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Standard Practice for Determining Degradation End Point in Degradable Polyethylene and Polypropylene Using a Tensile Test[1]

This standard is issued under the fixed designation D3826; 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 practice covers the determination of a degradation-end point *(a brittle point)* for degradable polyethylene/polypropylene films and sheeting less than 1.0 mm (0.04 in.) thick. This practice is not intended for determination of the rate of degree of degradation of a polyethylene/polypropylene film or sheet, but rather, to assess when in the course of its degradation under some condition, a brittle point is reached. If one wishes to monitor tensile elongation during the degradation process (such as when the tensile elongation is significantly greater than 5 %), Test Method D882 is recommended. This practice should not be considered the only way of determining a degradation-end point.

1.2 Tensile properties of plastics 1.0 mm (0.04 in.) or greater in thickness shall be determined in accordance with Test Method D638.

1.3 Use a static weighing-constant rate of grip separation test. This procedure employs a constant rate of separation of the grips holding the sample and a static load cell.

NOTE 1—This procedure is based on the use of grip separation as a measure of extension; however, the desirability of using extension indicators accurate to ±1.0 % or better as specified in Test Method D638 is recognized, and a provision for the use of such instrumentation is incorporated in the procedure.

1.4 This procedure has been successful for determining the degradation end point of ethylene-carbon-monoxide copolymers and has screened successfully two other additive-type polyethylenes in a round robin test.

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

1.6 *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.*

1.7 There is no equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D374 Test Methods for Thickness of Solid Electrical Insulation (Withdrawn 2013)[3]

D618 Practice for Conditioning Plastics for Testing

D638 Test Method for Tensile Properties of Plastics

D882 Test Method for Tensile Properties of Thin Plastic Sheeting

D5208 Practice for Fluorescent Ultraviolet (UV) Exposure of Photodegradable Plastics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 Definitions of terms and symbols relating to tension testing of plastics appear in the Annex to Test Method D638.

3.1.2 *line grips, n*—in tensile testing machines, grips having faces designed to concentrate the entire gripping force along a single line perpendicular to the direction of testing stress.

3.1.3 *tear failure, n*—in tensile testing of films, a failure characterized by fracture initiating at one edge of the specimen and progressing across the specimen at a rate slow enough to produce an anomalous load-deformation curve.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *film, n*—for the purpose of this practice, a piece of material not exceeding 0.250 mm (0.01 in.) in thickness.

3.2.2 *brittle point, n*—in degradable polyethylene/polypropylene film, that point in the history of a material when 75 % of the specimens tested have a tensile elongation at break of 5 % or less.

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics and Biobased Products.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

4. Significance and Use

4.1 The tensile elongation property determined by this practice is of value for the characterization of degradable materials. The tensile elongation property may vary with specimen thickness, method of preparation, speed of testing, type of grips used, and manner of measuring test extension. Consequently, where precise comparative results are desired, these factors must be carefully controlled.

4.2 The tensile elongation property may be utilized to provide data for research and development and engineering design as well as quality control specifications. However, data from such tests cannot be considered significant for applications differing widely from the load-time scale of the test employed.

4.3 Materials that fail by tearing give anomalous data that cannot be compared with those from normal failure.

4.4 Before proceeding with this test method, reference should be made to the specifications of the material being tested. Any test specimen preparation, conditioning, dimensions, or testing parameters or a combination thereof, covered in the material specifications shall take precedence over those mentioned in this test method. If there are no material specifications, then the default conditions apply.

5. Apparatus

5.1 *Testing Machines:*

5.1.1 Use a testing machine of the constant rate-of-jaw-separation type. The machine shall be equipped with a weighing system that moves a maximum distance of 2 % of the specimen extension within the range being measured. Also, there should be a device for recording the tensile load and the amount of separation of the grips; both of these measuring systems shall be accurate to ±2 %. The rate of separation of the grips shall be uniform and capable of adjustment from approximately 1.3 to 500 mm/min (0.05 to 20 in./min) in increments necessary to produce the strain rates specified in 9.2.

5.2 *Grips*—Use a gripping system that minimizes both slippage and uneven stress distribution with the test specimen.

NOTE 2—Grips lined with thin rubber, crocus-cloth, or pressure-sensitive tape as well as file-faced or serrated grips have been successfully used for many materials. The choice of grip surface depends on the material tested and thickness. More recently, line grips padded on the round face with 1.0 mm (40 mil) blotting paper have been found superior. Air-actuated grips have been found advantageous, particularly in the case of materials that tend to *neck* into the grips, since pressure is maintained at all times. In cases where samples frequently fail at the edge of the grips, it may be advantageous to increase slightly the radius of curvature of the edges where the grips come in contact with the test area of the specimen.

5.3 *Thickness Gage*—A dead-weight dial micrometer as prescribed in Method C of Test Methods D374, reading to 0.0025 mm (0.0001 in.) or less.

5.4 *Width-Measuring Devices*—Suitable test scales or other width-measuring devices capable of reading to 0.25 mm (0.010 in.) or less.

5.5 *Specimen Cutter*—Fixtures incorporating razor blades, suitable paper cutters, or other devices capable of safely cutting the specimens to the proper width and producing straight, clean, parallel edges with no visible imperfections. A device consisting of two parallel knives mounted firmly against a precision-ground base shear-block (similar to a paper cutter) has proved satisfactory. The use of striking dies is not recommended because poor and inconsistent specimen edges may be produced. It is imperative that the cutting edges be kept sharp and free from visible scratches or nicks.

5.6 *Extension Indicators*—If employed, extension indicators shall conform to requirements specified in Test Method D638. In addition, such apparatus shall be so designed as to minimize stress on the specimen at the contact points of the specimen and the indicator.

NOTE 3—A high-response speed in the recording system is desirable, particularly when relatively high strain rates are employed for rigid materials. The speed of pen response for recorders is supplied by manufacturers of this equipment. Take care to conduct tests at conditions such that response time (ability of recorder to follow actual load) produces less than 2 % error.

6. Test Specimen

6.1 Cut test specimens prior to exposure. Take utmost care in cutting specimens to prevent nicks and tears that are likely to cause premature failures (see Note 4). The edges shall be parallel to within 5 % of the width over the length of the specimen between the grips.

NOTE 4—A microscopic examination of the specimen may be used to detect flaws due to sample or specimen preparation.

6.2 Prepare the test specimen with uniform width and length. Examples of typical lengths and widths are:

Width, mm	Length, mm
13 (0.5 in.)	152 (6 in.)
25 (1.0 in.)	102 (4 in.)

6.2.1 The test specimen thickness is contingent upon the thickness of the end-use application. The test specimen thickness should be the same as that for the specific end-use application.

6.3 Wherever possible, select test specimen so that thickness is uniform to within 10 % of the thickness over the length of the specimen between the grips in the case of materials 0.25 mm/in. (0.010 in.) or less in thickness, and to within 5 % in the case of materials greater than 0.25 mm (0.010 in.) in thickness but less than 1.00 mm (0.040 in.) in thickness.

NOTE 5—In cases where thickness variations are in excess of those recommended in 6.3, results may not be characteristic of the material under test.

6.4 Whenever possible, cut test specimens and test in the machine direction only.

7. Number of Test Specimens

7.1 Take sufficient test specimens in the machine direction from each sample to ensure four acceptable measurements (see 4.3, 7.2). Samples that break during degradation exposure can be deemed to have absolute tensile elongations of less than 5 % (see 10.2).

NOTE 6—When exposing samples (especially outdoors) they will sometimes break up, and tensile tests can not be properly performed. For this reason, broken samples are deemed to have reached 5 % absolute elongation for the purpose of determining the brittle point.

7.2 Discard specimens that fail at some obvious flaw, or that fail outside the gage length, unless such flaws or conditions constitute a variable whose effect is being studied. However, jaw breaks (failures at the grip contact point) are acceptable if it has been shown that results from such tests are in essential agreement with values obtained from breaks occurring within the gage length.

NOTE 7—In the case of some materials, examination of specimens prior to and following testing under crossed optical polarizers (polarizing films) provides a useful means of detecting flaws which may be responsible for premature failure.

8. Conditioning

8.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D618.

8.2 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity.

9. Procedure

9.1 Set the initial grip separation that is appropriate for the dimensions of the test specimen.

9.2 Set the rate of grip separation to provide an initial strain rate of 0.1 mm/mm · min.

9.3 Measure the thickness to an accuracy of 0.0025 mm (0.0001 in.) or better or films less than 0.25 mm (0.010 in.) in thickness and to an accuracy of 1 % or better for specimens greater than 0.25 mm (0.010 in.) but less than 1.0 mm (0.040 in.) in thickness.

9.4 Place the test specimen in the grips of the testing machine, taking care to align the long axis of the specimen with an imaginary line joining the points of attachment of the grips to the machine. Tighten the grips evenly and firmly to the degree necessary to minimize slippage of the specimen during the test.

10. Calculation

10.1 Calculate the *percentage elongation at break* by dividing the elongation at the moment of rupture of the specimen by the initial gage length of the specimen and multiplying by 100. When the gage marks or extensometers are used to define a specific test section, use only this length. Report the result in percent to two significant figures.

10.2 The material is considered degraded to the brittle point when 75 % or more of the test specimens have a tensile elongation of 5 % or less.

11. Report

11.1 Report the following information:

11.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, previous history, and orientation of samples with respect to anisotropy (if any),

11.1.2 Method of preparing test specimens,

11.1.3 Thickness, width and length of test specimens,

11.1.4 Complete identification of exposure practice or procedure used to degrade test specimens,

11.1.5 Grip separation (initial),

11.1.6 Crosshead speed (rate of grip separation),

11.1.7 Gage length (if different from gage separation),

11.1.8 Type of grips used, including facing (if any),

11.1.9 Conditioning procedure (test conditions, temperature, and relative humidity, if non-standard),

11.1.10 Anomalous behavior such as tear failure and failure at a grip,

11.1.11 Percentage elongation at break of each sample and the number of samples that broke during exposure,

11.1.12 Whether or not the specimens tested reached the brittle point and the exposure time required to do so.

11.1.13 Indicate whether an extensometer is employed.

12. Precision and Bias

12.1 A round robin conducted and analyzed according to Practice E691 for three degradable polyolefin polymers produced the repeatability and reproducibility results for tensile elongation shown in Table 1. Each laboratory tested specimens that were unexposed and specimens that had been exposed for 240 h according to Practice D5208 (Cycle A).[4]

13. Keywords

13.1 brittle point; degradable plastics; plastics; polyethylene/polypropylene films/sheeting; tensile elongation

[4] Supporting data available at ASTM Headquarters. Request RR: RR:D20-1233.

TABLE 1 Tensile Elongation at Break Determined According to Practice D3826

Material Tested	Average	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
ECO,[A] unexposed	282.4	41.6	138.8	166.8	388.7
ECO, exposed 240 h[B]	2.5	1.8	2.1	5.1	6.0
clear LLDPE, [C] unexposed	371.4	40.1	231.3	112.3	647.7
clear LLDPE, exposed 240 h[B]	6.5	5.1	5.8	14.2	16.3
white LLDPE, unexposed	235.4	34.3	146.5	96.0	410.2
white LLDPE, exposed 240 h[B]	1.4	0.5	1.0	1.4	2.6

[A] ECO material is an ethylene/CO polymer that is known to degrade under UV exposure.
[B] LLDPE and White LLDPE are blown film linear low density polyethylene with an additive to promote degradation under sunlight. Clear LLDPE is natural color, and white LLDPE had some TiO_2 white pigment.