

# Standard Test Methods for Rubber Property—Brittleness Point of Flexible Polymers and Coated Fabrics<sup>1</sup>

This standard is issued under the fixed designation D2137; 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 ( $\varepsilon$ ) 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 the determination of the lowest temperature at which rubber vulcanizates and rubbercoated fabrics will not exhibit fractures or coating cracks when subjected to specified impact conditions.

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

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

# 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

- D751 Test Methods for Coated Fabrics
- D832 Practice for Rubber Conditioning For Low Temperature Testing
- D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

# 3. Summary of Test Methods

3.1 A specified number of specimens is given a singleimpact under specified impact and temperature conditions until the temperature is found at which no failures occur. This is defined as the brittleness temperature.

3.2 There are four test methods:

3.2.1 *Test Method A*—The determination of the lowest temperature at which rubber vulcanizates will not fracture or crack (refer to 9.1).

3.2.2 *Test Method B*—The determination of the lowest temperature at which rubber-coated fabrics will not fracture or exhibit coating cracks (refer to 9.2).

3.2.3 *Test Method C*—Testing at a specified temperature and testing of materials from a supplier (refer to 9.3). This method is used for the classification of materials and for specification purposes.

3.2.4 *Test Method D*—The determination of 50 % brittleness temperature (refer to 9.4). This is the temperature at which 50 % of the specimens fail.

3.3 These tests may be performed either in a liquid heat transfer media or in a gaseous media (refer to 9.1.1.9).

# 4. Significance and Use

4.1 These test methods cover the evaluation of rubber materials or fabrics coated therewith subjected to low-temperature flex with an impact under well-defined conditions of striker speed. The response is largely dependent on effects of low temperatures such as crystallization, incompatibility of plasticizer, or the inherent dynamic behavior of the material itself. Data obtained by these test methods may be used to predict the product behavior in applications where the conditions are similar to those specified in these test methods.

4.2 These test methods have been found useful for specification and development purposes but do not necessarily indicate the lowest temperature at which the material may be used.

# 5. Apparatus

5.1 *Specimen Clamp*, designed so as to hold firmly the specimen(s) as cantilever beams (Fig. 1).

5.2 *Striker*—The edge of the striker shall have a radius of  $1.6 \pm 0.1 \text{ mm} (0.063 \pm 0.005 \text{ in.})$ . The edge shall move relative to the specimen at a rectilinear speed of  $2.0 \pm 0.2 \text{ m/s}$  ( $6.6 \pm 0.6 \text{ ft/s}$ ) at impact and immediately after. The speed of the solenoid-activated striker should be frequently calibrated by the method described in the annex. Other types of testers

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<sup>&</sup>lt;sup>2</sup> 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.



FIG. 1 Specimen Clamp and Striker

shall be calibrated according to their appropriate methods and manufacturers' instructions. In order to have the required speed, care must be taken to ensure that the striking energy of at least 3.0 J per specimen is used.

Note 1—The striker may be motor-driven, solenoid-operated, gravityactivated or spring-loaded. The motor-driven tester should be equipped with a safety interlock to prevent striker motion when the cover is open.

5.2.1 *Position of Striking Edge*—The distance between the center line of the striking edge and the clamps shall be 8.0  $\pm$  0.3 mm (0.31  $\pm$  0.01 in.). The clearance between the striking arm and the clamp at and immediately following impact shall be:

5.2.1.1 Test Method A, C, and D—6.4  $\pm$  0.3 mm (0.25  $\pm$  0.01 in.)

5.2.1.2 Test Method B— Listed as follows:

Specimen Thickness, mm (in.)	Clearance, mm (in.)
1.65 to 2.20 (0.065 to 0.087)	$6.4 \pm 0.3 \ (0.25 \pm 0.01)$
1.05 to 1.64 (0.041 to 0.064)	5.7 ± 0.3 (0.22 ± 0.01)
0.55 to 1.04 (0.022 to 0.040)	$5.2 \pm 0.3 \ (0.20 \pm 0.01)$
0.10 to 0.54 (0.004 to 0.021)	$4.8 \pm 0.3 \ (0.19 \pm 0.01)$

NOTE 2—The dimensional requirements for Test Method B may be obtained by fabricating individual plates to fit the specimen holder illustrated in Fig. 1.

5.3 *Tank or Test Chamber*—A tank for liquid heat transfer media or a test chamber for gaseous media is required. To ensure thorough circulation of the heat transfer medium, a stirrer should be provided for liquids and a fan or blower for gaseous media.

#### 5.4 Heat Transfer Media:

5.4.1 *Liquid Heat Transfer Medium*—The recommended heat transfer media are listed below. Methanol is typically used, however, it is both flammable and toxic. Methanol, or any flammable or toxic media, shall be used only in a tank/test chamber which is specifically designed and manufactured to accommodate such media.

NOTE 3—Any other liquid heat transfer medium that remains fluid at the test temperature and will not appreciably affect the material tested may be used. The following materials have been used down to the indicated temperatures.

Dow Corning—200 fluids:	°C
5 mm <sup>2</sup> /s viscosity	-60
2 mm <sup>2</sup> /s viscosity	-76
Methanol	-90
Propyl Alcohol or methylcyclohexane	-120

Note 4—The desired temperature may also be obtained by filling the tank with the heat transfer medium and lowering its temperature by the addition of liquid carbon dioxide controlled by a solenoid-activated unit with an associated temperature control. Where temperatures below that obtainable by solid or liquid carbon dioxide are required, liquid nitrogen may be used.

5.4.2 *Gaseous Medium*— A gaseous medium may be used provided ample time is allowed for the specimens to reach temperature equilibrium with the temperature of the medium.

5.4.2.1 A gaseous medium may be used if the low temperature will not affect the operation of the impact mechanism and remains fluid at the specified temperature.

5.5 *Temperature Control*—Suitable means shall be provided for controlling the temperature of the heat transfer medium within  $\pm 0.5^{\circ}$ C ( $\pm 1^{\circ}$ F) if the medium is liquid and within  $\pm 1^{\circ}$ C ( $\pm 1.8^{\circ}$ F) with gaseous medium.

5.5.1 Temperature monitoring is done with a thermocouple or other temperature-sensing device with associated temperature indicator, digital or analog, having a resolution of  $0.5^{\circ}$ C (1°F) or greater and a range suitable for the temperatures at which the tests are to be made.

5.5.2 The thermocouple is preferably constructed of copperconstantan wire having a diameter between 0.2 and 0.5 mm (32 to 24 AWG) and shall be fusion-bonded at the junction. It shall be located as near the specimens as possible without making contact. A thermometer may also be used if it can be shown to agree with the thermocouple or other devices that respond rapidly and accurately to temperature change.

5.5.3 Automatic changes in temperature of a liquid medium may be obtained by means of a system consisting of an externally cooled tank connected to the test area with suitable tubing, a thermoregulator, a pump, an electric immersion heater or internal heat exchanger, and appropriate switches. The regulator, alternately activating both the pump and heating system through the switches, controls the amount of liquid coolant being pumped to the test chamber as well as the amount of heat coming from the heater.

5.5.4 Manual temperature changes for liquid media may be accomplished with powdered carbon dioxide (dry ice) and an electric immersion heater.

5.5.5 Devices employed to monitor temperature, thermocouples or liquid-in-glass thermometers, shall be calibrated at intervals recommended by the manufacturer. If no interval is recommended a one year interval is suggested.

5.5.6 Devices employed to control temperature, typically thermocouples and associated devices, shall be calibrated at intervals recommended by the manufacturer. If no interval is recommended a one year interval is suggested.

## 6. Time Lapse Between Vulcanization and Testing

6.1 For all test purposes, the minimum time between vulcanization and testing shall be 16 h.

6.2 For nonproduct tests, the maximum time between vulcanization and testing should be 672 h (four weeks), and for evaluation intended to be comparable, the tests should be carried out after the same time interval.

6.3 For product tests, whenever possible, the time between vulcanization and testing should not exceed 2160 h (three months). In other cases, tests should be made within 1440 h (two months) of the date of receipt by the customer.

### 7. Test Specimens

7.1 *Test Method A*—Die-cut specimens as illustrated in Fig. 2 shall be considered standard.



Note 1—The test piece thickness is  $2.0 \pm 0.2$  mm. FIG. 2 Test Method A (Modified T-50) Test Specimen (formerly referred to as Type B Specimen)

7.1.1 Test Method B specimens as illustrated in Fig. 3 may be used but will not necessarily provide comparable results. Their use shall be indicated in the test report.

7.1.2 Specimens of other than  $2.0 \pm 0.2 \text{ mm} (0.08 \pm 0.01 \text{ in.})$  thicknesses may be used provided it can be shown that they give equivalent results for the material being tested. Their use shall be indicated in the test report.

7.2 Test Method B—Die-cut specimens as illustrated in Fig. 3 shall be used. They should be die-cut with the longer dimensions parallel to the lengthwise direction of the coated fabric, unless otherwise specified, and be  $40 \pm 6 \text{ mm} (1.6 \pm 0.25 \text{ in.}) \log 6 \pm 0.5 \text{ mm} (0.25 \pm 0.02 \text{ in.}) \text{ wide, and } 2.0 \pm 0.2 \text{ mm} (0.08 \pm 0.01 \text{ in.})$  in thickness.

7.3 *Test Method C*—Die-cut specimens as illustrated in Fig. 1 or Fig. 2 may be used and reported accordingly.

7.4 *Test Method D*—Die-cut specimens as illustrated in Fig. 1 or Fig. 2 may be used and reported accordingly.

7.5 Regular calibration of the cutting dies to maintain the proper geometric dimensions and cutting edge sharpness is important in achieving repeatable and reproducible test outcomes. The cutting die knife edges should be regularly inspected for damage or wear under  $10\times$  or greater magnification. Any cutting die which exhibits nicks or disfigurement shall be removed from service. It is recommended that cutting dies be calibrated and sharpened once yearly or more frequently depending on the frequency and severity of use.

## 8. Conditioning

8.1 *Test Method A*—Condition the test specimens at 23  $\pm$  2°C (73.4  $\pm$  3.6°F) and 50  $\pm$  5 % relative humidity for no less than 16 h prior to testing.

8.2 *Test Method B*—The test specimens shall be conditioned prior to the test in accordance with the standard conditions in Test Methods D751.

8.3 Where long-term effects, such as crystallization, incompatibility, etc., of the material, are to be studied, the test specimens may be conditioned in accordance with Practice D832.



FIG. 3 Test Method B Test Specimen (formerly referred to as Type A Specimen)

8.4 *Test Method C*—Condition the specimens as described in Test Method A (8.1).

8.5 *Test Method D*—Condition the specimens as described in Test Method A (8.1).

## 9. Procedure

9.1 Test Method A:

9.1.1 Test with Liquid Heat Transfer Medium:

9.1.1.1 Prepare and bring the bath to a temperature below the expected lowest temperature of non-failure. Place sufficient liquid in the tank to ensure approximately 25 mm (1 in.) liquid covering the test specimens.

9.1.1.2 Mount five specimens as illustrated in Fig. 2 in the apparatus with the entire tab in the clamp. Immerse the specimens for  $5.0 \pm 0.5$  min at the test temperature. The immersion time shall be reported in 10.1.2.

(1) Alternatively, an immersion time of  $3.0 \pm 0.5$  min may be employed. The immersion time shall be reported in 10.1.2.

9.1.1.3 If five specimens as illustrated in Fig. 3 are used, a minimum of 6 mm (0.25 in.) of the specimen length must be held in the clamp.

9.1.1.4 The clamp shall be properly tightened so that each test specimen is held with approximately the same clamping torque. A clamping torque of 0.15 to 0.25 N is recommended.

9.1.1.5 If the energy capacity causes the speed of the striker to fall below 1.8 m/s (6 ft/s), a smaller number of specimens shall be mounted for testing so that the striker speed does not fall below 1.8 m/s (6 ft/s). This speed shall be maintained for at least 6.0 mm of travel following the impact.

9.1.1.6 After immersion for the specified time, record the actual test temperature and deliver a single impact to the specimens.

9.1.1.7 Examine each specimen to determine whether or not it has failed. Failure is defined as any crack, fissure, or hole visible to the naked eye, or complete separation into two or more pieces. When a specimen has not completely separated, bend it to an angle of  $90^{\circ}$  in the same direction as the bend caused by the impact, then examine it for cracks at the bend.

9.1.1.8 Repeat the test at the next higher temperatures at 10°C intervals using new specimens each time until no failure is obtained. Then decrease the bath temperature at 2°C intervals. Test at each temperature to determine the lowest temperature at which no failures occur. Record this temperature as the lowest temperature of non-failure.

9.1.1.9 Test with Gaseous Heat Transfer Medium:

(1) Adjust the refrigerating unit and bring the test chamber, test apparatus, and specimens to thermal equilibrium at the desired temperature (see Note 4). An alternative method is to place the striker and specimen clamp through the top of the refrigerating unit with the solenoid remaining outside the unit and insulated from the cold air.

(2) The actual testing is performed in the same manner as described in 9.1.1.

## 9.2 Test Method B:

9.2.1 Follow the instructions in accordance with 9.1.1.1 through 9.1.1.9, except that specimens as illustrated in Fig. 3 shall be used. The specimens shall be examined for any visible fracture or crack in the coating under  $5 \times$  magnification, after