Designation: F904 – 16

Standard Test Method for Comparison of Bond Strength or Ply Adhesion of Similar Laminates Made from Flexible Materials[1]

This standard is issued under the fixed designation F904; 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.

INTRODUCTION

It has been widely discussed in the literature that bond strength of flexible multi-ply materials is impossible to measure with current technology. The above is recognized and accepted, since all known methods of measurement include the force required to bend the separated layers, in addition to that required to separate them. However, useful information can be obtained when one realizes that the bending force is included and that direct comparisons between different materials, or even between the same materials of different thicknesses, cannot be made. Also, conditioning that affects the softness or moduli of the plies will be reflected in the bond strength measurement.

1. Scope

1.1 This test method covers a procedure for comparing the bond strength or ply adhesion of similar laminates made from flexible materials such as cellulose, paper, plastic film, and foil. This includes laminates made by various processes: adhesive laminates, extrusion coatings, extrusion laminates, and coextrusion.

1.2 Because of the impact of the bending force, direct comparisons between different materials or even between the same materials of different thickness cannot be made.

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

1.4 *This standard does not purport to address all of the safety problems, 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.* Specific precautionary statements are given in 7.1.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D882 Test Method for Tensile Properties of Thin Plastic Sheeting

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *adhesive failure*—failure at the interface of two adjacent layers.

3.1.2 *cohesive failure*—failure within one of the two adjacent layers comprising the bonded area under test.

3.1.3 *bond strength*—amount of force or energy required to separate plies of material or materials plus the force to bend the plies.

3.1.4 *necking*—localized reduction in cross section which may occur in a material under tensile stress.

3.1.5 *web*—refers to roll stock after it has been unwound from the roll.

4. Summary of Test Method

4.1 Ply separation is initially started mechanically by the application of heat or by using a solvent. The separated plies of the test specimen are placed into the grips of a tensile testing machine. The grips are then separated and the force required to further separate the plies is defined as bond strength. Alternatively, the energy may be used.

NOTE 1—The force to bend the separated plies is included.

[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Packaging and is the direct responsibility of Subcommittee F02.20 on Physical 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

5. Significance and Use

5.1 Laminates are made by bonding together two or more layers of material or materials. Their performance is often dependent on the ability of the laminate to function as a single unit. If the plies have not been properly bonded together, the performance may be adversely affected. Laminates may maintain adequate bond strength under standard test conditions, but under conditions of use may exhibit an increase or decrease in bond strength. Applying heat, such as in boilable pouch applications, may adversely affect bond strength, as may cold temperatures, such as those encountered in freezer storage of foods. Fats and oils may also influence bond strength as well as the softness or moduli of the plies. This test method can be used to compare bond strength of similar materials and to study changes under these and other conditions of end use, providing caution is used with respect to the effects of the included force to bend the separated plies.

6. Apparatus

6.1 *Grips*—A gripping system that minimizes both slippage and uneven stress distribution is required. Grips lined with thin rubber, crocus cloth or pressure sensitive tape, as well as file-faced or serrated grips have been successfully used for many materials. Air-actuated grips have been found advantageous, particularly in the case of materials that tend to "neck" in the grips, since pressure is maintained at all times.

6.2 *Testing Machine*—A tensile testing machine conforming to the requirements of Test Method D882.

6.3 *Specimen Cutter*—In accordance with Test Method D882.

7. Reagents and Materials

7.1 *Solvents*—Toluene, ethyl acetate, MEK (2-Butanone) THF (tetrahydrofuran) or other suitable solvent to weaken the bond between layers sufficiently so that delamination may be started.

7.1.1 **Warning—***Use of these solvents requires that appropriate safeguards be used to avoid hazards of skin contact, inhalation, and flammability.*

8. Sampling, Test Specimens, and Test Units

8.1 Sampling must be performed in a manner that will provide the desired information. No single procedure can be given for all situations. Therefore, Practice D1898 should be used as a guide in planning sampling procedures.

8.2 *Test Specimens*—Cut strips 1.0 in. (25 mm.) wide ± 5% and about 10 in. (250 mm) long. It is important that the test specimens are cut with clean, uniform edges so as not to affect the test results.

8.3 *Test Unit*—Test five specimens in the longitudinal (machine) direction. It may be desirable to test specimens in the transverse (cross-machine) direction for special purposes.

9. Preparation of Apparatus and Calibration

9.1 Equip the tensile testing machine according to manufacturer's instructions for tensile testing thin films.

9.2 Set full-scale load so that most test specimen scans fall in the center two thirds of the chart, and draw speed at 28.0 cm/min ± 10 % (10 or 12 in./min are included). A few trial runs may be required. Other draw speeds may be used if it can be shown that they yield the same results as those specified.

10. Conditioning

10.1 *Specimen Conditioning:*

10.1.1 Store specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h.

10.2 *End-Use Specimen Conditioning :*

10.2.1 Store specimens at the specific end-use temperature and humidity for not less than 40 h.

10.2.2 Accelerated testing conditions for "wet" materials packaging may be accomplished by placing the test specimens between paper towels saturated with distilled water, sealing in a moisture proof pouch and storing at 23 ± 2°C (73.4 ± 3.6°F) for 40 h.

10.2.3 Conditioning to simulate other conditions of end use shall be determined by specific application. Normally the conditioning period will be 40 to 96 h.

10.2.4 In some cases, conditioning will consist of treatment of test specimen prior to actual conditioning. Example: Boilable pouch applications. Test specimens may be immersed in boiling water for a period of time equal to normal end use and then conditioned for testing as specified in 10.1.1 (or perhaps tested shortly after boiling treatment if desired to simulate performance in use).

NOTE 2—At these conditions pouch and contents should be aged to allow time for contents to migrate into seal area.

10.3 *Test Conditions:*

10.3.1 Conduct tests in the standard laboratory atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity.

11. Procedure

11.1 Initiate separation of the plies of the test specimens mechanically if possible. That is, crinkle roughly or apply adhesive tape to both sides, or both, and pull apart. If not, initiate the separation by making a heat seal and pulling it apart. If both the above fail, determine which solvent seems best by trial and error and initiate the separation by immersion of the end of the strip in the solvent for as little time as possible to initiate the separation. Heat the solvent solution only if absolutely necessary. Dry well in room air without heat.

11.2 Clamp the separated ends of the test specimen in the jaws of the tensile testing machine using an original jaw distance of 25.4 mm (1.0 in.) and make certain the jaws are aligned vertically.

11.3 The unseparated portion of each test specimen shall be treated in one of the following ways:

11.3.1 Left loose to move around freely,

11.3.2 Supported at 90° to the direction of draw by hand, or

11.3.3 Mechanically supported at 90° to the direction of draw.

11.4 Activate the tensile testing machine and record the force to separate 3 in. of the test specimen at 280 mm/min ± 10 % (10 or 12 in./min). Repeat for each test specimen in the test unit.

12. Calculation of Results

12.1 Disregarding the initial peak, determine the average force to separate the next 2 in. of each test specimen. Express in newtons/metre, g/25.4 mm, or pounds force/inch.

12.2 Alternatively, determine the energy to separate this 2-in. segment of each test specimen. Express in joules/metre or foot pound-force/inch.

13. Report

13.1 The report shall include the following:

13.1.1 Complete sample identification.

13.1.2 Statement of conditioning.

13.1.3 Name and model number of tensile machine used.

13.1.4 Method of supporting unseparated portion of test specimen.

13.1.5 Film direction if different than longitudinal.

13.1.6 Method of separation.

13.1.7 Number of specimens tested.

13.1.8 Average force or energy to separate each ply of each test specimen.

13.1.9 Average force or energy, standard deviation, and confidence limits for each test unit.

13.1.10 Type of failure—adhesive or cohesive failure.

TABLE 1 Bond Strength

NOTE 1— Values expressed in units of g/25.4 mm

Material	Thickness or other Condition	Average	Sr[A]	SR[B]
2	0.5 mil Oriented Polyester/ metallization/2.5 mil Polyolefin	258.4	12.6	79.4
1	5.0 mil PVC/2.0 mil Ionomer	409.1	20.0	77.6
3	1.5 mil Nylon/PVdC/4.0 mil Poly-olefin	805.7	274.9	743.0

[A]Sr is the within-laboratory standard deviation.
[B]SR is the between-laboratories standard deviation.

14. Precision and Bias

14.1 *Precision*—Table 1 is based on a round robin conducted in 1989 in accordance with Practice E691, involving three materials tested by seven laboratories.[4] For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories which tested them. Each test result is an individual determination. Each laboratory obtained ten test results for each material.

14.2 *Bias*—There are no recognized standards by which to estimate bias of this test method.

15. Keywords

15.1 adhesion; bond strength; flexible barrier materials; laminates

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1008.

APPENDIX

(Nonmandatory Information)

X1. BACKING SAMPLES WITH CELLOPHANE TAPE BEFORE MEASURING BOND STRENGTH

X1.1 Sometimes bond strengths are sufficient to cause tensile breaks of the plies before delamination occurs. When this is experienced backing either the samples or test specimens with cellophane tape will often allow measurement of a value for bond strength. This technique is sometimes helpful when studying particular materials. However, it must be used with caution and the full knowledge that the force to bend the tape including any variations in both the thickness of the tape and its adhesion to the test specimen will be reflected in the bond strength values obtained.

X1.2 Until this technique is better established, it should be used for special studies only and not for establishing values between laboratories.