Designation: D1871 – 04 (Reapproved 2014)

Standard Test Method for Adhesion Between Tire Bead Wire and Rubber[1]

This standard is issued under the fixed designation D1871; 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 cover procedures for testing the strength of adhesion of single-filament wire to vulcanized rubber compounds. The method applies to, but is not limited to, wire made from brass, bronze, or zinc coated steel wire. The adhesion strength is expressed as the magnitude of the pull-out force for the single filament of wire.

1.2 This test method is applicable to single-filament wires used in reinforced rubber products as single filaments and is normally used to evaluate the adhesion of samples of wire to a standard rubber applied under specified conditions. It is primarily used to evaluate tire bead wire and may be applied, with modifications and by agreement between supplier and customer, to various wire types used in rubber product reinforcing.

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

1.4 *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. See 6.5.1.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D76 Specification for Tensile Testing Machines for Textiles

D123 Terminology Relating to Textiles

D1566 Terminology Relating to Rubber

D2906 Practice for Statements on Precision and Bias for Textiles (Withdrawn 2008)[3]

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D4392 Terminology for Statistically Related Terms (Withdrawn 1993)[3]

D6477 Terminology Relating to Tire Cord, Bead Wire, Hose Reinforcing Wire, and Fabrics

E456 Terminology Relating to Quality and Statistics

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms relating to tire cord, bead wire, hose wire, and tire cord fabrics, refer to Terminology D6477

3.1.1.1 The following terms are relevant to this standard: adhesion, curing, holland cloth, hose reinforcing wire, mill grain, rubber, rubber compound, as used in the manufacture of rubber articles, tire bead, tire bead wire, and vulcanization.

3.1.2 For definitions of terms relating to rubber, refer to Terminology D1566

3.1.3 For definitions of terms relating to testing and statistical concepts, refer to Terminology D4392 or E456D4392E456.

3.1.4 For definitions of other terms related to textiles, refer to Terminology D123.

4. Summary of Test Methods

4.1 The wires are vulcanized into a block or pad of rubber and the force necessary to pull the wires out of the rubber is measured. The direction of pull-out is axial, that is, along the wire.

5. Significance and Use

5.1 To contribute to the mechanical properties required in a product, tire bead wire must have good adhesion to the rubber matrix. This allows the rubber to absorb part of the energy, distributing it uniformly between the reinforcing material and the rubber compound. This test method is considered satisfactory for acceptance testing of commercial shipments of wire since it has been used extensively in the trade for this purpose. This test method may be used for purchase specification requirements or manufacturing control of bead wire.

5.1.1 If there are differences of practical significance between reported test results for two laboratories (or more), comparative tests should be performed to determine if there is

[1] Thistest method is under the jurisdiction of ASTM Committee D13 on Textiles and are the direct responsibility of Subcommittee D13.19 on Industrial Fibers and Metallic Reinforcements.

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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] The last approved version of this historical standard is referenced on www.astm.org.

a statistical bias between them, using competent statistical assistance. As a minimum, test samples should be used that are as homogeneous as possible, that are drawn from the material from which the disparate test results were obtained, and that are randomly assigned in equal numbers to each laboratory for testing. Other materials with established test values may be used for this purpose. The test results from the two laboratories should be compared using a statistical test for unpaired data, at a probability level chosen prior to the testing series. If a bias is found, either its cause must be found and corrected, or future test results for that material must be adjusted in consideration of the known bias.

5.2 The characteristics of single filament steel wires that affect the adhesion property are wire diameter, coating composition, and coating mass. The storage conditions, age, and vulcanization conditions of the rubber compound will affect the test results and must be specified by the supplier of the rubber compound.

6. Apparatus and Materials

6.1 *Mold,*[4] designed as shown in Fig. 1 for a 12.5-mm thick block of rubber, 200 mm long, and 50 mm wide, with 15 beveled slots across the width of the mold spaced 12.5 mm apart at the middle of the mold thickness, and with top and bottom plates for the mold. If more than five wires break when testing with the standard mold, the purchaser and the supplier may agree to use a mold cavity that is less than 50 mm wide.

6.2 *Tensile Testing Machine,* CRE (Constant-Rate-of-Extension) type, of such capacity of the load cell in use that the maximum force required to pull out the wires shall not exceed 85 % nor be less than 15 % of the rated capacity. The rate of travel of the power actuated grip shall be 50 ± 5 mm/min, or up to 150± 15 mm/min by agreement between the purchaser and the seller. The specifications and methods of calibration and verification shall conform to Specification D76.

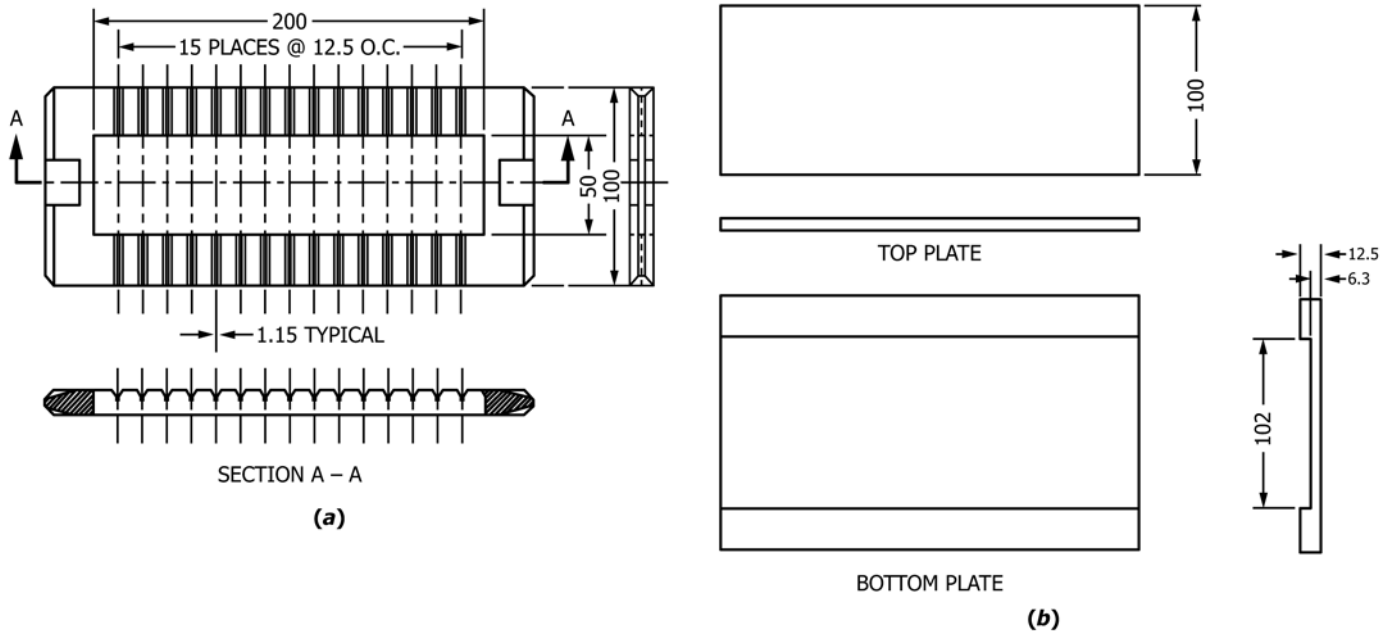
6.3 *Top Grip,*[4] designed as shown in Fig. 2 shall be a special holder made for the vulcanized block sample. The bottom grip may be any type clamp of sufficient capacity to handle the specimen and designed to prevent its slippage in the grip[5] or to prevent premature filament breakage.

6.4 *Vulcanizing Press,* large enough to accommodate the mold, and capable of exerting at least 70 kN total force on the mold.[6] Electrical or steam heat for the top and bottom platens shall be provided, of sufficient capacity for maintaining the mold components at a temperature within 3°C of the requirements for the rubber compound being used.

[4] Suitable molds and block holder are available from Bartell Machinery Systems Corp, Rome, NY 13440.

[5] Series 2710 screw action grips, Series 2716 wedge action grips from Instron Corp., 2500 Washington St., Canton, MA 02021, and Scott A420 clamps from GCA/Precision Scientific, 3737 W. Cortland St., Chicago, IL 60647, have been found practical for testing single filament wire.

[6] Suitable vulcanizing presses are manufactured by Given P-H-I, Pasadena Presses, 1100 John Reed Court, City of Industry, CA 91745.



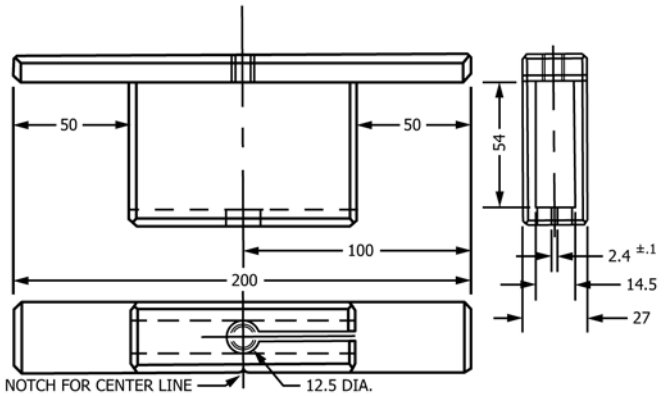
TOLERANCES
All dimensions ±0.2
Angular±½ °
Except where noted

NOTE 1—Material—Steel.

NOTE 2—Break all sharp corners.

NOTE 3—All dimensions in millimetres except where noted.

FIG. 1 Mold with Top and Bottom Plates



TOLERANCES
All dimensions ±0.2
Except where noted

NOTE 1—Material—Steel.

NOTE 2—Break all sharp corners.

NOTE 3—All dimensions in millimetres except where noted.

FIG. 2 Top Grip

6.5 *Solvent,* used for the preparation of the rubber and wire in this test method shall be such that the surface of the rubber will be freshened and the wire surface cleaned without adversely affecting the adhesion. If remilled or freshly milled compound is used, the use of a solvent can be left to mutual agreement between the user and the supplier of the compound.

6.5.1 A suitable solvent has been found to be lead-free gasoline (normal heptane) with a distillation range from 40 to 141°C and a maximum recovery of 97 %, available from most solvent suppliers. **Precaution**—Adequate health and safety precautions should be observed in the handling and use of any solvent selected for use in this test method.

6.6 *Rubber Compound,* shall be furnished by the purchaser of the wire, together with pertinent information on the temperature and time for the cure of that particular rubber as well as aging time limits for holding the block between vulcanizing and testing, but not less than 16 h. Since the adhesion between rubber and wire is influenced by the age and storage conditions of the uncured rubber compounds, the purchaser of the wire shall also specify the conditions of storage and any time limit for such storage of the batch. The rubber compound may be provided in sheet form, 7 mm thick on a non-hygroscopic backing, such as a plasticizer free plastic material, or may be provided unmilled in a form requiring milling immediately prior to use.

6.7 *Mold Release Lubricant,* A suitable mold release lubricant may be applied to the empty mold to facilitate test block removal. Excess lubricant shallbe wiped from the mold and particularly from the slots provided for the wires. The lubricant shall not be applied when exposed wires are in the area, and should only be used when absolutely necessary. Use of mold release lubricant should be recorded on the test report.

7. Sampling

7.1 *Lot Sample*—As a lot sample for acceptance testing, take at random the number of reels, coils, spools, or other shipping units of wire directed in an applicable material specification or other agreement between purchaser and the supplier. Consider reels, coils, spools, or other shipping units of wire to be the primary sampling units.

NOTE 1—A realistic specification or other agreement between the purchaser and the supplier requires taking into account the variability between and within primary sampling units so as to provide a sampling plan which at the specified level of the property of interest has a meaningful producer's risk, acceptable quality level, and desired limiting quality level.

7.2 *Laboratory Sample*—Use the primary sampling units in the lot sample as a laboratory sample. These samples of reels, coils, spools, or other shipping units of wire are customarily taken by the manufacturer and provided to the purchaser with the lot shipment.

7.3 *Test Specimens*—Prepare three specimens from each laboratory sample by cutting 250 to 300-mm lengths of the wire and laying them out on a clean surface such as cloth or paper. The wires should be touched only at their ends, in no case on that portion that is to be embedded in the test pad. Unless otherwise specified, the wires shall be tested "as is," representing the condition in which the wire lot and samples were received. If "washing" the surface of the wire before test is specified, gently wipe the wire with a soft cloth dampened with the solvent.

8. Procedure

8.1 *Rubber Compound*—Cut the rubber sheet to the size of the mold cavity, unless it has been precut to that size for storage; two pieces are required for each block. Lay these out and freshen their top surfaces with the solvent, applied with a soft cloth or brush. Plan the freshening for a drying time to be at least 10 but not over 20 min before the building step of the procedure. If the compound has been milled immediately prior to use, freshening is not required.

8.2 *Block Building*—Preheat the mold, including top and bottom plates, to the cure temperature of the rubber to be used.

8.2.1 Remove the mold from the preheating and take off the top plate.

8.2.2 With its freshened side up, press the bottom piece of rubber into the mold with a metal or wooden peg.

8.2.3 Lay the wires, one by one, into the tapered slots, making note of their identification, for example, the sample reel number, for later matching of the test results. Position the wires with about 25 mm sticking out from one side of the mold and about 150 mm from the other. Do not let the fingers touch the wires within the 50 mm length that will be in contact with rubber.

NOTE 2—Slots 1 and 15 should be filled with dummy lengths of wire which will be subsequently pulled, but their test values should not be recorded nor included in the calculations.

8.2.4 Place the top piece of rubber in the mold with its freshened side down and press it firmly in place with a metal or wooden peg.

8.2.5 Replace the top plate, put the mold in the press, and apply a force of at least 70 kN to the mold. This force when applied to the mold in Fig. 1 is equal to a pressure of 3.5 mPa. Excessive force is not necessary and may damage the mold.

Vulcanize the block under pressure for the time and temperature conditions of the rubber compound being used.

8.2.6 It is important that the time from removal of the mold from the heat and replacing it in the press not exceed 3 min. This time limit also applies during the building of blocks subsequent to the first one, where the mold is already hot and when a cured block must be removed from the mold before it can be filled.

8.2.7 It is an acceptable practice to prebuild the test pad in a cold mold following the above block building procedures (see 8.2.2 – 8.2.4), and apply sufficient pressure to hold the assembled pad together. This pressure may be applied by stitching layers together or applying pressure in a cold press. The prebuilt pad is then placed in a preheated mold for vulcanization. Maximum storage time of a prebuilt pad before vulcanization is 90 min.

8.3 *Preparation of Vulcanized Block*—Remove the mold from the press and push out the vulcanized block. If there are other blocks to prepare, proceed with the building operations as outlined in 8.2. Let the cured blocks condition at room temperature, 23 ± 2°C, for the required time limits specified by the rubber supplier, but not less than 16 h. Cut off the 25-mm lengths of protruding wire close to the edge of the block. Extraneous flash adjacent to the sample wires should be cut off using a razor-blade or surgical knife, taking care not to damage the wire. Trim off the extraneous flash from both edges of the block.

8.4 *Testing*—Push the block into the top grip of the testing machine until the first wire is in the center of the 12.5-mm hole. Note that for ease of wire centering the grips may be reversed from the description contained in this section. Make sure the load-reading attachment of the tester is zero. Then clamp the first wire in the bottom grip and start the machine. When the wire pulls out, stop the machine, note the pull-out force to the nearest 5 N and release the wedges. Pull the tested wire out of the block by hand and slide the block in the holder until the next wire is centered. Clamp it in the wedges, start the machine, pull the wire out, and note its pull-out force. Repeat the procedure with the other wires of the block. Continue sliding each subsequent wire into the wedges until they move too far to grasp the wire length. Then return the moving grip to its starting position and start it again. The moving grip may be returned to its starting position after each pull if this is found to be more convenient for adequate sample positioning.

9. Calculation

9.1 Calculate the average pull-out force for the wires representing one laboratory sample, and for the samples representing one lot.

9.2 Calculate the lot sample standard deviation and coefficient of variation, if requested by the purchaser.

10. Report

10.1 State that the tests were performed as directed in Test Methods D1871. Describe the material or product sampled and tested.

10.2 Report the following information:

10.2.1 Identification of wire samples, individual test results, the sample averages, and standard deviation and coefficient of variation, if calculated,

10.2.2 Identification of rubber compound, and its vulcanizing conditions,

10.2.3 Type of tensile test machine used, and rate of extension, and

10.2.4 Any deviation from the standard test procedure, including using a mold that is less than 50 mm (2 in.) wide.

11. Precision and Bias[7]

11.1 *Interlaboratory Test Data*—An interlaboratory evaluation was conducted in 1992 in which randomly drawn samples of two wire diameters of tire bead wire and one rubber compound were tested in 16 laboratories in accordance with Test Methods D1871. Each laboratory used two operators, each of whom tested the two materials on two separate days. In addition, separate testing was performed at each laboratory to identify assignable variation to specific parts of the test method. Initial analysis of the data indicated that the results from one of the laboratories was a statistical outlier and upon investigation the cause for this condition was determined to be failure to follow the test method properly. The data for the one laboratory was deleted prior to further analysis. In addition, some laboratories reported wire breaks in the test grip before pull-out from the test pad. For this reason, the 0.965 mm sample was considered a no test and critical difference values are reported only for the 1.295 mm diameter wire.

11.2 *Precision*— Two test results should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences listed in Table 1.

NOTE 3—The tabulated values of the critical differences and confidence limits should be considered to be a general statement, particularly with respect to between-laboratory precision. Before a meaningful statement can be made about two specific laboratories, the amount of statistical bias, if any, between them must be established, with each comparison being based on recent data obtained so as to be as nearly homogeneous as

[7] Supporting data are available from ASTM Headquarters. Request RR:D13-1085.

TABLE 1 Critical Differences for Two Averages for the Conditions Noted, 95 % Probability Level, Pull-Out Force (Newtons)

Wire Diameter	Number of Test Results in Each Average	Single-Operator Precision	Within-Laboratory Precision	Between-Laboratory Precision
1.295 mm	2	289	308	439
	3	174	204	374
	4	145	179	360
	5	129	167	354

possible and then randomly assigned in equal numbers to each of the laboratories.

11.3 *Bias*—The procedure in this test methods has no known bias because the value of adhesion can be defined only in terms of a test method.