Designation: F521 – 16

Standard Test Methods for Bond Integrity of Transparent Laminates[1]

This standard is issued under the fixed designation F521; 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 These test methods cover determination of the bond integrity of transparent laminates. The laminates are usually made of two or more glass or hard plastic sheets held together by an elastomeric material. These test methods are intended to provide a means of determining the strength of the bond between the glass or plastic and the elastomeric interlayer under various mechanical or thermal loading conditions.

1.2 The test methods appear as follows:

Test Methods	Sections
Test Method A—Flatwise Bond Tensile Strength	5 – 11
Test Method B—Interlaminar Shear Strength	12 – 17
Test Method C—Creep Rupture	18 – 25
Test Method D—Thermal Exposure	26 – 30

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D952 Test Method for Bond or Cohesive Strength of Sheet Plastics and Electrical Insulating Materials

2.2 *ANSI Standard:*[3]

B1.1 Standard for Unified Screw Threads

3. Terminology

3.1 *Definitions:*

3.1.1 *delamination, n*—a visible separation between two layers of bonded material.

3.1.2 *face plies, n*—transparent glass or plastic outer materials joined together with an interlayer.

3.1.3 *interlayer, n*—transparent material used as the bonding agent between two or more hard, transparent materials.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *number of plies, n*—a three-ply laminate is one having two transparent glass or plastic plies and one interlayer ply. A five-ply laminate has three glass or plastic plies and two interlayer plies.

4. Significance and Use

4.1 These test methods provide a means to measure quantitatively the bond integrity between the outer layers of the transparency and the interlayer, or to measure the cohesive properties of the interlayer, under various loading conditions.

4.2 These test methods provide empirical results useful for control purposes, correlation with service results, and as quality control tests for acceptance of production parts.

4.3 Test results obtained on small, laboratory-size samples shown herein are indicative of full-size part capability, but not necessarily usable for design purposes.

TEST METHOD A—FLATWISE BOND TENSILE STRENGTH

5. Summary of Test Method

5.1 The bond is subjected to a mechanical load in a direction perpendicular to the plane of the bond. The adhesive or cohesive strength between the interlayer and the outer layers (flatwise tensile strength) is determined, and expressed in terms of pascals (or pounds-force per square inch).

6. Apparatus

6.1 *Metal Blocks*—A pair of 50-mm (2-in.) square metal blocks of 24 ST aluminum alloy, each having a maximum height of 50 mm (2 in.). Each block shall have in one end a hole (see Fig. 1) tapped 22.2 mm (⅞ in.) in accordance with ANSI B1.1, to accommodate threaded 22.2-mm (⅞-in.) studs of convenient length (see Test Method D952). Alternative metal blocks utilize an aluminum "T" section, cut to 50 mm (2 in.) square. A hole shall be drilled in the upright section of each "T" block (see Fig. 2) to accommodate a metal pin or holding device compatible with the test machine used.

6.2 *Testing Machine*—Any suitable machine of the constant-rate-of-crosshead movement type. The testing machine shall be

[1] These test methods are under the jurisdiction of ASTM Committee F07 on Aerospace and Aircraft and are the direct responsibility of Subcommittee F07.08 on Transparent Enclosures and Materials.

Current edition approved April 1, 2016. Published April 2016. Originally approved in 1977. Last previous edition approved in 2010 as F521 – 83 (2010). DOI: 10.1520/F0521-16.

[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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

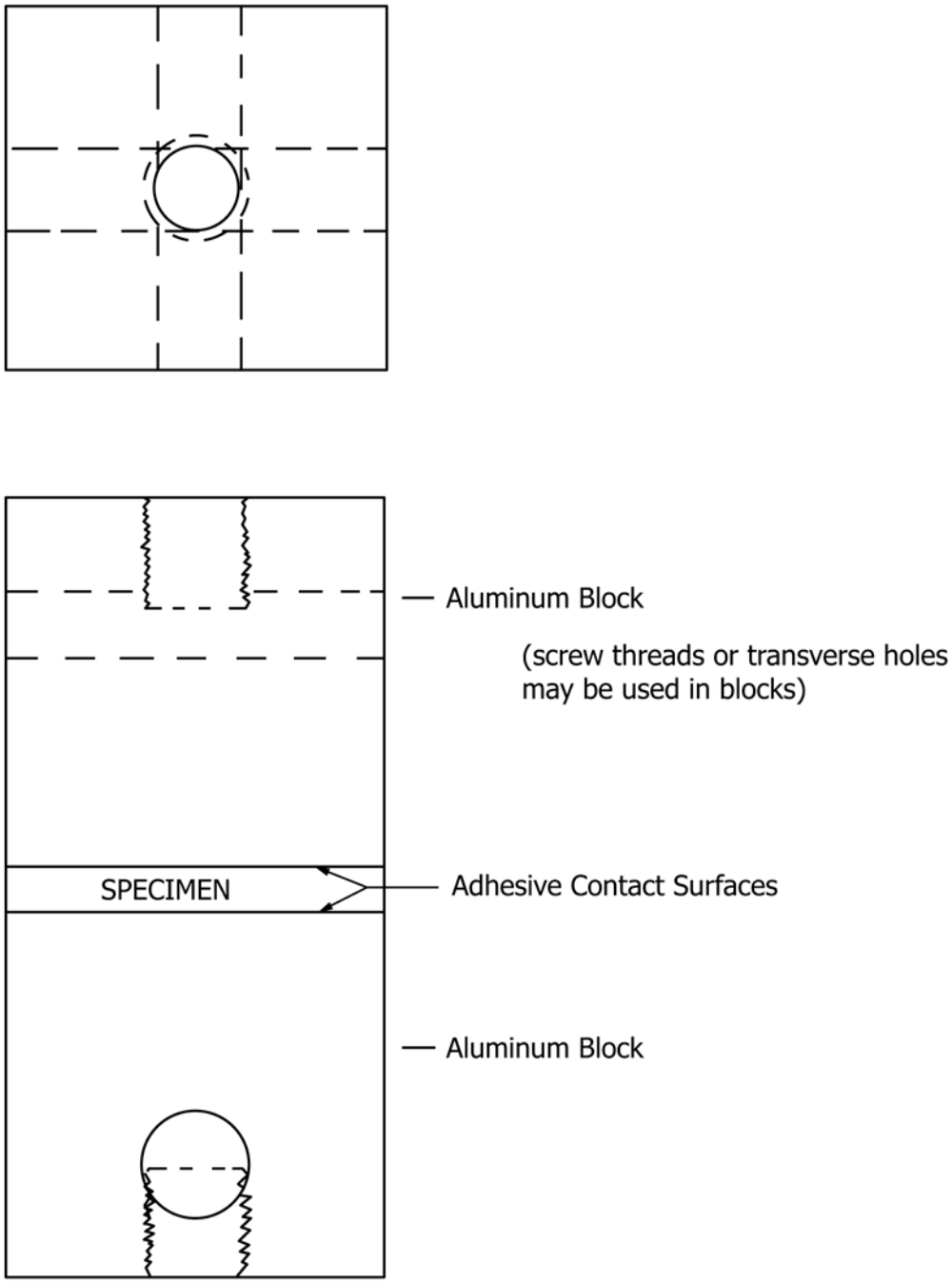


FIG. 1 Test Assembly for Flatwise Tensile Strength Test

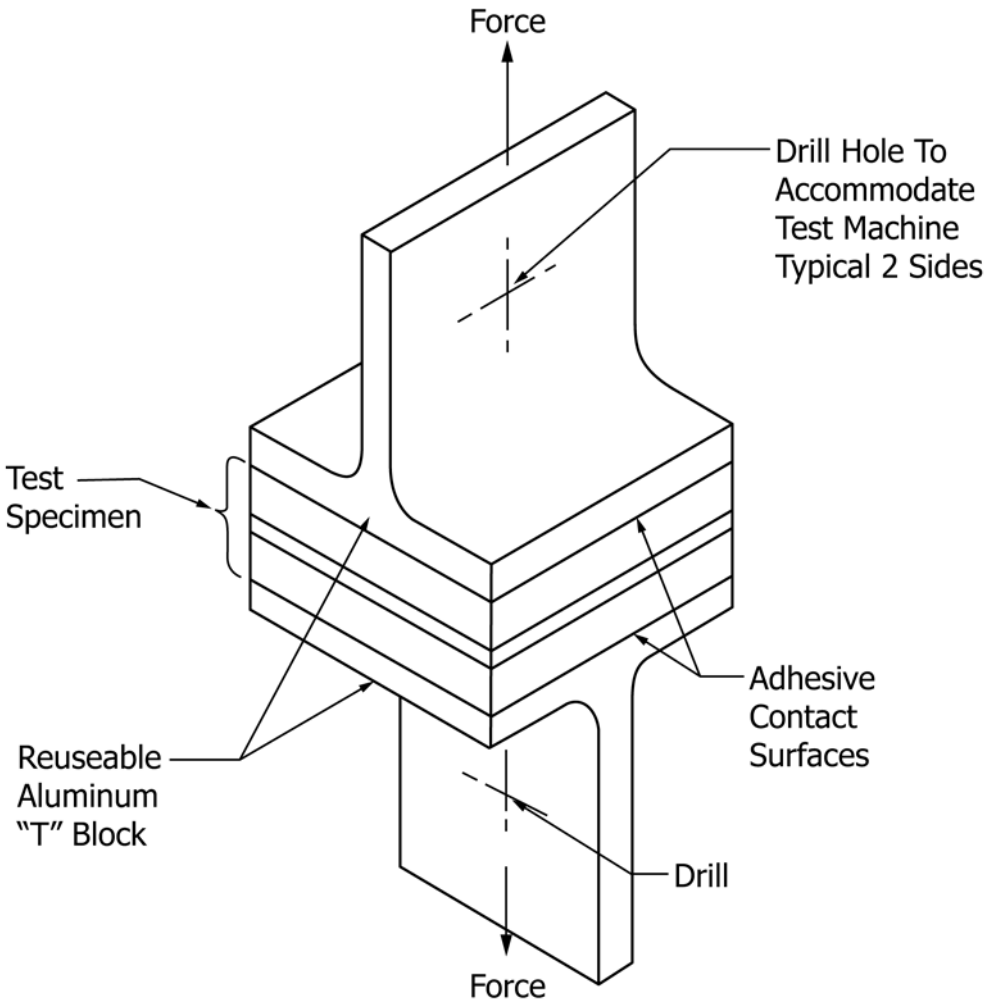


FIG. 2 Optional Tensile Strength Test Specimen

equipped with the necessary drive mechanism for imparting to the crosshead a uniform, controlled velocity with respect to the base. The testing machine shall also be equipped with a load-indicating mechanism capable of showing the total load applied to the test specimen. This mechanism shall be essentially free from inertial-lag at the specified rate of testing and shall indicate the load with an accuracy of ±1.0 % of the indicated value, or better.

6.3 *Adhesive*—Any suitable adhesive.[4]

7. Test Specimen

7.1 The test specimen shall consist of a 50-mm (2-in.) square sample of laminate prepared in such a manner as to produce smooth edges to minimize the possibility of edge chipping during testing. The thickness of the specimen shall be the thickness of the laminate. The upper and lower surfaces shall be parallel to each other and reasonably flat. Test five specimens.

8. Preparation of Apparatus

8.1 Determine the cross-sectional area of the test specimen in a plane parallel to the surface.

8.2 Gently abrade the bonding surfaces of the metal blocks and the specimen (except glass—see Note 1) using 200–400 grit paper or light sandblasting. Do not abrade the edges and corners of the specimen or the metal blocks. Do not round the corners.

NOTE 1—Do not abrade glass surfaces unless absolutely necessary to obtain adhesion to the thoroughly cleaned surface.

8.3 Clean all contact surfaces of the specimens and metal or "T" blocks with a soft cloth saturated with a suitable solvent or clean dry air blast. Thereafter, do not touch the cleaned surfaces with the hands. Apply a thin coating of adhesive to both contact surfaces being careful to remove all air bubbles from the adhesive. Place the specimen between the coated blocks, being certain the blocks are aligned, then clamp the assembly until the adhesive is cured.

[4] Hysol Adhesive 907, a two-part epoxy adhesive available from E. V. Roberts Co., 9601 West Jefferson Blvd., Culver City, CA 90230, has been found satisfactory for use in this test. The instructions in Section 8 for preparation of the test assembly are based on the use of this material. Any adhesive that is found to perform satisfactorily under this test may be used provided that the procedure for the preparation of the test assembly is suitably modified to follow the manufacturer's recommendation for the use of the adhesive.

9. Conditioning

9.1 Condition the test specimen at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 24 h prior to testing.

9.2 Conduct tests in the Standard Laboratory Atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity unless otherwise specified.

10. Procedure

10.1 Unless otherwise specified, test five specimens. Insert the specimen assembly in the tension testing machine with self-aligning holders and load to failure at a rate of 1.25 mm (0.05 in.)/min.

10.2 If block adhesive failure occurs, discard the test and test another specimen.

NOTE 2—If aluminum blocks are to be reused, one method of removing the adhesive is to insert the blocks in an oven at 150°C (300°F) for 1.5 h. When the blocks have cooled, the remaining portion of the test specimen is easily removed by a surface sanding wheel or sandblast. In order to maintain a plane surface, it is recommended that the metal blocks be finished on a flat emery surface.

11. Report

11.1 The report shall include the following:

11.1.1 Complete identification of the material tested, including type or grade of substrate and interlayer, thickness, manufacturing history, and so forth,

11.1.2 The block adhesive used,

11.1.3 The atmospheric conditions in the test room,

11.1.4 The total load, in newtons (or pounds-force), required to break each specimen,

11.1.5 The unit stress, in pascals (or pounds-force per square inch), required for failure (calculate the unit stress by dividing the load by the area of the test specimen), and

11.1.6 Failure mode (such as within the interlayer, or at which interface).

TEST METHOD B—INTERLAMINAR SHEAR STRENGTH

12. Summary of Test Method

12.1 The bond is subjected to mechanical load in the direction of the plane of the interlayer. The maximum adhesive or cohesive strength between the interlayer and the outer plies (shear strength) is determined, and is expressed in pascals (or pounds-force per square inch).

13. Apparatus

13.1 *Shear Tool*—A shear test fixture of the sliding type which is so constructed that the specimen faces are firmly supported between the stationary and movable blocks to minimize peel effects. Suitable forms of shear tools are shown in Figs. 3 and 4, depending on specimen type.

13.2 *Testing Machine*—See 6.2.

14. Test Specimen

14.1 The test specimens shall be either three-ply or five-ply construction as shown in Figs. 5 and 6. The five-ply construction is preferred, especially for specimens with relatively thick interlayers of 2.5 mm (0.1 in.) or more.

14.2 The test specimen shall be 50 mm (2 in.) square minimum. Increasing specimen size will give slightly better accuracy up to the point where the face plies begin to fracture. Prepare the specimens in such a manner as to produce smooth edges to minimize premature edge chipping during testing.

14.3 Orient the samples to duplicate the actual loading conditions in service whenever possible.

14.4 *Number of Test Specimens:*

14.4.1 Test at least five specimens for each sample in the case of isotropic materials.

14.4.2 Test ten specimens, five normal to, and five parallel with the principal axis of anisotropy, for each sample of anisotropic material.

14.4.3 Discard specimens that break at some obvious flaw and retest, unless such flaws constitute a variable whose effect is desired for study.

15. Conditioning

15.1 Condition the specimens in accordance with Section 9.

16. Procedure

16.1 Measure and record the length and width of the bond area with a suitable micrometer to the nearest 0.025 mm (0.001 in.).

16.2 Place the specimen in the test fixture, taking care to align the loaded end of the specimen parallel to the loading bar.

16.3 Set the speed of testing at 1.25 mm (0.05 in.)/min and start the testing machine.

16.4 Record the maximum load carried by the specimen up to the point of rupture.

16.5 Remove and examine the test specimen for evidence of premature failure due to edge chipping or slippage of the specimen in the fixture. If premature failure has occurred, discard the sample and retest another sample.

16.6 Calculate the bond stress by dividing the maximum load by the bond area. For three-ply tests, the bond area is the area of one of the bond-line surfaces; for five-ply tests, the area is two times the area of one of the bond-line surfaces.

17. Report

17.1 The report shall include the following:

17.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, configuration principal dimensions, and previous history,

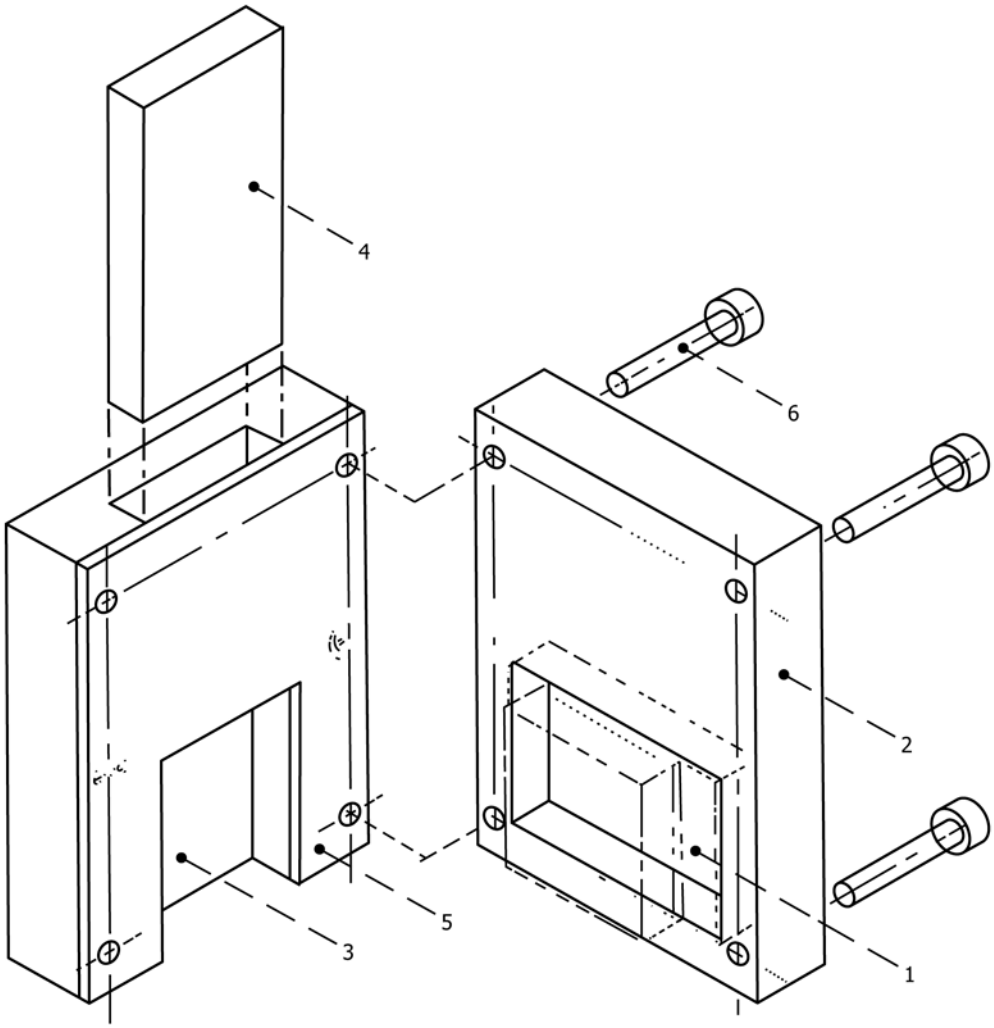
17.1.2 The size of the specimen and direction of loading,

17.1.3 The conditioning procedure,

17.1.4 The total load, in newtons (or pounds-force), required to break each specimen,

17.1.5 The bond shear stress, in pascals (or pounds-force per square inch), and

17.1.6 Failure mode (such as within the interlayer or at which interface).



1. Three-ply shear test specimen.
2. Female steel housing.
3. Male steel housing.
4. Loading bar (hardened steel).
5. Shim (same thickness as the interlayer).
6. Bolts.

FIG. 3 Three-Ply Shear Test Fixture

TEST METHOD C—CREEP RUPTURE

18. Summary of Test Method

18.1 The bond is subjected to a specified duration of load application under a variety of environmental conditions. The time to failure or mode of failure, with a given load, is determined.

19. Significance and Use

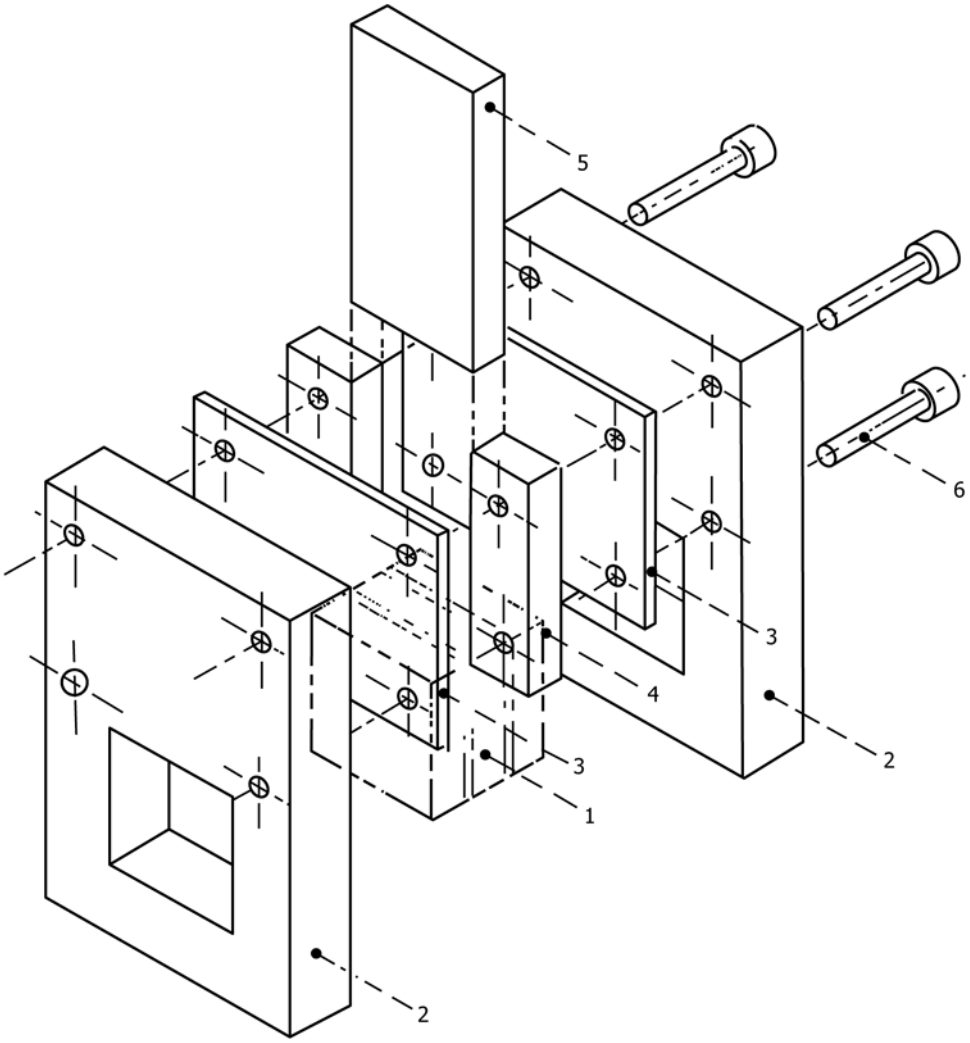
19.1 Data from creep tests are of considerable importance in predicting the performance of materials with variations of design or interlayer materials. Variations include elevated or low-temperature testing, incorporation of specific edge design configurations such as bus bars, separator films, or coated substrates and symmetric or asymmetric loading conditions.

19.2 The test is generally not suitable for quality control acceptance testing due to the extended time periods required to obtain results.

20. Apparatus

20.1 *Metal Blocks*—See 6.1.

20.2 *Testing Machine*—A tension testing machine with a constant load setting and a load indicator is suitable for performing this testing. This type of loading affords a wide range of applied loads, but due to the time-consuming nature of



1. Five-ply shear test specimens.
2. Steel housing.
3. Shim (same thickness as the interlayer).
4. Steel spacer.
5. Loading bar (hardened steel).
6. Bolts.

FIG. 4 Five-Ply Shear Test Fixture

the test, limits the number of specimens that can be tested within a period of time. The preferred testing machine is either a commercial creep machine, or a weighted lever mounted on a steel frame (see Fig. 7). It is possible to construct test systems with several stations and a variation of loads by appropriately positioning a slotted lead weight along the loading lever arm.

20.3 *Adhesive*—Any suitable adhesive.[4]

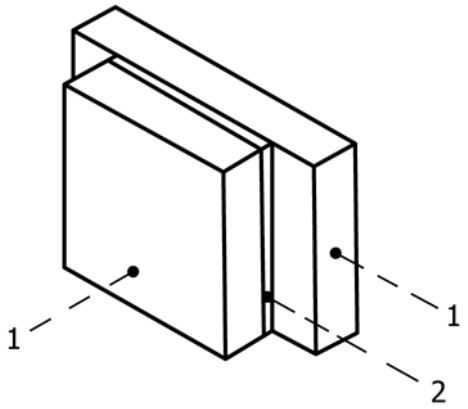
21. Test Specimen

21.1 See Section 7.

22. Preparation of Apparatus

22.1 Prepare assembly in accordance with Section 8. If eccentric loading is desired, prepare assembly in accordance with Note 3 and Fig. 8.

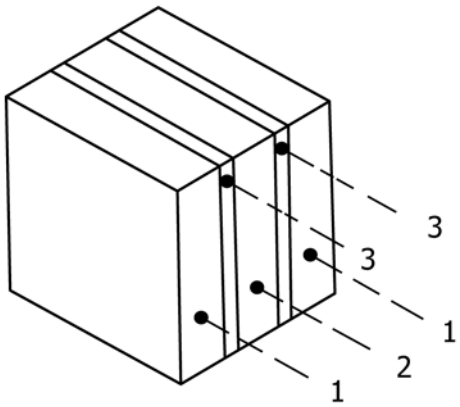
NOTE 3—In reference to Fig. 8, the specimen shall be mounted concentrically (solid lines) or eccentrically (dotted lines) according to the loading area. Instead of the eccentrically mounted specimen, the holes in the "T" block are moved on a horizontal centerline toward each edge of the "T" block to produce an eccentric load when the specimen is concentrically mounted. Variations of the specimen such as separators or inserts in the interlayer are evaluated by this test method.



1. Face plies.

2. Interlayer.

FIG. 5 Three-Ply Shear Test Specimen



1. Face plies.

2. Center ply.

3. Interlayer.

FIG. 6 Five-Ply Shear Test Specimen

23. Conditioning

23.1 Condition test specimens for creep testing to obtain consistent moisture content and temperature. Unless otherwise specified, condition specimens in accordance with Section 9, with conditions remaining constant during the test.

24. Procedure

24.1 When the adhesive has cured, insert the specimen assembly in the tension machine or test frame and apply the specified dead-weight load. Record time at application of the load, at initiation of failure, and at complete failure. Initiation is often indicated by the appearance of a small delamination, void, or bubble. This void or bubble is observed through the polished sides of the specimen.

24.2 If the specimen does not fail after the specified time interval, remove the load from the test assembly.

24.3 For testing the material specified, this test method is so designed that failure between the adhesive and metal shall not occur. If failure does occur between adhesive and metal, discard the test and test another specimen.

25. Report

25.1 The report shall include the following:

25.1.1 Complete identification of the laminate tested, including type or grade of substrate and interlayer, thickness of each component, manufacturing history, supplier, and so forth,

25.1.2 The block adhesive used,

25.1.3 The total load applied,

25.1.4 The preconditioning data,

25.1.5 The testing environment (temperature and humidity),

25.1.6 The unit stress in pascals (or pounds-force per square inch),

25.1.7 Time to failure or strain measurement, or both, up to failure, and

25.1.8 Failure mode.

TEST METHOD D—THERMAL EXPOSURE

26. Summary of Test Method

26.1 The bond is subjected to extreme temperature changes (thermal shock). This usually refers to low temperatures since most multilayer fabrication is done at elevated temperatures. The resistance of the transparency to cracking, spalling, or delamination (thermal exposure resistance) is determined.

27. Apparatus

27.1 Suitable cooling and heating apparatus shall be used to effect the desired rate of change of temperature. Cooling is usually accomplished by refrigeration. Heating methods include a circulating air oven, or by built-in heaters on the transparency.

28. Test Specimen

28.1 Because thermal expansion stresses are related to specimen size, the sample shall be the full-size transparency, unless otherwise agreed upon between the supplier and the user.

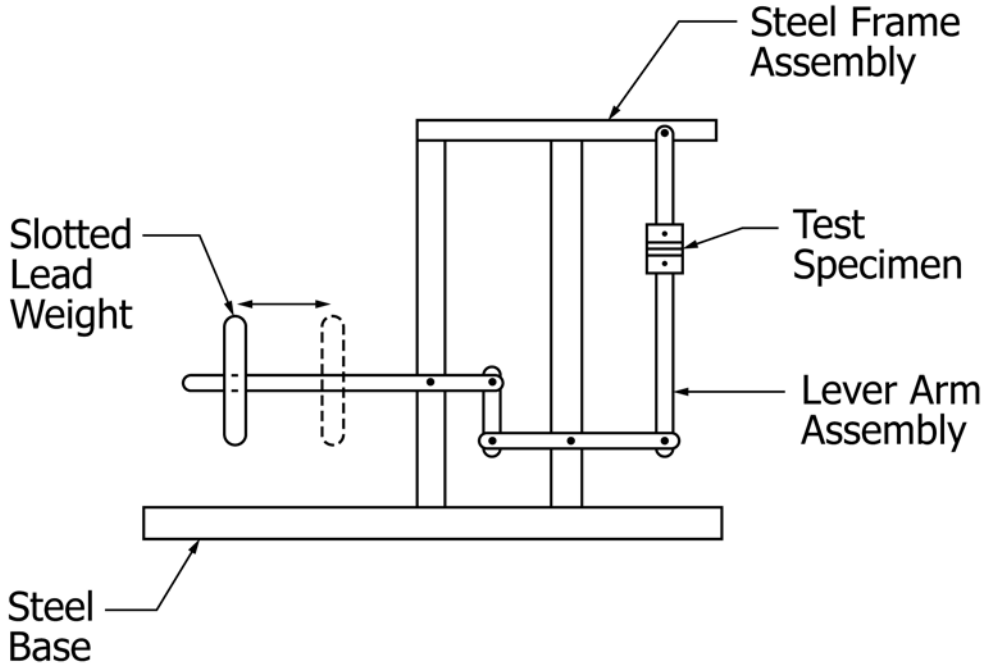


FIG. 7 Lever Loading Frame for Dead Weight Specimen

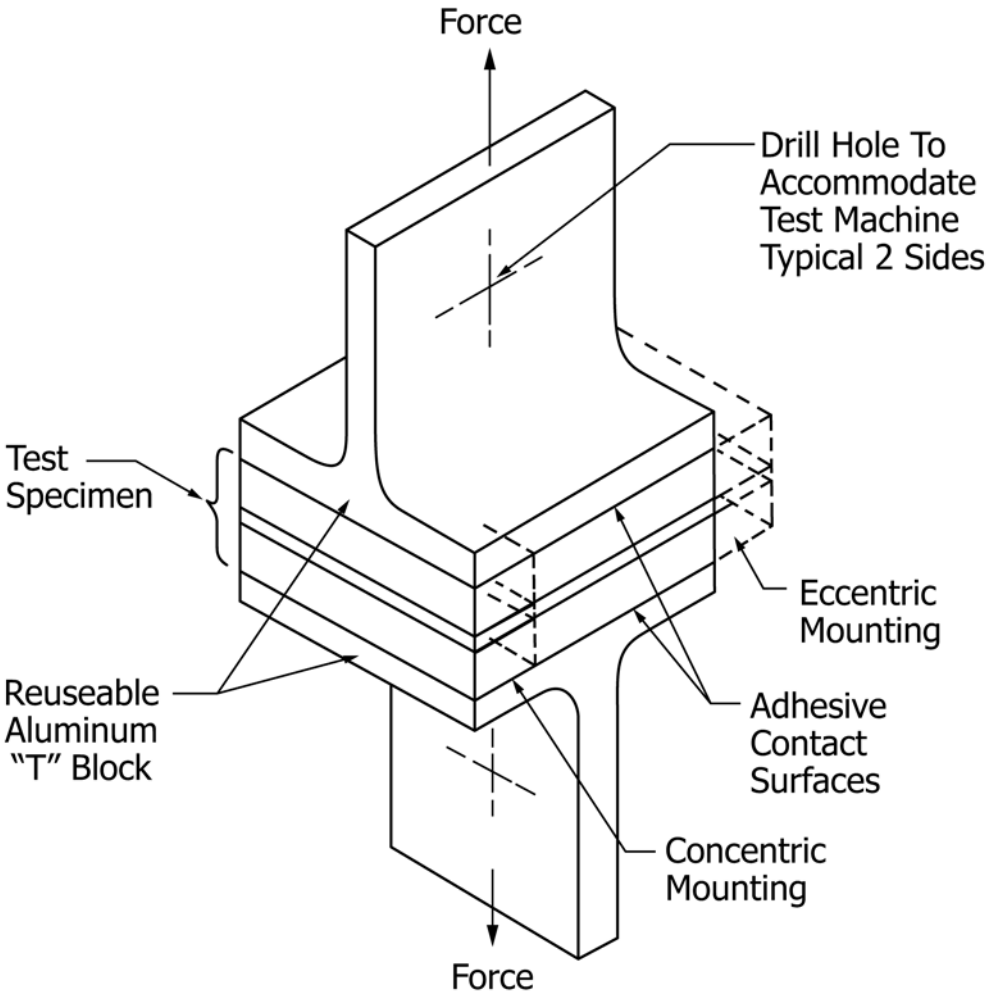


FIG. 8 Creep Test Specimen

29. Procedure

29.1 Rates of heating and cooling vary with each usage. Therefore, the exact procedure to be used to determine the thermal shock resistance shall be as agreed upon between the supplier and the user. Some guidelines which shall be considered are:

29.2 Mechanically restraining the part so as not to relieve stresses or break a component by bending.

29.3 Soaking the part at the lowest soak temperature anticipated in service for a time necessary to reach steady state (thickness dependent, usually less than 4 h).

29.4 Application of full aircraft voltage to the anti-ice coating until control temperature is achieved. Sometimes a ramp warm-up feature is included in the controller function, which is duplicated in the test. In the event an oven is used, the test part temperature at insertion and the rate of rise shall be as agreed upon between the supplier and the user.

30. Report

30.1 The report shall include the following:

30.1.1 Complete identification of the laminate tested, including type or grade of substrate and interlayer, thickness of each component, manufacturing history, supplier, and so forth,

30.1.2 Complete description of apparatus, set-up method of measurement, temperature, tolerances, and so forth,

30.1.3 Method of exposure to temperature extreme (including thermal profiles and number of exposures),

30.1.4 Time of failure (if any), and

30.1.5 Description of failure mode.

31. Precision and Bias

31.1 *Precision*—The task force responsible for this test method is planning to initiate an interlaboratory test to obtain data for this precision section. Upon completion this test method will be submitted for ballot with the new precision section. Interested parties are encouraged to contact the task force chairman through ASTM headquarters to obtain the latest status on this endeavor.

31.2 *Bias*—Bias cannot be evaluated as there are no absolute or accepted standards for the properties measured.

32. Keywords

32.1 adhesion; bond integrity; bond strength; interlayer; shear strength; tensile strength; transparent laminates