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Standard Practice for Separation of Plies for Bond Strength of Laminated Flexible Materials¹

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1. Scope

1.1 This practice describes techniques for separating plies of laminates made from flexible materials such as cellulose, paper, plastic film, and foil to enable the measurement of the bond strength or ply adhesion of the laminate. This includes laminates made by various processes: adhesive laminates, extrusion coatings, extrusion laminates, and coextrusions.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 6.1.1.

1.4 *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

[F88 Test Method for Seal Strength of Flexible Barrier Materials](#)

[E171 Practice for Conditioning and Testing Flexible Barrier Packaging](#)

¹ This practice is under the jurisdiction of ASTM Committee F02 on Primary 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. Terminology

3.1 *Definitions:*

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

4. Summary of Practice

4.1 This practice describes three general techniques that can be used to initiate separation of any two plies to facilitate a bond strength measurement. Three techniques are described for use with samples that do not have a non-laminated edge. Technique A describes various forms of mechanical separation. Techniques B and C involve the use of heat or solvents to separate the plies. The separated plies of the test specimen may be placed into the grips of a tensile testing machine to determine the force (or energy) required to further separate the plies, which is defined as bond strength.

5. Significance and Use

5.1 Laminates are made by bonding together two or more layers of material or materials, where each layer might be a single or multi-layer material. When the bonding agent is reactive and requires time to reach full performance, the bond strength is typically measured as a green (un-cured) bond and a cured bond. For processes that intentionally create a non-laminated edge, that edge is generally used to initiate the bond strength measurement. The techniques described in this practice can be used to initiate separation of plies when a non-laminated edge is not present.

6. Reagents and Materials

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

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

7. Sampling and Test Specimens

7.1 Depending on the nature of the sample being evaluated, the size of the sample, and the reason for evaluation, separating the plies in more than one location may be warranted.

8. Conditioning

8.1 Conditioning of samples is not normally necessary provided that any reactive bonding agents have been allowed to fully cure.

8.2 If conditioning before testing is desired and appropriate, then see Practice **E171** for guidance.

9. Procedure

9.1 The following techniques (or a combination thereof) may be employed to initiate separation between the plies of a flexible laminated material.

9.2 *Technique A – Mechanical Separation:*

9.2.1 Initiate separation of the plies of the test specimens mechanically by crinkling roughly or applying adhesive tape to both sides, or both, and pull apart. Alternatively, nick the edge of the film, and tear or stretch the film to initiate separation, or use a tool, such as a pick.

9.3 *Technique B – Thermal Separation:*

9.3.1 If mechanical separation is unsuccessful, and if one of the layers of the lamination can be heat-sealed to itself or another substrate, it may be possible to initiate separation by making a heat seal and pulling it apart.

9.4 *Technique C – Solvent Separation:*

9.4.1 If both of the above methods fail, select an appropriate solvent 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. If the appropriate solvent is not known, start with the least aggressive solvent, and only move to more aggressive solvents if separation is not initiating with the less aggressive solvent. Dry well in room air without heat in a fumehood. Use appropriate safe laboratory practices when using solvents.

NOTE 1—Care should be taken in interpreting results when solvents are used as solvent may wick into the adhesive past where film separation is apparent.

9.5 Once separation has been achieved, a bond strength test using the procedures outlined in Test Method **F88** may be

performed. Sample size and width may vary depending on the sample being tested and should be noted in a report.

9.5.1 Often, 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 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.

9.5.2 Exposure of the laminate to products, environmental conditions, or intended uses may impact its bond strength (such as applying heat in boilable pouch applications, sterilization, or exposure to cold temperatures in freezer storage of foods). Fats and oils may also influence bond strength. Additionally, if the flexible laminated film has come in contact with other chemicals, such as those used in pharmaceutical or diagnostic applications, bond strengths may be impacted as well.

9.6 Additionally, other tests such as FT-IR may be performed to evaluate the adhesive layer between the layers of interest in a laminated structure.

10. Report

10.1 The report shall include the following:

10.1.1 Complete sample identification.

10.1.2 Statement of conditioning, if any.

10.1.3 Method used to separate plies, including specific solvents used.

10.1.4 Subsequent testing performed on sample (for example, bond strength, FT-IR, etc.).

10.1.5 Number of specimens evaluated.

10.1.6 Data collected should be reported in conformance with the requirements of the appropriate test method.

11. Keywords

11.1 adhesion; bond strength; flexible barrier materials; laminates; ply adhesion; ply separation

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