Designation: D5289 – 12

Standard Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters[1]

This standard is issued under the fixed designation D5289; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a method for the measurement of selected vulcanization characteristics of rubber compounds using unsealed and sealed torsion shear cure meters. The two types of instruments may not give the same results.

NOTE 1—An alternative method for the measurement of vulcanization characteristics is given in Test Method D2084.

1.2 The values stated in SI units are to be regarded as the 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:*[2]

D1349 Practice for Rubber—Standard Conditions for Testing

D1566 Terminology Relating to Rubber

D2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *rotorless cure meter, n*—a name for a class of cure meters that uses one of the two specimen shaping members or dies to sense the torque or stress during strain application. Rotorless cure meters do not have a third member in the form of a rotor (see definitions of cure meter in Terminology D1566 and Test Method D2084).

3.1.2 *S' torque, n—for an oscillating shear rotorless cure meter*, the value measured by a torque transducer at the peak strain amplitude of the oscillating cycle; represents the elastic response of the test material.

3.1.3 The following measurements may be taken from the recorded curve of torque as a function of time (see Fig. 1):

3.1.3.1 *minimum S' torque*—measure of the elastic stiffness of the unvulcanized test specimen at the specified vulcanizing temperature, taken at the lowest point in the vulcanization curve.

3.1.3.2 *maximum, plateau,* or *highest S' torque*—measure of the elastic stiffness of the vulcanized test specimen at the specified vulcanizing temperature, measured within a specified period of time.

3.1.3.3 *time to a percentage of full cure*—measure of cure based on the time to develop some percentage of the difference in S' torque from the minimum to the maximum.

3.1.3.4 *time to incipient cure (scorch time)*—measure of the time at which a specified small increase in S' torque has occurred; it indicates the beginning of vulcanization.

3.1.4 *S" torque, n—for an oscillating shear rotorless cure meter*, the value measured by a torque transducer at zero strain amplitude of the oscillating cycle; represents the viscous response of the test material (see Fig. 2).

3.1.5 *S* torque, n—for an oscillating shear rotorless cure meter*, the maximum value measured by a torque transducer during the oscillating cycle (see Fig. 2).

3.1.6 The relationship between S*, S', and S" for any oscillating cycle is:

$$S^* = \sqrt{(S')^2 + (S'')^2} \quad (1)$$

3.1.7 *phase angle δ, n—for an oscillating shear cure meter*, the angle of shift between the sinusoidal strain and the sinusoidal S* torque during the oscillating cycle (see Fig. 2).

3.1.8 *Tan δ, n—for an oscillating shear rotorless cure meter*, the tangent of the phase angle δ.

3.1.8.1 *Discussion*—The relationship between Tan δ, S', and S" is:

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.12 on Processability Tests.

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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.

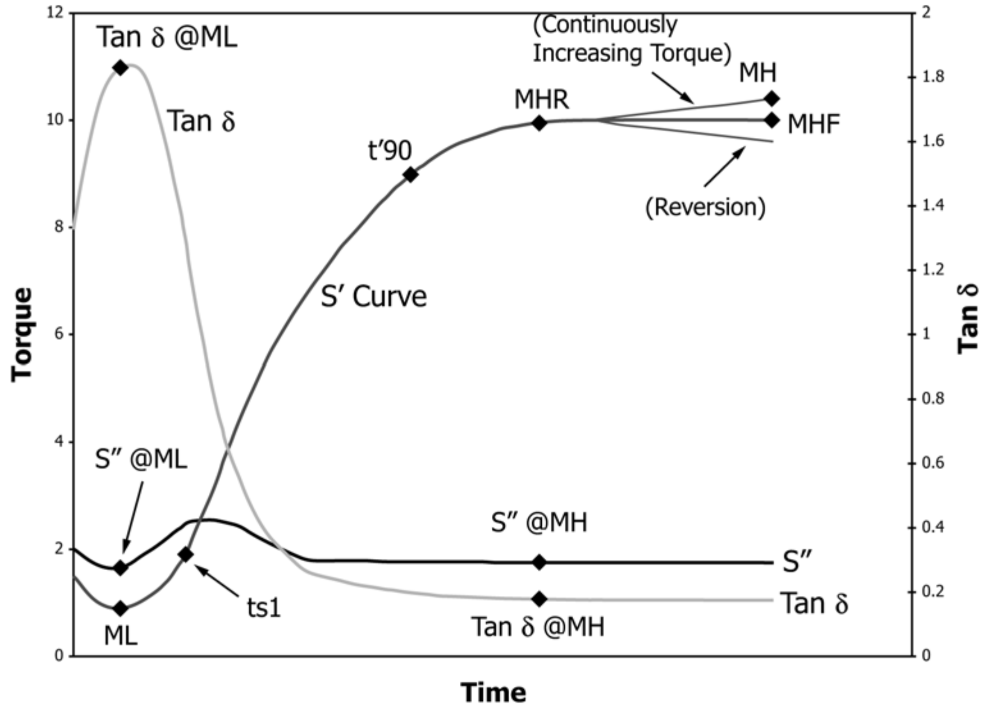

FIG. 1 Typical Vulcanization Curves

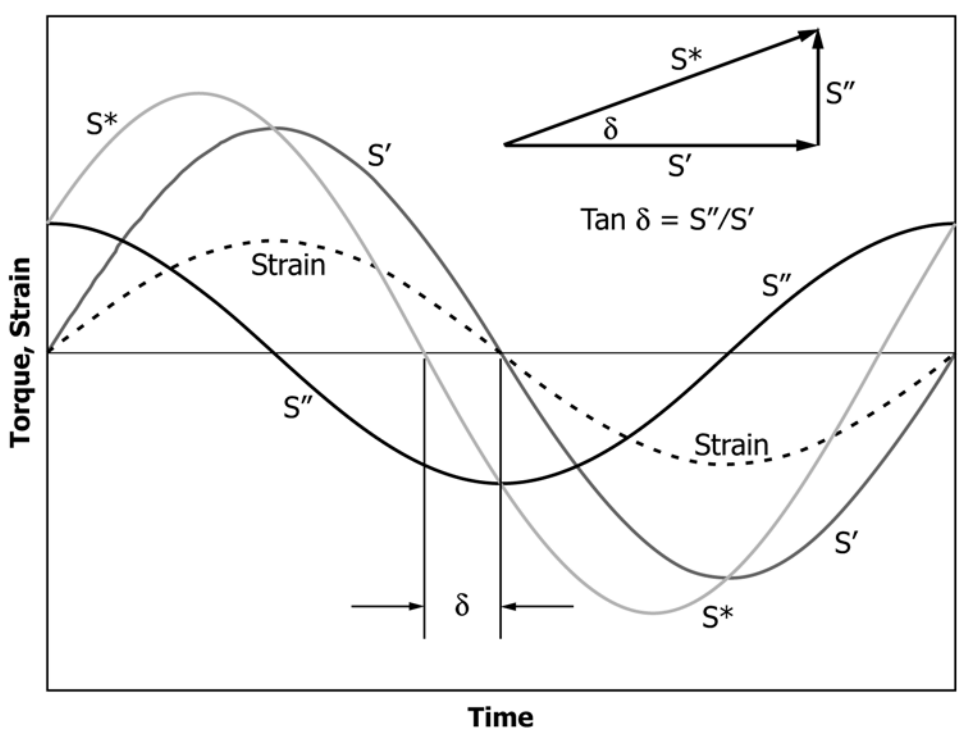

FIG. 2 Example of Torque Responses for an Oscillation Cycle

$$Tan\ \delta = \frac{S''}{S'} \quad (2)$$

3.1.9 The following measurements may be taken from the recorded S" and Tan δ curves as a function of time (see Fig. 1):

3.1.9.1 *S" @ML*—value of S" torque when the minimum S' torque is taken.

3.1.9.2 *Tan* δ *@ML*—value of Tan δ when the minimum S' torque is measured.

3.1.9.3 *S" @MH*—value of S" torque when the maximum S' torque is taken.

3.1.9.4 *Tan* δ *@MH*—value of Tan δ when the maximum S' torque is measured.

4. Summary of Test Method

4.1 A rubber test piece is contained in a die cavity which may be closed or almost closed and maintained at an elevated temperature. The cavity is formed by two dies, one of which is oscillated through a small rotary amplitude. This action produces a sinusoidal alternating torsional strain in the test piece and a sinusoidal shear torque which depends on the stiffness (shear modulus) of the rubber compound.

4.2 The stiffness of the rubber test piece increases as vulcanization proceeds. The test is completed when the recorded torque rises to either an equilibrium or maximum value, or when a predetermined time has elapsed (see Fig. 1). A curve representing the torque at peak strain in one direction of the oscillation cycle is continuously recorded as a function of time.

5. Significance and Use

5.1 This test method is used to determine the vulcanization characteristics of (vulcanizable) rubber compounds.

5.2 This test method may be used for quality control in rubber manufacturing processes, for research and development testing of raw-rubber compounded in an evaluation formulation, and for evaluating various raw materials used in preparing (vulcanizable) rubber compounds.

5.3 The test specimen in a rotorless cure meter approaches the test temperature in a shorter time and there is a better temperature distribution in the test specimen due to the elimination of the unheated rotor found in oscillating disk cure meters.

5.4 Several manufacturers produce rotorless cure meters with design differences that may result in different torque responses and cure times for each design. Correlations of test results between cure meters of different designs should be established for each compound tested, and for each set of test conditions.

6. Apparatus

6.1 Rotorless cure meters of two types can be used. In each case, an oscillation of small amplitude is applied to one die.

6.1.1 *Unsealed Torsion Strain Rotorless Cure Meter*—This type of cure meter measures the torque produced by an angular strain of constant amplitude in a cavity that is not completely closed (see Fig. 3(a)).

6.1.2 *Sealed Torsion Strain Rotorless Cure Meter*—This type of cure meter measures the torque produced by an angular strain of constant amplitude in a cavity that is completely closed and sealed (see Fig. 4(a)).

6.2 *Die Cavity*—The die cavity is formed by two dies. In the measuring position, the two dies are fixed a specified distance apart so that the cavity is almost closed (see Fig. 3(b)), or closed and sealed (see Fig. 4(a)).

6.2.1 The dimensions for typical torsional shear curemeters include biconical-shaped dies having a diameter of 40 ± 2 mm (1.57 ± 0.08 in.), and an angle of separation ranging from 7 to 18°, depending on the manufacturer's design. In the center of the dies, a separation equal to 0.5 mm (0.02 in.) plus the die gap should be maintained (see Fig. 3(b) or Fig. 4(b)). Manufacturer's guidelines should be followed to determine if the dies have been excessively worn and should be replaced.

6.2.2 *Die Gap*—The gap between the edges of the dies in the closed position shall be between 0.05 and 0.20 mm (0.002 to 0.008 in.), preferably 0.1 mm (0.004 in.) for unsealed cavities. For sealed cavities, no gap should exist at the edges of the dies.

6.2.3 *Die Closing Mechanism*—A pneumatic cylinder or other device shall close the dies and hold them closed during the test with a force of not less than 8.0 kN (1820 lbf).

6.3 *Die Oscillating System*—The die oscillating system imparts a torsional oscillating movement to one of the dies, in the plane of the cavity.

6.3.1 The amplitude of the oscillation should be ±0.1 to ±3.0°, preferably ±0.5° of arc for torsional shear cure meters.

6.3.2 The frequency of oscillation should be between 0.5 and 2 Hz, preferably 1.7 ± 0.1 Hz.

6.4 *Torque Measuring System*—A torque measuring system shall measure the resultant torque.

6.4.1 The torque measuring device shall be rigidly coupled to one of the dies and any deformation shall be negligibly small and shall generate a signal which is proportional to the torque. The total error resulting from zero point error, sensitivity error, linearity, and reproducibility errors shall not exceed 1 % of the measuring range selected.

NOTE 2—The elastic deformation of the oscillating and measuring system should not be more than 1 % of the oscillating amplitude; otherwise, the curemeter curves must be corrected.

6.4.2 The torque recorder device shall be used to record the signal from the torque measuring device. It shall record the S' torque at maximum oscillation as a function of time.

6.4.3 The torque recorder device shall be used to record the signal from the torque measuring device. It shall record the torque at maximum oscillation continuously as a function of time (see Fig. 1) and shall have a response time for full-scale deflection on the torque scale of 1 s or less. The torque shall be recorded with an accuracy of ±0.5 % of the range. Torque recording devices may include analog chart recorders, printers, plotters, or computers.

6.5 Torque calibration equipment is required to measure the angular strain amplitude and to calibrate the torque measuring device. Examples of calibration equipment are shown in Fig. 5 and Fig. 6. The amplitude of oscillation of the device shall be checked with no test specimen in it. A displacement transducer shall be used to measure the amplitude and torque measurements shall be checked against standard masses using a device as shown in Fig. 5. An alternate technique shall use a torque standard.

6.5.1 For calibrating torsion shear curemeters, either a displacement transducer and wire-mass calibration or a torque standard shall be used.

6.5.1.1 A displacement transducer for checking angular displacement shall be coupled by a knife-edge bearing in contact with a rod fixed to one of the dies (see Fig. 5). The force measuring system shall be checked by loading a wire, attached to the die or block by a pulley, with masses corresponding to the full-scale force being measured. The torque