



Designation: **D 4498 – 9500**

Standard Test Method for Heat-Fail Temperature in Shear of Hot Melt Adhesives¹

This standard is issued under the fixed designation D 4498; 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 standard.

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:*

D 907 Terminology of Adhesives²

¹ This test method is under the jurisdiction of ASTM Committee D-14 on Adhesives and is the direct responsibility of Subcommittee D14.50 on Hot Melt and Pressure Sensitive Adhesives.

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- E 28 Test Method for Softening Point by Ring-and-Ball Apparatus³
- E 145 Specification for Gravity-Convection and Forced Ventilation Ovens⁴
- E 171 Specification for Standard Atmospheres for Conditioning and Testing Flexible Barrier Materials⁵

3. Terminology

3.1 Definitions:

3.1.1 ~~Many~~ **Definitions**—Many terms in this test method are defined in accordance with Terminology D 907.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *heat-fail temperature*—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 $\pm 25.4 \mu\text{m}$ (± 1 mil) tolerances.

5.2 *Standard Substrate*—NIST Standard Reference Material 1810 (Liner-Board)⁶

5.3 *Heat Sealing Device*—Sentinel heat sealer or equivalent capable of maintaining selected sealing temperature within $\pm 2.5^\circ\text{C}$ ($\pm 5^\circ\text{F}$).

5.4 *Forced-Ventilation Oven*, manual or programmed. Oven shall be capable of maintaining selected temperatures within $\pm 1\%$ of the differential between oven and ambient temperatures in accordance with Specification E 145, with the programmable oven capable of attaining smooth temperature increases of $30^\circ\text{C}/\text{h}$ 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 $76 \pm 25 \mu\text{m}$ (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 ~~1~~ **25.4 by 25.4 mm (1 by 1 in.)** inside two strips of standard substrate. Place this assembly 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.

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

Adhesive Type	Laboratory	1	2	3	4	5	6	7	8	9	10	10
		P ^A	M	M	P	M	M	M	M	M	M	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.

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.

² *Annual Book of ASTM Standards*, Vol 15.06.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.024.

⁵ *Annual Book of ASTM Standards*, Vol 15.09.

⁶ Available from National Institute of Standards and Technology, Office of Standard Reference Materials, Gaithersburg, MD 20899.

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 $76.2 \mu\text{m}$ (1 and 3 mils). Condition the bonded specimens for 24 h according to Specification E 171 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 $500 \pm 5 \text{ g}$ ($1.1 \pm 0.01 \text{ lb}$). Although the weight designated is recommended, other weights can be used. If so, report.

7.2 Set the initial oven temperature at approximately ~~45~~ 25 to ~~70°F~~ (~~25~~ 40°C (~~45 to 40°C~~) 70°F) below the softening point of the adhesive, as previously determined in accordance with Test Method E 28. Increase the oven temperature sequentially; for manual controls ~~10°F~~ (~~5°C~~) 5°C (10°F) at 10-min intervals, for programmed controls ~~60°F~~ (~~30°C/h~~); 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 $500 \pm 5 \text{ g}$ ($1.1 \pm 0.01 \text{ lb}$) 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*—Tables 1 and 2 indicate the between-laboratories variability. Additional data, including within-laboratory

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

variability is to be provided in succeeding interlaboratory testing.

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

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