control, but with a specified limit on time to failure from withdrawal from the conditioning chamber. Record any modifications to the test environment.
- Note 3—When testing a conditioned specimen at elevated temperature with no fluid exposure control, the percentage moisture loss of the specimen prior to test completion may be estimated by placing a conditioned traveler of known weight within the test chamber at the same time the specimen is placed in the chamber. The traveler should be configured to mimic the specimen, such that moisture evaporation is comparable to that of the test specimen. Upon completion of the test, the traveler is removed from the chamber, weighed, and the percentage weight calculated and reported.
- 11.5 The specimen shall be placed in the fixture so that its longitudinal direction is perpendicular to the loading rollers (see subsection 7.2.5).
- 11.6 Loading for all CC and fracture tests shall be performed in displacement control at a nominal rate of 0.5 mm/min [0.02 in./min], although rates between 0.10 and 0.80 mm/min [0.004 to 0.031 in./min] are acceptable. Unless otherwise specified, unloading shall also be in displacement control at a rate between 0.10 and 1.6 mm/min [0.004 to 0.063 in./min].
- 11.7 Peak forces during CC are equal to 50% of the expected value of the critical force  $(P_c)$  at that particular crack length; these are chosen to correspond to approximately 25% of  $G_{IIc}$ . That is, the peak CC force varies with crack length. The method of determining the peak force for each crack length during CC is presented in Annex A2.
- 11.8 Compliance calibration tests are performed by loading the specimen to the peak CC force as defined in subsection 11.7 and then unloading. The force and deflection data are to be recorded continuously or at frequent and regular intervals during the loading portion only; a sampling rate of 5 Hz or greater and a target minimum of 500 data points per test are recommended.
- 11.9 Values of  $G_{IIc}$  shall be obtained from the insert and from the precrack. All toughness values are obtained using the

CC method. Subsection 11.9.1 is to be used if both NPC and PC toughness values are to be obtained from the same specimen and the recommended precracking procedure is to be adopted; otherwise, subsections 11.9.2 and 11.9.3 are to be used. Peak forces expected during these fracture tests may be estimated by the method in Annex A1.

Note 4—If desired, a five-point CC may be used. In this case, CC is performed for both the NPC and PC tests with crack lengths, a, equal to 20, 25, 35, and 40 mm [0.8, 1.0, 1.4, and 1.6 in.), and the fracture test is still performed with a = 30 mm (1.2 in.). Data from all five crack lengths are then used to obtain the CC coefficients (subsection 13.2).

11.9.1 Non-Precracked and Precracked Toughness from the Same Specimen—In the approach that follows, crack advance during the NPC test creates the precrack that is used for the PC test. The approach has been shown to produce accurate NPC and PC toughnesses with a PC  $G_{IIc}$  that is within or approaching the "minimum toughness plateau" that some materials evidence, i.e., when  $G_{IIc}$  decreases to a minimum plateau value with the amount of dynamic advance that occurs during precracking (2-4). The approach also ensures that any differences between the location of the true and calculated crack tip do not affect the accuracy of  $G_{IIc}$  (2).

11.9.1.1 Non-Precracked CC—With reference to Fig. 2, the specimen is placed in the fixture so that the CC mark that is farthest from the cracked end is aligned with the center of the support roller at the cracked end. The first CC test is then performed with a crack length, a, equal to 20 mm [0.8 in.], following the procedure defined in subsection 11.8. The specimen is then repositioned such that a = 40 mm [1.6 in.], i.e., so that the CC mark that is closest to the cracked end is aligned with the center of the support roller at the cracked end. The second CC test is then performed as defined in subsection

11.9.1.2 Non-Precracked Fracture Test—Following NPC CC, the specimen shall be repositioned in the fixture so that a = 30 mm. This shall correspond to placing the center CC mark over the center of the support roller that is at the cracked end. The specimen is then loaded until the delamination advances, as seen by visual assessment on the specimen or by a drop in force on the force versus displacement plot. The specimen shall be unloaded at a nominal rate of 0.5 mm/min [0.02 in./min], although rates between 0.10 and 0.80 mm/min [0.004 to 0.031 in./min] are acceptable (i.e., in order to decrease the total time required for the test). The force and

displacement data are to be recorded continuously or at frequent and regular intervals during the entire test; a sampling rate of 5 Hz or greater and a target minimum of 1000 data points per test are recommended.

11.9.1.3 Determination of Crack Length for the Precracked Test—The unloading data from the non-precracked fracture test of subsection 11.9.1.2 is used to compute a value of  $a_{calc}$  using the method of subsection 13.6. This value of  $a_{calc}$  is measured from the existing center CC mark. A new "PC crack tip mark" shall be placed at this location. Three new "PC CC markings" shall then be placed at 20, 30, and 40 mm [0.8, 1.2, and 1.6 in.] from the PC crack tip mark as shown in Fig. 5. The center mark, with distance from the crack tip equal to 30 mm [1.2 in.] is for the fracture test and the other two marks are for CC testing.

If desired, the location of the crack tip may also be determined visually as the average of the locations found on the two edges. If the visually determined crack tip,  $a_{vis}$ , is (1) past (to the right of) the loading roller and (2) longer than  $a_{calc}$ , then  $a_{vis}$  may be used in place of  $a_{calc}$  for the placement of the PC crack tip mark.

11.9.1.4 Precracked CC—Prior to the PC fracture test, the compliances from two different crack lengths are obtained by appropriate placement of the specimen in the fixture. The first CC test is performed with a = 20 mm [0.8 in.] and the second with a = 40 mm [1.6 in.]. This is performed using the PC CC markings of subsection 11.9.1.3 and following the procedure of subsection 11.9.1.1.

11.9.1.5 Precracked Fracture Test—Following PC CC, the specimen shall be repositioned in the fixture so that a = 30 mm [1.2 in.]. This shall correspond to placing the center PC CC mark over the center of the support roller that is at the cracked end. The specimen is then loaded until the delamination advances, as seen by visual assessment on the specimen or by a drop in force on the force versus displacement plot. The specimen is then unloaded. The force and deflection data are to be recorded continuously or at frequent and regular intervals during the loading portion only; a sampling rate of 5 Hz or greater and a target minimum of 750 data points per test are recommended.

11.9.2 Non-Precracked Toughness Only—This test is performed as described in subsections 11.9.1.1 and 11.9.1.2. Unloading data and determination of  $a_{calc}$  (subsection 11.9.1.3) are not required.

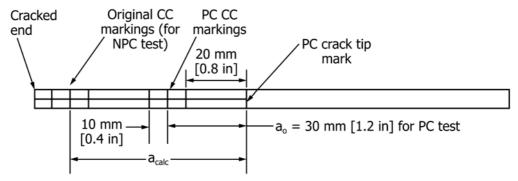


FIG. 5 Configuration of Specimen for Precracked Test When the Same Specimen is Used for NPC and PC Testing (nominal dimensions shown)

11.9.3 Precracked Toughness Only—The method that follows is valid provided that a record of the precracked delamination front shape prior to the PC test can be obtained. This record must be sufficiently detailed to allow for quantitative measurements of crack length as a function of position at five locations in the width direction. These five locations shall consist of the two edges, 50% of the distance from either edge towards the center of the specimen, and in the specimen's center.

11.9.3.1 Subsequent to precracking and prior to testing, the location of the crack front is to be determined by an appropriate method and marked on the edge of the specimen. This comprises the "PC crack tip mark."

11.9.3.2 Three "PC CC markings" shall be placed at distance of 20, 30, and 40 mm (0.8, 1.2, and 1.6 in.) from the PC crack tip mark.

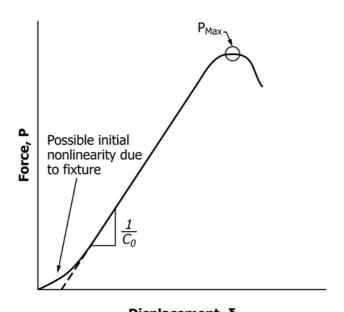
11.9.3.3 The PC CC and PC fracture test are performed following the procedures for the NPC test and as specified in subsection 11.9.2.

# 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 as needed to replace results from specimens where values are not calculated.

### 13. Calculations

13.1 Interlaminar Fracture Toughness Calculations—NPC and PC initiation values of  $G_{IIc}$  are to be obtained from the maximum force  $(P_{Max})$  (Fig. 6).



Displacement, δ
FIG. 6 Illustration of Compliance and Maximum Load Point Deter-

mination

13.2 Compliance Calibration Coefficients—CC coefficients from each NPC and PC test are to be determined using the method described in subsections 13.2.1 and 13.2.2 (1,2,4).

13.2.1 Non-precracked CC Coefficients—Plot the three compliances from the NPC test versus crack length cubed. The three compliances are those from the two CC tests (at a = 20and 40 mm [0.8 and 1.6 in.]) and from the fracture test ( $C_0$ , Fig. 6, which is at crack length  $a_o = 30 \text{ mm} [1.2 \text{ in.}]$ ). At each crack length, the compliance is determined by a linear least squares regression analysis to obtain the slope of the displacement versus force ( $\delta$  versus P) data. For the two CC tests, this regression analysis (curve-fit) shall include all data for which the force is greater than or equal to 90 N [20 lb], including the peak force used during the test. The 90 N [20 lb] force is chosen to be sufficiently large such that the curve-fit excludes any data affected by an initial nonlinearity (Fig. 6). For the fracture test, the regression analysis (curve-fit) shall include all data for which the force is greater than or equal to 90 N [20 lb] and less than or equal to 50% of the maximum force from the fracture test (this yields  $C_0$ ). The CC coefficients, A and m, are to be determined using a linear least squares linear regression analysis of the compliance, C, versus crack length cubed  $(a^3)$ data of the form:

$$C = A + ma^3 \tag{2}$$

where A and m are the CC coefficients or, more specifically, A is the intercept and m is the slope obtained from the regression analysis. The correlation coefficient,  $r^2$ , for the curve-fit is also to be determined. The linear least squares regression analysis used to determine the compliance at any crack length from the  $\delta$  versus P data as well as the values of A, m and  $r^2$  is described in Annex A3.

13.2.2 Precracked CC Coefficients—PC CC coefficients are to be determined in the same fashion as the NPC CC coefficients (subsection 13.2.1), except that the three compliances from the PC test and their associated crack lengths are to be used.

13.3 Determination of Candidate Toughness—A candidate toughness,  $G_Q$ , is first determined and checked for validity. If it is valid, then  $G_{IIc} = G_Q$ . Otherwise, the results from the test are discarded (in which case  $G_Q$  is generally used to modify the CC forces for subsequent testing). The NPC candidate toughness is determined using the method of subsection 13.3.1. If the recommended precracking method of subsection 11.9.1 is adopted, then the PC candidate toughness is determined identically. Otherwise, the PC candidate toughness is determined as described in subsection 13.3.2.

13.3.1 Non-Precracked Tests, and Precracked Toughness Tests Per Subsection 11.9.1—The candidate toughness is determined using

$$G_{\mathcal{Q}} = \frac{3mP_{Max}^2 a_0^2}{2B} \tag{3}$$

where m is the CC coefficient,  $P_{Max}$  is the maximum force from the fracture test as defined in subsection 13.1,  $a_o$  is the crack length used in the fracture test (30 mm [1.2 in.]), and B is the specimen width. When determining the NPC  $G_O$ , these

parameters are taken from the NPC CC and fracture tests. They are taken from tests of the precracked specimen when determining the PC  $G_O$ .

13.3.2 Precracking By a Method Other Than Subsection 11.9.1:

13.3.2.1 Crack Front Assessments—Following completion of the PC test, the actual crack lengths at the five widthwise locations specified in subsection 11.9.3 shall be determined by suitable means, for example, from pre-test c-scan images, or if a precracking method is used that leaves visually evident crack front markings, by splitting the specimen in half and making appropriate measurements. The shortest of these five lengths shall be subtracted from the longest to determine  $\Delta s$ , a measure of deviation from straightness (2-4). The actual crack length used for the PC test,  $a_{PC}$ , shall be determined by the equivalent area method (3,5). This method defines an area of growth equivalent to that which occurred, but with a straight front normal to the edges, and the location of this front defines the length of the crack. The method is employed by using the shape of the precrack to determine the area bounded by the delamination front, the two longitudinal edges, and the location of the support roller at the cracked end during the precracked test, and then dividing this area by the specimen's width.

13.3.2.2 The candidate toughness is determined using:

$$G_{\mathcal{Q}} = \frac{3mP_{\text{Max}}^2 a_{PC}^2}{2B} \tag{4}$$

13.4 Candidate Toughness Evaluation—The NPC candidate toughness is evaluated using the method of subsection 13.4.1. If the recommended precracking method of subsection 11.9.1 is adopted, then the PC candidate toughness evaluation is performed identically. Otherwise, the PC candidate toughness evaluation is performed as described in subsection 13.4.2.

13.4.1 Non-Precracked Tests, and Precracked Toughness Tests Per Subsection 11.9.1—The percentages of  $G_Q$  (% $G_Q$ ) that were achieved during compliance calibration are calculated using:

$$\%G_{Q,j} = \left[\frac{100(P_j \ a_j)^2}{(P_{Max} \ a_o)^2}\right]; j = 1,2$$
 (5)

where  $\%G_{Q,j}$  are the two values of  $G_Q$  associated with the two compliance tests,  $P_{Max}$  is taken from the fracture test as defined in subsection 13.1,  $P_j$  is the peak value of the force achieved during CC at  $a_j$ , and  $a_j$  is the  $j^{\text{th}}$  crack length used during CC. For each NPC and PC test, the two values of  $\%G_Q$  are computed at  $a_1 = 20$  mm [0.8 in.] and  $a_2 = 40$  mm [1.6 in.]. If both values satisfy  $15 \le \%G_Q \le 35$ , then the candidate toughness is accepted. Otherwise, it is recommended that the results from this test are discarded and, if necessary, CC forces are modified appropriately for additional testing (2).

13.4.2 Precracking By a Method Other Than Subsection 11.9.1—The percentages of  $G_Q$  (% $G_Q$ ) that were achieved during PC compliance calibration are calculated by the approach described in subsection 13.4.1, except that  $a_{PC}$  (subsection 13.3.2.1) is used in place of  $a_o$  in Eq 5. If all values of % $G_Q$  satisfy  $15 \le %G_Q \le 60$  and  $\Delta s \le 4$  mm [0.16 in.], then the candidate toughness is accepted. Otherwise, the results from this test are discarded and, if necessary, CC forces are modified appropriately for additional testing (2).

13.5 *Toughness Determination*—For any specimen where the candidate toughness is accepted (NPC or PC):

$$G_{Uc} = G_O \tag{6}$$

13.6 To determine the value of  $a_{calc}$  (subsection 11.9.1.3), the compliance of the unloading line at the end of the NPC test (i.e., after delamination growth has occurred, subsection 11.9.1.2), is computed by a linear least squares regression analysis of the displacement versus force data using the method in Annex A3. The data shall be curve-fit over the same range of force as is used to obtain  $C_0$  (subsection 13.2.1). The compliance of the NPC test unloading line,  $C_u$ , is used in the equation:

$$a_{calc} = \left(\frac{C_u - A}{m}\right)^{1/3} \tag{7}$$

where A and m are the CC coefficients from the NPC test (subsection 13.2.1).

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

$$\bar{x} = \left(\sum_{i=1}^{n} x_i\right)/n \tag{8}$$

$$S_{n-1} = \sqrt{\left(\sum_{i=1}^{n} x_i^2 - n \,\bar{x}^2\right) / (n-1)}$$
 (9)

$$CV = 100 \times S_{n-1}/\bar{x} \tag{10}$$

where:

 $\bar{x}$  = sample mean (average),  $S_{n-1}$  = sample standard deviation,

CV = sample coefficient of variation, in percent,

n = number of specimens, and $x_i = \text{measured or derived property.}$ 

### 14. Report

14.1 Report the following information, or report 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 5—Guides E1309, E1434, and E1471 contain data reporting recommendations for composite materials and composite materials mechanical testing,

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 Identification of the material tested including: material specification, material type, material designation, manufacturer, manufacturer's lot or batch number, source (if not from manufacturer), date of certification, expiration of certification, filament diameter, tow or yarn filament count and twist, sizing, form or weave, fiber areal weight, matrix type, prepreg matrix content, and prepreg volatiles content,

14.1.6 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,

- 14.1.7 Ply orientation (stacking sequence) of the laminate,
- 14.1.8 If requested, report density, reinforcement volume fraction, and void content test methods, specimen sampling method and geometries, test parameters, and test data,
  - 14.1.9 Average ply thickness of the material,
  - 14.1.10 Results of any nondestructive evaluation tests,
- 14.1.11 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,
- 14.1.12 Calibration dates and methods for all measurement and test equipment,
- 14.1.13 Type of test machine, alignment data, and data acquisition sampling rate and equipment type,
  - 14.1.14 Measured dimensions for each test specimen,
- 14.1.15 Conditioning parameters and results, and the procedure used if other than that specified in the test method,
- 14.1.16 Relative humidity and temperature of the testing laboratory,
- 14.1.17 Environment of the test machine environmental chamber (if used) and soak time at environment,
- 14.1.18 Loading and support nose type and dimensions for loading fixture, fixture roller diameters,  $r_1$  and  $r_2$ , and fixture span length, 2L,
  - 14.1.19 Number of specimens tested,
  - 14.1.20 Speed of testing,
- 14.1.21 If applicable, transducer placement on the specimen, transducer type, and calibration data for each transducer used,
- 14.1.22 Tabulated data of force versus displacement, and force-displacement curves, for each specimen, and
- 14.1.23 Tabulated data of force versus displacement, and force-displacement curves for each system compliance test.
- 14.2 A set of recommended data sheets are given in Appendix X1. These sheets may be used as a template, but they are not intended to be inclusive to all items listed in 14.1. Thus, additional data sheets may be required. The recommended data sheets contain the items listed below.
- 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 methods used to determine fiber volume fraction and void content.
- 14.2.2 Coupon Data—Type and thickness of insert, average width (B) and thickness (2h) of each specimen, and maximum width and thickness variation (as defined in subsection 8.3.6) along the length of each specimen.
- 14.2.3 *Precracking Prodeedure*—Identification of whether the recommended procedure was used and, if not, complete description of the precracking process, and crack length versus width measurement procedure (subsections 11.9.3 and 13.3.2.1).
- 14.2.4 Test Geometry—Values of  $a_o$  used for NPC and PC tests, and values of  $a_1$  and  $a_2$  used for CC.
- 14.2.5 *Fixture Compliance*—Compliance of the test fixture as determined by the method of subsection 7.2.3.

- 14.2.6 Test Results:
- 14.2.6.1 Force versus displacement curves from fracture tests indicating force and displacement at the maximum load point.
- 14.2.6.2 Values of m, A, and  $r^2$  for each NPC and PC specimen tested.
- 14.2.6.3 Values of  $%G_Q$  for each NPC and PC specimen tested, with those results that were found to be unacceptable clearly indicated.
- 14.2.6.4 For cases where the recommended precracking procedure was not followed, the five measured values of crack length, the resulting value of  $a_{PC}$ , and the maximum measured difference in crack length along the delamination front of the precrack,  $\Delta s$  (subsection 13.3.2.1), for each PC test.
- 14.2.6.5 Tabulated results for those NPC and PC tests for which the candidate toughness was accepted indicating the test type (NPC or PC), the specimen number, the corresponding value of  $G_{IIc}$ , and the mean, normal standard deviation, and coefficient of variation (standard deviation divided by the mean, expressed as a percentage) for each data set (NPC and PC).
- 14.2.6.6 Other observations from testing that may have affected the test outcomes. Examples include, but are not limited to, specimen or manufacturing imperfections that are visually evident, relatively loud "cracking" noises well before crack advance, large amounts of nonlinearity in the loading curve, permanent deformation after unloading, a postmortem check that reveals tears, folds, or an irregular shape at the end of the insert (i.e., the insert was not straight and parallel), deviation of the precrack from the midplane, occurrence of fiber-bridging, or sticking of the insert foil.

# 15. Precision and Bias<sup>4</sup>

15.1 Interlaboratory Study—An interlaboratory study (ILS) for precision data was conducted on this test method in 2011. Nine laboratories participated in the evaluation of three material systems provided by a single supplier. Each laboratory tested two materials at ambient laboratory conditions. For a specific material, each laboratory was supplied with five test specimens. These were tested by a single operator. Each specimen was used to determine a single value of the non-precracked toughness and a single value of the precracked toughness. All specimen preparation, testing, and data reduction conformed to the requirements and specifications of this standard. Except for the limited number of materials that were tested, Practice E691 was followed for the design and analysis of the data.

15.1.1 Materials Considered:

15.1.1.1 *IM7/977-3 carbon/epoxy*, comprised of a continuous, high performance, intermediate modulus (276 GPa [40 msi]), PAN based carbon fiber and a 177°C [350°F] curing toughened epoxy resin with a 177°C [350°F] dry and 132°C [270°F] wet service capability.

15.1.1.2 *G40-800/5276-1 carbon/epoxy*, comprised of a continuous, high performance, intermediate modulus (290 GPa

<sup>&</sup>lt;sup>4</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D30-1006. Contact ASTM Customer Service at service@astm.org.

[42 msi]) carbon fiber and a highly toughened epoxy resin with a 177°C (350°F) curing temperature and a service temperature range of -59 to 121°C [-75 to 250°F].

15.1.1.3 *S2/5216 glass/epoxy*, comprised of S-2 glass fibers and a 93 to 121°C [200 to 250°F] curing modified epoxy resin with a service temperature range of -55 to 82°C [-67 to 180°F].

15.1.2 Test Specimens—For both the IM7/977-3 and G40-800/5276-1 materials, all specimens were cut from a single plate, whereas specimens were cut from two plates for S2/5216. Specimen thicknesses (2h) of the two carbon/epoxy materials were essentially the same with a mean value of 3.4 mm [0.134 in.], whereas the mean thickness of the glass/epoxy specimens was 4.1 mm [0.161 in.].

15.1.3 *ILS Results*—Key results from the ILS are presented in Table 2 and Table 3. Table 2 presents results from testing of non-precracked specimens, and Table 3 presents the precracked results. The first five columns of each table present the material name, the number of laboratories that participated in testing of that material, the total (combined) number of replicates tested by all participating laboratories, the mean toughness obtained from all test results combined, and the average coefficient of variation (CV), obtained by averaging the CVs reported by each laboratory. The remaining columns present the repeatability (r) and reproducibility (R) limits that were obtained by statistical analyses of the data. Additional information is provided in Research Report RR:D30-1006.

15.2 *Precision*—There are two types of precision: within-laboratory (the repeatability, r) and between-laboratory (the reproducibility, R). Practice E691 suggests that for a 95% confidence interval the maximum difference between an indi-

vidual observation and the average should be within 2.0 standard deviations, while the maximum difference between any two observations should be within 2.8 standard deviations. The final four columns of Table 2 and Table 3 present the latter forms of r and R only, as the former can be derived from these. That is, two test results that are obtained (by the same operator using the same equipment on the same day) from an individual laboratory for the same material shall be judged not equivalent if they differ by more than the "r" value for that material; two test results obtained by different operators using different equipment in different laboratories shall be judged not equivalent if they differ by more than the "R" value for that material. Repeatability and reproducibility limits for comparisons between individual observations and mean values may be obtained by multiplying the values in the last four columns of Table 2 and Table 3 by 2.0/2.8. The results of Table 2 and Table 3 indicate that the precision of this test method is relatively insensitive to minor variations in testing practices.

Note 6—Due to the number of participating laboratories and materials and specimens tested, the repeatability and reproducibility limits of Table 2 and Table 3 should be considered as general guides, and the associated 95% probabilities as only a rough indicator of what can be expected.

Note 7—To judge the equivalency of two test results of materials different than those in Table 2 and Table 3, it is recommended to choose the material from the table that is closest in characteristics to the test material.

15.3 *Bias*—No other test method for determining the mode II interlaminar fracture toughness of composite laminates has been evaluated to the extent where it can be used to determine inherent bias in the ENF test.

**TABLE 2 Non-Precracked Results from ILS** 

Material	No. Labs	Total Replicates	Mean <i>G<sub>IIc</sub></i> (kJ/m²	Mean CV (%) <sup>A</sup>	r (kJ/m²	r (% of mean)	R (kJ/m²	R (% of mean)
		•	[inlbf/in.2])		[inlbf/in. <sup>2</sup> ])		[inlbf/in. <sup>2</sup> ])	
IM7/977-3	4	20	0.908 [5.18]	3.1	0.0836 [0.477]	9.2	0.0871 [0.497]	9.6
G40-800/5276-1	5	25	2.11 [12.0]	3.9	0.246 [1.40]	11.6	0.285 [1.63]	13.5
S2/5216	9	44	1.17 [6.68]	6.5	0.222 [1.27]	19.0	0.222 [1.27]	19.0

<sup>&</sup>lt;sup>A</sup>Average of the coefficients of variation reported by each participating lab.

**TABLE 3 Precracked Results from ILS** 

Material	No. Labs	Total Replicates	Mean <i>G<sub>IIc</sub></i> (kJ/m² [inlbf/in.²])	Mean CV (%) <sup>A</sup>	r (kJ/m² [inlbf/in.²])	r (% of mean)	<i>R</i> (kJ/m² [inlbf/in.²])	R (% of mean)
IM7/977-3	4	20	0.620 [3.54]	5.4	0.103 [0.588]	16.6	0.105 [0.600]	17.0
G40-800/5276-1	5	25	1.99 [11.4]	3.8	0.219 [1.25]	11.0	0.239 [1.36]	12.1
S2/5216	9	43	0.930 [5.31]	5.9	0.159 [0.908]	17.1	0.188 [1.07]	20.2

<sup>&</sup>lt;sup>A</sup>Average of the coefficients of variation reported by each participating lab.

# 16. Keywords

16.1 composite materials; delamination; end notched flexure; ENF; interlaminar fracture toughness; mode II; shear

### **ANNEXES**

(Mandatory Information)

### A1. CRITICAL FORCE AND FLEXURAL MODULUS ESTIMATION

# A1.1 Overview

A1.1.1 Values of the mode II fracture toughness and flexural modulus,  $E_{If}$ , for the material of interest are required in order to estimate the critical force,  $P_c$ . The flexural modulus may be obtained using Test Method D7264/D7264M or, although somewhat less accurate, the longitudinal modulus,  $E_{II}$ , may be used in its place.  $E_{If}$  may also be determined from CC data using the method described in subsection A1.2.2. Initial estimates of  $G_{IIc}$  may be obtained from data on similar composite systems, from previous testing experience on the material of interest, or from an "exploratory test" as described in subsection A2.1.

### A1.2 Calculation

A1.2.1 The critical force at fracture can be approximated using classical beam theory (CBT) as (1):

$$P_{c} = \frac{4B}{3a_{o}} \sqrt{G_{IIC} E_{IJ} h^{3}}$$
 (A1.1)

A1.2.2 Flexural Modulus—If  $E_{If}$  is to be extracted from the results of a test, CBT yields: (1)

$$E_{tf} = \frac{L^3}{4ABh^3} \tag{A1.2}$$

where *A* is the CC coefficient obtained during CC testing of that specimen.

### A2. FORCE DETERMINATION FOR COMPLIANCE CALIBRATION

# A2.1 Overview

A2.1.1 Values of  $G_{IIc}$  and  $E_{If}$  are required in order to determine the force for compliance calibration and are to be obtained by one of the methods described in Annex A1. Note that proper choice of the CC forces will affect whether or not the candidate toughness,  $G_Q$ , is acceptable. Thus, in certain cases, the first test of a new material will produce a value of  $G_Q$  that is not acceptable. When this occurs, this first test (NPC and/or PC) can be classified as "exploratory," the value of  $G_Q$  that was obtained can be used as an improved approximation for  $G_{IIc}$ , the value of  $E_{If}$  may be extracted from the test data as described in subsection A1.2.2, and these values of  $G_{IIc}$  and  $E_{If}$  can be used to determine new values for the CC forces. Further, it is recommended that the data from the first test of a new

material that produces an acceptable value of  $G_Q$  are used to update the CC forces for subsequent testing of that material.

### **A2.2** Calculation

A2.2.1 Peak forces during CC are determined using CBT as  $0.5P_c$  at each crack length. This is chosen to produce a value of G at each crack length that is 25% of  $G_{IIc}$ . This yields: (1,4)

$$P_{j} = \frac{2B}{3a_{j}} \sqrt{G_{IIc} E_{Ij} h^{3}}$$
 (A2.1)

where  $P_j$  is the peak value of the force to be used during CC at  $a_j$ , and  $a_j$  is the  $j^{\text{th}}$  crack length used during CC. For each NPC and PC test there are two CC forces and two associated crack lengths:  $a_1 = 20 \text{ mm} [0.8 \text{ in.}]$  and  $a_2 = 40 \text{ mm} [1.6 \text{ in.}]$ .

# A3. LINEAR LEAST SQUARES REGRESSION ANALYSIS

### A3.1 Overview

A3.1.1 Linear least squares regression analyses are used to obtain the compliance, *C*, from the displacement versus force data at any crack length (subsections 13.2.1, 13.2.2, and 13.6). They are also used to obtain the CC coefficients that define the equations for non-precracked and precracked compliance versus crack length (subsections 13.2.1 and 13.2.2). The equations that follow are standard expressions and should correspond to those used in commercially available software packages that fit a linear equation to a set of data. However, this should be

verified and the equations defined herein are to be utilized in the event of any differences.

# A3.2 Calculation

A3.2.1 Consider a set of n data points  $(x_i, y_i)$  that are to be curve-fit with a linear equation of the form y = bx + d. Thus, when fitting displacement versus force  $(\delta \text{ vs } P)$  data,  $(x_i, y_i)$  correspond to the discrete values of  $(P_i, \delta_i)$ , and b will correspond to the compliance at that crack length. When fitting compliance versus crack length (C vs a) data to Eq. 2,  $(x_i, y_i)$ 

correspond to the discrete values of  $(a_i^3, C_i)$ , b will correspond to the slope, m, and d is the intercept A. A linear least squares regression analysis minimizes the sum of the squared residuals, where a residual is defined as the difference between the curve-fit (y = bx + d) and the actual data point at each known value of the independent variable. Applying this criterion results in the following equations:

$$b = \frac{n\Sigma x_i y_i - \Sigma x_i y_i}{n\Sigma x_i^2 - (\Sigma x_i)^2}$$
 (A3.1)

$$d = \frac{\sum y_i - m\sum x_i}{n} \tag{A3.2}$$

The correlation coefficient,  $r^2$ , is given by:

$$r^{2} = \frac{(n \; \Sigma \; x_{i} \; y_{i} \; - \; \Sigma \; x_{i} \; \Sigma \; y_{i})^{2}}{[n \; \Sigma \; x_{i}^{2} \; - \; (\Sigma \; x_{i})^{2}][n \; \Sigma \; y_{i}^{2} \; - \; (\Sigma \; y_{i})^{2}]}$$
(A3.3)

All summations in equations Eq A3.1-A3.3 are taken over the range i = 1 to n.

# **APPENDIX**

(Nonmandatory Information)

### X1. RECOMMENDED DATA SHEETS

# X1.1 Recommended Data Sheets

X1.1.1 Data Sheets

Panel Data			
Material	Producer	Date of manufacture	Date of cutting into specimens
Panel No.	Specimen numbers from this	panel	
Insert material	Insert thickness	FAW	Vf

FIG. X1.1

Specime	n Geo	metry	for Par	iel No:	1		Materia	l:					
Date of me	asureme	nt:					Operato	or:					
Specimen V	Vidth and	d Thickne	ess Data						Thickness	, 2h (mm)	)		
Specimen		. v	/idth, B (m	m)		Locat	tion 1	Loca	tion 2	Loca	tion 3		
Number	Loc. 1	Loc. 2	Loc. 3	Avg.	Δ Max.	Left	Right	Left	Right	Left	Right	Avg.	Δ Max.
XX-YY-01 XX-YY-02													
Mean values													

FIG. X1.2

Specime	n Geo	metry	for Par	nel No:	1		Materia	:					
Date of me	asureme	nt:					Operato	r:					
Specimen V	Vidth and	d Thickne	ess Data						Thickness	s, 2h (in.)			
Specimen			Vidth, B (ir	າ.)	_	Locat	ion 1	Locat	tion 2	Locat	tion 3		
Number	Loc. 1	Loc. 2	Loc. 3	Avg.	Δ Max.	Left	Right	Left	Right	Left	Right	Avg.	Δ Max.
XX-YY-01 XX-YY-02													
Mean values													

FIG. X1.3

Mater	rial	Date of	f Tests	Ope	rator	a <sub>0</sub> (	mm):	a <sub>1</sub> [a <sub>m</sub>	in for CC]	(mm):	a <sub>2</sub> [a <sub>n</sub>	nax for CC	] (mm):	r <sub>1</sub> (mm)	r <sub>2</sub> (mm)
DATA															
Spec.		ids (N)	_	liances (n	<del></del>	А	m		P <sub>Max</sub>	G <sub>Q</sub>	%GQ	%GQ	Accept?	G <sub>IIC</sub>	a <sub>calc</sub>
No.	At a <sub>1</sub>	At a <sub>2</sub>	C(a <sub>1</sub> )	C(a <sub>2</sub> )	C(a <sub>0</sub> )	(mm/N)	1/(Nmm <sup>2</sup> )	r <sup>2</sup>	(N)	(kJ/m <sup>2</sup> )	(a=a <sub>1</sub> )	(a=a <sub>2</sub> )	(Y/N)	(kJ/m <sup>2</sup> )	(mm)
X-YY-01 X-YY-02															
												9	ean SD CV		

FIG. X1.4

Mate	rial	Date of	Tests	Ope	rator	a <sub>0</sub>	(in.):	a <sub>1</sub>	[a <sub>min</sub> for	CC] (in.):	a <sub>2</sub> [a <sub>r</sub>	<sub>nax</sub> for CC	[] (in.):	r <sub>1</sub> (in.)	r <sub>2</sub> (in.)
DATA															
Spec.	CC loa	ds (lbf)	Comp	oliances (i	n./lbf)	А	m		P <sub>Max</sub>	G <sub>Q</sub>	%G <sub>Q</sub>	%G <sub>o</sub>	Accept?	G <sub>IIC</sub>	a <sub>calc</sub>
No.	At a <sub>1</sub>	At a <sub>2</sub>	C(a <sub>1</sub> )	C(a <sub>2</sub> )	C(a <sub>0</sub> )		1/(lbf-in. <sup>2</sup> )	r <sup>2</sup>	(lbf)	(inlbf/in. <sup>2</sup> )	(a=a <sub>1</sub> )	(a=a <sub>2</sub> )	(Y/N)	(inlbf/in. <sup>2</sup> )	(in.)
XX-YY-01															
XX-YY-02															
													ean		
													SD.		
												l	CV Spac		
													Spec. ax		
												l	lin		

FIG. X1.5

Mate	erial	Date	Oper	ator:	a <sub>0</sub> (ı	mm):	a <sub>1</sub> [a <sub>min</sub>	for CC] (n	nm):	a <sub>2</sub> [a <sub>max</sub>	for CC] (mi	m):	r <sub>1</sub> (mm)	r <sub>2</sub> (mm)
Descript	ion of pr	ecracking	g method:	:										
DATA														
Spec.	CC loa	ads (N)	Comp	liances (m	nm/N)	A	m		P <sub>Max</sub>	Go	%G <sub>Q</sub>	%G <sub>Q</sub>	Accept?	$G_{IIC}$
No.	At a <sub>1</sub>	At a <sub>2</sub>	C(a <sub>1</sub> )	C(a <sub>2</sub> )	C(a <sub>0</sub> )	(mm/N)	1/(Nmm <sup>2</sup> )	r <sup>2</sup>	(N)	G <sub>Q</sub> (kJ/m²)	(a=a <sub>1</sub> )	(a=a <sub>2</sub> )	(Y/N)	(kJ/m <sup>2</sup> )
(X-YY-01 (X-YY-02														
												No.	ean SD CV Spec. 1ax 1in	

FIG. X1.6

		est Res	T											
Mate	erial	Date	Oper	rator:	a <sub>0</sub> (	(in.):	a <sub>1</sub> [a <sub>min</sub>	for CC]	(in.):	a <sub>2</sub> [a <sub>max</sub>	for CC] (i	n.):	r <sub>1</sub> (in.)	r <sub>2</sub> (in.)
Descript	ion of pr	ecracking	g method	:						I				l
DATA														
Spec.	CC loa	ds (lbf)	Comp	oliances (in	./lbf)	A	m		P <sub>Max</sub>	G <sub>Q</sub>	%G <sub>Q</sub>	%G <sub>Q</sub>	Accept?	G <sub>IIC</sub>
No.	At a <sub>1</sub>	At a <sub>2</sub>	C(a <sub>1</sub> )	C(a <sub>2</sub> )	C(a <sub>0</sub> )	(in./lbf)	1/(lbf-in. <sup>2</sup> )	r <sup>2</sup>	(lbf)	(inlbf/in.2)	(a=a <sub>1</sub> )	(a=a <sub>2</sub> )	(Y/N)	(inlbf/in. <sup>2</sup>
xx-yy-01 xx-yy-02														
												No.	ean SD CV Spec. Max Min	

# FIG. X1.7

Fixture compliance (mm/N) per subsection 7.2.3:		
Minimum specimen compliance (mm/N) from tabulated results:		
Ratio of fixture compliance to minimum specimen compliance:	≤ 0.03? (Y/N)	Acceptable? (Y/N)
·	, ,	1 ' '
Fixture compliance (in /lbf) per subsection 7.00:	I	
Fixture compliance (in./lbf) per subsection 7.2.3:		I
Fixture compliance (in./lbf) per subsection 7.2.3:  Minimum specimen compliance (in./lbf) from tabulated results:	I	I

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