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# Standard Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Three-Point Loading at Room Temperature[1]

This standard is issued under the fixed designation D7972; 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 determination of the flexural strength of manufactured carbon and graphite articles using a square, rectangular or cylindrical beam in three-point loading at room temperature.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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]

C78 Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)

C559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles

C1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature

C1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics

C1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics

D7775 Guide for Measurements on Small Graphite Specimens

E4 Practices for Force Verification of Testing Machines

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

## 3. Terminology

3.1 *Definitions:*

3.1.1 *flexural strength*—a measure of the ultimate load carrying capacity of a specified beam in bending.

3.1.2 *grain—in manufactured (synthetic) carbon and graphite*, particle of filler material (usually coke or graphite) in the starting mix formulation. Also referred to as granular material, filler particle, or aggregate material.

## 4. Significance and Use

4.1 This test method provides a framework for material development, quality control, characterization, and design data generation purposes. The user needs to assess the applicability of the method on the specific material and for the intended use, as shown by the interlaboratory study.

4.2 This test method determines the maximum loading on a graphite specimen with simple beam geometry in three–point bending, and it provides a means for the calculation of flexural strength at ambient temperature and environmental conditions.

4.3 The flexure stress is computed based on simple beam theory with assumptions that the material is isotropic and homogeneous, the moduli of elasticity in tension and compression are identical, and the material is linearly elastic. For materials with large grains, the minimum specimen dimension should be significantly larger than the maximum grain size (see Guide D7775).

4.4 Flexural strength of a group of test specimens is influenced by several parameters associated with the test procedure. Such factors include the loading rate, test environment, specimen size, specimen preparation, and test fixtures. Specimen sizes and fixtures should be chosen to reduce errors due to material variability or testing parameters, such as friction and non-parallelism of specimen surfaces.

4.5 The flexural strength of a manufactured graphite or carbon material is dependent on both its inherent resistance to fracture and the size and severity of flaws. Variations in these cause a natural scatter in test results for a sample of test specimens. Fractographic analysis of fracture surfaces, although beyond the scope of this standard, is highly recommended for all purposes, especially if the data will be used for design as discussed in Practices C1239 and C1322.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite 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.

4.6 The three-point test configuration exposes only a very small portion of the specimen to the maximum stress. Therefore, three-point flexural strengths are likely to be much greater than four-point flexural strengths. Three-point flexure has some advantages. It uses simpler test fixtures, allowing small specimen testing and fracture toughness measurements. However, four-point flexure is preferred and recommended for most characterization purposes.

## 5. Apparatus

5.1 *Loading*—Specimens may be loaded in any suitable testing machine provided that uniform rates of loading can be maintained. The testing machine shall be equipped with a means for retaining read-out of the maximum force applied to the specimen. The accuracy of the testing machine shall be in accordance with Practice E4.

5.2 *Fixture*—The three-point loading fixture shall consist of bearing blocks or cylindrical bearings spaced in a three-point loading configuration (see Test Method C1161). A hardened steel bearing block or its equivalent is necessary to prevent distortion of the loading member.

5.2.1 The fixture shall ensure that forces applied to the beam are normal only and without eccentricity through the use of spherical bearing blocks (see Test Method C78) or articulating roller bearing assemblies (see 5.3 and Test Method C1161).

5.2.2 The cylindrical bearing length shall be such that the test specimen width is fully supported, and the cylindrical bearing diameter shall be 0.75 to 1.5 times the specimen thickness/diameter.

5.2.3 The lower support bearings shall be free to rotate in order to relieve frictional constraints. The middle load bearing of the three-point fixture need not rotate. The three bearings shall be parallel over their length. The load application bearing (upper bearing) shall be centered with respect to the two lower support bearings within ±0.10 mm.

5.3 The directions of loads and reactions may be maintained parallel by judicious use of linkages, rocker bearings, and flexure plates. Eccentricity of loading can be avoided by the use of spherical bearing blocks or articulating roller bearings.

5.3.1 *Semi-articulated Three-point Fixture*—Specimens prepared in accordance with the parallelism requirements of 6.1 may be tested in a semi-articulated fixture. The middle bearing shall be fixed and not free to roll. The two outer bearings shall be parallel to each other over their length. The two outer bearings shall articulate together as a pair to match the specimen surface, or the middle bearing shall articulate to match the specimen surface. All three bearings shall rest uniformly and evenly across the specimen surface. The fixture shall be designed to apply equal load to the two outer bearings.

5.3.2 *Fully-articulated Three-point Fixture*—Specimens that do not meet the parallelism requirements of 6.1 shall be tested in a fully-articulated fixture. Well-machined specimens may also be tested in a fully-articulating fixture. The two support (outer) bearings shall be free to roll outwards. The middle bearing shall not roll. Any two of the bearings shall be capable of articulating to match the specimen surface. All three bearings shall rest uniformly and evenly across the specimen surface. The fixture shall be designed to apply equal load to the two outer bearings.

**TABLE 1 Specimen Sizes and Testing Configurations in the Interlaboratory Study**

| Configuration | Nominal Specimen Size (mm) | Specimen Thickness, d (mm) | Support Span, L (mm) | Crosshead Speed, mm/s (mm/m) |
|---|---|---|---|---|
| I | 10 × 10 × 64 | 10 | 50.00 | 0.0042 (0.25) |
| II | 9.5 × 4.8 × 64 | 4.8 | 50.00 | 0.0087 (0.52) |
| III | Ø10 × 64 | 10 | 50.00 | 0.0042 (0.25) |
| IV | 25 × 25 × 150 | 25 | 100.00 | 0.0067 (0.40) |
| V | Ø25 × 150 | 25 | 100.00 | 0.0067 (0.40) |

## 6. Test Specimen

6.1 *Specimen Size*—The size and geometry of the test specimens used in this interlaboratory study are shown in Table 1. It is recommended that the size of the test specimen is selected such that the minimum dimension of the specimen is greater than 5 times the largest particle dimension. It is recommended that the test specimen has a length to thickness/diameter ratio of at least 6, and a width to thickness ratio not greater than 2.

6.1.1 For test specimens that do not meet this ratio for strength testing, see Ref **(1)**[3] and Guide D7775.

6.2 *Preparation*—The test specimen shall be prepared to yield a parallelepiped of square or rectangular cross section or a cylinder. The faces of the parallelepiped specimens shall be parallel and flat within 0.025 mm/mm. In addition, the samples having a maximum particle size less than 0.15 mm in diameter must be finished so that the surface roughness is less than 3 µm Ra. Sample edges should be free from visible flaws and chips.

NOTE 1—For ease of machining to conventional standards, 3 µm Ra is equivalent to 125 µin. AA. For finishing of specimens with maximum particle sizes of greater than 0.150 mm, grain structure and porosity can limit the accurate measurement of roughness. In these cases, the surface roughness should be visually equivalent to 3 µm Ra as estimated based on the visible surface of the graphite.

NOTE 2—Surface preparation of test specimens can introduce machining microcracks which may have a pronounced effect on flexural strength. Machining damage imposed during specimen preparation can be either a random interfering factor, or an inherent part of the strength characteristic to be measured. With proper care and good machining practice, it is possible to obtain fractures during strength testing from the material's natural flaws. Surface preparation can also lead to residual stresses. Universal or standardized test methods of surface preparation do not exist. It should be understood that final machining steps may or may not negate machining damage introduced during the early course or intermediate machining.

6.3 *Measurements*—All dimensions shall be measured to an accuracy of 0.5 % (see Test Method C559).

6.4 *Orientation*—The specimen shall be marked or otherwise identified to denote its orientation with respect to the parent stock.

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

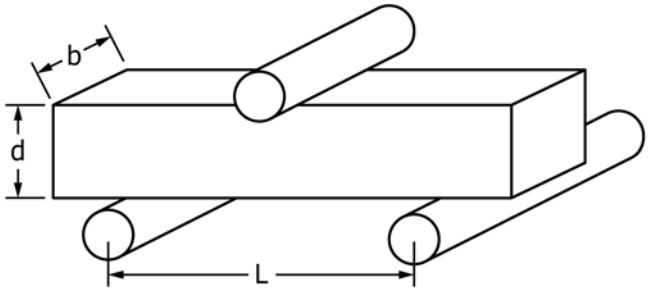


**FIG. 1 The Three-Point Fixture Configuration**

6.5 *Drying*—Each specimen must be dried in a vented oven at 110°C for a period of 2h (see Test Method C559). The sample must then be stored in a dry environment or a desiccator and held there prior to testing.

NOTE 3—Water, either in the form of liquid or as humidity in air, can have an effect on flexural mechanical behavior. Excessive adsorbed water can result in a reduced failure stress due to a decrease in fracture surface energy.

## 7. Procedure

7.1 Place test specimens on their appropriate fixtures in specific testing configurations, as shown in Fig. 1. A fully articulating fixture is required if the specimen parallelism requirements cannot be met.

7.2 Position the specimen on the support bearings on the three-point test fixture so that there is an approximately equal amount of overhang of the specimen beyond the support bearings.

7.3 Position the specimen front to back so that the specimen is directly centered below the axis of the applied load.

7.4 The load bearing shall make contact with the upper surface of the test specimen. The support bearing blocks must be parallel to each other and perpendicular to the test surfaces.

7.5 Load the specimen at a uniform rate such that breakage occurs from flexure rather than impact. As guideline, breaking should not occur in less than 10 s.

7.6 Preserve the fractured specimens until released by the responsible engineer.

## 8. Test Data Record

8.1 All dimensions measured according to Test Method C559 shall be recorded.

8.2 Record the test duration, the break force and fracture location.

8.3 The load at failure must be recorded to an accuracy of better than ±2 % of the full-scale value. A full-scale value of 5 kN would require recording to an accuracy of at least ±100 N.

## 9. Calculation

9.1 If the fracture occurs directly underneath the load bearing, calculate the flexural strength as follows:

9.1.1 For square cross-section specimens:

$$\sigma = (3\ P\ L)/(2\ d^3) \quad (1)$$

where:

$P$ = break force,
$L$ = support span, and
$d$ = specimen thickness.

9.1.2 For rectangular cross-section specimens:

$$\sigma = (3\ P\ L)/(2\ b\ d^2) \quad (2)$$

where:

$b$ = specimen width.

9.1.3 For circular cross-section specimens:

$$\sigma = (8\ P\ L)/(\pi\ D^3) \quad (3)$$

where:

$D$ = specimen diameter.

9.2 If the fracture does not occur directly underneath the load bearing block, the location of the fracture shall be recorded as such, and the results of the test shall be reported.

9.3 If fracture occurs in less than 10 s, the results shall be discarded but reported.

NOTE 4—It should be recognized that the above equations do not necessarily give the stress that was acting directly on the origin that caused failure. The equations do not account for subsurface origins or breaks away from the area under maximum flexure stress (directly below the load bearing), nor do they correct for the potential tension/compression inequality in modulus (behavior that is not linear elastic) commonly accepted in graphite. For conventional Weibull analysis, use the calculated maximum stress in the specimen at failure from the equations as shown.

## 10. Report

10.1 The report of each test shall include the following:

10.1.1 Sample identification,

10.1.2 Average width and thickness or diameter to better than 0.025 mm,

10.1.3 Average weight, g, and density, g/cm$^3$, to within 0.5 %,

10.1.4 Support span length, mm,

10.1.5 Rate of loading, mm/min, and test duration, s,

10.1.6 Maximum applied load, N,

10.1.7 Flexural strength calculated to the nearest 10 kPa,

10.1.8 Defects in specimen,

10.1.9 Orientation and location of specimen,

10.1.10 Failure location, and

10.1.11 Environmental conditions, that is, humidity and temperature.

10.2 Description of test machine and three-point test fixture, including pictures or schematics.

**TABLE 2 Statistical Analysis of Measurements of Specimen Density**

| Material (Config.) | Mean Density ($\bar{x}$) (g/cm$^3$) | St. Dev ($S_{\bar{x}}$) (g/cm$^3$) | Repeatability (within lab) $CV_r$ (%) | Reproducibility (between labs) $CV_R$ (%) | 95 % Limit of Repeatability r (%) | 95 % Limit of Reproducibility R (%) |
|---|---|---|---|---|---|---|
| Material A (I) | 1.810 | 0.004 | 0.166 | 0.276 | 0.497 | 0.773 |
| Material B (II) | 1.805 | 0.005 | 0.720 | 0.720 | 1.939 | 2.050 |
| Material C (III) | 1.808 | 0.004 | 0.664 | 0.664 | 1.881 | 1.936 |
| Material D (IV) | 1.857 | 0.004 | 0.808 | 0.808 | 2.262 | 2.208 |
| Material E (V) | 1.859 | 0.009 | 0.538 | 0.699 | 1.506 | 1.937 |
| Material F (IV) | 1.712 | 0.064 | 1.460 | 3.972 | 4.089 | 11.098 |
| Material G (V) | 1.707 | 0.062 | 2.168 | 4.218 | 6.151 | 11.716 |

10.3 Description of the machining and specimen surface preparation technique or the estimated surface roughness.

## 11. Precision and Bias[4]

11.1 The flexure strength of graphite is not a deterministic quantity, but will vary from one specimen to another. There will be an inherent statistical scatter in the results for finite specimen populations (for example, 30 specimens). Weibull statistics can model this variability as discussed in Practice C1322 and Ref (**2**).

11.2 *Experimental Errors:*

11.2.1 The experimental errors in the flexure test have been thoroughly analyzed and documented in Refs (**1, 3**). The specifications and tolerances in this test method have been chosen such that the individual errors are typically less than 0.5 % each and the total error is estimated to be less than 3 %. A conservative upper limit is of the order of 5 %. This is the maximum possible error in stress for an individual specimen.

11.2.2 Configurations III and V (cylindrical samples) are somewhat more prone to error in three-point loading. For this reason, this configuration is not recommended for design purposes, but only for characterization and materials development.

11.2.3 The mean flexural strength of the cylindrical samples is somewhat higher than the mean flexural strength of the other specimen types of the same graphite grade. Similarly, the variance of the flexural strength of the cylindrical samples is generally higher than that of the other specimen types of the same graphite grade because of the limited specimen volume under maximum stress.

11.3 A total of eight laboratories have participated in this interlaboratory study. Each laboratory was provided with 70 samples in seven distinct groups depending on material type, size and cross section. Three types of graphite were measured in the interlaboratory study with maximum grain size ranging from 0.025 mm to 1.8 mm. The laboratories were asked to carry out three-point bend tests using equipment available to them and in accordance with the specific requirements discussed in the previous sections. These requirements ensured that each laboratory followed a standardised procedure for each sample type and included: pre-test drying and weighing, sample parallelism requirements and the choice of test fixture design (semi or fully articulated), the alignment accuracy and size of the test fixture rollers and the test procedure to be followed.

11.3.1 An analysis of all the data, according to Practice E691, to determine the consistency statistics between the participating laboratories and within any one laboratory was carried out. The statistical analysis using the h-statistics (analysis of the means) and the k-statistics (analysis of the variance) showed good agreement of the three-point bend strength data within lab and between labs within the 95 % confidence levels.

11.3.2 Out of the 56 h-statistics values (eight laboratories and seven materials) calculated, only one was above the h critical value. Out of the 56 k-statistics values calculated, only two were above the k critical value. These two cases do not coincide with the case that failed the h-statistic.

11.3.3 These three cases were further investigated. Moreover, the same statistical analysis was carried out for the density measurements of the same specimens to investigate a possible relationship between variability in strength and variability in density measurements. This exercise demonstrated that that the combinations of material and laboratory that failed the h- and k- statistics for the flexural strength measurements are different from the combinations that failed the corresponding statistics for the density measurements. Hence, the task group has decided to retain all data for the calculation of the repeatability and reproducibility of the technique.

11.3.4 The repeatability (within laboratory) coefficient of variance ranged from 4.03 % to 19.54 %, depending on the material.

11.3.5 The reproducibility (between laboratories) coefficient of variance ranged from 4.93 % to 18.98 %, depending on the material.

11.3.6 The 95 % repeatability limit, r (within laboratory), ranged from 11.28 % to 54.73 %, depending on the material.

11.3.7 The 95 % reproducibility (between laboratories) limit, R, ranged from 13.79 % to 53.14 %, depending on the material.

11.3.8 The statistics for density and flexural strength are summarised in Table 2 and Table 3, respectively. It can be seen that the very high values of the coefficient of variance in the flexural strength measurements are obtained from the two material groups (F and G) that have very low density and unusually high coefficient of variance in the density measurements. This indicates an inherent material variability in these two specimen groups.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1792. Contact ASTM Customer Service at service@astm.org.

**TABLE 3 Statistical Analysis of Measurements of Three-Point Flexure Strength**

| Material (Config.) | Mean Density ($\bar{x}$) ($g/cm^3$) | St. Dev ($S_{\bar{x}}$) ($g/cm^3$) | Repeatability (within lab) $CV_r$ (%) | Reproducibility (between labs) $CV_R$ (%) | 95 % Limit of Repeatability r (%) | 95 % Limit of Reproducibility R (%) |
|---|---|---|---|---|---|---|
| Material A (I) | 50.307 | 1.561 | 4.031 | 4.926 | 11.285 | 13.789 |
| Material B (II) | 49.491 | 0.962 | 6.227 | 6.219 | 17.440 | 17.415 |
| Material C (III) | 55.144 | 1.094 | 5.210 | 5.326 | 14.587 | 14.912 |
| Material D (IV) | 31.109 | 1.184 | 6.535 | 7.274 | 18.294 | 20.367 |
| Material E (V) | 34.855 | 1.235 | 6.868 | 7.416 | 19.231 | 20.766 |
| Material F (IV) | 11.774 | 0.385 | 12.179 | 12.010 | 34.109 | 33.625 |
| Material G (V) | 12.592 | 0.510 | 19.544 | 18.980 | 54.725 | 53.145 |

11.4 *Bias*—No true statement on bias can be made because no reference carbon or graphite material exists.

## 12. Keywords

12.1 carbon; flexural strength; graphite

## REFERENCES


**(1)** Metcalfe, M.P. and Tzelepi, A., "The Effect of Test Specimen Size on Graphite Strength," ASTM Special Technical Publication 1578.

**(2)** Duffy, S., "Quality Control Using Inferential Statistics in Weibull Based Reliability Analyses," ASTM Special Technical Publication 1578.

**(3)** Wheatley, C. J., "The scaling of the strength of nuclear graphite with particular emphasis on statistical aspects and implications for testing," ASTM Special Technical Publication 1578.


## RELATED MATERIAL

ASTM Test Method C1684 for Flexural Strength of Advanced Ceramics at Ambient Temperature—Cylindrical Rod Strength

ASTM Practice E177 for Use of the Terms Precision and Bias in ASTM Test Methods