Designation: C 1144 – 89 (Reapproved 1997)

Standard Test Method for Splitting Tensile Strength for Brittle Nuclear Waste Forms[1]

This standard is issued under the fixed designation C 1144; 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 is used to measure the static splitting tensile strength of cylindrical specimens of brittle nuclear waste forms. It provides splitting tensile-strength data that can be used to compare the strength of waste forms when tests are done on one size of specimen.

1.2 The test method is applicable to glass, ceramic, and concrete waste forms that are sufficiently homogeneous (Note 1) but not to coated-particle, metal-matrix, bituminous, or plastic waste forms, or concretes with large-scale heterogeneities. Cementitious waste forms with heterogeneities >1 to 2 mm and <5 mm can be tested using this procedure provided the specimen size is increased from the reference size of 12.7 mm diameter by 6 mm length, to 51 mm diameter by 100 mm length, as recommended in Test Method C 496 and Practice C 192.

NOTE 1—Generally, the specimen structural or microstructural heterogeneities must be less than about one-tenth the diameter of the specimen.

1.3 This test method can be used as a quality control check on brittle waste forms and may be useful for optimizing waste form processing. Meaningful comparison of waste forms, however, requires data obtained on specimens of one size.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard may involve hazardous materials, operations, and equipment. 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.* For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

C 39 Test Method for Compressive Strength of Cylindrical Concrete Specimens[2]

C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory[2]

C 496 Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens[2]

C 773 Test Method for Compressive (Crushing) Strength of Fired Whiteware Materials[3]

D 2938 Test Method for Unconfined Compressive Strength of Intact Rock Core Specimens[4]

E 4 Practices for Force Verification of Testing Machines[5]

2.2 *Society of Manufacturing Engineers:*

Geometrical Tolerance Interpretations, SME Tool and Manufacturing Engineers Handbook[6]

3. Summary of Test Method

3.1 A right-circular cylinder of the waste solid is loaded diametrally between two hardened, parallel bearing blocks positioned between the specimen and the two test machine platens, one of which is moving at a constant speed relative to the other (Fig. 1).

3.2 As the load increases, the resultant stress eventually reaches the fracture strength of the material, and the specimen splits along the vertical diameter, usually with some subsidiary fracture at other locations. The splitting tensile strength, T (MPa), is calculated from the measured fracture load as follows:

$$T = 2P/\pi LD \quad (1)$$

where:
P = applied force, or fracture load, at initiation of fracture, N,
L = specimen length, mm, and
D = specimen diameter, mm.

3.3 The splitting tensile-strength test uses a compressive loading to effect a tensile stress. The stress state in the specimen during the test is well documented by both theoretical and experimental stress analysis. The stress state is intended to be biaxial with a uniform tensile stress normal to the loading axis across the anticipated fracture plane (the vertical diameter between loading points). The loading pads tend to prevent compressive-stress failure near the loading points. In a valid test, failure is initiated near the axis of the cylinder and

[1] This test method is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.07 on Waste Materials.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 15.02.

[4] *Annual Book of ASTM Standards*, Vol 04.08.

[5] *Annual Book of ASTM Standards*, Vol 03.01.

[6] Available from Society of Manufacturing Engineers, P.O. Box 930, One SME Dr., Dearborn, MI 48121.

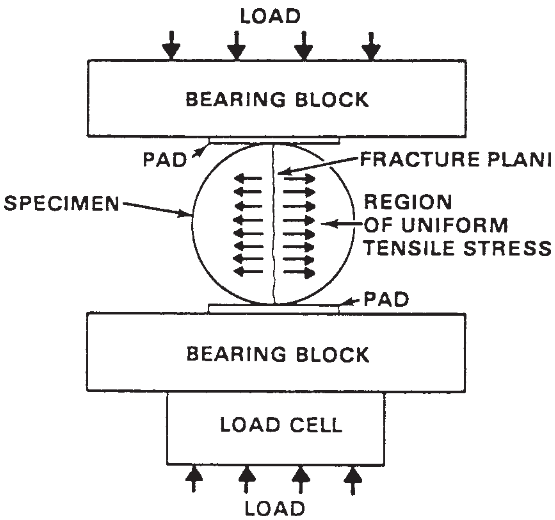
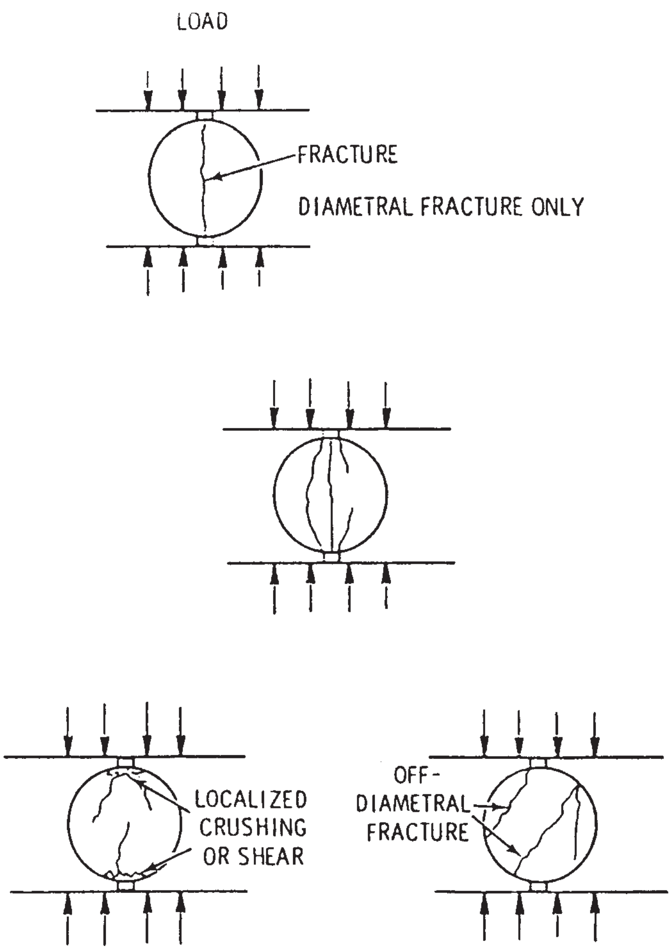


FIG. 1 Diametral Test Specimen and Apparatus

propagates on the plane defined by the lines of contact of the bearing blocks with the specimen (see Fig. 2(*a*) and Section 5).



(*a*) Normal Tensile Failure (Valid Test)
(*b*) Triple-Cleft Failure (Valid Test)
(*c*) Compression and Shear Failures (Invalid Test)

FIG. 2 Failure Modes

4. Significance and Use

4.1 The splitting tensile-strength test can be used only on brittle waste materials such as ceramics, glass, concrete, or other materials that also have tensile fracture strengths that are less than one third of the compression strengths.

4.2 The test cannot be used for metal-matrix, bituminous, plastic, or coated-particle waste forms.

4.3 The strength values derived from this test cannot be applied to compressive-stress impact failure. The results apply only to tensile-stress failure. A separate compression-strength test, in which a cylindrical specimen is loaded on the flat surfaces, is required to determine compression strength along the lines of Test Methods C 39, D 2938, and C 773. Failures caused by impact must be determined in a separate test.

4.4 This test method is applicable only to brittle solids because these are the only materials that fail under a definable stress state for the test specimen geometry and loading. For instance, extensive local shearing at or near the loading points that will also occur for plastically deformable solids, such as ductile metals or viscous polymers, will change the stress distribution sufficiently to invalidate the elastic-stress calculation used to obtain the tensile stress across the vertical fracture plane. Ductile materials will not, in many cases, fracture in the test.

4.5 The effect of specimen size on the measured strength of brittle materials is not determined by this test method. In some materials, such as concretes, heterogeneities may be so large that tests on larger specimens are more representative. Testing along the lines of Test Method C 496 may then be appropriate to measure splitting tensile strength.

4.6 This test method does not determine the effects of time and environment on strength, nor does it address failure under long-duration static loading.

4.7 This test method can be used as a quality-control check and for optimizing waste form processing.

5. Interferences

5.1 Visually inspect the specimen after fracture. Disqualification is based on the occurrence of compression and shear failure or failure at an observable surface flaw. See 5.3, 5.4, and 5.5 for guidance in identification of the failure mode. Report identification of the failure mode in terms relatable to these sections.

5.2 There are two fracture modes that indicate a valid test, normal tensile failure and triple-cleft failure, both of which can be followed by additional severe fragmentation of the center vertical region of the specimen. A third type of failure, or fracture, called compression and shear failure, invalidates the test results. Because of the possible varied fractures and because there is no satisfactory way to predict which will occur, the specimen must be examined after the test to qualify the results.

5.3 *Normal Tensile Failure*—In normal tensile failure, the specimen splits along the loaded diameter (see Fig. 2(*a*)). This is the ideal failure and can be used to compute splitting tensile strength. The fracture may not completely extend from one bearing block to the other initially. The load to initiate the fracture is used to calculate strength.

5.4 *Triple-Cleft Failure*—Triple-cleft failure is a variation on the normal tensile failure, and the specimen splits into four approximately equal-sized pieces, two on each side of the loaded diameter (see Fig. 2(*b*)). Tests exhibiting this failure also yield valid values of splitting tensile strength. Additional fragmentation can occur when the fracture is initiated on the diametral plane, as in glasses where the stresses on the central unsupported vertical region (after initial splitting) cause fragmentation of that region.

5.5 *Compression and Shear Failure*— In compression and shear failures, the specimen is crushed near the bearing blocks without fracturing through the diameter, or the specimen may fail near the loading pads due to a local crushing or by fracturing at any angle away from the loaded diameter (see Fig. 2(*c*)). In some cases, the specimen may change shape before fracture or may not fracture at all. Tests with these types of failure or deformation cannot be used to compute splitting tensile strengths, and stresses calculated from such tests are not reportable as tensile strengths. Choice of loading pad may avoid these types of failure in some cases.

6. Apparatus

6.1 *Test Temperature*—Conduct the test at room temperature and report the test temperature.

6.2 *Testing Machine*—Use a constant crosshead-speed machine at a speed of 8×10^{-4} mm/s$\pm$ 50 % (Note 2). A fixed loading rate machine is not acceptable. The machine can be either screw driven or otherwise controlled to give a fixed speed. The stiffness of the various members of the loading system shall be sufficiently high, such that the total deflection per unit force is less than 10^{-8} m/*N*, not including the specimen.

NOTE 2—Deviations in crosshead speeds of this magnitude will not affect test results.

6.3 *Bearing Blocks*—Bearing blocks with Rockwell hardness >60 HRC are required. Any permanent indentation of the bearing block invalidates the test. Suitable materials are tool steels hardened from 60 to 65 HRC by conventional heat treatments and ground to obtain a smooth loading surface. The surfaces of the bearing blocks in contact with the pads shall be flat to within ±0.03 mm, parallel within ±0.03 mm/mm (1.7°) measured on each of two perpendicular directions, and perpendicular to the loading axis within ±0.03 mm/mm (1.7°).

6.4 *Pad Materials*—The choice of pad material depends on the strength and elastic modulus of the material tested. A suitable pad material is one that prevents contact between the test specimen and the bearing blocks but is soft enough to distribute the load over a small area. If the specimen and bearing block contact during the test (determined by visual inspection of the pad after testing), the test result is invalid. In general, balsa wood is a suitable pad material for testing glass and other materials with splitting tensile strengths less than approximately 100 MPa. The grain of the wood shall be aligned perpendicular to the line of contact between specimen and bearing block with the grain parallel to the bearing block. The thickness of the balsa wood shall be 1.6 ± 0.2 mm (Note 3). Fully annealed OFHC copper foil 0.13 by 0.01-mm thick (Note 3) is suitable for higher strength waste forms.

NOTE 3—Deviations in pad material sizes of this magnitude will not affect test results.

6.5 *Load-Measurement System*—Use a strip-chart or x-y recorder to obtain a record of the loading force versus time. The recorder must be capable of responding to sudden changes in load (response time <1 s full scale). Use the strip-chart or x-y recorder to record the calibration loads during a check of the load-measurement system with dead weights or an electrical method prior to testing. Use a load cell that has been verified according to Practice E 4. The chart speed shall be appropriate for displaying the elastic or straight-line portion of the loading at an angle no greater than 80° to the time axis. It is imperative to have a continuous recording of load to ensure that the fracture is not missed.

6.6 *Number of Tests*—Initially, five valid test results should be obtained. Calculate the percent relative standard deviation of the five measured tensile strengths as follows:

$$\text{RSD (\%)} = \frac{s}{\bar{T}} \cdot 100 \quad (2)$$

where:

T_i = tensile strength for the ith test, and

$\bar{T}$ = the sample mean tensile strength $= \frac{1}{5}\sum_{i=1}^{5} T_i$, and

s = the sample standard deviation $= \frac{1}{5}\sum_{i=1}^{5} (T_i - \bar{T})^2$ $^{1/2}$.

6.6.1 If the percent relative standard deviation is less than 20.1 %, no additional tests are required. If the RSD (%) exceeds 20.1 %, use Table 1 to determine the number of additional tests required. If the RSD (%) is greater than 27.1 %, the material has variations in strength that are unusually large. Report the results of the ten tests in this case.

6.6.2 The criterion for the sample sizes given in Table 1 is based on the desire that the half-width of the 95 % confidence interval for the average tensile strength be no greater than 25 % of that value. If the tensile strength measurements come from a normal distribution, this should be approximately true. Naturally, the actual confidence-interval statements made will be based on the observed sample values, not the desired result.

7. Hazards

7.1 Specimens of brittle materials under stress can fracture and produce flying fragments. In addition to other precautions, take precautions against injury by placing a shield around the specimen to stop such fragments.

TABLE 1 Minimum Number of Required Tests (Based on the Sample % Relative Standard Deviation from Five Tests)

Sample Relative Standard Deviation, RSD (%)	Number of tests required, (*n*)	Number of additional tests
≤20.1	5	0
20.2 to 22.1	6	1
22.2 to 23.9	7	2
24.0 to 25.5	8	3
25.6 to 27.0	9	4
≥27.1	10	5

8. Test Specimens

8.1 Use a specimen that is 12.7 ± 0.3 mm in diameter and 6.4 ± 0.15 mm in length, round within a tolerance of 0.025 mm, straight within a tolerance of 0.050 mm, and with the ends square to the cylinder axis within a tolerance of 0.075 mm; the ends of the specimen are to be parallel within 0.150 mm (Note 4). Use geometrical tolerance interpretations given by the SME Tool and Manufacturing Engineers Handbook. When larger specimens are used, such as in Test Method C 496, the tolerances given above can be increased by the ratio of the larger specimen diameter to 12.7 mm. For cementitious waste forms that have heterogeneities less than about 5 mm, use a specimen that is 50.0 ± 1.2 mm in diameter and 100.0 ± 2.5 mm in length with corresponding larger tolerances on straightness, squareness, and parallelism.

NOTE 4—Deviations in pad material sizes of this magnitude will not affect test results significantly.

8.2 Core drill specimens to obtain 12.7-mm diameter rods from large pieces of the waste form. To obtain an acceptable surface finish on the cylindrical surface, use centerless grinding with 200-grit SiC or a tool-post grinder on a lathe using either a 60-grit Al_2O_3 wheel (32 Norton VBE or equivalent) or 100-grit diamond wheel. Then produce the flat surfaces with a diamond cut-off saw with 200-grit particles or with a surface grinder with 100 or 220-grit diamond wheel of 75 diamond concentration. Use water, when appropriate, as a coolant in the cutting or grinding operations. When water is inappropriate, use dry cutting or grinding. Reject any specimens that have chips or surface flaws with a largest dimension >1 mm. Uncharacteristic minor chips or surface flaws (<1 mm) at the specimen edges on the curved surfaces can be tolerated only if these are positioned >6 mm away from the loading points and only if fracture does not initiate at these locations. If fracture occurs at these locations, discard the results and repeat the test. Flaws as large as 1 mm are permitted on the flat surfaces only if they are at least 4 mm from the vertical diameter. Only flaws smaller than 0.1 mm are permitted within 4 mm of the vertical diameter.

9. Calibration and Standardization

9.1 To ensure accuracy of stress calculations, the calibration of the load-measurement system and specimen dimensions must be traceable to NIST standards. Enter and maintain records of calibrations and dates of calibrations in laboratory notebooks. A summary of applicable references is given in Table 2.

10. Procedure

10.1 *Quality Assurance Requirements*— This procedure must conform to all applicable quality assurance requirements of the laboratory performing the test.

10.2 *Testing an Individual Specimen*:

10.2.1 Record the room temperature.

10.2.2 Record the specimen thickness and diameter to the nearest 0.01 mm and verify that each specimen is within specified dimensional tolerances (Section 8). Examine specimen surface for flaws or chips> 0.1 mm.

TABLE 2 Required Calibration

Measurement	Instrument and Sensitivity	Calibration Reference
Specimen dimensions	caliper micrometer, 0.01 mm or better	NIST traceable gage blocks—every six months
Specimen straightness, roundness, and parallelism	caliper micrometer, 0.01 mm or better LVDT gage, 0.01 mm or better	NIST traceable gage blocks and surface plate± 0.001 mm or better—every six months
Bearing-block flatness and parallelism	LVDT gage, 0.01 mm or better	NIST traceable gage blocks and surface plate± 0.001 mm or better—every six months
Load measurement system	load cell, 1 % of testing range or better	verified by Practice E 4 procedure—every 2 years or immediately following any repairs or load excursions above the rated capacity
	load-recording system, 1 % of full scale or better	Load indication checked with NIST referenced standard weights or NIST referenced, shunt calibration resistor on load cell before and after each 6-h test period

10.2.3 Verify that bearing blocks are properly aligned (see 6.3).

10.2.4 Check load-measurement system with standard weights or an electrical method before testing using Practice E 4. Choose the full-scale load indication of the recorder so that the fracture load is indicated at a position that is >20 % of full scale.

10.2.5 Position new pads for each test between the specimen and bearing blocks. Position the specimen on the lower pad with the flat surfaces parallel to the load axis. If flaws or chips exist, position the specimens accordingly (see Section 8). Apply a light load (<50 *N*) by manual control of the crosshead; the load shall not exceed 10 % of the final fracture load during the set-up of the specimen under manual control. The first test on a material for which the fracture load is unknown can be loaded only enough to hold the specimen in position. This will lengthen the duration of the test because of the time required to compress the pad but may avoid too high an initial loading on a material with unknown strength. Subsequent tests can be preloaded to values near 10 % of the fracture load. If the preload exceeds 10 % of the fracture load, the test result cannot be used.

10.2.6 Start the recording strip-chart recorder and then the crosshead so that the loading curve is recorded.

10.2.7 Examine the test record to determine the fracture load and record the value. Take as the fracture load the maximum load before the first drop in load or the maximum load if no prior drop in load occurred.

10.2.8 Stop or reverse the crosshead upon fracture.

10.2.9 Examine the specimen to document that fracture occurred as required (see Section 5). Note the condition of the loading pads and whether the pads prevented contact of specimen and bearing blocks. Reject the test result if contact occurred or if failure initiated at an observable chip or surface flaw.

10.2.10 Keep the strip chart as a record of testing.

10.2.11 Check the load-recording system with standard weights at the end of testing for each 6-h test period (see Table 2).

10.2.12 Photograph each fractured specimen at 3× magnification to show detail and fracture mode.

10.2.13 Calculate the splitting tensile strength according to Eq 1 and 3.2. Use the fracture load as determined in 10.2.7.

11. Report

11.1 Report the following information:

11.1.1 Material tested and its identification number,

11.1.2 Test method,

11.1.3 Name of investigator,

11.1.4 Affiliation of investigator, and

11.1.5 Date report was completed.

11.2 *Material Preparation and Composition*:

11.2.1 *Material Preparation*—Report significant processing details, temperature-time cycles, curing times, and age and storage conditions of waste form.

11.2.2 *Material Composition*—List the as-analyzed chemical composition.

11.3 *Specimen Preparation, Test Conditions, and Calibration*:

11.3.1 *Specimen Preparation*—Describe abrasives used, cutting techniques, and any thermal or cleaning treatments given. Report any surface flaws >0.1 mm.

11.3.2 *Test Conditions*—Tabulate the dates of tests. Identify test material, test temperature, pad material, and the rationale for its selection, test machine, and crosshead speed (mm/s).

11.3.3 *Calibration*—Document the date and procedure for calibrations of the load cell, load-measurement system, and caliper micrometer. Describe procedures used to determine that specimens and bearing blocks conformed to specified tolerances, flatness, and parallelisms.

11.4 *Fracture Strength Data*:

11.4.1 *Test Specimen Results*—Tabulate thickness and diameter (mm) for each specimen, fracture load (*N*) for each test, and calculate fracture strength (MPa) for each test. Report average strength, standard deviation, and relative standard deviation for results of five or more tests.

11.5 *Description of Fracture*:

11.5.1 Describe the fracture for each specimen. If the fracture is similar in all specimens, one photograph of 3× ± 10 % magnification is adequate; otherwise, include a photograph of each fracture type.

11.5.2 Report whether the pads between the specimen and bearing blocks prevented contact of specimen and bearing block.

12. Precision and Bias

12.1 No between-laboratory precision data are available. Based on available within-laboratory results, the relative standard deviation of the sample mean of the splitting tensile strength is expected to be less than the maximum allowable (Table 1) for most waste forms.

12.2 No data on bias are available; no standards are known to exist for splitting tensile strength.