Designation: D4498 – 07 (Reapproved 2015)

Standard Test Method for Heat-Fail Temperature in Shear of Hot Melt Adhesives[1]

This standard is issued under the fixed designation D4498; 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 is intended to determine the temperature at which specimens bonded with hot melt adhesive delaminate under static load in shear.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D907 Terminology of Adhesives

E28 Test Methods for Softening Point of Resins Derived from Pine Chemicals and Hydrocarbons, by Ring-and-Ball Apparatus

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

E171 Practice for Conditioning and Testing Flexible Barrier Packaging

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

3. Terminology

3.1 *Definitions*—Many of the terms found in this test method are defined in Terminology D907.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *heat-fail temperature, n*—the temperature at which delamination occurs under static loading in shear.

4. Significance and Use

4.1 Heat-fail temperature establishes a limiting temperature above which the adhesive is not to be exposed in service under shear load.

5. Apparatus

5.1 A device capable of producing adhesive films of uniform thickness with ±25.4 µm (±1 mil) tolerances.

5.2 *Standard Substrate*—NIST Standard Reference Material 1810 (Liner-Board)[3]

5.3 *Heat Sealing Device*—Sentinel heat sealer or equivalent capable of maintaining selected sealing temperature within ±2.5°C (±5°F).

5.4 *Forced-Ventilation Oven,* manual or programmed. Oven shall be capable of maintaining selected temperatures within ±1 % of the differential between oven and ambient temperatures in accordance with Specification E145, with the programmable oven capable of attaining smooth temperature increases of 30°C/h over a range of 25 to 150°C.

5.5 *Thermometric Device,* for monitoring oven temperatures.

5.6 *TFE-fluorocarbon Cloth,* silicone release paper, 500 g weights and clamping devices for suspending weights and specimens in the oven.

6. Sampling, Test Specimens, and Test Units

6.1 The test sample is to be representative of the adhesive being tested.

6.2 Prepare test specimen films of representative adhesive at a thickness of 76 ± 25 µm (3 ± 1 mil). Inspect the cooled films and reject any containing voids or other imperfections. Cut the films into pieces measuring 25.4 by 25.4 mm ± 1.6 mm (1 by 1 in. ± 0.0625 in.). Measure the thickness of the adhesive film to the nearest 0.013 mm (0.0005 in.). Cut strips of standard substrate measuring 25.4 ± 1.6 by 76 mm (1 ± 0.0625 by 3 in.) with 76-mm (3-in.) dimension in the machine direction.

6.3 Prepare a lap joint measuring 25.4 by 25.4 mm (1 by 1 in.) inside two strips of standard substrate. Place this assembly

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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] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

TABLE 1 Heat-Fail Temperature Round Robin Data, °F

Adhesive Type	Laboratory	1 P[A]	2 M	3 M	4 P	5 M	6 M	7 M	8 M	9 M	10 M	10 P
EVA		145	160	138	149	. . .	145	. . .	140	135	149	132
Polyester		265	270	268	270	275	277	259/265	. . .	260	253	262
Polyamide		261	275	254	259	265	264	250	250	243	253	262
Polypropylene		188	170	124	176	. . .	153	170	152	. . .	158	176
Polyethylene		195	200	184	191	195	192	180	181	163	196	192

[A] P is an abbreviation for programmed oven. M is an abbreviation for manual control oven.

TABLE 2 Summary of Results (10 Laboratories)

Adhesive Type	Av. Heat-fail Temperature, °F	Standard Deviation, °F
EVA	142	9.3
Polyester	263	11.7
Polyamide	256	11.5
Polypropylene	160	19.5
Polyethylene	187	11.2

between a folded sheet of silicone-coated release paper or Teflon cloth (Note 1). Insert the assembly between the platens of the heat sealer having only the upper platen heated. Make the seal under the following conditions and seal at least 6 specimens of each adhesive for test.

6.3.1 *Temperature*—As needed to activate hot-melt adhesive (Note 2).

6.3.2 *Pressure*—103.4 kPa (15 psi).

6.3.3 *Dwell Time*—1.5 s.

NOTE 1—Use of the coated release paper or TFE-fluorocarbon cloth may require a higher than expected sealing temperature because of thermal insulating effects.

NOTE 2—The operator should test at least two bonds after cooling to room temperature to ensure that adequate adhesion has been obtained. Adjust sealing temperature to obtain that adhesion.

6.4 Measure the thickness of the two pieces of substrate, calculate, and record the adhesive thickness, which is to be between 25.4 and 76.2 µm (1 and 3 mils). Condition the bonded specimens for 24 h according to Specification E171 before testing.

7. Procedure

7.1 Randomly select and suspend three bonded replicates in the oven. Attach the weight to the bottom tab of substrate so that the total load is 500 ± 5 g (1.1 ± 0.01 lb). Although the weight designated is recommended, other weights can be used. If so, report.

7.2 Set the initial oven temperature at approximately 25 to 40°C (45 to 70°F) below the softening point of the adhesive, as previously determined in accordance with Test Method E28. Increase the oven temperature sequentially; for manual controls 5°C (10°F) at 10-min intervals, for programmed controls 30°C (60°F)/h. Continue to increase the oven temperature until all specimens fail.

7.3 Record the temperature at which each specimen fails. Average the results for replicate specimens and report to the nearest degree as the heat-fail temperature. Also report three individual results.

8. Report

8.1 Complete identification of the adhesive tested, including type, source, manufacturer's code, lot number, and form in which received.

8.2 Complete identity of substrates used if different from the standard substrate.

8.3 Activation temperature used to make lap joint specimens.

8.4 Weight used if other than 500 ± 5 g (1.1 ± 0.01 lb) test weight.

8.5 A tabulation of test results, including the measured thickness of adhesive for each test specimen as determined in accordance with 6.2.

9. Precision and Bias

9.1 *Precision*—Table 1 and Table 2 show data from round robin testing from an early study. An interlaboratory study using Practice E691 will be conducted to generate data to revise Section 9 to bring it into conformance with the Precision and Bias section in the ASTM Form and Style Manual.

9.2 *Bias*—No statement is made about the bias of this test method since the result merely records a temperature at which failure of bond under shear load is observed.

10. Keywords

10.1 heat fail temperature; hot-melt adhesive; shear