



Standard Test Methods for Rubber Property—Adhesion to Rigid Substrates¹

This standard is issued under the fixed designation D429; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover procedures for testing the static adhesional strength of rubber to rigid materials (in most cases metals).

Method A—Rubber Part Assembled Between Two Parallel Metal Plates.

Method B—90° Stripping Test—Rubber Part Assembled to One Metal Plate.

Method C—Measuring Adhesion of Rubber to Metal with a Conical Specimen.

Method D—Adhesion Test—Post-Vulcanization (PV) Bonding of Rubber to Metal.

Method E—90° Stripping Test—Rubber Tank Lining—Assembled to One Metal Plate.

Method F—Rubber Part Assembled Between Two Parallel Convex-Shaped Metal Plates

Method G—Measuring Bond Durability for Rubber-to-Metal Bonded Components with a Double Shear Cylindrical Specimen

Method H—Measuring Bond Durability for Rubber-to-Metal Bonded Components with a Quadruple Shear Specimen

1.2 While the test method may be used with a wide variety of rigid materials, use of materials other than metals is the exception. For this reason, we have used the word “metal” in the text rather than “rigid materials.”

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

¹ These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.25 on Rubber Adhesive Systems.

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2. Referenced Documents

2.1 ASTM Standards:²

B117 Practice for Operating Salt Spray (Fog) Apparatus
D395 Test Methods for Rubber Property—Compression Set
D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D471 Test Method for Rubber Property—Effect of Liquids
D572 Test Method for Rubber—Deterioration by Heat and Oxygen

D573 Test Method for Rubber—Deterioration in an Air Oven

D1149 Test Methods for Rubber Deterioration—Cracking in an Ozone Controlled Environment

E4 Practices for Force Verification of Testing Machines
G153 Practice for Operating Enclosed Carbon Arc Light Apparatus for Exposure of Nonmetallic Materials

2.2 *ASTM Adjuncts:*
Holding Fixture Drawings³

3. Significance and Use

3.1 These test methods are designed primarily for specimens prepared in a laboratory under standardized conditions such as may be used to provide data for development and control of rubber compounds and methods of manufacture. With slight modifications as indicated, Methods A, B, C, D, and E are also used for obtaining comparative adhesion test values of production parts whenever the design permits preparation of suitable test specimens. Methods A, B, C, and D are applicable in the case of many products in which rubber is used for controlling vibration.

METHOD A—RUBBER PART ASSEMBLED BETWEEN TWO PARALLEL METAL PLATES

4. Adhesion Failure Terminology

4.1 *R* indicates the failure is in the rubber.

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 Complete detail drawings of the fixture are available from ASTM International Headquarters. Order Adjunct No. **ADJD0429**. Original adjunct produced in 1959.

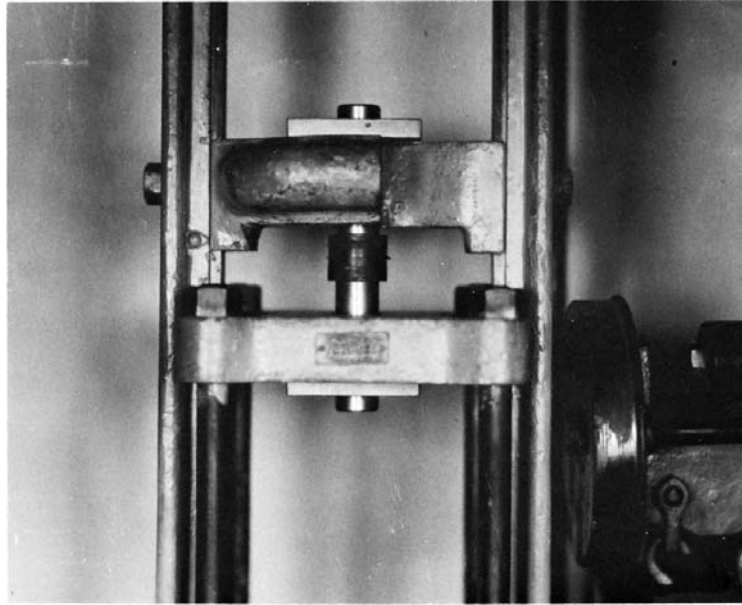


FIG. 1 An Example of an Adhesion Test Specimen Attached in a Testing Machine

4.2 *RC* indicates the failure is at the rubber-cover cement interface.

4.3 *CP* indicates the failure is at the cover cement-prime cement interface.

4.4 *M* (can also be denoted as *CM*) indicates the failure is at the metal-prime cement interface.

NOTE 1—Example—The percentages of the various types of failure may be estimated as in the following examples:

R-50, RC-50 means that roughly one half or 50 % of the area showed failure in the rubber and the other 50 % showed failure at the rubber cover cement interface.

R-25, RC-25, M-50 means three types of failure were present with the *M* indicating 50 % failure at the metal-primer interface. Determining the locus of failure can normally be determined visually, but may require analytical test methods.

5. Significance and Use

5.1 Method A is used to determine adhesion values in instances where users feel this design is more reflective of an actual product and where control tests have been set up as a result. The sample also lends itself to testing the effectiveness of different processing techniques and various adhesive systems.

6. Apparatus

6.1 *Testing Machine*—A tension testing machine conforming to the requirements of Practices **E4** shall be used for measuring the strength of adhesion. The moving head of the machine shall travel at the uniform rate of 0.40 ± 0.04 mm/s (1.0 ± 0.1 in./min). The machine shall be provided with a recording device to give the total force in Newtons or pounds-force at the conclusion of the test. A machine with a capacity no greater than 44.5 kN (10 000 lbf) will be found suitable in most cases.

6.2 *Grips*—The fixtures for holding the specimen in the testing machine will depend on the type of specimen, but in all cases shall be provided with ball seats, or another device, to permit centering of the load during the test. A typical adhesion test assembly is shown in **Fig. 1**. Specially molded laboratory specimens shall be attached by means of threaded studs on the metal parts as shown in **Fig. 2**. Specimens cut from production parts may be clamped as illustrated in **Fig. 3**.

7. Standard Test Specimens

7.1 After assembly, the standard test specimen shall consist of a cylinder of cured rubber 3.2 ± 0.1 mm (0.125 ± 0.005 in.) thick and with a diameter of 39.9 ± 0.1 mm (1.597 ± 0.005 in.), which results in a surface area of 1250 ± 5 mm² (2.00 ± 0.01 in.²). This will be attached to the faces of two metal plates, each at least 9.5 mm (0.37 in.) in thickness and of the same diameter as the rubber cylinder. The metal plates shall have flat faces that shall be parallel in the finished specimen. The cylindrical shape is used to eliminate sharp corners and to give uniform distribution of the pulling force.

NOTE 2—While a 1250 mm² (2 in.²) area test specimen shall be considered the standard for reference purposes, it shall be permissible to use a specimen of only 625 mm² (1 in.²) in area, when it is desirable to prepare and cure a large number of test specimens at one time. The diameter of this test specimen shall be 28.21 ± 0.02 mm (1.129 ± 0.001 in.) and the unvulcanized piece shall be cut to dimensions of approximately 24 mm ($1\frac{5}{16}$ in.) in diameter and approximately 5 mm ($\frac{3}{16}$ in.) in thickness (approximately 10 % volume excess).

8. Laboratory Preparation of Standard Test Specimens

8.1 Prepare the standard test specimen to be used for development and control purposes in the laboratory as described in **8.2** through **8.6**. Methods other than those specified in **8.2** to **8.6** may be applied and must be described in the test report.

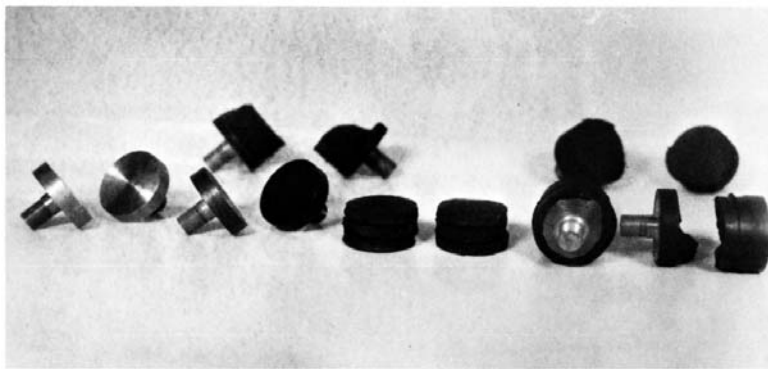


FIG. 2 Views of a Typical Test Specimen Showing Individual Metal Parts and Rubber Cylinders

8.2 Machine circular metal parts of standard dimensions from rolled bar steel SAE No. 1020⁴ for standard reference pieces. However, other metals may be used if in conformity with the essential dimensions. Prepare smoothly machined test faces of the metals in accordance with any test method for securing adhesion that may be under investigation.

8.3 Cut unvulcanized rubber pieces to dimensions of approximately 35 mm (1 3/8 in.) in diameter and approximately 5 mm (3/16 in.) in thickness so as to give maximum pressure of the rubber against the metal surface during vulcanization (see Fig. 2). Clean or treat surfaces to be adhered in accordance with the method being investigated.

8.4 Assemble the metal parts and rubber pieces for vulcanization in the mold. A typical mold and test piece are shown in Fig. 4. The assembly is illustrated in Fig. 2. After vulcanization, approximately 0.039 mm (0.0015 in.) of rubber should be over the edge of the metal substrate. Prior to vulcanization, take great care to keep the surfaces to be adhered clean and free of dust, moisture, or other foreign material. Do not touch surfaces.

8.5 Vulcanize by heating the mold for a definite time at a controlled temperature in a hydraulic vulcanization press. Choose the time and temperature of cure according to the rubber compound used. Take care in removing the specimens from the mold to avoid subjecting the adhered surfaces to stress before the specimens have cooled.

8.6 After vulcanization, store the specimens at a temperature of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) at least 16 h prior to testing.

9. Test Specimens from Production Parts

9.1 For comparative test of adhesion in production parts, select a portion of the production-made part so that a specimen consisting of a rubber piece sandwiched between two parallel plates of metal may be cut. Hollow-mill a specimen out of the production part and finish the piece either by turning or grinding down to size in such a manner that the surfaces of the rubber and metal shall be free of imperfections, which have a tendency to start a tear in the rubber part during the application of tension while testing.

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1040.

9.2 In routine production testing, it is sometimes necessary to test specimens of different size and shape than those specified as standard. In such cases, the test method of preparation shall be similar to that given in 9.1, but the test results secured shall not be compared with those obtained using the standard test specimen.

10. Procedure

10.1 Mount the vulcanized standard test specimen or the specimen cut from a production part in the testing machine, as shown in Fig. 1, so that the tension shall be uniformly distributed. Test at a rate of 0.40 ± 0.04 mm/s (1.0 ± 0.1 in./min), or at 0.083 ± 0.08 mm/s (2.0 ± 0.2 in./min), until the rubber either separates from the metal surface or ruptures. Record the total force at the time of failure. Typically 3 specimens are tested per condition, but other sample sizes can be used, if reported.

10.2 In case of rupture in the rubber, the strength of the adhesion bond is obviously not measured, but is shown to be greater than the strength of the rubber itself. Record the type or types of failure, and express in accordance with the adhesion failure terminology described in Section 4. Estimate and record the percentages of the various types of failures.

11. Calculation

11.1 Express the adhesion value in pascals or pounds-force per square inch. Calculate by dividing the tension force causing failure by the original area of the adhered surface. In case of rubber failure, the adhesion value is recognized as being higher than that reported.

12. Report

12.1 The report shall include the following:

12.1.1 The result, calculated in accordance with Section 11,

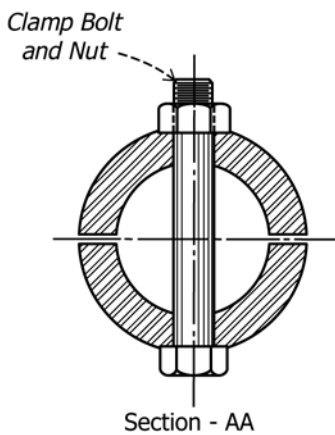
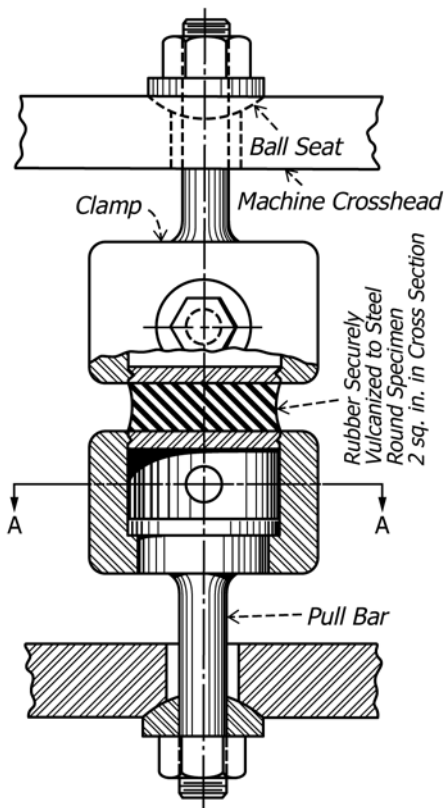
12.1.2 All observed and recorded data including the number of test pieces involved,

12.1.3 Notation of type or types of failure, and the estimated percentages of the various types of failures (Section 10),

12.1.4 A description of the specimen and a notation indicating whether the specimen was molded in the laboratory or prepared from a production part,

12.1.5 Dimensions of the test specimen (comparisons may be made only among specimens of the same size and shape),

METHOD B—90° STRIPPING TEST—RUBBER PART ASSEMBLED TO ONE METAL PLATE



2 in.² = 12.9 cm²

FIG. 3 Method of Testing Adhesion When Sample Section is Taken from Production Parts

14. Significance and Use

14.1 This test is intended to determine the adhesive strength of rubber-to-metal bonding agents. The results are obtained by measuring the force necessary to separate a rubber from a metal surface. The data obtained indicates the strength of adhesion along a line across the width of the rubber strip being separated from a metal plate at a 90° angle. The test provides valuable data for development and control of rubber compounds and test methods of bonding. It also serves as a screening test for the evaluation of various bonding agents, techniques, or both.

NOTE 3—It is permissible to use a 45° angle to bring about separation, but if this is done, the same should be indicated on the form on which the data are recorded. If this notation does not appear, it must be assumed that the 90° angle has been used. The angle of pull, in this case 45°, merely means that the equipment has been so designed that the angle made by that face of the rubber being stripped from the metal and the metal to which it has been adhered is 45°. Experience indicates a lower force is obtained for the 45° angle than for the 90° angle, and also a break significantly closer to the bond interface.

15. Adhesion Failure Terminology

15.1 *R* indicates the failure is in the rubber.

15.2 *RC* indicates the failure is at the rubber-cover cement interface.

15.3 *CP* indicates the failure is at the cover cement-prime cement interface.

15.4 *M* (can also be denoted as *CM*) indicates the failure is at the metal-prime cement interface.

NOTE 4—Example—The percentages of the various types of failure may be estimated as in the following examples:

R-50, *RC*-50 means that roughly one half or 50 % of the area showed failure in the rubber and the other 50 % showed failure at the rubber-cover cement interface.

R-25, *RC*-25, *M*-50 means that three types of failure are present with the *M* indicating 50 % failure at the metal-primer interface.

16. Apparatus

16.1 *Testing Machine*—A power driven test machine equipped to produce a uniform rate of grip separation shall be used for measuring the strength of adhesion. The head of the machine shall travel at the uniform rate of 0.83 ± 0.08 mm/s (2 ± 0.2 in./min).

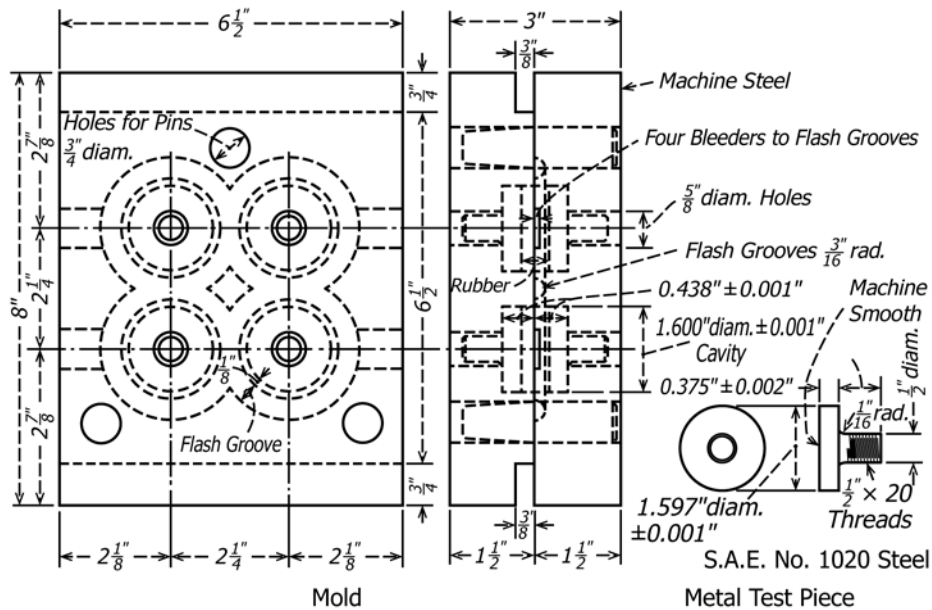
16.2 *Fixture*—Any suitable fixture for holding the test specimen to the upper head of the machine may be used provided the direction of pull to cause separation is as perpendicular as possible at all times during the test. This means making a 90° angle with the top holding fixture. The test fixture shown in operation in Fig. 5 complies with the above and provides the ease and simplicity of testing.³

16.3 *Grips*—Any suitable grip may be used, provided it does not slip or rupture the rubber strip. The grip shown in Fig. 5 complies and provides ease and simplicity of operation.

- 12.1.6 Test method used for determining adhesion specified in this standard, including the test rate,
- 12.1.7 Surface finish of substrate, if known,
- 12.1.8 Date of manufacture or vulcanization, if known,
- 12.1.9 Time and temperature of vulcanization, if known,
- 12.1.10 Temperature and humidity of test room,
- 12.1.11 Date of test, and
- 12.1.12 Any deviations from this test method.

13. Precision and Bias⁴

13.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.



| in. | mm |
|---------------|--------------|
| 1/16 | 2 |
| 1/8 | 3 |
| 3/16 | 5 |
| 3/8 | 10 |
| 1/2 | 13 |
| 5/8 | 16 |
| 3/4 | 19 |
| 1 1/2 | 38 |
| 2 1/8 | 54 |
| 2 1/4 | 57 |
| 2 7/8 | 73 |
| 3 | 76 |
| 6 1/2 | 165 |
| 8 | 203 |
| 0.375 ± 0.002 | 9.53 ± 0.05 |
| 0.125 ± 0.005 | 3.2 ± 0.1 |
| 1.597 ± 0.001 | 15.16 ± 0.03 |
| 1.600 ± 0.001 | 40.64 ± 0.03 |

FIG. 4 Detailed Drawing of a Mold for Adhesion Test Pieces

17. Standard Test Specimen

17.1 The standard test specimen shall consist of a strip of rubber 6.3 ± 0.1 mm (0.250 ± 0.005 in.) in thickness, 25 ± 0.5 mm (1.000 ± 0.02 in.) in width, and 125 mm (5 in.) in length adhered to the face of a metal strip 1.6 ± 0.1 mm (0.062 ± 0.004 in.) in thickness, 25 ± 0.5 mm (1.000 ± 0.02 in.) in width, and 60 ± 1 mm (2.36 ± 0.04 in.) in length. The test specimen shall be so prepared that the bonded area of 25 by 25 mm (1 by 1 in.) is fixed approximately in the middle of the metal member as shown in Fig. 6.

NOTE 5—In cases where the 1.6 -mm (0.062 -in.) thick metal is permanently deformed during testing, a thicker metal may be used when agreed upon between the purchaser and the supplier.

18. Preparation of Standard Test Specimen

18.1 Prepare the standard test specimen as described in 18.1.1 to 18.1.6:

18.1.1 Rectangular metal parts of the standard dimensions described in Section 17 shall have the area to be bonded prepared in accordance with the test method for determining

adhesion that may be under investigation. Mask both ends with pressure-sensitive tape so that only the area described in Section 17 will be available for adhesion.

18.1.2 Cut unvulcanized rubber slabs approximately 8 mm (0.3 in.) in thickness to dimensions of the required size for the mold, so as to provide maximum pressure of the rubber against the metal surfaces during vulcanization. (See Fig. 6 for suitable mold design.) The inside mold dimensions parallel to the longitudinal axis of the metal part shall be 125 mm for nominal 5 in. The dimension along the transverse axis of the metal part can be altered according to the number of pieces required for any one cure. This will leave sufficient length of rubber strip for fastening in the grip shown in Fig. 5. Solvent-wash rubber surface to be adhered or treat in accordance with the method being investigated. Freshly milled rubber need not be solvent wiped.

18.1.3 Assemble the metal parts and rubber pieces for vulcanization as shown in Fig. 6, properly spacing the metal parts along one edge of the rubber slab and then placing the assembly, metal parts down, in the mold cavity. Cure the whole

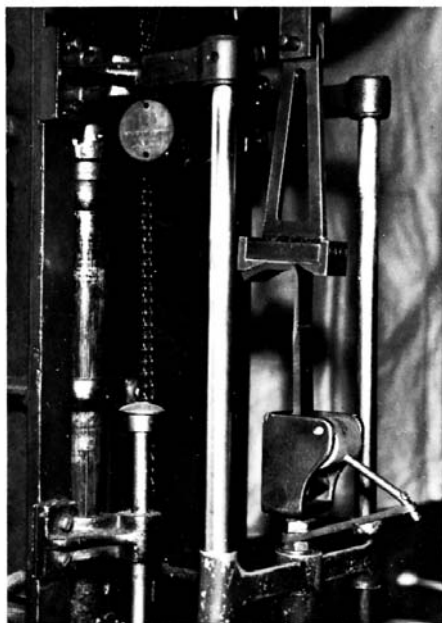


FIG. 5 Adhesion Tensile Testing Fixture in Operation

assembly in accordance with the method being investigated. Prior to assembly and vulcanization, take great care to keep the surfaces to be adhered clean and free of dust, moisture, or other foreign material.

18.1.4 Vulcanize by heating the mold under pressure for a definite time at a controlled temperature in a hydraulic vulcanizing press. The time and temperature of cure shall be in accordance with the system being investigated. At the conclusion of the cure, take care in removing the specimens from the mold to avoid subjecting the adhered surfaces to stress before the specimens have cooled.

18.1.5 After vulcanization, store the specimens at a temperature of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) for at least 16 h.

18.1.6 Separate the specimens from each other in preparation for testing. This is done by cutting with a scissors, handknife, or other suitable equipment. The edges of the specimen may then be buffed on a belt sander to bring the edge of the rubber flush with the edge of the metal strips (Note 6). Take care not to overheat the metal parts or the rubber and not to reduce the width of the sample being tested beyond the tolerances described.

NOTE 6—Present experience indicates negligible differences in results between samples buffed clean and samples tested without buffing, because edges of metal parts are not specifically prepared for adhesion and the edge contact area, given a metal part thickness of 1.6 mm (0.062 in.) is not significant.

19. Procedure

19.1 Place the test specimen (Note 7) symmetrically in the jaws attached to the fixed grip with the separating edge toward the operator, as shown in Fig. 5. Place the tab in the grip and apply a steady load at the rate of 50 mm (2.0 in.)/min until separation is complete. Use the autographic recorder in order to show graphically the adhesion values over the full length of the test specimen.

NOTE 7—This specimen lends itself to the determination of the relative effect of environmental conditions on possible bond deterioration. Specimens may be exposed unstressed or under a constant, relatively low bond stress. It is recommended that one of the standard ASTM environmental exposure conditions be used. Among those applicable are the following: Test Methods D471, D572, D573, D1149, Practice G153, and Practice B117. If environments other than the above are used, they shall be described completely. This should include the conditions of the exposure such as time, temperature, etc.

20. Calculations

20.1 Express the adhesion value in newtons per millimetre or pounds-force per inch of width.

21. Report

21.1 The report shall include the following, preferably reported on an Adhesion Test Data Sheet illustrated in Table 1:

21.1.1 The results calculated in accordance with Section 20.

21.1.2 All observed and recorded data,

21.1.3 Notation of the type or types of failures and the estimated percentages of the various types of failures (Section 15),

21.1.4 Description of the specimen including statement of method of determining adhesion, if known,

21.1.5 Date of vulcanization, if known,

21.1.6 Time and temperature of vulcanization, if known,

21.1.7 Temperature of test room, and

21.1.8 Date of test.

22. Precision and Bias⁴

22.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.

METHOD C—MEASURING ADHESION OF RUBBER-TO-METAL WITH A CONICAL SPECIMEN

23. Significance and Use

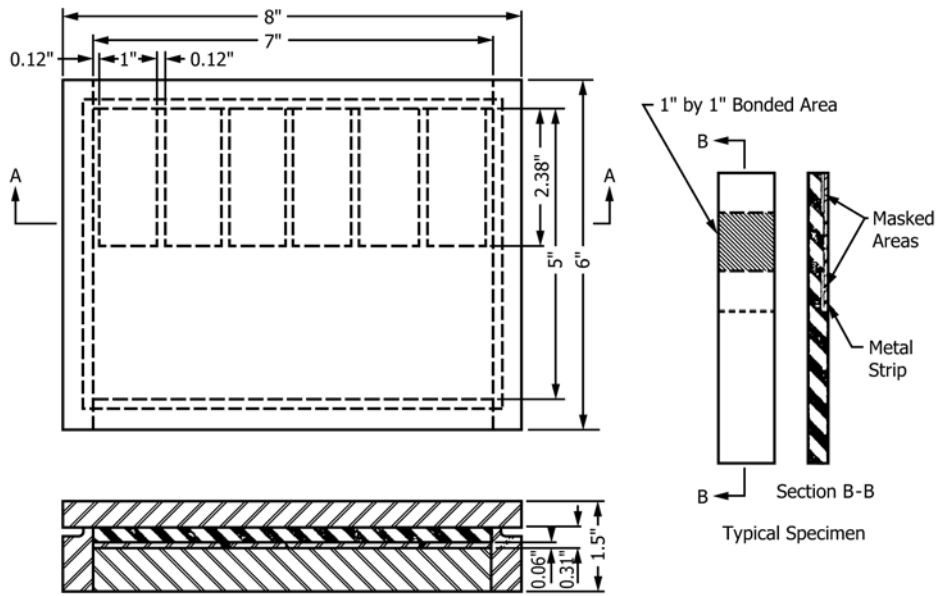
23.1 The test method is designed primarily to test pieces prepared in the laboratory under standard conditions to provide data for development and control of bonding systems and their components, such as cements or special rubber compounds, and of test methods of manufacture. While intended to be applied where rubber is bonded to supporting rigid pieces, it may not cover such cases where the support, a high modulus material, has low rigidity due to small transverse dimensions, as in the case of rubber bonded to metal wires, cords, or thin sheets. The particular geometry of the test piece produces, in most cases, an interfacial failure between the rubber and conical part, because of a stress concentration at the tip of the cone.

24. Adhesion Failure Terminology

24.1 Express the type of adhesion failure as determined by examination of broken test pieces by one or more of the following symbols:

24.2 *R* indicates failure in the rubber.

24.3 *RC* indicates failure in the rubber-cover cement.



| | | Section A-A | | | | | | | | | |
|-----|----|-------------|------|------|----|-----|------|-----|-----|-----|-----|
| | | 0.06 | 0.12 | 0.31 | 1 | 1.5 | 2.38 | 5 | 6 | 7 | 8 |
| in. | mm | 1.5 | 3 | 7.9 | 25 | 38 | 60.5 | 127 | 152 | 178 | 203 |

FIG. 6 Adhesion Test Specimen Mold

TABLE 1 Example of Form for Reporting Adhesion test Results

| Stock | Cure | Metal and Metal Finishing Used | Pull | | Type of Break, % | | | |
|------------|------|--------------------------------|-----------------|------------------|------------------|----|----|---|
| | | | Sample, lbf/in. | Average, lbf/in. | R | RC | CP | M |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| Date _____ | | | | | By _____ | | | |

24.4 CP indicates failure in the cover cement-prime cement.

24.5 M (can also be denoted as CM) indicates failure in the prime cement-metal interface.

NOTE 8—Example—The percentages of the various types of failure may be estimated as in the following examples:

R-50, RC-50 means that roughly one half or 50 % of the area showed failure in the rubber and the other 50 % showed failure at the rubber-cover cement interface.

R-25, RC-25, M-50 means three types of failure present with the M indicating 50 % failure at the metal primer interface.

25. Apparatus

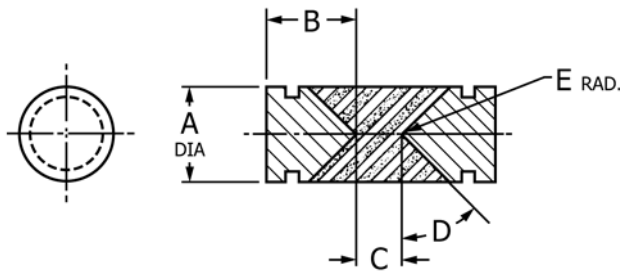
25.1 Testing Machine—A tension testing machine conforming to the requirements of Practices E4 shall be used for measuring the strength of adhesion. The moving head of the machine shall travel at the uniform rate of 0.83 ± 0.08 mm/s (2.0 ± 0.2 in./min). The machine shall be provided with a recording device to give the total force in newtons or pounds-force at the conclusion of the test. A machine with a capacity no greater than 44.5 kN (10 000 lbf) will be found suitable in most cases.

25.2 Grips—The fixtures for holding the specimen in the test machine will depend on the type of specimen but in all cases shall be provided with ball seats to permit proper centering of the applied load during the test. These grips can be of two types: (1) a simple slip-in type which engages in the grooved section of the conical specimen, or (2) a threaded stud with a locknut. This could be a combination fixture.

26. Standard Test Specimen

26.1 A standard test specimen shall consist of a rubber compound bonded to two conical metal end pieces. The diameter of the test specimen shall be 25 ± 0.5 mm (1.00 ± 0.2 in.). The distance between the opposed conical end pieces shall be 11.5 ± 1.2 mm (0.45 ± 0.05 in.). For the test specimen in Fig. 7, the height of each conical end piece shall be 23.4 ± 0.5 mm (0.92 ± 0.02 in.) (Note 9). The half-angle of the cone vertex shall be 45 ± 1° and the tip shall not be rounded to radius greater than 0.8 mm (0.03 in.).

NOTE 9—Theoretically, the height of end pieces could be any length. The end pieces in Fig. 7 have been the most practical size for molding and testing.



| | mm | in. |
|----------------|------------|-------------|
| A | 25.0 ± 0.5 | 1.00 ± 0.02 |
| B ^A | 23.5 ± 0.5 | 0.92 ± 0.02 |
| C | 11.5 ± 1.2 | 0.45 ± 0.05 |
| D | 45° ± 1 | |
| E (radius) | 0.8 max | 0.03 max |

^A Suggested dimension.

FIG. 7 Test Specimens with Conical Adhesion Areas

27. Laboratory Preparation of Standard Test Specimens

27.1 The standard test specimen is formed by two cylindrical rigid parts terminated by opposite conical ends, and a cylinder of rubber bonded to the conical ends. Prepare in the laboratory in accordance with 27.1.1 through 27.1.7.

27.1.1 Machine conical metal parts from rolled bar steel SAE No. 1020⁴ or SAE No. 1010 for standard reference pieces. However, other rigid materials may be used if in conformity with the essential dimensions to determine the effect of adhesion on other rigid materials. Prepare the test face of the cone in accordance with any test method for securing adhesion that may be under investigation.

27.1.2 Vulcanize the specimens in a transfer mold. For the six-cavity transfer mold shown in Fig. 8, the unvulcanized rubber piece shall be approximately 20 mm (0.8 in.) in thickness and approximately 110 mm (4.3 in.) in diameter. Sufficient rubber stock should be used to fill the pot and provide some excess after filling the mold cavities.

27.1.3 Clean or treat the surface of the conical ends in accordance with the adhesion system under investigation. Coat the treated metal surface with the adhesive over the conical area only.

27.1.4 Place the rigid parts and the rubber compound in the preheated mold for vulcanization.

27.1.5 Vulcanize with conditions of time, temperature, and pressure being regulated by their interrelationship effect on adhesion to be determined. It may be necessary to surround the mold with an insulating shield to maintain proper temperature during cure.

27.1.5.1 At the conclusion of the cure, take great care when removing the test specimens from the mold to avoid subjecting the bonded surfaces to undue stress before the test specimens have cooled.

27.1.6 After vulcanization, store the specimens at a temperature of 23 ± 2°C (73.4 ± 3.6°F) for at least 16 h prior to testing.

27.1.7 Fig. 8 shows a mold, unvulcanized rubber compound, molded test specimens, and adhesive coated conical pieces.

28. Procedure

28.1 Mount the test specimens in the grips of the test machine using the appropriate fixtures. Take care in centering and adjusting the test specimen so the tension is symmetrically distributed in the cross section during the test. Apply the tension load until the rubber either separates from the adhesive surface or ruptures. Record the total load at the time of failure. Record type or types of failure and express in accordance with the standard adhesion failure terminology (see Section 24).

29. Calculations

29.1 Express the adhesion value in Newtons or pounds-force required to produce failure. In cases where the failure is in the rubber, the adhesion value is recognized as being higher than that recorded.

30. Report

30.1 The report shall include the following, preferably reported on an Adhesion Test Data Sheet illustrated in Table 1:

- 30.1.1 Type and code number of the rubber compound,
- 30.1.2 Cure time and temperature,
- 30.1.3 Type of metal or rigid substrate used and surface preparation,
- 30.1.4 Adhesive and dilution,
- 30.1.5 The adhesion value for each test piece,
- 30.1.6 Type and percentage of failure,
- 30.1.7 Date of vulcanization and date of test, and
- 30.1.8 Temperature of test room.

31. Precision and Bias

31.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.

32. Salvaging of Bonded Metal Parts

32.1 Bonded metal parts may be salvaged by the usual burning or chemical stripping techniques. Mechanical or chemical surface treatments are used to re-establish a clean bonding surface. The sharpness of the conical tip may be reduced during salvaging. This affects reproducibility of the test results and care must be taken to re-establish the sharpness of this cone to 0.8-mm (0.03-in.) radius (maximum).

METHOD D—ADHESION TEST: POST-VULCANIZATION (PV) BONDING OF RUBBER TO METAL

33. Significance and Use

33.1 Methods A, B, and C of this standard cover procedures for testing the strength of adhesion of rubber to metal when the bond is formed during vulcanization. Method D may be used to determine the strength of adhesion of rubber to metal when the bond is formed after the rubber has been vulcanized. In the rubber industry, this is commonly referred to as post-vulcanization (PV) bonding.

33.2 In the test, a vulcanized circular disk that has been adhesive coated on both ends, is assembled between two parallel metal parts. The rubber-metal assembly is then placed



FIG. 8 Mold and Conical Test Specimens

in a fixture under compression and heated in an oven for a specified time at a specified temperature.

33.3 When tested to rupture in an axial direction, a measurement of rubber-to-metal adhesion is obtained. The rupture force and type of failure are recorded.

34. Apparatus

34.1 *Testing Machine*, A tension testing machine conforming to the requirements of Practices E4 shall be used for measuring the strength of adhesion. The crosshead of the machine shall travel at the uniform rate of 0.83 ± 0.08 mm/s (2.0 ± 0.2 in./min). The machine shall be provided with a device to record the maximum force in Newtons or pounds-force at the conclusion of the test. A machine of at least 44.5-kN (10 000-lbf) capacity is suitable.

34.2 *Grips*—The fixtures for holding the specimen in the testing machine will depend on the type of specimen but in all cases shall be provided with ball seats, or a suitable mechanism, to permit proper centering of the load during the test. These grips can be of the type described in 6.2 of Method A and are illustrated in Fig. 1 and Fig. 3.

34.3 Details of the apparatus used to hold and compress the rubber metal bonded assembly during oven cure are shown in Fig. 9 (outside plates) and Fig. 10 (center aligning plate). The center aligning plate, which controls compression of the bonded assembly to 10.0 ± 0.1 %, is held to the outside plates by means of two bolts, washers, and nuts. The bolts are tightened until the metal pieces compress the rubber properly.

35. Standard Test Specimens

35.1 Prepare the vulcanized rubber test specimen in accordance with the procedure given in Test Methods D395.

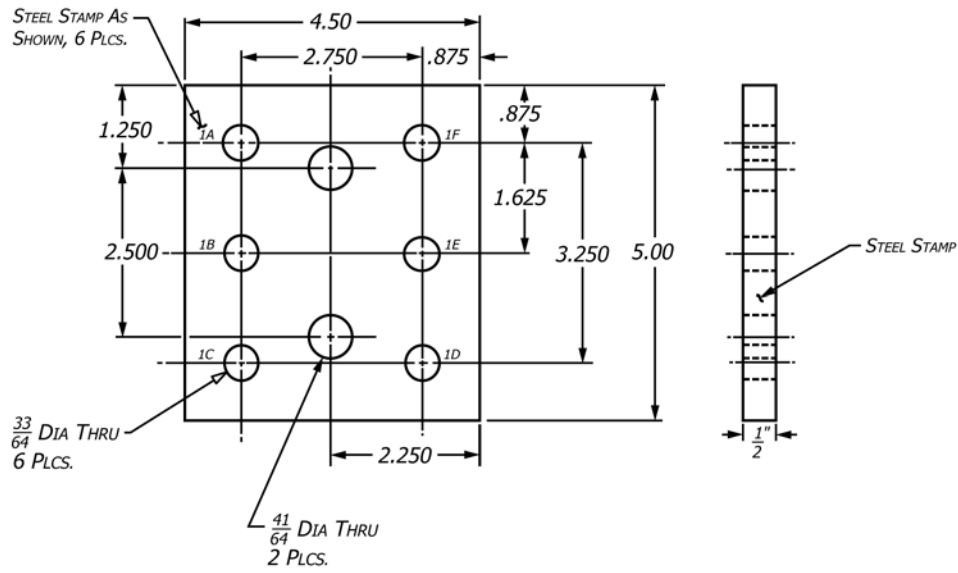
35.2 The metal test specimen is identical to the standard metal test specimen described in Section 6, Method A, of this standard.

36. Preparation of Standard Test Specimens

36.1 Soak lint-free paper towel in methyl ethyl ketone (MEK), or another suitable cleaning solvent, and lay flat on a clean surface. Wipe the rubber surfaces to be bonded several times across the paper to remove any residual surface contaminants. Following air-drying for at least 15 min, post-dry the cleaned rubber specimens for 10 min at 70°C in a circulating air oven. Remove the rubber specimens from the oven and allow to condition at Standard Laboratory Temperature ($23 \pm 2^\circ\text{C}$) for at least 30 min prior to adhesive coating, preferably by brush application.

36.2 Dip each surface of the adhesive-coated specimen alternately into a suitable assembly oil⁵ to a depth slightly greater than the surface to be covered. Place the specimen on its side and allow excess oil to drain off for about 5 min prior to assembly.

⁵ Rubber assembly oil—Ex. Seagul Type R, Clark Oil and Chemical Co., Cleveland, OH.



| in. | mm |
|-------|--------|
| 1/2 | 12.7 |
| 33/64 | 13.10 |
| 41/64 | 16.27 |
| 0.875 | 22.23 |
| 1.250 | 31.75 |
| 1.625 | 41.28 |
| 2.250 | 57.15 |
| 2.500 | 63.50 |
| 3.250 | 82.55 |
| 4.50 | 114.30 |
| 5.00 | 127.0 |

FIG. 9 Outside Plates (Two Required) Low-Carbon Steel

36.3 Assemble the test unit in accordance with Fig. 11. Required compression is automatically obtained by the spacer as the unit is tightened.

36.4 Place the test unit in a circulating air oven at 150°C for a period of 60 ± 10 min, or at a time and temperature recommended by the adhesive supplier. Remove the unit and immediately cool in room temperature tap water for at least 20 min. Following cooling, disassemble the test unit and store the individual test specimens at a temperature of 23 ± 2°C at least 48 h prior to testing.

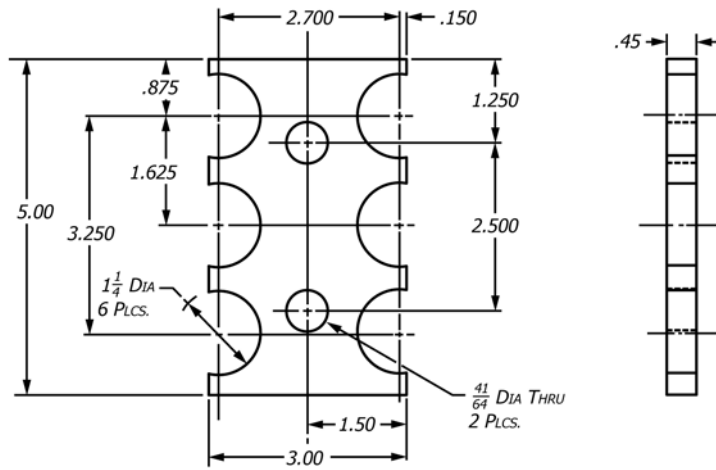
37. Procedure

37.1 Attach the bonded assembly to the tension testing machine using the appropriate fixtures or grips. Apply a force

until destruction of the bonded assembly is complete. Record the type of failure, expressed in accordance with the adhesion failure terminology described in Section 4 under Method A of this standard.

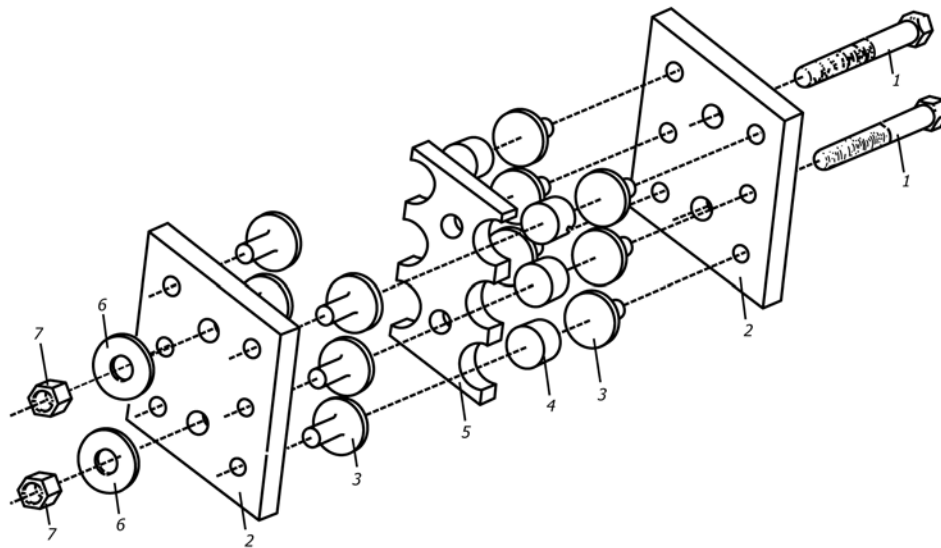
38. Calculations

38.1 Express the adhesion value in pascals (or kPa, MPa) or pounds-force per square inch and calculate by dividing the tension force by the original area of the adhered surface. In the case of rupture of the rubber, the adhesion value is recognized as being higher than the rupture strength that is calculated as described above.



| in. | mm |
|-------|--------|
| 0.150 | 3.81 |
| 0.45 | 11.40 |
| 41/64 | 16.27 |
| 0.875 | 22.23 |
| 1 1/4 | 31.75 |
| 1.250 | 31.75 |
| 1.50 | 38.10 |
| 1.625 | 41.28 |
| 2.500 | 63.50 |
| 2.700 | 68.58 |
| 3.00 | 76.20 |
| 5.00 | 127.00 |

FIG. 10 Center Aligning Plate (One Required) Low-Carbon Steel



| No. | Description | No. Req'd |
|-----|---------------------------------|-----------|
| 1 | 5/8-16 UNC 3-in. long hex bolt | 2 |
| 2 | Fig. 9 - outside plate | 2 |
| 3 | Metal test specimen | 12 |
| 4 | Vulcanized rubber puck | 6 |
| 5 | Fig. 10 - center aligning plate | 1 |
| 6 | Washer | 2 |
| 7 | 5/8-16 UNC nut | 2 |

FIG. 11 Total Assembly

39. Report

39.1 The report shall include the following, preferably reported on an Adhesion Test Data Sheet illustrated in **Table 1**:

- 39.1.1 Type and code number of the rubber compound,
- 39.1.2 Bonded assembly preparation; cure time and temperature,
- 39.1.3 Type of metal and surface preparation,
- 39.1.4 Adhesive and dilution,
- 39.1.5 Method of application,
- 39.1.6 Failure or rupture stress measured in pascals or pounds-force per square inch,
- 39.1.7 Type of failure, and
- 39.1.8 Date of test.

40. Precision and Bias⁴

40.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.

METHOD E—90° STRIPPING TEST—RUBBER TANK LINING—ASSEMBLED TO ONE METAL PLATE

41. Significance and Use

41.1 This test is intended to determine the adhesive strength of rubber tank lining to tank wall. The results are obtained by measuring the pull necessary to separate a rubber strip from a metal surface. The data obtained indicate the strength of adhesion along a line across the width of the rubber strip being separated from a plate at a 90° angle. The test provides valuable data for development and control of rubber compounds and methods of bonding. It also serves as a screening test for the evaluation of various bonding agents, techniques, or both.

42. Adhesion Failure Terminology

- 42.1 *R* indicates the failure is in the rubber.
- 42.2 *RC* indicates the failure is at the rubber-cover cement interface.
- 42.3 *CP* indicates the failure is at the cover cement-prime cement interface.
- 42.4 *M* (can also be denoted as *CM*) indicates the failure is at the metal-prime cement interface.

NOTE 10—*Example*—The percentages of the various types of failure may be estimated as in the following examples:

R-50, RC-50 means that roughly one half or 50 % of the area showed failure in the rubber and the other 50 % showed failure at the rubber – cover cement interface.

R-25, RC-25, M-50 means that three types of failure are present with the *M* indicating 50 % failure at the metal – primer interface.

43. Static-Mass Method (SM)

43.1 Apparatus:

43.1.1 The apparatus required for the adhesion test by the static-mass test method consists of a supporting frame, testing clamps, calibrated masses, and a mass carrier.

43.1.2 The frame has sufficient height to permit the mass carrier to be suspended from the clamp holding the test specimen and to hang freely during the progress of the test.

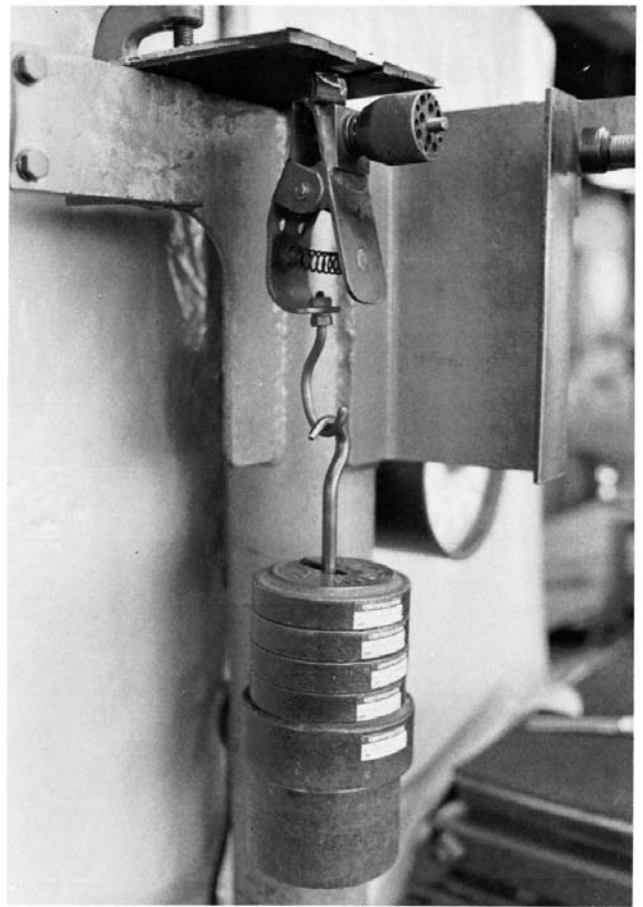


FIG. 12 Test Apparatus For Static Mass Adhesion Test on Rubber Tank Lining

43.1.3 A photograph of a suitable support frame, together with test specimen, clamp, mass carrier, and calibrated masses, is shown in **Fig. 12**.

44. Standard Test Specimen

44.1 A standard test specimen is shown in **Fig. 13** and the 25 ± 0.05 -mm (1.000 ± 0.002 -in.) wide strip of rubber is fixed approximately in the center of the metal plate. Overall size of the prepared test specimen from which the 25 mm wide strip is taken is 150 ± 3 mm (6.000 ± 0.15 in.) square. One end of the cemented plate is to be masked with approximately 25 mm (1 in.) wide pressure-sensitive tape.

45. Laboratory Preparation of Standard Test Pieces

45.1 The standard test specimens shall be as described in **45.1.1 to 45.1.4**.

45.1.1 Prepare contact area of hot-rolled, mild black steel in accordance with the method for securing adhesion that is specified by supplier, or that may be under investigation. Size of metal plate is approximately 150 mm^2 (6 in.^2) and at least 3.4 mm (0.13 in.) in thickness. Mask one end with approximately 25 mm (1.0 in.) wide pressure-sensitive tape.

NOTE 11—In cases where the 3.18-mm (0.135-in.) thick metal is permanently deformed during testing, a thicker metal plate may be used. Likewise, a different grade of steel may be specified when agreed upon

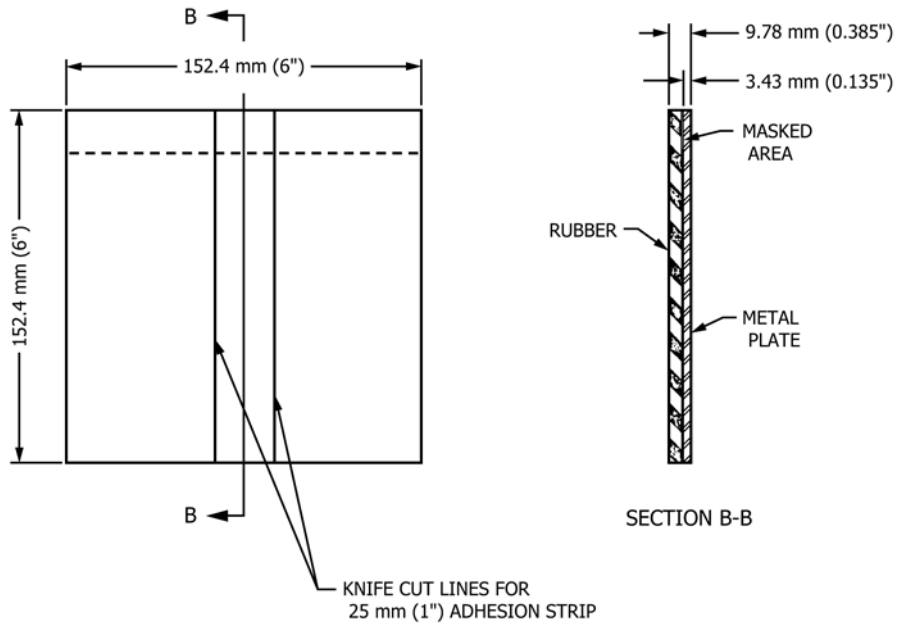


FIG. 13 Adhesion Test Specimen for Rubber Tank Lining



FIG. 14 An Example of a Rubber Tank Lining Adhesion Specimen in a Testing Machine

between the purchaser and the supplier.

45.1.2 Apply unvulcanized rubber slabs, 6.3 ± 0.5 mm (0.25 ± 0.02 in.) thick to the prepared surface of the metal plate.

NOTE 12—Other thickness of rubber may be used to correspond to actual ordered gage of tank lining stock if so noted on test report.

45.1.3 Vulcanize the rubber in the same manner as recommended by the rubber tank lining manufacturer; that is, cured by exhaust steam, steam pressure, or chemical cure at the recommended time, temperature, and procedure.

45.1.4 After vulcanization, store the specimens at a temperature of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) at least 16 h. Cut a 25-mm wide strip as shown in Fig. 9.

46. Procedure

46.1 Separate the part to be tested by hand at the masked area end and at a sufficient distance to permit jaws of the testing clamp to be attached. Mount the plate on a support frame, attach a clamp to the test specimen at separated end. Suspend a mass carrier to the clamp and apply known masses

to the carrier until the mass causes a constant rate of travel of 0.4 mm/s (1 in./min) throughout the test.

47. Machine Method (M)

47.1 Apparatus:

47.1.1 *Testing Machine*—Paragraph 6.1 of Test Methods **D412** describes the testing machine that shall be used for measuring the strength of adhesion. The head of the machine shall travel at the uniform rate of 50 mm (2.0 in.)/min.

47.1.2 *Fixture*—Any suitable fixture for holding the test specimen to the upper head of the machine may be used provided the direction of pull to cause separation shall be as nearly perpendicular as possible at all times during the test, that is, making a 90° angle with the top holding fixture. The test fixture shown in operation in **Fig. 5** complies with the above and provides ease and simplicity of testing.

47.1.3 *Grips*—Any suitable grip may be used, provided it does not slip or rupture the rubber strip. The grip shown in **Fig. 5** complies and provides ease and simplicity of operation.

48. Standard Test Specimen

48.1 Refer to **44.1** (same as used for static-mass test method).

49. Laboratory Preparation of Standard Test Pieces

49.1 Refer to **45.1** through **45.1.4**. (Same as used for static-mass test method).

50. Procedure

50.1 Place the test specimen (**Note 13**) symmetrically in the jaws attached to the fixed grip with the separating edge toward the operator, as shown in **Fig. 5**. Place the tab in the grip and apply a steady load at the rate of 50 mm (2.0 in.)/min until separation is complete. Use the autographic recorder to show graphically the adhesion values over the full length of the test specimen.

NOTE 13—This specimen lends itself to the determination of the relative effect of environmental conditions on possible bond deterioration. Specimens may be exposed unstressed or under a constant, relatively low bond stress. It is recommended that one of the standard ASTM environmental exposure conditions be used. Among those applicable are the following: Test Methods **D471**, **D572**, **D573**, **D1149**, Practice **G153**, and Practice **B117**. If environments other than the above are used, they shall be described completely. This should include the conditions of the exposure such as time, temperature, etc.

51. Precautions

51.1 Tearing:

51.1.1 If, during a test, one of the parts begins to tear instead of separating from the other part of the specimen, cut the material being torn with a knife up to the surface of contact between the two parts and start the test again.

51.1.2 If one of the parts repeatedly tears, instead of separating it from the other part, a satisfactory result may be secured in the static-mass test method by reducing the amount of the mass used if this is permitted. In case of repeated tearing when using the machine test method, take as the result of the test the average load at which tearing occurs.

51.2 *Effect of Temperature*—The results of this test are affected by temperature. Elevating the temperature causes a faster rate of stripping under a given load. The temperature during test shall therefore be $23 \pm 2^\circ\text{C}$ ($73.1 \pm 3.6^\circ\text{F}$) and the actual temperature shall be recorded.

52. Calculations

52.1 For the static-mass test method, report the adhesion value as separation rate per unit width under a stated force, as follows:

$$\begin{array}{l} \text{___ mm/s. for 25-mm width under ___ N force} \\ \text{___ in./s for 1-in. width under ___ lbf} \end{array}$$

If a width other than 25 mm (1 in.) was used, correct the separation by multiplying the rate by the quotient of actual width per desired width as follows:

$$\begin{array}{l} \text{or ___ mm/25 mm} \\ \text{___ in./1 in.} \end{array}$$

Include both the rate and the actual width in the report.

For the machine test method, the autographic chart constitutes the report, but the value of the adhesion is determined by drawing on the chart the best average line between the maximum and minimum force values. The force so indicated, expressed in Newtons per metre (or pounds-force per inch) is divided by the width measured in **10.1** for separation at the rate used and is reported as the adhesion value, as follows:

$$N/m \text{ (lbf/in.)} = \text{force/actual width} \quad (1)$$

Show both the force and actual width in the report.

53. Interpretation of Results

53.1 In the static-mass test method, no stripping is started unless a certain mass is used. Small increments in mass do not progressively cause proportional increases in rate of separation. The rate initially increases slowly, but increases rapidly as the mass increases. Rate of separation must therefore be interpreted carefully. A specimen required to separate not more than 0.4 mm/s (1 in./min) under a force of 90 N (20 lbf) might be regarded as very inferior if the test result was 2 mm/s (5 in./min). The same specimen might not strip at all under a force of 67 N (15 lbf) and might meet the requirement with a force of 80 N (18 lbf).

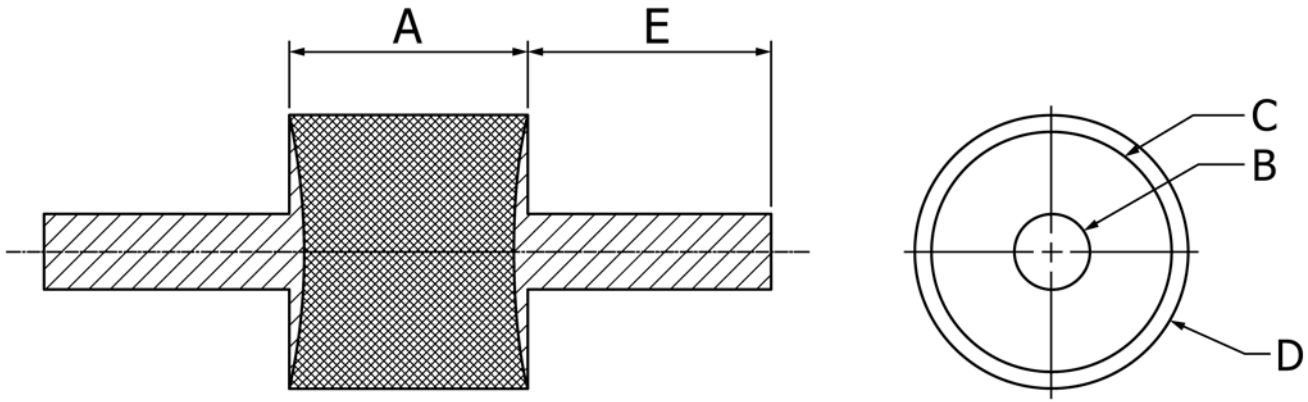
53.2 The true adhesion value is not determined in the case where the specimens repeatedly tear. The test, however, indicates that the strength of adhesion exceeds the strength of the material, and that the adhesion value is not less than the result obtained. In cases of tearing, the results should be so designated.

53.3 Adhesion values may differ between different plies of the same article of plied construction and also at different points along the same ply. The adhesion value of one ply to another is taken as the average result of the test over a reasonable distance of separation. When possible, a distance of separation of at least 100 mm (4 in.) shall be used.

54. Report

54.1 The report shall include the following:

54.1.1 Results of the adhesion test expressed in accordance with Sections **53** and **52**,



| Dimension | Size (mm) |
|--------------------------|---------------------------|
| A | 25.0 mm ± 0.05 mm |
| B | 8 mm × 1.25 thread |
| C | 25.0 mm ± 0.76 mm |
| D | 28.7 mm ± 0.76 mm |
| E | 25.4 mm ± 0.76 mm |
| Radius of Surface | 41.3 mm ± 0.25 mm |

FIG. 15 Test Specimen

54.1.2 All observations and recorded data on which the results are based,

54.1.3 Date of manufacture or vulcanization of rubber, if known,

54.1.4 Date of test,

54.1.5 Statement of the test method used (static-mass or machine),

54.1.6 Temperature of test room, and

54.1.7 Dimensions of the test specimen.

55. Precision and Bias⁴

55.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.

METHOD F—RUBBER PART ASSEMBLED BETWEEN TWO PARALLEL CONVEX-SHAPED METAL PLATES

56. Significance and Use

56.1 Method F is used for determining the adhesive strength of rubber-to-metal bonding agents. The adhesion values are obtained by measuring the force in an axial direction that is necessary to separate the bonded rubber from the metal component. The metal component is composed of two parallel metal test specimens that have a convex shape on the bonded side. (Fig. 15). This particular geometry produces in many

instances, interfacial failure(s) and thus is a discriminatory screening test for the agents.

57. Adhesion Failure Terminology

57.1 Express the type of adhesion failure as determined by examination of broken test pieces by one or more of the following symbols:

57.2 *R* Indicates failure in the rubber (substrate failure).

57.3 *RC* indicates failure in the rubber-cover cement (interfacial failure).

57.4 *CP* indicates failure in the cover cement-prime cement (interfacial failure).

57.5 *M* or *CM* indicates failure in the prime cement-metal interface (interfacial failure).

NOTE 14—*Example*—The percentage of the various types of failure may be estimated as in the following examples:

R-60, RC40 means that 60 % of the bonded area showed failure in the rubber and the other 40 % showed failure at the rubber-cover cement interface.

R-25, RC-25, M-50 means that three types of failure are present with *R* indicating 25 % failure in the rubber, *RC*, 25 % failure between the rubber and the cement(adhesive)interface, and 50 % failure at the metal-primer interface, 25 % in the rubber.

58. Apparatus

58.1 *Testing Machine*—A testing machine as described in 6.1 of Test Methods D412 shall be used for measuring the

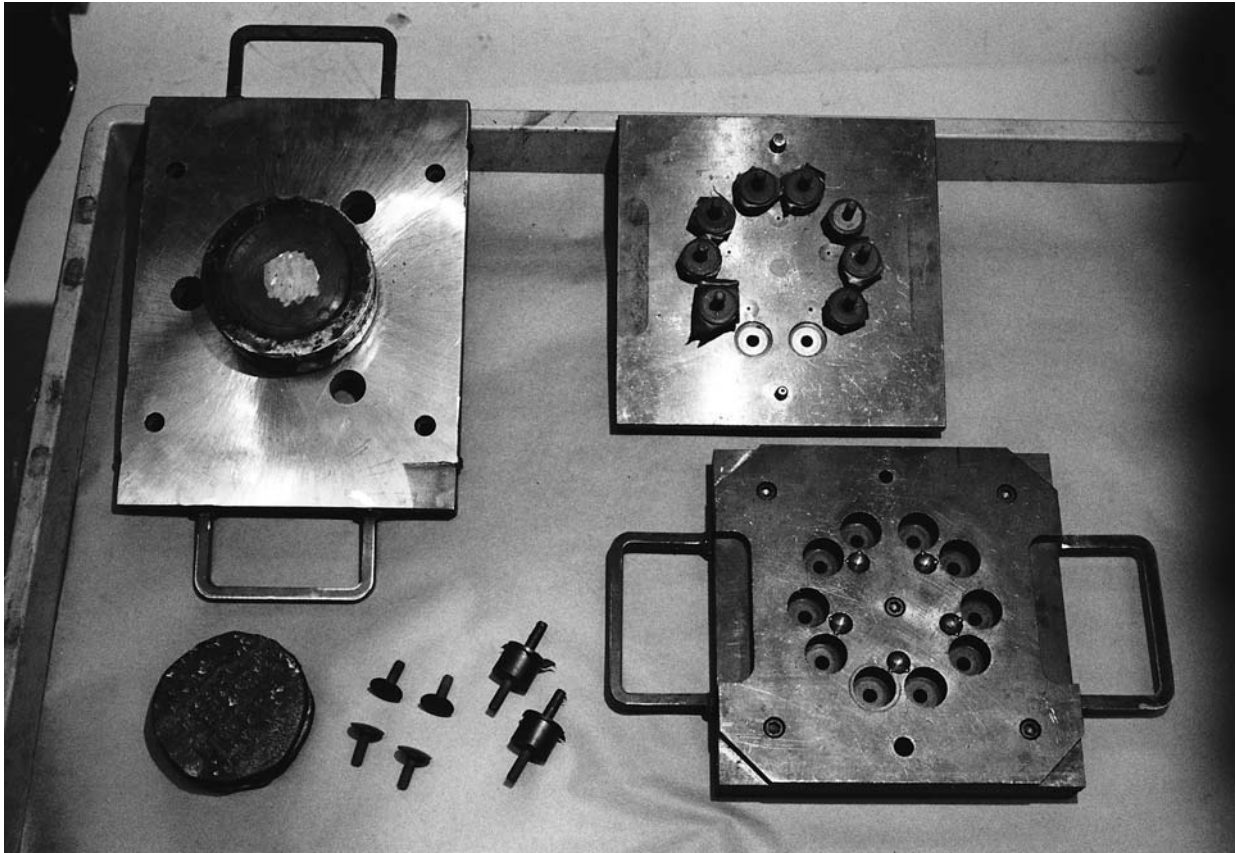


FIG. 16 Test Mold and Test Specimens

strength of adhesion. The head of the machine shall travel at the uniform rate of 0.83 ± 0.08 mm/s (2 ± 0.2 in./min). The machine shall be provided with a recording device to give the force in newtons or pounds-force at the conclusion of the test.

58.2 *Fixture*—The fixtures for holding the specimen in the test machine shall be threaded so the threaded studs of the test specimen can be screwed into the grip. Fixtures may be provided with ball seats, or a similar mechanism, to permit proper centering of the load during the test.

59. Standard Test Specimen

59.1 A standard test specimen shall consist of a rubber compound bonded to two convex-shaped metal end pieces. The diameter of each test specimen shall be 28.70 ± 0.76 mm. The distance between the pieces shall be 25.00 ± 0.76 mm, the radius 41.3 ± 0.25 mm. (see Fig. 15).

60. Laboratory Preparation of Test Specimens

60.1 The standard test specimen is formed by vulcanizing the desired type of rubber in a mold containing the adhesive coated metal test parts. Injection molding, transfer molding, or compression molding may be used. Prepare in the laboratory in accordance with 60.1.1 – 60.1.6.

60.1.1 Machine the metal test part from rolled bar steel SAE No. 1020 or SAE No. 1010 for standard reference pieces. Other rigid materials may be used if in conformity with the essential dimensions.

60.1.2 Prepare the test face of the specified convex specimen in accordance with any test method for securing adhesion that may be under investigation (for example, phosphated treated, mechanical blasted, etc.).

60.1.3 Coat the prepared metal surface with the adhesive system to be tested in accordance to the manufacturers recommendations.

60.1.4 Place the dry adhesive coated specimens in the mold. For compression or transfer molding, place the rubber preforms in the mold cavity. For injection molding, inject the rubber into the mold.

60.1.5 Vulcanize with conditions of time, temperature, and pressure in accordance with the system being investigated.

60.1.6 After a suitable vulcanization cycle, remove the specimens with care to avoid subjecting the adhered surfaces to stress before the specimens have cooled. Store the specimens at a temperature of $23 \pm 2^\circ\text{C}$ ($73 \pm 4^\circ\text{F}$) for at least 16 h prior to testing.

60.2 Fig. 16 shows a photograph of a mold and inserts.

61. Procedure

61.1 Mount the test specimens in the threaded grips of the test machine. Apply the tension load until the rubber either separates from the adhesive surface or the rubber ruptures. Record the total load at the time of failure. Observe the record the type or types of failure and express in accordance with the standard adhesion failure terminology (see Section 57).

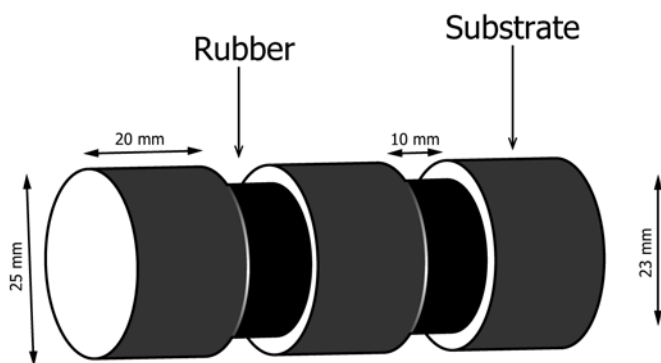


FIG. 17 Double Shear Test Piece

62. Calculation

62.1 Express the adhesion value in newtons (N) or pounds-force (lbs) required to produce failure. In cases where the failure is in the rubber, the adhesion value is recognized as being higher than that recorded.

63. Report

63.1 The report shall include the following, preferably reported on an Adhesion Test Data Sheet illustrated in Table 1:

- 63.1.1 Type and code number of the rubber compound,
- 63.1.2 Cure time and temperature,
- 63.1.3 Type of metal or rigid substrate used and surface preparation,
- 63.1.4 Molding method used (compression, injection, etc.)
- 63.1.5 Adhesive used, dilution, and application method,
- 63.1.6 The adhesion value for each test piece,
- 63.1.7 Type and percentage of failure,
- 63.1.8 Date of vulcanization and date of test, and
- 63.1.9 Temperature of test room and humidity.

64. Precision and Bias

64.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.

METHOD G—MEASURING BOND DURABILITY FOR RUBBER-TO-METAL BONDED COMPONENTS WITH A DOUBLE SHEAR CYLINDRICAL SPECIMEN

65. Significance and Use

65.1 Method G is used to determine relative bond durability between various adhesives. The adhesives to be compared are used to bond rubber-to-metal using a double shear cylindrical specimen (see Fig. 17) which is then placed in a fixture to impart a shear stress on the bond line. This assembly (see Fig. 18) is then placed in the prescribed test medium and the time to obtain a failure depth of 5 mm at the rubber-metal bond interface is noted. This test measures the durability of a specific adhesive as a function of changes in the rubber formation. Different adhesives may be examined to find the best for a given rubber recipe. Also, different metal preparations and their effect on bond durability may be investigated.

66. Adhesion Failure Terminology

66.1 Express the type of adhesion failure as determined by the visual examination of the of the failed area using one or more of the following symbols:

66.2 *R* indicates failure in the rubber.

66.3 *RC* indicates failure in between the rubber and the adhesive.

66.4 *CP* indicates failure between the adhesive top coat and the primer in the case of a two coat system.

66.5 *M* (or *CM*) indicates failure between the primer (or adhesive in the case of a one coat system) and the substrate, for example, metal.

NOTE 15—*Example*—The percentage of the various types of failure may be estimated as in the following examples:

R-60, RC-40 means that 60 % of the bonded area showed failure in the rubber and the other 40 % showed failure at the rubber-adhesive interface.

R-25, RC-25, M-50 means that three types of failures are present with R indicating 25 % failure in the rubber, RC, 25 % failure between the rubber and the cement (adhesive) interface, and 50 % failure at the metal-primer interface.

67. Apparatus

67.1 *Test Specimens*—Fig. 17 shows a molded test specimen. The diameter of the substrate component (for example, steel, aluminum, plastic, etc.) shall be 25 ± 0.1 mm and the length 20 ± 0.1 mm. The rubber sections shall be 23 ± 0.1 mm in diameter and 10 ± 0.1 mm in length. A single- or multi-cavity mold can be used to make the bonded specimen, and it may be a mold for compression, transfer, or injection molding.

67.2 *Test Fixture*—Fig. 18 shows the test fixture for holding the bonded specimens in a strained position. The suggested strain is 100 %, however, some elastomers may have an ultimate shear strain less than 100 % and thus a fixture must be made to accommodate the lower shear strain value. A guideline is as follows:

| Ultimate Shear Strain (%) | Test Strain (%) |
|---------------------------|-----------------|
| >200 | 100 |
| 100 to 200 | 50 |
| 50 to 100 | 25 |

While the test fixture frames may be steel, acetal plastic is preferred. The plastic does not corrode or otherwise contribute to bond failure. Tests have shown consistent results with acetal frames. The positioning of the hole in the frame's center piece determines the percent strain.

67.3 *Test Apparatus*—Fig. 19 shows one apparatus that may be used for immersing the fixtured test specimens. While the exposure conditions may be varied to suit individual programs, the following have been used successfully:

Media: 5 % sodium chloride solution in distilled water
 Temperature: $35 \pm 1^\circ\text{C}$
 Dissolved Oxygen: 17 ± 1 mg/L

68. Laboratory Preparation of Test Specimens

68.1 Prepare the test face of the cylinders with any test method for securing adhesion that may be under investigation, for example, phosphate treated, mechanical blast, etc.

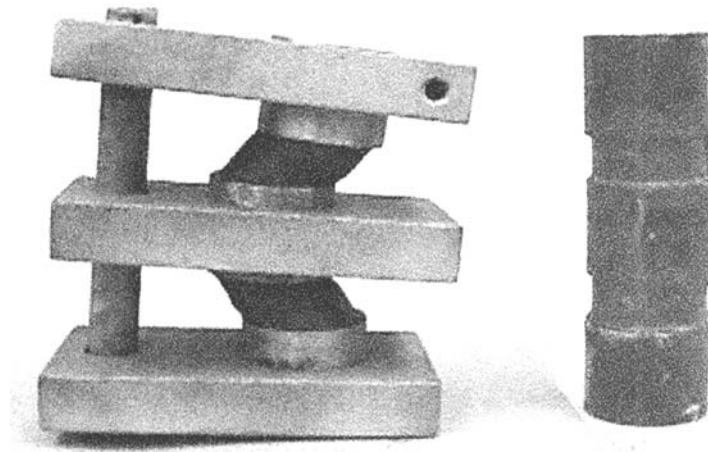


FIG. 18 Test Specimen in a 100 % Strain Fixture (Frame)

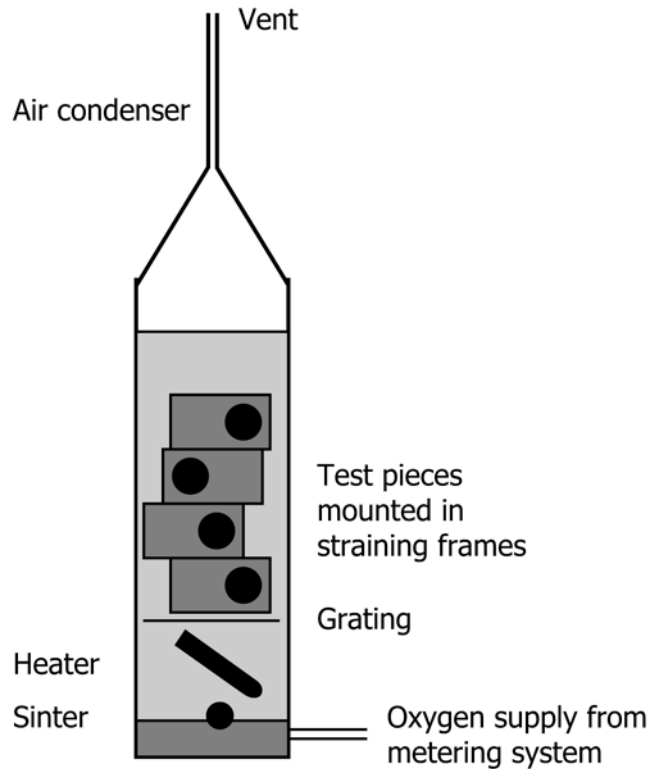


FIG. 19 Test Samples in the Environmental Chamber

68.2 Coat the prepared metal surface with the adhesive system to be tested in accordance with the manufacturer's recommendations, for example, film thickness, dry time.

68.3 Place the dry adhesive coated specimens in the mold. Fig. 20 illustrates the basic mold design for a transfer mold. For compression or transfer molding, place the rubber preform in the mold cavity or transfer pot. For injection molding, inject the rubber into the mold.

68.4 The test specimens are formed by vulcanizing the desired type of rubber in a mold containing the adhesive coated metal cylinders. The cure conditions (temperature and time) are determined by the elastomer compound being used.

68.5 After a suitable vulcanization cycle, remove the specimens. Store the specimens at a temperature of $23 \pm 2^\circ\text{C}$ ($73 \pm 4^\circ\text{F}$) for at least 16 h prior to testing.

69. Procedure

69.1 Pull one testpiece of each molded batch to determine that 100 % adhesion, for example 100R, is obtained prior to immersion.

69.2 Secure the bonded test specimens in the test fixture with the chosen % strain.

69.3 It is suggested that 5 samples (20 interfaces) are tested for each experiment.

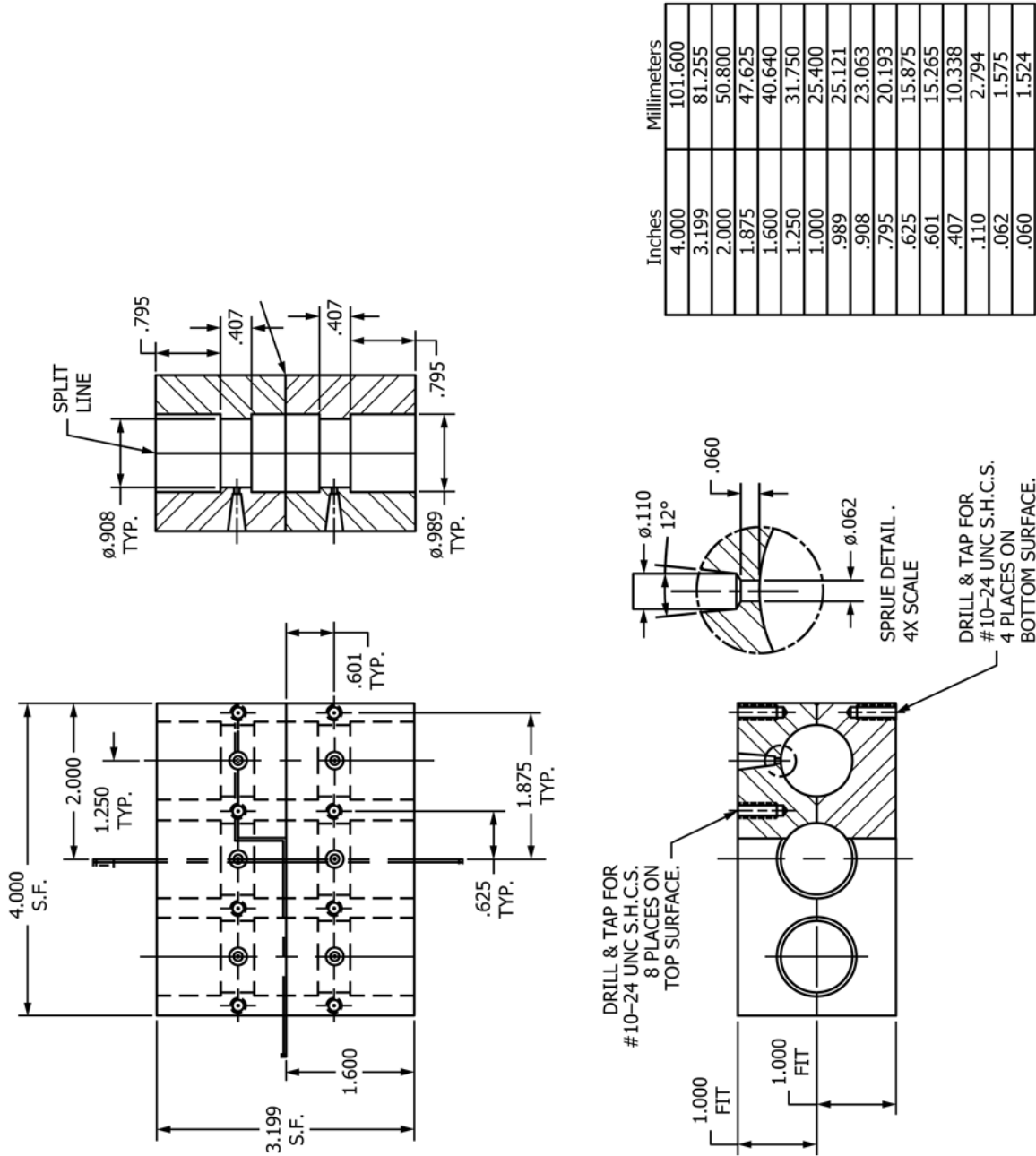
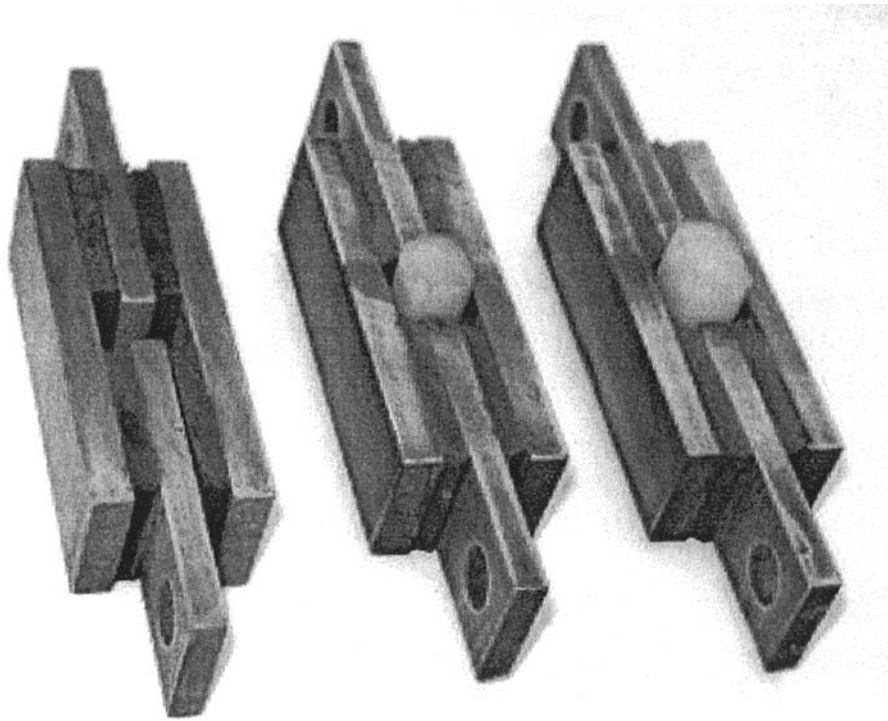


FIG. 20 Basic Mold Design for Making Adhesion Test Pieces

NOTE 1—All dimensions in mm unless otherwise noted.



NOTE 1—Prestrains 0, 50, and 100 % (left to right)

FIG. 21 Quadruple Shear Test Pieces

69.4 Immerse these fixtured parts into the test chamber which has been prepared in terms of temperature and salt content. Begin the oxygen bubbling at the selected rate. At various time intervals, remove the fixtured samples, and using a short steel ruler or other calibrated scale, measure the debond depth. Continue the test until the debond depth of 5 mm is noted for each bond.

70. Calculation

70.1 Measure the amount of debond in mm and the corresponding time.

71. Report

71.1 The report shall include the type and code number of the rubber compound,

71.2 Cure time and temperature,

71.3 Molding method (for example, compression, transfer, injection),

71.4 Type of substrate and its surface preparation,

71.5 Test conditions (salt concentration, dissolved oxygen level, temperature, test shear strain),

71.6 The failure criterion (for example, 5 mm of bond failure),

71.7 The average time required by the replicate bonds to meet the failure criterion, along with standard deviation and 95 % confidence interval,

71.8 Molding method used,

71.9 The adhesive used, dilution, application method and film thickness,

71.10 The type and percentage of bond failure, and

71.11 Date of vulcanization and date of failure measurement.

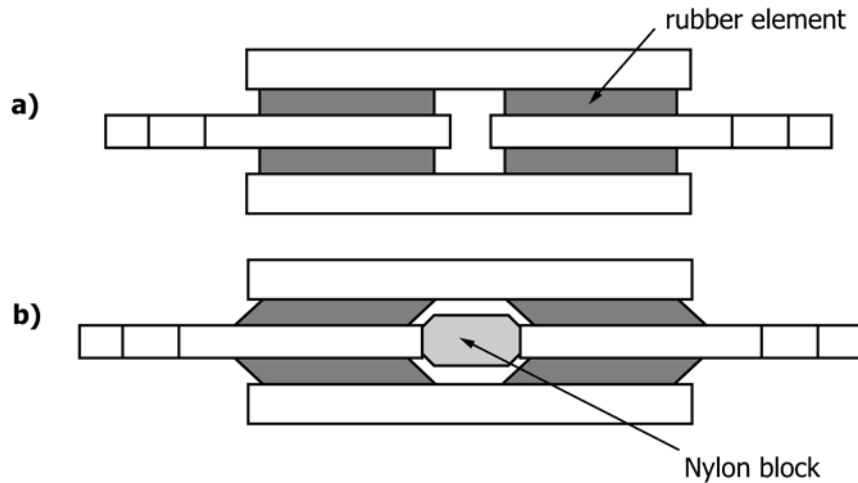
72. Precision and Bias

72.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.

METHOD H—MEASURING BOND DURABILITY OF RUBBER-TO-METAL BONDED COMPONENTS WITH QUADRUPLE LAP SHEAR SPECIMEN

73. Significance and Use

73.1 This method has all the attributes of Method G except that no fixture for imparting the shear stress is required, only a simple block inserted into the test piece once the pre-strain has been applied. The test piece dimensions are similar to an existing standard adhesion test piece ISO 1827: 1991 (BS903: A14: 1992(1998)). This results in a compact test piece, pre-strained to a selected pre-strain, for exposure to prescribed environments such as salt spray, elevated temperature and humidity, boiling water, hot glycol or brine. See Fig. 21. Note that the use of a pre-strain of 100 %, and immersion in boiling water, has provided a highly accelerated test for aqueous bonding agents subjected to water immersion at ambient temperature.



NOTE 1—a.) Unstrained; b.) 100 % strain.

FIG. 22 Quadruple Shear Test Piece

74. Adhesion Failure Terminology

74.1 Express the type of adhesion failure as determined by the visual examination of the of the failed area using one or more of the following symbols:

74.1.1 R indicates the failure is in the rubber.

74.1.2 RC indicates the failure is at the rubber-cover cement interface.

74.1.3 CP indicates the failure is at the cover cement-prime cement interface.

74.1.4 M indicates the failure is at the metal-prime cement interface.

NOTE 16—Example—The percentage of the various types of failure may be estimated as in the following examples: R-60, RC -40 means that 60 % of the bonded area showed failure in the rubber and the other 40% showed failure at the rubber-adhesive interface. R-25, RC-25, M-50 means that three types of failures are present with R indicating 25 % failure in the rubber, RC, 25 % failure between the rubber and the cement (adhesive) interface, and 50 % failure at the metal-primer interface.

75. Apparatus

75.1 Test pieces. Fig. 21 shows a photo of the molded test piece.

75.2 The test piece consists of four identical rubber elements, $4\text{mm} \pm 1\text{mm}$ thick, $20\text{mm} \pm 5\text{mm}$ wide and $25\text{mm} \pm 5\text{mm}$ long. The rubber is bonded to rigid plates of the same width and appropriate lengths to obtain a symmetrical double-sandwich arrangements. The central plates may have a hole at each protruding end to accommodate pins to fixture the assembly to the test machine. See Fig. 22.

75.3 Suitable blocks (for example, nylon) to maintain the desired prestrain. The shape of the block shall be such that, in addition to setting the prescribed prestrain, there is no contact between the block and the strained rubber elements (see Fig. 22).

75.4 Testing machine: To test the adhesion before/after an immersion test, a power driven test machine equipped to produce a uniform rate of grip separation shall be used for measuring the strength of adhesion. The head of the machine

shall travel at the uniform rate of 0.83 ± 0.08 mm/s. (2 ± 0.2 in./min.). The machine shall be provided with a recording device to give the force in Newtons or pounds-force at the conclusion of the test.

76. Laboratory Preparation of Test Specimens

76.1 Masking is required to ensure that the bonding agent is applied only to the portion of the plates where bonding is required. The bonding agent is applied and dried according to the manufacture's suggestions.

76.2 The bonding shall be done using compression molding, transfer molding or injection molding of the rubber into a suitable mold holding the adhesive coated samples.

76.3 A minimum of two replicates (eight interfaces) shall be used for each prestrain and environmental condition, plus two control replicates for each prestrain. In addition, one test piece of each batch shall be prepared as a quality control for the bonding procedure.

77. Procedure

77.1 Pull a quality control test piece from each molded batch to determine that 100 % adhesion (100R) is obtained prior to environmental exposure or laboratory storage with a pre-strain.

77.2 Test pieces are pre-strained by pulling in a test machine to just above the desired pre-strain and inserting a nylon block of suitable dimensions (14×9 mm for 100 % strain) between the central metal plates to maintain the prescribed prestrain.

77.3 Subject the test pieces to the chosen environmental conditions, for example, those shown in 67.3 (Method G). Monitor these conditions to ensure they are stable (for example, for aqueous immersion, the solutions should be stirred adequately and the pH and oxygen concentration should be checked regularly). Control test pieces are to be kept under standard laboratory temperature and humidity.

77.4 Inspect test pieces for signs of corrosion or incipient bond failure at appropriate periods (for example, twice hourly

for eight h; twice daily for 1 week; weekly for 1 month then 4 weekly). Using a short steel ruler or other calibrated scale, measure the de-bond depth.

77.5 After a predetermined period of time, pull to failure each test piece which remains sufficiently intact, including those exposed to the chosen environmental conditions and the control test pieces, and record the peak force required to do this.

77.6 Measure the amount of de-bond in mm and classify the loci of failure according to Section 74. Note that the loci may be different according to whether the failure took place during the environmental exposure or during the pull to failure.

78. Report

78.1 The report shall include the type and code of the rubber compound.

78.2 Cure time and temperature.

78.3 Molding method (for example, compression, transfer, or injection).

78.4 Type of substrate and surface preparation.

78.5 Bonding agent used, application method and film thickness.

78.6 Environmental test conditions (for example, immersion medium, temperature), prestrain(s) and maximum duration of exposure.

78.7 The failure criterion (for example, 5 mm bond failure during environmental exposure).

78.8 An estimate of the time it would take to reach the failure criterion under the prescribed environmental conditions. Ideally this will be the average time taken by replicates to reach the failure criterion, but for effective bonding agents extrapolation may be desirable to avoid unacceptably long test times.

78.9 Type and percentage of bond failure during final shear test and of incipient bond failure during environmental exposure; shear load to failure of test pieces at the end of the prescribed period of environmental exposure.

78.10 Dates of vulcanisation, environmental exposure, incipient failure inspections and tensile machine pull to failure.

78.11 As an option, load-deflection plots for all shear tests to failure, and for the prestrain applied as in 77.2, may be measured and reported.

79. Precision and Bias

79.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.

80. Keywords

80.1 adhesion; adhesive; bond durability; double-shear test piece; elastomer; metals; rubber; rubber-to-metal bonding

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