

Designation: D8336 - 21

Standard Test Method for Characterizing Tack of Prepregs Using a Continuous Application-and-Peel Procedure¹

This standard is issued under the fixed designation D8336; 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 covers measurement of adhesion (tack) between partially cured (B-staged) composite prepreg and a substrate in a peel test, under specified conditions. The test may be conducted to measure tack between a flexible layer of prepreg and another prepreg layer bonded to a rigid substrate (Method I) or a rigid metal substrate (Method II). This test method is primarily geared towards material characterization for automated material layup but can be modified for use with other processes. It is well known that material tack is a function of multiple processing and environmental variables. Permissible composite prepreg materials include carbon, glass, and aramid fibers within a B-staged thermoset resin.

1.2 Measured tack is specified in terms of a peel force at a given specimen width.

1.3 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.5 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

- A480/A480M Specification for General Requirements for Flat-Rolled Stainless and Heat-Resisting Steel Plate, Sheet, and Strip
- **D883** Terminology Relating to Plastics
- D1781 Test Method for Climbing Drum Peel for Adhesives
- D3167 Test Method for Floating Roller Peel Resistance of Adhesives
- D3878 Terminology for Composite Materials
- E4 Practices for Force Verification of Testing Machines
- E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E456 Terminology Relating to Quality and Statistics

3. Terminology

3.1 *Definitions*—Terminology D3878 defines terms relating to composite materials. Terminology D883 defines terms relating to plastics. Terminology E456 and Practice E177 define terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over the other documents.

4. Summary of Test Method

4.1 A continuous application-and-peel method is employed to quantify tack at given test conditions (such as temperature) between one B-staged prepreg layer and a second B-staged prepreg layer which is bonded to a rigid substrate, or between a B-staged prepreg specimen and a rigid substrate. During a test, a prepreg specimen laid up on a substrate with or without another prepreg layer is fed through the test fixture continuously (see Fig. 1). The compaction roller presses the prepreg against the substrate (and against the peel roller) at a controllable force, bonding the prepreg to the substrate at a set application rate. Simultaneously, the prepreg is peeled from the substrate at a peel rate which is identical to the application rate. Data is collected over two phases where only the second phase is related to adhesion. The peel force is determined from the difference in the average force between the two phases. Measuring the peel force during a test gives a measure for the strength of adhesion between prepreg and substrate at the given peel rate.

¹This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.03 on Constituent/Precursor 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.



FIG. 1 Schematic of Continuous Application-and-Peel Test Method (Example: Method II)

5. Significance and Use

5.1 Characterizing tack for different prepreg materials, test parameters, surface combinations, and environmental conditions provides insight for optimizing process parameters (particularly deposition rate and deposition temperature) for industrial automated material placement processes.

5.2 Results obtained through employing the continuous application-and-peel method, as described in studies (1-3),³ reflect the effects of adhesion forming between prepreg layers or between prepreg and metal substrate, and loss of cohesion within the resin in the prepreg, upon tack. This test method allows the adhesive properties of B-staged resin to be explored in a manner relevant for dynamic material deposition processes, where timescales for bonding of prepreg to the substrate or previously placed prepreg layers are short prior to curing. In contrast, Test Methods D3167 and D1781 determine the peel resistance of adhesive bonds for adhesion measurement and process control of laminated or bonded adherends.

5.3 The test method is suitable to quantify tack of prepregs for acceptance and process control and can be extended to determine resin shelf life or to adjust process parameters to resin out-time. Direct comparison of different resins/prepreg or processes can only be made when specimen preparation and test conditions are identical.

6. Interferences

6.1 *Material Test Face*—The manufacturing of prepregs may cause differences in resin content and resin distribution

between the two sides or faces of the prepreg, which may have an effect on tack. Hence, tack tests must be carried out consistently on the same prepreg face to minimize interference due to the possible difference in material properties on the prepreg surfaces. It is recommended that the specimen layup assembly should represent the layup relevant to the intended use of the prepreg.

6.2 *Material Out-Time*—Prepreg out-time from frozen storage can change the characteristics of the material. Follow the guidelines provided by the material manufacturer for material storage and out-life. The operator must record and report changes to material out-time where applicable, in line with 13.1.

6.3 *Rigid Substrate Test Surface*—For tests following Method II, contaminants on the metal substrate surfaces can affect prepreg tack. Ensure that the surfaces are clean and dry as specified in 11.3, prior to application of the prepreg.

6.4 Environmental Control and Measurement—Variation in test temperature will affect tack; the recommendations for temperature control in the environmental chamber are specified in 7.1.11. Relative humidity has also been shown to affect prepreg tack (2). Unless specific conditioning of the prepreg is required (see Section 10), the relative humidity during the test shall be recorded as specified in 11.4.4 for each test temperature.

7. Apparatus

7.1 The test fixture (Fig. 2) holds two pairs of stiff rollers, arranged horizontally. One pair of rollers supports the specimen during a test. In the second pair, the top roller (peel roller) is fixed in position, while springs apply a vertical force on the

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

🖽 D8336 – 21



FIG. 2 Tack Testing Fixture: (a) Exploded View of Assembly, (b) Fully Assembled, and (c) Initial Rollers Positions Prior to a Test Whereby the Vertical Distance Indicates the Travel Range Between the Two Rollers to Accommodate Various Thicknesses of Prepreg and Substrate

bottom roller (compaction roller), allowing it to press the prepreg onto the substrate. All components of the fixture shall be of sufficient stiffness not to deform during a test. The fixture is mounted on the base of a test machine.

7.1.1 The base plate shall be wide enough to accommodate the rollers.

7.1.2 The side plates shall be attached to the base plate. The side plates shall have holes for the peel roller axle and the top support roller axle, slots (vertical) for the compaction roller axle, as well as slots for the bottom support roller axle (which can be adjusted to ensure sufficient clearance between the support rollers to accommodate specimens of different thickness).

7.1.3 The lower column shaft shall fit the base of the testing machine. Its dimensions shall position the peel roller directly below the moving cross-head (Fig. 2(b)) to allow the prepreg to be peeled from the substrate at an angle of 90° . Positioning is critical to ensure clearance between all parts of the set-up during a test. For tests conducted in non-ambient environments, the lower column shaft shall position the fixture in the center of the environmental chamber.

7.1.4 The peel roller shall be in a fixed position as determined by holes in the side plates. The roller surface shall be stiff and not deform when a compaction load is applied. Rotary bearings shall be used to minimize frictional losses in the roller. The recommended roller width is 80 mm (3.15 in.), and the recommended diameter is 25 mm (0.98 in.), although other dimensions may be utilized.

7.1.5 The compaction roller shall have the same design as the peel roller. It is suspended on springs and can move within the vertical slots in the side plates.

7.1.6 The support rollers shall have the same design as the peel roller. The bottom support roller shall be positioned at the same height as the compaction roller (adjustable height). The recommended horizontal distance from the compaction roller is 40 mm (1.57 in.).

7.1.7 The compaction springs shall be sufficiently robust to withstand overloading. Their behavior shall be linear around the target compaction load.

7.1.8 The spring brackets shall be attached to the side plates. They shall be long enough to allow appropriate loading (extension or relaxation) of the compaction springs through adjusting jacking screws

7.1.9 The material clamp shall fit the connector at the load cell of the testing machine. It shall be wider than the specimens but fit in the gap between the spring brackets. Clamping of the flexible prepreg layer prevents slip during a test and holds the layer against the face of the peel roller.

7.1.10 A handle assembly is connected to a lever mechanism which allows opening a gap between the compaction and peel rollers for loading of a substrate with a specimen.

7.1.11 An environmental test chamber is required for test environments other than ambient testing laboratory conditions. This chamber shall be capable of maintaining the test specimen at the required test environment during the mechanical test.

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, as in the case of a designed experiment. For statistically significant data, the procedures outlined in Practice E122 should be consulted. For specimens obtained from a prepreg roll, it is recommended that prepreg surfaces are identified as Face 1 and Face 2 when unrolled from the prepreg roll as shown in Fig. 3 to aid sample preparation. The method of sampling shall be reported in accordance with 13.1.

8.2 Geometry:



FIG. 3 Identification of Prepreg Surfaces When Unrolling From the Prepreg Roll

8.2.1 Specimens shall be cut from a tape or sheet of prepreg. If a uni-directional prepreg tape is to be tested, the fiber orientation in the test specimen must be parallel to the long axis of the substrate. If a prepreg with bi-directional reinforcement is tested, one of the fiber directions needs to be parallel with the long axis of the substrate. Specimen length shall be at least 215 mm (8.46 in.), and the recommended specimen width shall be 75 mm (2.95 in.). If a different specimen width is used, it must be ensured that the specimen width is narrower than both the rollers and the substrate. The specimen width shall be recorded as specified in 13.1.9. The specimen surface whose tack is not being evaluated shall be covered with the protective film normally provided with a prepreg roll. The purpose of the film is to prevent resin from the prepreg to transfer to the fixture rollers. A suitably flexible film that will not wrinkle when wrapped around the roller can be used for this purpose. Fig. 4 illustrates the specimen layup assemblies for Methods I and II consisting of the prepreg specimen, protective films, and rigid substrate.

8.2.2 Rigid substrates are recommended to be made from stainless steel with Specification A480/A480M No. 2B finish. If other suitably rigid substrate materials are used, with a surface finish allowing the substrate to pass smoothly over the compaction roller, this must be recorded as specified in 13.1.8. The substrate shall be at least 140 mm (5.51 in.) in length, 80 mm (3.15 in.) in width, and 0.8 mm (0.03 in.) thick. The edges of the substrates shall be deburred if cut from a larger sheet.

8.3 If tack between two layers of prepreg is to be tested (Method I), an additional layer of prepreg shall be cut from a tape or sheet. This layer does not need to be the same type of prepreg as the layer to be peeled (described in 8.2.1), but the specimen layup assembly should represent the layup relevant to the intended use of the prepreg. The orientation of this prepreg layer does not need to be parallel with the long axis of the substrate. The angle between the fiber orientation in uni-directional prepreg tapes and the substrate axis (that is, peel direction) or the angle between the fabric warp-direction in prepreg fabrics and the substrate axis can be set as in the production layup. The length of this layer shall correspond to the length of the substrate; the width is the same as for the other specimen layer within a tolerance specified during slitting of the material, in the absence of which a general tolerance of ± 1 mm shall be used. One surface of the additional layer of prepreg shall be covered with protective film.

Note 1—Within the recommendations stated in 8.2, other dimensions of specimen and of rigid substrate may be used, provided that the environmental chamber and tack test fixture can accommodate the specimen dimensions for the application-and-peel motion.

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.

9.2 Calibration of Test Apparatus:

9.2.1 The calibration is performed during installation of the fixture prior to initial use to obtain the desired compaction force. The following procedure provides a calibration curve to

a) SIDE VIEW METHOD I 40 mm (1.57 in) 40 mm (1.57 in) 60 mm (2.36 in) 75 mm (2.95 in) DISTANCE BETWEEN LENGTH TO BEND AROUND THE PHASE 2 PHASE 1 SUPPORT AND PEEL ROLLER AND TO GRIP THE END COMPACTION ROLLERS WITH MATERIAL CLAMP PROTECTIVE FILM 135 mm (5.31 in) PREPREG SPECIMEN 80 mm (3.15 in) 140 mm (5.51 in) SUBSTRATE METHOD II 135 mm (5.31 in) PROTECTIVE FILM ~ PREPREG SPECIMEN 140 mm (5.51 in) SUBSTRATE b) TOP VIEW (FOR BOTH METHODS I & II) 215 mm (8.46 in) 80 mm 75 mm (3.15 in) (2.95 in)

D8336 - 21

FIG. 4 Specimen Assembly for Both Methods I and II: (a) Side View and (b) Top View

determine the required number of turns of the jacking screws to set a given compaction force.

9.2.2 The test machine must have an established reference surface and procedure permitting alignment of test specimen within 2.5 mm (0.10 in.) of the loading axis of the machine. A calibrated testing machine shall be used which can be operated at constant crosshead speed over the specified range. The test machine mechanism shall be essentially free from inertial lag at the crosshead speeds specified. The machine shall be equipped with an appropriate force-measuring device (for example, a load cell). The accuracy of the test machine shall be in accordance with Practices E4.

9.2.3 Loosen the jacking screws, such that the compaction roller is suspended loosely by the springs, and the axles of the peel roller and the compaction roller are parallel.

9.2.4 Connect a stiff L-shaped steel bracket to the load cell of the test machine using the material clamp (Fig. 5). Position the short flange of the bracket directly on the compaction roller. Zero the load cell reading with no force applied. The jacking screws are tightened or loosened to find the start point for the calibration (where the reading of the load cell starts to increase from zero). Both jacking screws shall be adjusted by the same amount.



9.2.5 To acquire a force calibration curve, incrementally tighten both jacking screws (by the same number of turns) and record the corresponding force readings for each increment within a relevant range of force readings.

9.2.6 Remove the L-shaped bracket from the material clamp.

10. Conditioning

10.1 Ensure prepreg removed from frozen storage is warmed to ambient conditions before being removed from a sealed bag to avoid moisture condensation on the prepreg surfaces.

Note 2—Prepreg can be conditioned by exposure to defined temperatures for extended periods of time or to defined level of humidity, or both (2, 4).

11. Procedure

11.1 General Instructions:

11.1.1 Report any deviations from this test method, whether intentional or inadvertent.

11.2 Adjustment of Compaction Force:

11.2.1 Pull down the handle to lower the compaction roller. Insert a substrate with (Method I) or without (Method II) an attached layer of prepreg. Release the handle such that the compaction roller presses the surface of the substrate or of the attached prepreg layer against the peel roller.

11.2.2 Loosen both jacking screws until the substrate (Method II) or the attached prepreg layer (Method I) is just in contact with the peel roller and the compaction roller. The axles of the compaction roller and the peel roller shall be parallel. This state corresponds to zero compaction force.

11.2.3 Tighten both jacking screws by the appropriate number of turns to set the target compaction force according to the calibration curve. A force of 100 N (22.48 lbf) is recommended for a specimen width of 75 mm (2.95 in.).

11.2.4 Pull down the handle to lower the compaction roller. Remove the substrate.

11.3 Preparation of Substrates and Prepreg Specimens:

11.3.1 Both surfaces of the metal substrates need to be cleaned thoroughly (with acetone or a solvent-based cleaner) using lint free cloth to remove any contamination. Ensure enough time for solvent to evaporate.

11.3.2 For Method I tests, bond a layer of prepreg to the substrate at an orientation required to imitate the layup in a production scenario using a suitable adhesive such as double-sided adhesive tape. The method used to bond the prepreg to the substrate must produce a stronger bond than that between

two layers of prepreg at the test temperature. The exposed surface of the prepreg layer shall be bonded to the substrate.

11.3.3 Trim an additional layer of protective film to a recommended length of 135 mm (5.31 in.). The width corresponds to the width of the prepreg specimen.

11.3.4 Partially cover the exposed face of the prepreg layer with the trimmed layer of protective film, such that the prepreg surface stays exposed on a recommended length of 80 mm (3.15 in.) as illustrated in Fig. 6 (Method I).

11.3.5 Place the prepreg layer on the surface of the prepreg layer bonded to the substrate (Method I) or on the substrate (Method II), such that the exposed surface is in contact with the substrate or the exposed part of the other prepreg layer. The end of the exposed prepreg surface shall be flush with a short edge of the substrate. The covered part of the prepreg surface extends over the other short edge of the substrate (see Fig. 4 and Fig. 6). No compaction pressure is to be applied on the prepreg surface. Unlike in Test Methods D3167 and D1781, the prepreg layer attached to the substrate (Method II) or to the prepreg layer attached to the substrate (Method I). Avoid contamination of exposed surfaces while handling specimens and substrates.

11.4 Tack Testing:

11.4.1 Pull down the handle to lower the compaction roller. Insert the specimen / substrate layup, such that the specimen is on top. The specimen/substrate layup needs to be centered between the side plates of the fixture. The end of the substrate with the extending prepreg layer needs to be positioned between compaction and peel roller (Fig. 7) such that the metal plate does not protrude past the vertical tangent to both roller surfaces. Release the handle and ensure that the compaction roller is pressing the specimen against the substrate.

11.4.2 Bend the covered part of the top prepreg layer around the peel roller and insert the end into the material clamp (see Fig. 7). Tighten the material clamp.

11.4.3 Balance the load and displacement channels on the testing machine and set the cross-head speed for upward movement.

11.4.4 For tack testing at temperatures higher or lower than ambient temperature, set the target temperature on the control unit of the environmental chamber. Using a non-contact method to measure the specimen surface temperature (for example, infra-red thermometer), commence the test when the surface temperature is within ± 1 °C of the target temperature, and record the surface temperature and the relative humidity within the environmental chamber.

Note 3—Tolerance on surface temperature is tight due to significant variation of tack with temperature.

11.4.5 Start the test to apply-and-peel the top prepreg layer from the layup assembly at a constant cross head speed. Record the experiment time, cross-head displacement, and load at a minimum rate of ten readings per second



FIG. 6 Layup of Different Material Layers for Prepreg-prepreg (Method I) and Prepreg-substrate (Method II) Tack Tests With Recommended Dimensions