Designation: D4497 – 10

Standard Test Method for Determining the Open Time of Hot Melt Adhesives (Manual Method)[1]

This standard is issued under the fixed designation D4497; 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 covers the manual determination of the open-time range of hot-melt adhesives under the stated conditions.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units in parentheses are provided 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

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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 *open time, n*—the time available between application of the adhesive to the first substrate and the mating of the second substrate which still results in a minimum of 50 % fiber tear when tested at ambient conditions.

4. Summary of Test Method

4.1 In this test method, open time is determined by bonding together with the adhesive, pieces of standard paper at intervals of 5 s sequentially after the molten adhesive has been applied using a heated film applicator on one surface. The bonded specimens are examined later to determine the longest time period, between application of the adhesive to one surface and mating with a second surface, which allows substantial bonding.

5. Significance and Use

5.1 This test method allows comparing the open time of several hot melt adhesives.

5.2 This test method is not intended to provide the open-time observed for a specific adhesive in a specific manufacturing operation. This test method can be used to compare the performance of adhesives which are represented to be identical or to rank the open-time of a group of hot melt adhesives.

6. Apparatus

6.1 *Oven,* which meets the precision required by the procedure, should be used to heat the adhesive sample and the applicator.

6.2 *Film Applicator*—Multiple clearance 0.125 to 1.25 mm (5 to 50 mil) square P.G. and T. Number 2 Film Applicator[3] capable of providing an applied film of adhesive having a nominal width of 50 mm (2 in.).

6.3 *Hand Thermometer,* preferably metal, capable of measuring the temperature of the hot-melt with an accuracy of ±1°C (±2°F).

6.4 *Primary Standard Substrate,* 200 by 280 mm (8 by 11 in.), NBS Standard Reference Material 1810 (Liner Board).[4] Substrates are to be conditioned as described in Practice E177.

6.5 *Secondary Standard Substrate,* 125 mm (5 in.) length in the material direction cut to 19 mm (0.75 in.) wide, NBS Standard Reference Material 1810 (Liner Board).[3]

6.6 *Glass Beakers* (600 mL), for melting the adhesive.

6.7 *Watchglass,* to cover beaker.

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[1] This test method is under the jurisdiction of ASTM Committee D10 on Packaging and is the direct responsibility of Subcommittee D10.14 on Tape and Labels.

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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] Film Applicator No. 2 is available from Precision Gage and Tool Company, 375 Gargrave Rd., Dayton, OH 45449, www.precisiongage-toll.com.

[4] Available from National Institute of Standards and Technology (NIST), 2320 Measurement Services Division, Gaithersburg, MD 20899-2320, www.nist.gov.

6.8 *Beakers, Tongs, and Protective gloves,* for handling hot vessels and applicators.

6.9 *Transparent Pressure Sensitive Tape.*

6.10 *Stop Watch or Other Timer,* graduated to at least 0.5 s.

6.11 *Weighted Rectangular Block,* having at least one smooth and flat wood face, 50 by 12.5 mm (2 by 0.5 in.) weighing 100 g (3.5 oz), the mass being uniformly distributed.

6.12 *Micrometer or Thickness Gage,* capable of measuring to the nearest 0.025 mm (1 mil).

7. Sampling

7.1 The test sample should be representative of the adhesive being tested.

8. Procedure

8.1 Add 300 ± 10 g of the adhesive to the 600-mL glass beaker and cover with a watch glass. Place the beaker containing the adhesive into the oven and heat until at the application temperature recommended by the manufacturer.

8.2 Concurrently heat the film applicator in the same oven to condition it to the sample temperature as in 8.1.

8.3 Perform testing in a relatively draft-free room. While the sample is melting, prepare the substrates for testing as shown in Figs. 1 and 2.

8.3.1 Tape the long sides of a piece of primary standard substrate (see 6.4), minimum sheet size 100 by 280 mm (4 by 11 in.), to a flat smooth surface of low thermal conductivity, specifically to a larger piece of chipboard (such as from the back of a tablet). Then tape the chipboard to an underlying surface.

8.3.2 Place the strips of the secondary standard substrate (see 6.5) across the width of the primary substrate, as shown in Fig. 1, leaving approximately 6 mm (¼ in.) between the strips. The top strip should be approximately 75 mm (3 in.) below the top edge of the primary substrate. Using pressure sensitive tape, attach one end of the strips to the primary substrate. Fold the unattached end of each strip back over the attached end so that the primary substrate is exposed for application of the adhesive (see Fig. 2).

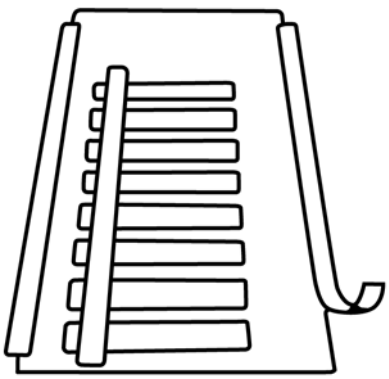

FIG. 1 Secondary Substrate Strips on Primary Substrate Before Hot Melt Application

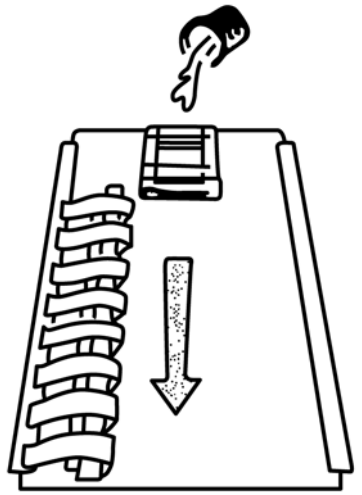

FIG. 2 Strips Drawn Back to Allow Hot Melt Application

8.4 Remove the sample from the oven, and by stirring with the metal thermometer, quickly verify that the temperature is within ±3°C (±5°F) of the manufacturers recommended application temperature.

8.5 Quickly remove the adhesive applicator from the oven, and center it above the top strip of the secondary substrate. The 0.5 mm (20 mil) gap should face down and be near the top end of the primary substrate.

8.6 Fill the applicator reservoir to approximately a ¼-in. level with conditioned hot adhesive.

8.7 Move the applicator and the contained molten adhesive with one smooth motion over the length of the paper taking approximately 2 s to complete the drawdown.

NOTE 1—Approximately 2 s is satisfactory. The purpose is to standardize on a reasonable rate and eliminate an excessively fast or slow drawdown which will affect the results.

NOTE 2—Trial applications should be made to determine the proper applicator gap to lay down a specified film thickness. Normally, a gap of 0.50 mm (20 mil) will lay down a film of 0.25 mm (10 mil). Variations in applied film thickness will occur because of differences in the hot flow properties of the adhesive.

8.8 Immediately after the film has been drawn down, start the timer.

8.9 At 5 s on the timer, or a longer time if it is known that an adhesive will have an open time longer than about 30 s, lay the top piece of secondary substrate across the cooling film, and continue sequentially at 5 s intervals. Use the weighted block to apply immediately a momentary load of 5 s duration to press the strips onto the adhesive, moving the block to successive strips as they are laid onto the adhesive. See Fig. 3. The wood face of the block faces the substrate. Repeat this operation until it is obvious the adhesive film has solidified and will not bond to the secondary substrate.

8.10 Allow the adhesive film to cool at ambient temperature for 30 to 60 min before measuring.

8.11 Measure the combined thickness of the adhesive film and the two substrates. Subtract the thickness of the two

TABLE 1 Open Time (Seconds)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	S_R	r	R
Ethylene-Vinyl Acetate	7.8	1.2	2.7	3.3	7.5
Polyester	6.7	1.8	3.2	5.1	9.1
Polyethylene	16.1	1.7	2.9	4.7	8.0
Polyamide	17.5	2.6	5.2	7.4	14.4
Polypropylene	20.8	2.0	8.4	5.7	23.5

[A] The average of the laboratories' calculated averages.

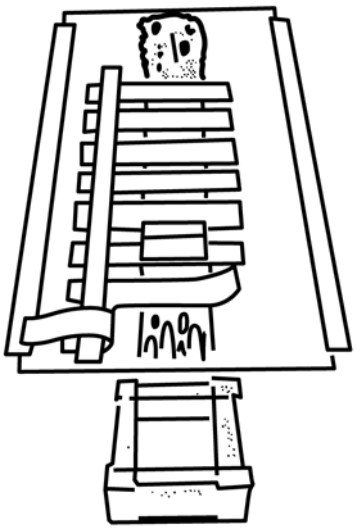

FIG. 3 Strips Successively Placed onto Hot Melt

substrates to determine the nominal adhesive thickness to the nearest 0.025 mm (1 mil).

8.12 With a uniform motion, peel off each strip of secondary substrate at an angle of 90° to the surface. Take about 10 s to peel off each strip. If the paper tears near the initial edge, repeat the peel from the opposite end of the strip. Record the percent area of fiber tear for each strip and the time in seconds at which that strip was laid onto the cooling adhesive.

8.13 Repeat the entire test procedure in 8.1 – 8.12 for a total of three determinations.

9. Report

9.1 Report the mean open-time of the three replicate determinations as the longest time, to the nearest 5 s, at which a minimum of 50 % fiber tear is obtained.

9.2 Report the ambient temperature and relative humidity.

9.3 Report nominal adhesive thickness, to the nearest 0.025 mm (1 mil), as the mean of the three replicate determinations.

9.4 Report the application temperature used (see 8.4).

10. Precision and Bias

10.1 The precision of this test method is based on an interlaboratory study of D4447, Test Method for Determining the Open Time of Hot Melt Adhesives (Manual Method). Each of six laboratories tested five different materials. Every "test result" represents an individual determination, and all participants reported triplicate test results. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D14-1016.[5]

10.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more that the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

10.1.1.1 Repeatability limits are listed in Table 1.

10.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

10.1.2.1 Reproducibility limits are listed in Table 1.

10.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

10.1.4 Any judgment in accordance with statements 10.1.1 and 10.1.2 would have an approximate 95 % probability of being correct.

10.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

10.3 The precision statement was determined through statistical examination of 87 results, from six laboratories, on five materials.

10.4 To judge the equivalency of two test results, it is recommended to choose the material in characteristics to the test material.

11. Keywords

11.1 application temperature; open time

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D14-1016.