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Standard Test Method for Raw Rubber or Unvulcanized Compounds—Determination of Tensile Green Strength[1]

This standard is issued under the fixed designation D 6746; 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 describes a method to evaluate a characteristic of raw rubber or unvulcanized rubber compounds, or both, that is designated as tensile green strength. This special strength property for uncured rubbers is an important processing performance attribute in rubber product manufacturing.

1.2 *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] 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. Referenced Documents

2.1 *ASTM Standards:* [2]

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D 1349 Practice for Rubber—Standard Temperatures for Testing

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

3. Terminology

3.1 *Definitions:*

3.1.1 *green strength*, *n*—a characteristic property of a raw or unvulcanized rubber compound that indicates the capability of retaining mechanical or structural integrity when the rubber is subjected to deformation of any type.

3.1.1.1 *Discussion*—The word "green" is a synonym for uncured or unvulcanized.

3.1.2 *tensile green strength*, *n*—green strength of a raw or unvulcanized rubber compound that is evaluated by the application of tensile stress or strain.

4. Summary of Test Method

4.1 The tensile stress-strain characteristics of a dumbbell or another recommended test piece of raw or unvulcanized compounded rubber are determined on a tensile testing machine capable of maintaining a constant rate of separation of the moving jaws.

4.2 In evaluating tensile green strength, several types of stress-strain behavior may be observed, depending on the rubber and the composition of the unvulcanized compound. (See Section 5.) Fig. 1 indicates three typical stress-strain curves. Type 1 has a yield point and exhibits elongational flow beyond the yield point, culminating in rupture at a higher stress and elongation compared to the initial yield stress. Type 2 also has a yield point, but beyond this point follows a decreasing stress behavior as the maximum rupture elongation is attained. Type 3 has a stress-strain curve that is similar to cured rubbers, with no yield or elongation beyond the point of rupture at maximum stress.

5. Significance and Use

5.1 The stress-strain properties of unvulcanized rubber (either a prepared mix or in the raw state) are important to certain processing operations in the rubber industry. These unvulcanized rubber properties are frequently referred to as "tensile green strength," denoting that the final vulcanization cycle has not yet been achieved.

5.2 Tensile green strength is determined primarily by the physical and chemical characteristics of polymers, such as molecular weight, tendency to crystallize, degree of branching, and so forth. It is also related to the compound formulation, particularly filler and plasticizer content, and the presence of peptizers. Tensile green strength can be a good indication of processing behavior. It is a particularly important characteristic for all processing operations in which elongation predominates.

5.3 Tensile green strength is dependent on the test piece preparation, rate of extension, and test temperature. Therefore, a single-point method cannot be expected to give correlation between tensile green strength and processing behavior over the whole range of processing conditions.

6. Interferences

6.1 For reliable test results, it is important that test specimens are of accurate dimensions and are free of air inclusions, blisters, and contaminants.

6.2 A defective cutting die or slippage of the test specimen in the clamps may cause incorrect results.

6.3 Exposure to excessive ultraviolet radiation or chemical vapors may also affect the results.

7. Apparatus

7.1 *Tensile Testing Machine*—The tensile testing machine shall be in accordance with the requirements of Test Methods D 412. It shall be capable of maintaining a constant rate of separation of the jaws at 100 ± 10 mm/min or, for special cases, at a value within the range of 20 to 1000 mm/min. It should have means to test the force on the test piece and the elongation by the distance between the gauge marks on the dumbbell. It should be capable of recording the force/elongation curve obtained during the test. If using an automatic extensometer, a non-contacting type of extensometer is preferred.

7.2 *Mold*—The mold shall meet the requirements of Practice D 3182.

8. Hazards

8.1 There are no hazards inherent to the techniques described. Caution should be exercised and the user should be aware of any possible pinch points.

[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*; Vol 09.01. volume information, refer to the standard's Document Summary page on the ASTM website.

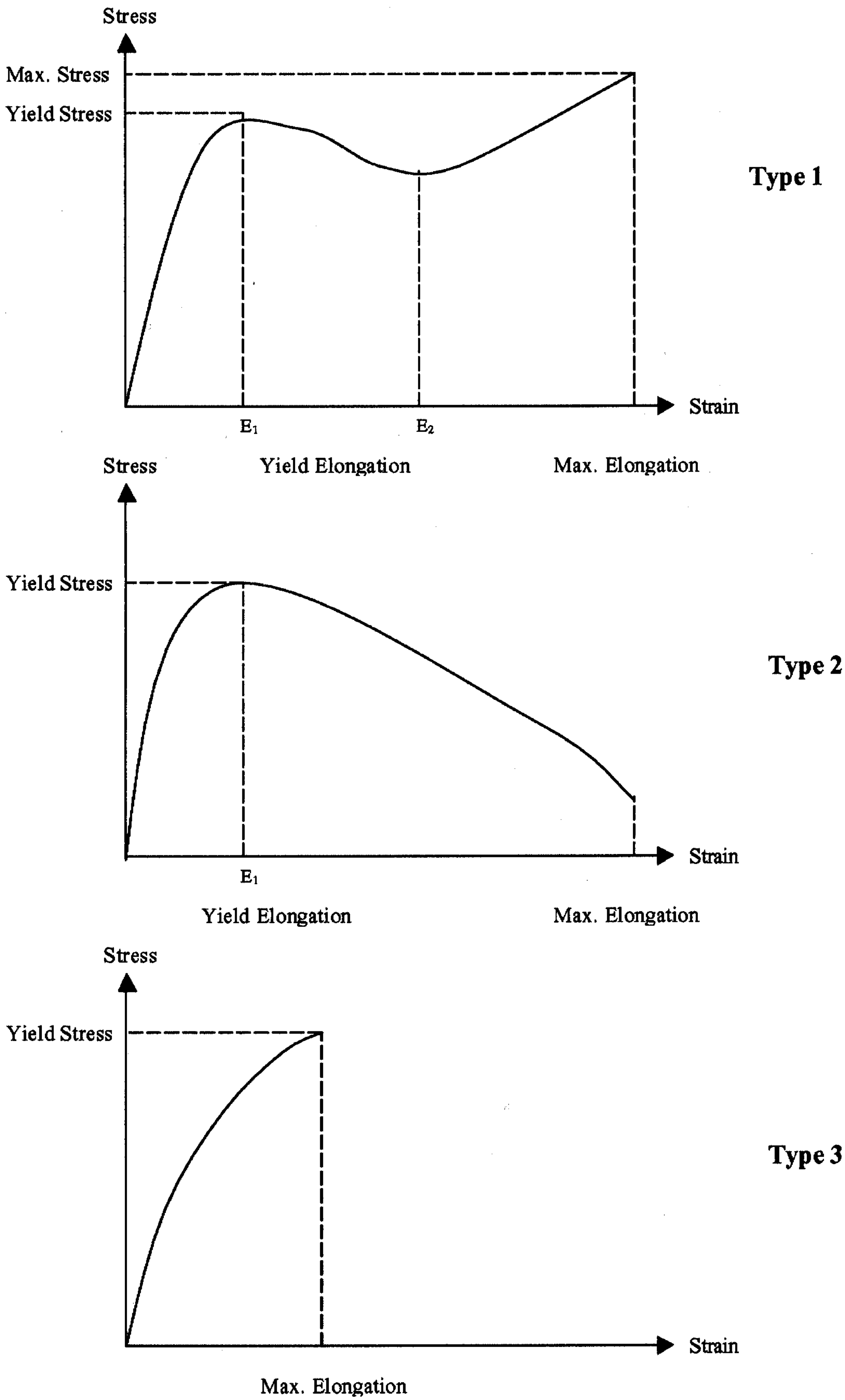

FIG. 1 Typical Tensile Stress-Strain Curves

8.2 Normal safety precautions, including the use of personal protective equipment and good laboratory practice, should be observed when using any equipment. This is especially true when performing tests at elevated temperatures.

9. Test Specimen

9.1 The preferred test specimen is the dumbbell Die C specimen in accordance with Test Methods D 412. Other types of test specimens may also be used. Since surface conditions can affect the results, there may be cases where a wider specimen would be preferred, such as Die A from Test Methods D 412.

9.2 Since different types of test specimens do not necessarily give the same values, comparison of results from different specimen types should be avoided.

10. Preparation

10.1 Raw rubber and unvulcanized compounds shall be sheeted out to approximately 2.5-mm thickness and placed in the mold with the grain direction oriented so as to have the grain direction parallel with the length of the dumbbells. It is also permissible to test against the grain, but this must be noted in the report section. (See Section 15.) Only fresh stock shall be used.

10.2 Polyester film (0.05-mm thickness) shall be placed between the mold walls and the rubber in order to promote mold release. The sample shall be compressed for 5 ± 1 min at 100 ± 2°C under a minimum of 2.5 MPa platen pressure, allowed to cool to laboratory temperature for 30 ± 2 min under adequate pressure to keep test specimen flat, then removed. The cooling shall be done out of the press using room temperature metal plates.

10.3 For some raw rubbers, longer times or higher temperatures may be necessary in order to obtain a smooth sheet free from porosity. For some compounds, a lower temperature may be necessary when there is a danger of scorch at the preferred temperature.

10.4 A sample may also be acquired during processing by sheeting off a mill, calender, or by extrusion and then cut with a die. Special care should be taken to ensure that the sample is free of trapped air.

10.5 Cut three test specimens from the sheet, using a suitable die. (See 9.1.)

11. Calibration and Standardization

11.1 The tensile testing machine shall be calibrated according to the procedure in Test Methods D 412.

12. Conditioning

12.1 The test specimens shall be conditioned at a temperature of 23 ± 2°C for at least 16 h and not more than 24 h before testing.

12.2 Some slow crystallizing polymers, such as polychloroprene, may need a longer conditioning period in order to accurately measure the maximum green strength.

13. Procedure

13.1 Place the dumbbell in the grips of the testing machine in accordance with the procedure in Test Methods D 412. Adjust the rate of displacement of the moving jaw to 100 ± 10 mm/min and start the tensile test. If the sample breaks at the grips, that result shall be discarded and a retest carried out.

13.1.1 The preferred rate of separation of the jaws is 100 ± 10 mm/min. In special cases, other rates may be used, but only tests carried out at the same rate can be compared. Rollover or self-tightening grips may help prevent slippage.

13.2 The test shall be carried out on three test specimens. If the standard deviation is greater than 20 % of the mean value, then five specimens should be tested.

13.3 Unless otherwise specified, the standard test temperature shall be 23 ± 2°C.

13.4 When it is necessary to test at other temperatures, use one of the standard temperatures listed in Practice D 1349. Specimens shall be conditioned at these test temperatures for 15 ± 2 min prior to testing.

14. Calculation or Interpretation of Results

14.1 Using the typical stress-strain curves given in Fig. 1, determine the yield stress or maximum stress in MPa. Other parameters may be determined, such as yield elongation (E_1) or the stress at a definite reference elongation corresponding to the deformation entailed by a subsequent processing operation. The median value is the preferred value to report.

14.2 Calculate the tensile stress at any specified elongation as follows:

$$T_{(xxx)} = F_{(xxx)}/A \tag{1}$$

where:

$T_{(xxx)}$ = tensile stress at (*xxx*) % elongation, MPa,
$F_{(xxx)}$ = force at specified elongation, MN, and
A = cross-sectional area of unstrained specimen, m^2.

14.3 Calculate the yield stress as follows:

$$Y_{(stress)} = F_{(y)}/A \tag{2}$$

where:

$Y_{(stress)}$ = yield stress, that stress level where the yield point occurs, MPa,
$F_{(y)}$ = magnitude of force at the yield point, MN, and
A = cross-sectional area of unstrained specimen, m^2.

14.4 Evaluate the yield strain as that strain or elongation magnitude, where the rate of change of stress with respect to strain goes through a zero value.

14.5 Calculate the elongation (at any degree of extension) as follows:

$$E = 100[L - L_{(o)}]/L_{(o)} \quad (3)$$

where:

E = the elongation in percent (of original bench mark distance),
L = observed distance between bench marks on the extended specimen, and
$L_{(o)}$ = original distance between bench marks (use same units for L and $L_{(o)}$).

14.6 The maximum elongation is evaluated when L is equal to the distance between benchmarks at the point of specimen rupture.

15. Report

15.1 Report the following information:

15.1.1 Full description of the sample and its origin,

15.1.2 Method of preparation of the test specimen, that is, time and temperature of molding, if not at standard conditions,

15.1.3 Type and dimensions of test specimen, if not a Die C dumbbell,

15.1.4 Grain direction, if not at the standard condition of testing with the grain,

15.1.5 Date of test,

15.1.6 Rate of extension, if not at standard conditions,

15.1.7 Temperature and humidity of test room, if not at standard conditions,

15.1.8 Time of conditioning,

15.1.9 Temperature of test if at other than 23 ± 2°C,

15.1.10 Number of test specimens tested if not three, and

15.1.11 Median of all results (yield stress, maximum stress, yield elongation, maximum elongation).

16. Precision and Bias

16.1 *Introduction*— The interlaboratory test program (ITP) for precision evaluation for both yield stress and yield strain was conducted in 2003 using the precision procedures and guidelines as described in the newly revised version of Test Method D4483 (scheduled for publication in 2004) which replaces the previous version of Test Method D4483. Refer to the new Test Method D4483 for additional background on precision terminology.

16.2 The precision results as determined by this ITP may not be applied to acceptance or rejection testing for any group of materials or products without documentation that the results of this precision evaluation actually apply to the products or materials tested.

16.3 Four rubbers (compounds) were used in the ITP; EPDM, NR, SBR and NBR. Four laboratories participated in the testing program. Mixed compounds of each rubber were sent to the participating laboratories and in each laboratory each compound was milled, test sheets molded and test specimens cut for testing. This procedure is essentially equivalent to a Type 2 precision where a complete set of operational steps are required to generate a test specimen. A full or complete Type 2 precision would require that each laboratory mix each compound and conduct the other remaining preparation steps. Thus the precision as evaluated is designated as a modified Type 2 precision. See Test Method D4483, sec 5.1.5 for more on Type 1 vs Type 2 precision.

16.4 The testing was conducted on a test day 1 vs test day 2 basis, one day apart. On each test day milling, molding and specimen cutting were conducted as well as stress-strain testing. As stipulated in the method, three specimens were tested on each day and the mean was used as a test result. All analysis was conducted using test results. Due to the small number of participating laboratories, Test Method D4483 precision analysis Option 2 was used to respond to outliers as detected by the h and k analysis operations. Option 2 uses outlier replacement rather than outlier deletion which reduces the degrees of freedom for precision evaluation. See Sections 7 and 8, 9 and 10 of Test Method D4483 for more details on outlier detection and on the AOT outlier replacement operations.

16.5 *Precision Results*—The precision results for both yield stress and yield strain are given in Table 1 on the basis of ascending order of the mean test result value. General statements for the use of the precision results are cited below. These are given in terms of both the absolute precision, **r** or **R** and also for relative precision (**r**) and (**R**).

16.5.1 *Repeatability*— The repeatability, or local domain precision, for each material or rubber for each of the test methods has been established by the values found in Table 1. Two individual test results (obtained by the proper use of this standard) that differ by more than the tabulated values for **r** , in measurement units and (**r**), in percent, shall be considered as suspect, for example, to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.

16.5.2 *Repeatability*— The reproducibility, or global domain precision, for each material or rubber for each of the test methods

TABLE 1 Precision for Yield Stress and Strain

Modified Type 2 Precision[A]

Part 1 — Yield Stress, MPa								Total
Within Labs					Between Labs			No Outliers
Material	Mean	Sr	r	(r)	SR	R	(R)	Replaced[B]
2 (NR)	0.25	0.007	0.019	7.5	0.04	0.113	45.4	1
3 (SBR)	.033	0.014	0.039	11.6	0.027	0.075	22.6	2
4 (NBR)	0.35	0.007	0.019	5.4	0.011	0.03	8.6	2
1 (EPDM)	0.78	0.035	0.098	12.5	0.065	0.183	23.4	1
		Overall Avg		9.25			25.0	
Part 2 – Yield Strain, %								Total
Within Labs					Between Labs			No Outliers
Material	Mean	Sr	r	(r)	SR	R	(R)	Replaced[B]
1 (EPDM)	35.6	3.06	8.57	24.1	16.4	45.9	129	none
4 (NBR)	40.4	8.12	22.7	56.3	10.9	30.6	75.8	1
2 (NR)	47.5	2.78	7.79	16.4	8.5	23.8	50.1	2
3 (SBR)	53.3	6.76	18.9	35.6	19.2	53.7	101	none
		Overall Avg		33.1			89.0	

Note: See text of Precision Clause for discussion of precision results of this table.

Sr = within-laboratory standard deviation
r = repeatability (in measurement units)
(r) = repeatability (in percent of mean level)
SR = between-laboratory standard deviation (for total between laboratory variation in measurement units)
R = reproducibility (in measurement units)
(R) = reproducibility (in percent of mean level)

[A] The precision was a modified Type 2; each mixed compound was sent to each lab; each labe milled and pressed the test sheet and cut the test specimens. See subsection 5.1.5 of Test Method D 4483 for definitions of Type 1 and 2 precision.
[B] Due to small number of participating labs, Option 2, outlier replacement, was used for outliers.

has been established by the values found in Table 1. Two individual test results obtained in different laboratories (by the proper use of this standard) that differ by more than the tabulated values for R , in measurement units and (R), in percent, shall be considered as suspect, for example, to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.

16.6 *Analysis Comments*—The average relative precision of yield stress, both (**r**) and (**R**) , is almost one fourth of that for yield strain; a 1 to 3.6 ratio. This difference may be due to inherent test precision differences or it may be due to the fact that a greater number of outliers were found for the original database for yield stress , a total of six for all four materials. Replacement of these outliers generates a revised database with less variation. Only three total outliers were found for the yield strain testing. The differences noted for yield stress vs yield strain precision may also be due to a combination of both factors.

16.7 *Bias*—Bias is the difference between a measured average test result and a reference or true value for the measurement in question. Reference values do not exist for this test method and therefore bias cannot be evaluated.

17. Keywords

17.1 green; raw rubber; rubber; tensile green strength; unvulcanized compounds