Designation: ~~D7998 – 15~~ D7998 – 19

Standard Test Method for Measuring the Effect of Temperature on the Cohesive Strength Development of Adhesives using Lap Shear Bonds under Tensile Loading[1]

This standard is issued under the fixed designation D7998; 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 concerns bonding and testing of wood adhesives and related adhesives using small scale tensile lap-shear samples in a manner that emphasizes transient cohesive strength as a function of bonding time and temperature.

1.2 Use of thin adherends enables bondlines to be rapidly heated to elevated temperatures and maintained at those temperatures for a range of times at a controlled pressure before testing.

1.3 Optional rapid forced air cooling of bonds after pressing and immediately before testing enables the effect of testing temperature on transient strength to be evaluated.

1.4 Bond overlap distance is specified to ensure that failure occurs in the bondline rather than in unbonded portions of adherend strips, and also to minimize the effect of shear stress non-uniformity along the overlap during tensile testing.

1.5 Standard wood or alternative non-standard materials must be of specified high quality and uniformity of structure and dimension to minimize variability of bonding and maximize stress transfer into the bonds during testing.

1.6 The effect of wood variability and type, or of the properties of alternative non-wood materials, on bond strength development may be explored using the method.

1.7 Optional hermetic sealing of bond overlaps during their heated pressing enables the effect of moisture on bonding to be evaluated.

1.8 Thermal damage, either of pre-formed bonds or by prolonging bond forming times, may be evaluated as a function of time and elevated temperature using this test method.

1.9 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.10 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use. Some specific hazards statements are given in Section ~~10~~10 on Hazards.*

1.11 *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]

D907 Terminology of Adhesives

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

3. Terminology

3.1 See Terminology D907 for other terms related to adhesives.

3.2 *Definitions of Terms Specific to This Standard:*

[1] This test method is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.30 on Wood Adhesives.

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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.

3.2.1 *adherend strip, n*—one of two adherend pieces that constitute a test specimen.

3.3 *adhesive spread rate, n*—the mass of adhesive applied per unit surface area of the overlap region of the adherend.

3.4 *contact heating, n*—the transfer of heat energy to the test specimen by the application of precisely temperature-controlled heads pressing on the test specimen's surfaces with controlled force.

3.5 *forced air cooling, n*—the rapid cooling of the overlap region of a test specimen after bonding at elevated temperature by the controlled application of air jets onto both external faces of the overlap portion of the specimen.

3.6 *test specimen, n*—the lap shear sample made up of an adhesive between two pieces of adherend.

3.7 *thermal damage, n*—the decrease in measured bond strength due to exposure to a specified elevated temperature for a specified time.

3.8 *transient bond strength, n*—the strength of a partially formed test specimen bond (sometimes referred to as "green strength") upon being tested.

4. Significance and Use

4.1 The test method enables strength values for wood and other materials bonded with an adhesive under a range of controlled bonding temperature, time, and pressure conditions to be evaluated. Bond formation and subsequent testing is affected in a coordinated fashion, and this enables transient strength values of sets of similar bond types to be explored with diverse parameters as independent variables. Principal among these variables is the temperature at which bonds are formed and the time that selected temperatures are maintained prior to testing. The use of controlled methods of adhesive application, the rapid attainment of stable bond formation conditions, and the rapid transition to the bond testing mode enables snapshots of bond strength to be attained as bonds progress from limited strength (or initial tack) to maximum strength. Derived data may be used to evaluate and compare the strength development characteristics of diverse types and formulations of adhesive. The method may thus be used to aid in tailoring and matching adhesives to the manufacture of diverse bonded products that involve heating.

4.2 The method may also be used to evaluate the co-dependent effect of temperature and time on the degradation of sample bonds. Pressing temperatures up to 265°C (509°F) may be necessary for such investigations of thermal degradation. Specimens are pressed for a range of times and temperatures and very shortly thereafter tested either at elevated temperature or immediately following rapid forced air cooling. Alternatively, thermal damage of pre-formed bond samples may be evaluated by subjecting them to controlled temperature and time sequences prior to testing.

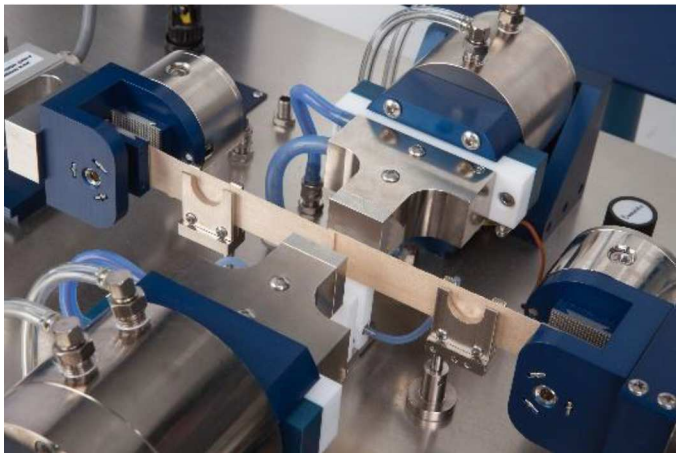
4.3 The method may also be used to evaluate the effect of wood type and variability, or of non-wood materials, on bond strength development.

4.4 By hermetically sealing the overlap region of sample bonds during their formation, the method may also be used to evaluate the effect of moisture and other resident volatile fluids on bond strength development.

4.5 The method may also be used to evaluate the effect that the temperature at which variously formed bonds are tested has on their strength. Controlled rapid forced air cooling immediately after bond formation but before testing is necessary for such investigations. This approach may be employed to explore the thermoplastic characteristic of thermosetting adhesives and also the strength of hot melt adhesives as a function of pressing and testing temperatures.

5. Apparatus

5.1 *Integrated Bond Forming and Testing Apparatus:*



A typical test sample mounted in an apparatus for rapid bond heating and pressing followed by bond pulling.

FIG. 1 Typical Test Sample[3]

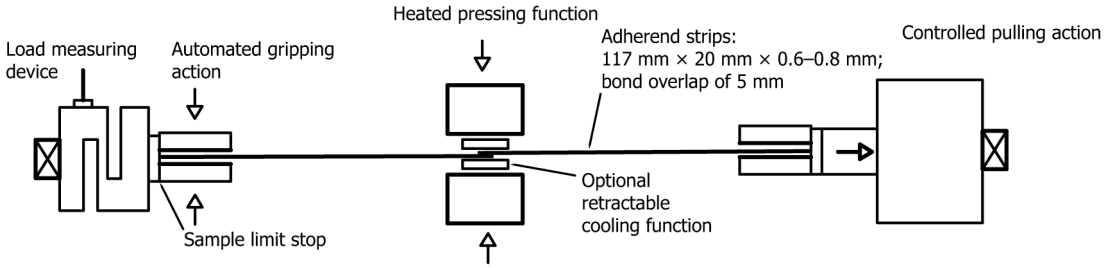


FIG. 2 Schematic of the Bonding and Testing Concept

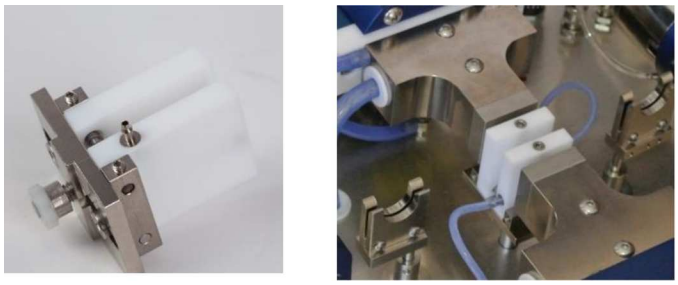
5.1.1 *Sample Forming Function*—A small laboratory scale apparatus that has two horizontally opposed temperature-controlled pressing heads measuring approximately 25 by 25 mm (1.0 by 1.0 in.) is required. The heads must close synchronously to a controlled force and dwell time in order to form adhesively bonded specimens in a lap fashion. The specimens must be in line with the gripping mechanisms of the apparatus (Fig. 1[3] and Fig. 2) and position stops must be provided within those mechanisms to ensure accuracy of bond overlap distance during sample positioning. Temperature control of the pressing heads between ambient and 150°C (302°F) must be maintained within ±1.5°C (2.7°F). Pressing force must be controllable between 20 and 800 ± 2 N (4.5 to 180 ± ~~0.45lbf),~~ 0.45 lbf), and press closure from the open position must be achievable within 2 s.

5.1.2 *Bond Testing Function*—For measuring transient bond strengths, the apparatus requires an integrated mechanical loading function which acts in an axis perpendicular to that of the sample forming function. Automated gripping of the end portions of test specimens is necessary to avoid any disruption of partially formed bonds prior to their being pulled. The apparatus must be capable of applying tensile testing force at a controlled rate of ~~500N/s~~ 500 N/s ± 25 N/s (112 ± ~~5.6lbf/s)~~ 5.6 lbf/s) up to at least ~~1kN (224lbf),~~ 1 kN (224 lbf), and load must be digitally sampled at a frequency of not less than 2 kHz during sample pulling in order to capture the peak load at bond failure with an accuracy of ~~± 2N (0.45lbf).~~ ± 2 N (0.45 lbf). The apparatus must be capable of affecting the transition from press opening to the onset of pulling within 2 s. Alternatively, if forced air cooling is employed after press opening according to Alternative Test Method I and 5.1.3, then the onset of pulling must be possible within 2 s of the cessation of bond cooling.

5.1.3 *Rapid Bond Cooling Function*—Bonds may be tested after rapid cooling to ambient temperature by employing Alternative Method I, 9.1. A small retractable forced air attachment is required to affect rapid and controlled temperature reduction of test bonds when the pressing heads are retracted at the end of the bond forming stage and immediately prior to the bond pulling stage. The transition between press opening and the activation of cooling must be achievable within 2 s. The cooling function must be capable of reducing the bondline temperature so as not to exceed 31°C (88°F) within 5 s. A possible cooling device is given in Fig. 3.

6. Adherend Type and Preparation

6.1 *Adherend Type*—Unless otherwise noted in the report, uniform, planar and defect-free hard maple (*Acer saccharum spp.*) or European beech (*Fagus sylvatica spp.*) of 0.6 to 0.8 mm (0.024 to 0.031 in.) thickness is to be used. The selected thickness must be maintained within ± 0.04 mm (0.0016 in.) and the measured thickness must be reported. The surfaces to be adhered must be knife-cut and not sanded. Other species and non-wood materials may be employed, and must be reported fully. In such cases, thickness must be minimized to ensure rapid heat transfer into bondlines to achieve target temperatures up to 150 ± 2°C (302 ± 3.6°F) within 15 s, while having sufficient tensile strength to ensure failure in the bondline rather than in the unbonded portions of adherend strips.



An attachment for rapid air-jet cooling which may be raised, activated and lowered automatically.

FIG. 3 Attachment for Rapid Air-jet Cooling

[3] Manufactured and available from Adhesive Evaluation Systems, Inc., 155 SW Madison Ave, Corvallis, OR 97333, www.adhesiveevaluationsystems.com.

6.2 *Adherend Preparation*—Wood or related material is to be loosely stacked and equilibrated at 21 ± 2°C (70 ± 3.6°F) and 50 ± 5 % relative humidity for at least 12 h before adherend strips are cut to size. Photo-degradation due to exposure to sunlight and UV light must be minimized. The strips are to be cut from portions of wood veneer having clear grain to provide strips of 120 ± 0.2 mm (4.724 ± 0.008 in.) length parallel to the grain ±4° and width of 20 mm ± 0.5 mm (0.787 ± 0.02 in.) across the grain. The specified precision in length is required to ensure accuracy of overlap distance when the two adherend strips are mounted in the bond forming and testing apparatus.

7. Adhesive Type and Application

7.1 *Adhesive Type*—A wide range of adhesive types may be evaluated using this test method as long as they may be applied in an accurate and uniform fashion onto the overlap portion of adherend strips. The method is well suited to the evaluation of the effect of temperature on the strength development of thermosetting adhesives. It may also be employed with hot melt adhesives in accordance with Alternative Method III, 9.3. Adhesive type and method of preparation must be reported.

7.2 *Adhesive Application and Spread Rate*—A wide range of adhesive spread rates may be employed, depending upon the adhesive type. A spread rate of 50 ± 5 g/m^2 (6.14 lb/1000 ft^2) is standard. A standard adhesive application method is not specified, but methods could include shielded spraying, contact or transfer printing, spatula, metered droplet deposition and dispersion methods, among others. Care should be taken to maximize uniformity of adhesive distribution over the bonding area. The application method employed and spread rate accuracy achieved must be reported in accordance with 7.3.

7.3 *Adhesive Spread Rate Measurement and Reporting*—Weight gain of adherend strips due to adhesive application using the reported method should be reported. Five randomly selected strips are to be weighed before and after adhesive application over a measured area. Variability in the delay between adhesive application and weighing must be minimized in order to minimize the effect of evaporation on the measurement. In any case, this delay must not exceed 10 s. Mean and standard deviation values of spread rate must be reported in the units of g/m^2 ~~(lb/1000ft~~(lb/1000 ft^2).

8. Bond Formation and Testing

8.1 *Bond Formation*—Adherend pairs are to be mounted in the bond forming and testing apparatus following the application of adhesive to the endmost portion of one of the adherend strips. A non-standard alternative is to apply adhesive to both adherend strips. A bond overlap of 5 ± 0.4 mm (0.197 ± 0.016 in.) is standard (see 8.1.1). The delay between adhesive application and closure of the heated pressing heads onto the overlap should be 7 ± 3 s in order to minimize the effects of adhesive pre-cure, penetration and evaporation. Deviations from these limits must be reported, including when open assembly time is an independent variable. A pressing pressure of 2 ± ~~0.2MPa~~ 0.2 MPa (290 ± 29 psi) is standard. This pressure requires a force of 200 ± ~~20N~~ 20 N (50 ± ~~5lbf)~~ 5 lbf) on the standard bond with a 20 by 5 mm (.787 by .196 in.) overlap. Deviations from this pressure may be necessary, particularly to reduce compression when non-standard wood or related materials of reduced hardness are employed or when pressing pressure is an independent variable. Minimum pressing time must be sufficient to ensure the selected temperature is attained at the bondline within ±2°C (3.8°F). For the standard adherend, this minimum pressing time is 15 s.

8.1.1 Non-standard overlap values must be reported in accordance with Alternative Method V, 9.5. Care must exercised if overlap distances greater than standard are employed since shear stress non-uniformity may be increased, a significant bending moment may be induced during pulling, and failure outside the bond zone may occur. Large overlap distances may be appropriate to explore tack properties (low shear strength) of adhesives.

8.2 *Bond Testing*—The transition from press opening at the end of a selected pressing period to the onset of pulling force rise must be within 2 s. Tensile testing force is to be applied at a rate of ~~500N/s ± 25N/s~~ 500 N/s ± 25 N/s (112 ± ~~5lbf/s).~~ 5 lbf/s). Slower rates may be employed in non-standard tests where the effect of loading rate and duration of load studies are conducted; these methods must be reported in accordance with Alternative Method V of 9.5. Load must be digitally sampled at a frequency of not less than 2 kHz during sample pulling in order to capture the peak load at bond failure within ± ~~2N (.45lbf).~~ 2 N (.45 lbf). Bond forming time, bond strength calculated by dividing peak force by bond area, and pressing temperature should be recorded for each bond formed and tested.

8.3 *Selecting Temperatures, Pressing Times and Numbers of Tests*—The method may be employed to explore transient strengths for combinations of bonding temperature and time for a given type of thermosetting adhesive. A principal focus of the method is to assemble data sufficient to construct scatter plots of bond strength versus pressing time for a range of selected temperatures. Each such scatter plot may exhibit the sequential accumulation of strength and, where such trends are substantially linear, regression may enable a rate of strength development value to be derived. Selection of bond pressing temperatures and times depends on the objectives of the study being undertaken and the reactivity of the adhesive being evaluated. A set of standard conditions will here be identified while acknowledging that a diversity of conditions may often be employed and should be reported in accordance with Alternative Method V in 9.5.

8.3.1 *Temperature Selection*—Five temperatures are identified as the standard method. Preliminary trials may be necessary to ascertain the range of temperatures appropriate for a given adhesive. The highest pressing temperature for a given adhesive is limited by transient strength accumulation detected for pressing times above 15 s, since useful data may only be obtained for bonds tested once stable temperature is attained within the allowable limit of ±2°C (3.8°F). The maximum allowable temperature is that