# NOTICE:¬This¬standard¬has¬either¬been¬superseded¬and¬replaced¬by¬a¬new¬version¬or discontinued.¬Contact¬ASTM¬International¬(www.astm.org)¬for¬the¬latest¬information.

€∰) De

Designation: C 1161 – 94 (Reapproved 1996)

AMERICAN SOCIETY FOR TESTING AND MATERIALS 100 Barr Harbor Dr., West Conshohocken, PA 19428 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

# Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature<sup>1</sup>

This standard is issued under the fixed designation C 1161; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope

1.1 This test method covers the determination of flexural strength of advanced ceramic materials at ambient temperature. Four-point–1/4 point and three-point loadings with prescribed spans are the standard. Rectangular specimens of prescribed cross-section sizes are used with specified features in prescribed specimen-fixture combinations.

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:

- E 4 Practices for Force Verification of Testing Machines<sup>2</sup> E 337 Test Method for Measured Humidity with a Psy-
- chrometer (The Measurement of Wet- and Dry-Bulb Temperatures)<sup>3</sup>
- 2.2 *Military Standard:*
- MIL-STD-1942 (MR) Flexural Strength of High Performance Ceramics at Ambient Temperature<sup>4</sup>

#### 3. Terminology

3.1 Definitions:

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

3.1.2 *four-point*—l/4 *point flexure*—configuration of flexural strength testing where a specimen is symmetrically loaded at two locations that are situated one quarter of the overall span, away from the outer two support bearings (see Fig. 1).

3.1.3 *three-point flexure*—configuration of flexural strength testing where a specimen is loaded at a location midway





```
B: L = 40 \text{ mm}
C: L = 80 \text{ mm}
```



between two support bearings (see Fig. 1).

# 4. Significance and Use

4.1 This test method may be used for material development, quality control, characterization, and design data generation purposes.

4.2 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. The average grain size should be no greater than one fiftieth of the beam thickness. The homogeneity and isotropy assumption in the standard rule out the use of this test for continuous fiber-reinforced ceramics.

4.3 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.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee C-28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Properties and Performance.

Current edition approved July 25, 1994. Published February 1995. Originally published as C 1161 – 90. Last previous edition C 1161 – 90.

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 03.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 11.03.

<sup>&</sup>lt;sup>4</sup> Available from Standardization Documents, Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

Specimen sizes and fixtures were chosen to provide a balance between practical configurations and resulting errors, as discussed in MIL-STD 1942 (MR) and Refs (1) and (2).<sup>5</sup> Specific fixture and specimen configurations were designated in order to permit ready comparison of data without the need for Weibullsize scaling.

4.4 The flexural strength of a ceramic material is dependent on both its inherent resistance to fracture and the presence of defects. Analysis of a fracture surface, fractography, though beyond the scope of this test method, is highly recommended for all purposes, especially for design data as discussed in MIL-STD-1942 (MR) and Refs (2–5).

#### 5. Interferences

5.1 The effects of time-dependent phenomena, such as stress corrosion or slow crack growth on strength tests conducted at ambient temperature, can be meaningful even for the relatively short times involved during testing. Such influences must be considered if flexure tests are to be used to generate design data.

5.2 Surface preparation of test specimens can introduce machining flaws 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. 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. Apparatus

6.1 Loading—Specimens may be loaded in any suitable testing machine provided that uniform rates of direct loading can be maintained. The load-measuring system shall be free of initial lag at the loading rates used and shall be equipped with a means for retaining read-out of the maximum load applied to the specimen. The accuracy of the testing machine shall be in accordance with Practices E 4 but within 0.5 %.

6.2 *Four-Point Flexure*—Four-point-1/4 point fixtures (Fig. 1) shall have support and loading spans as shown in Table 1.

6.3 *Three-Point Flexure*—Three-point fixtures (Fig. 1) shall have a support span as shown in Table 1.

6.4 *Bearings*—Three- and four-point flexure:

6.4.1 Cylindrical bearing edges shall be used for the support of the test specimen and for the application of load. The cylinders shall be made of hardened steel which has a hardness no less than HRC 40 or which has a yield strength no less than

 $^{\rm 5}$  The boldface numbers in parentheses refer to the references at the end of this test method.

TABLE 1 Fixture Spans

Configuration	Support Span (L), mm	Loading Span, mm
A	20	10
В	40	20
С	80	40

1240 MPa ( $\sim$ 180 ksi). Alternatively, the cylinders may be made of a ceramic with an elastic modulus between 2.0 and 4.0 × 10<sup>5</sup> MPa (30–60 × 10<sup>6</sup> psi) and a flexural strength no less than 275 MPa ( $\sim$ 40 ksi). The portions of the test fixture that support the bearings may need to be hardened to prevent permanent deformation. The cylindrical bearing length shall be at least three times the specimen width. The above requirements are intended to ensure that ceramics with strengths up to 1400 MPa ( $\sim$ 200 ksi) and elastic moduli as high as  $4.8 \times 10^5$ MPa (70 × 10<sup>6</sup> psi) can be tested without fixture damage. Higher strength and stiffer ceramic specimens may require harder bearings.

6.4.2 The bearing cylinder diameter shall be approximately 1.5 times the beam depth of the test specimen size employed. See Table 2.

6.4.3 The bearing cylinders shall be carefully positioned such that the spans are accurate within  $\pm 0.10$  mm. The load application bearing for the three-point configurations shall be positioned midway between the support bearing within  $\pm 0.10$  mm. The load application (inner) bearings for the four-point configurations shall be centered with respect to the support (outer) bearings within  $\pm 0.10$  mm.

6.4.4 The bearing cylinders shall be free to rotate in order to relieve frictional constraints (with the exception of the middle-load bearing in three-point flexure which need not rotate). This can be accomplished by mounting the cylinders in needle bearing assemblies, or more simply by mounting the cylinders as shown in Fig. 2 and Fig. 3. Note that the outer-support bearings roll *outward* and the inner-loading bearings roll *inward*.

6.5 *Semiarticulating–Four-Point Fixture*—Specimens prepared in accordance with the parallelism requirements of 7.1 may be tested in a semiarticulating fixture as illustrated in Fig. 2. The bearing cylinders themselves must be parallel to each other to within 0.015 mm (over their length).

6.6 *Fully Articulating–Four-Point Fixture*—Specimens that are as-fired, heat treated, or oxidized often have slight twists or unevenness. Specimens which do not meet the parallelism requirements of 7.1 shall be tested in a fully articulating fixture as illustrated in Fig. 3.

6.7 The fixture shall be stiffer than the specimen, so that most of the crosshead travel is imposed onto the specimen.

# 7. Specimen

7.1 Specimen Size—Dimensions are given in Table 3 and shown in Fig. 4. Cross-sectional dimensional tolerances are  $\pm 0.13$  mm for B and C specimens, and  $\pm 0.05$  mm for A. The parallelism tolerances on the four longitudinal faces are 0.015 mm for A and B and 0.03 mm for C. The two end faces need not be precision machined.

7.2 Specimen Preparation—Depending upon the intended application of the flexural strength data, use one of the following four specimen preparation procedures:

ring Diameters

Configuration	Diameter, mm
A	2.0 to 2.5
В	4.5
C	9.0

(新) C 1161



B: L = 40 mm

#### NOTE 2—Load is applied through a ball which permits the loading member to tilt as necessary to ensure uniform loading FIG. 2 Schematic of a Semiarticulated Four-Point Fixture Suitable for Flat and Parallel Specimens

7.2.1 *As-Fabricated*—The flexural specimen shall simulate the surface condition of an application where no machining is to be used; for example, as-cast, sintered, or injection-molded parts. No additional machining specifications are relevant. An edge chamfer is not necessary in this instance. As-fired specimens are especially prone to twist or warpage and might not meet the parallelism requirements. In this instance, a fully articulating fixture (6.6 and Fig. 3) shall be used in testing.

7.2.2 Application-Matched Machining—The specimen shall have the same surface preparation as that given to a component. Unless the process is proprietary, the report shall be specific about the stages of material removal, wheel grits, wheel bonding, and the amount of material removed per pass.

7.2.3 *Customary Procedures*—In instances where a customary machining procedure has been developed that is completely satisfactory for a class of materials (that is, it induces no unwanted surface damage or residual stresses), this procedure shall be used.

7.2.4 *Standard Procedures*—In the instances where 7.2.1 through 7.2.3 are not appropriate, then 7.2.4 shall apply. This procedure shall serve as minimum requirements and a more stringent procedure may be necessary.

7.2.4.1 All grinding shall be done with an ample supply of appropriate filtered coolant to keep workpiece and wheel constantly flooded and particles flushed. Grinding shall be in at least two stages, ranging from coarse to fine rates of material removal. All machining shall be in the surface grinding mode, and shall be parallel to the specimen long axis shown in Fig. 5. No Blanchard or rotary grinding shall be used.

7.2.4.2 The stock-removal rate shall not exceed 0.03 mm (0.001 in.) per pass to the last 0.06 mm (0.002 in.) per face. Final (and intermediate) finishing shall be performed with a diamond wheel that is between 320 and 500 grit. No less than 0.06 mm per face shall be removed during the final finishing phase, and at a rate of not more than 0.002 mm (0.0001 in.) per

pass. Remove approximately equal stock from opposite faces.

7.2.4.3 Materials with low fracture toughness and a greater susceptibility to grinding damage may require finer grinding wheels at very low removal rates.

7.2.4.4 The four long edges of each specimen shall be uniformly chamfered at  $45^{\circ}$ , a distance of  $0.12 \pm 0.03$  mm as shown in Fig. 4. They can alternatively be rounded with a radius of  $0.15\pm 0.05$  mm. Edge finishing must be comparable to that applied to the specimen surfaces. In particular, the direction of machining *shall be parallel* to the specimen long axis. If chamfers are larger than the tolerance allows, then corrections shall be made to the stress calculation (1). Alternatively, if a specimen can be prepared with an edge that is free of machining damage, then a chamfer is not required.

7.2.5 *Handling Precautions*—Care should be exercised in storing and handling of specimens to avoid the introduction of random and severe flaws, such as might occur if specimens were allowed to impact or scratch each other.

7.3 *Number of Specimens*—A minimum of 10 specimens shall be required for the purpose of estimating the mean. A minimum of 30 shall be necessary if estimates regarding the form of the strength distribution are to be reported (for example, a Weibull modulus). The number of specimens required by this test method has been established with the intent of determining not only reasonable confidence limits on strength distribution parameters, but also to help discern multiple-flaw population distributions. More than 30 specimens are recommended if multiple-flaw populations are present.

#### 8. Procedure

8.1 Test specimens on their appropriate fixtures in specific testing configurations. Test specimens Size A on either the four-point A fixture or the three-point A fixture. Similarly, test B specimens on B fixtures, and C specimens on C fixtures. A

C: L = 80 mm

(新) C 1161



NOTE 1—Configuration:

A: L = 20 mm

B: L = 40 mm

C: L = 80 mm

Note 2—Bearing A is fixed so that it will not pivot about the x axis. The other three bearings are free to pivot about the x axis. FIG. 3 Schematic of a Fully Articulating Four-Point Fixture Suitable for Twisted or Uneven Specimens

TABLE 3 Specimen Size				
Configuration	Width ( <i>b</i> ), mm	Depth ( <i>d</i> ), mm	Length ( <i>L<sub>T</sub></i> ), min, mm	
A	2.0	1.5	25	
В	4.0	3.0	45	
С	8.0	6.0	90	

fully articulating fixture is required if the specimen parallelism requirements cannot be met. An alternative procedure with a D specimen is given in the Appendix.

8.2 Carefully place each specimen into the test fixture to preclude possible damage and to ensure alignment of the specimen in the fixture. In particular, there should be an equal amount of overhang of the specimen beyond the outer bearings and the specimen should be directly centered below the axis of the applied load.

8.3 Slowly apply the load at right angles to the fixture. The maximum permissible stress in the specimen due to initial load shall not exceed 25 % of the mean strength. Inspect the points

of contact between the bearings and the specimen to ensure even line loading and that no dirt or contamination is present. If uneven line loading of the specimen occurs, use fully articulating fixtures.

8.4 Mark the specimen to identify the points of load application and also so that the tensile and compression faces can be distinguished. Carefully drawn pencil marks will suffice.

8.5 Put cotton, crumbled tissues, or other appropriate material around specimen to prevent pieces from flying out of the fixtures upon fracture. This step may help ensure operator's safety and preserve primary fracture pieces for subsequent fractographic analysis.

8.6 Loading Rates—The crosshead rates are chosen so that the strain rate upon the specimen shall be of the order of  $1.0 \times 10^{-4} s^{-1}$ .

8.6.1 The strain rate for either the three- or four-point $-\frac{1}{4}$  point mode of loading is as follows:

$$\boldsymbol{\epsilon} = 6 \, ds/L^2 \tag{1}$$

🖤 C 1161







where:

 $\tilde{\mathbf{\epsilon}}$  = strain rate,

d = specimen thickness,

s = crosshead speed, and

L = outer (support) span.

8.6.2 Crosshead speeds for the different testing configurations are given in Table 4.

TABLE 4 Crosshead Speeds for Displacement-Controlled Testing Machine

Configuration	Crosshead Speeds, mm/min	
A	0.2	
В	0.5	
С	1.0	

8.6.3 Times to failure for typical ceramics will range from 3 to 30 s. It is assumed that the fixtures are relatively rigid and that most of the testing-machine crosshead travel is imposed as strain on the test specimen.

8.7 *Breakload*—Measure the breakload with an accuracy of  $\pm 0.5$  %.

8.8 Specimen Dimension—Determine the thickness and width of each specimen to within 0.0025 mm (0.0001 in.). In order to avoid damage in the critical area, it is recommended that measurement be made after the specimen has broken at a point near the fracture origin. It is highly recommended to retain and preserve all primary fracture fragments for fractographic analysis.

8.9 Determine the relative humidity in accordance with Test Method E 337.

8.10 The occasional use of a strain-gaged specimen is recommended to verify that there is negligible error in stress, in accordance with 11.2.

## 9. Calculation

9.1 The standard formula for the strength of a beam in four-point-  $\frac{1}{4}$  point flexure is as follows:

$$S = \frac{3 PL}{4 bd^2}$$

where:

$$P = breakload,$$

- L =outer (support) span,
- b = specimen width, and
- d = specimen thickness.

9.2 The standard formula for the strength of a beam in three-point flexure is as follows:

$$S = \frac{3 PL}{2 bd^2} \tag{2}$$

9.3 Eq. 1 and Eq. 2 shall be used for the reporting of results

and are the common equations used for the flexure strength of a specimen.

NOTE 1—It should be recognized however, that Eq. 1 and Eq. 2 do not necessarily give the stress that was acting directly upon the flaw that caused failure. (In some instances, for example, for fracture mirror or fracture toughness calculations, the fracture stress must be corrected for subsurface origins and breaks outside the gage length.)

NOTE 2—The conversion between pounds per square inch (psi) and megapascals (MPa) is included for convenience (145.04 psi = 1 MPa; therefore, 100 000 psi = 100 ksi = 689.5 MPa.)

#### 10. Report

10.1 Test reports shall include the following:

10.1.1 Test configuration and specimen size used.

10.1.2 The number of specimens (n) used.

10.1.3 All relevant material data including vintage data or billet identification data. (Did all specimens come from one billet?) As a minimum, the date the material was manufactured shall be reported.

10.1.4 Exact method of specimen preparation, including all stages of machining.

10.1.5 Heat treatments or exposures, if any.

10.1.6 Test environment including humidity (Test Method E 337) and temperature.

10.1.7 Strain rate or crosshead rate.

10.1.8 Report the strength of every specimen in megapascals (pounds per square inch) to three significant figures.

10.1.9 Mean ( $\overline{S}$ ) and standard deviation (SD) where:

$$\bar{S} = \frac{\sum_{1}^{n} S}{n} \tag{3}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (S - \sum_{i=1}^{n} S)^{2}}{(n-1)}}$$
 (4)

10.1.10 Report of any deviations and alterations from the procedures described in this test method.

#### **11. Precision and Bias**

11.1 The flexure strength of a ceramic 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 sample sizes (for example, 30 specimens). Weibull statistics can model this variability as discussed in Refs (1) and (6–10). This test method has been devised so that the precision is very high and the bias very low compared to the inherent variability of strength of the material.

11.2 Experimental Errors:

11.2.1 The experimental errors in the flexure test have been thoroughly analyzed and documented in Ref (1). 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 probably less than 3% for four-point configurations B and C. (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 The error due to cross-section reduction associated with chamfering the edges can be of the order of 1 % for configuration B and less for configuration C in either three or four-point loadings, as discussed in Ref (1). The chamfer sizes in this test method have been reduced relative to those allowed in MIL-STD-1942 (MR). Chamfers larger than specified in this test method shall require a correction to stress calculations as discussed in Ref (1).

11.2.3 Configuration A is somewhat more prone to error which is probably greater than 5 % in four-point loading. Chamfer error due to reduction of cross-section areas is 4.1 %. For this reason, this configuration is not recommended for design purposes, but only for characterization and materials development.

11.3 An intralaboratory comparison of strength values of a high purity (99.9 %) sintered alumina was held (7). Three different individuals with three different universal testing machines on three different days compared the strength of lots of 30 specimens from a common batch of material. Three different fixtures, but of a common design, were used. The mean strengths varied by a maximum of 2.4 % and the Weibull moduli by a maximum of 27 % (average of 11.4). Both variations are well within the inherent scatter predicted for sample sizes of 30 as shown in Refs (1), (7), and (9).

11.4 An interlaboratory comparison of strength of the same alumina as cited in 11.3 was made between two laboratories. A1.3 % difference in the mean and an 18 % difference in Weibull modulus was observed, both of which are well within the inherent variability of the material.

11.5 An interlaboratory comparison of strength of a different alumina and of a silicon nitride was made between seven international laboratories. Reference (7) is a comprehensive report on this study which tested over 2000 specimens. Experimental results for strength variability on B specimens, in both three- and four-point testing, were generally consistent with analytical predictions of Ref (9). For a material with a Weibull modulus of 10, estimates of the mean (or characteristic strength) for samples of 30 specimens will have a coefficient of variance of 2.2 %. The coefficient of variance for estimates of the Weibull modulus is 18 %.

#### 12. Keywords

12.1 advanced ceramics; flexural strength; four-point flexure; three-point flexure

#### APPENDIX

#### (Nonmandatory Information)

#### **X1. ALTERNATIVE PROCEDURE**

X1.1 An alternative procedure is given in the following paragraphs. This alternate procedure may be used when the procedures in the main text are not suitable.

X1.2 *Fixture Spans*—A support span of 38.10 mm (1.5 in.) shall be used for three- or four-point loading, and a loading span of 19.05 mm (0.75 in.) shall be used for the four-point loading.

X1.3 *Bearing Diameter*—A bearing diameter of 4.5 to 5.0 mm diameter shall be used.

X1.4 Specimen Size—The specimen size D shall be as given in Fig. X1.1. The width is 6.35 mm (0.25 in.); the

thickness, 3.18 mm (0.125 in.) and the length greater than 45 mm (1.8 in.).

X1.5 *Crosshead Speed*—Crosshead speed shall be 0.5 mm/min (0.02 in./min).

X1.6 All other testing procedures and tolerances are as specified in the main text for the B configuration.

X1.7 *Precision and Bias*—Data on precision and bias obtained during an interlaboratory round robin study of the flexure strength of a sintered silicon nitride will be published soon. This study was conducted as a subtask of a larger International Energy Agency (IEA) round robin effect (**11**).



FIG. X1.1 The Alternative 'D' Test Specimen

# REFERENCES

- (1) Baratta, F. I., Quinn, G. D., and Matthews, W. T., "Errors Associated With Flexure Testing of Brittle Materials," U.S. Army MTL TR 87-35, July 1987.
- (2) Quinn, G. D., Baratta, F. I., and Conway, J. A., "Commentary on U.S. Army Standard Test Method for Flexural Strength of High Performance Ceramics at Ambient Temperature," U.S. Army AMMRC 85-21, August 1985.
- (3) Hoagland, R., Marshall, C., and Duckworth, W., "Reduction of Errors in Ceramic Bend Tests," *Journal of the American Ceramic Society*, Vol 59, No. 5–6, May–June, 1976, pp. 189–192.
- (4) Quinn, G. D., and Baratta, F. I., "Flexure Data, Can It Be Used For Ceramics Part Design?" Advanced Materials and Processes, December 1985, pp. 31–35.
- (5) Quinn, G. D., "Properties Testing and Materials Evaluation," *Ceramic Engineering and Science Proceedings*, Vol 5, May–June 1984, pp. 298–311.

- (6) Quinn, G. D., "Fractographic Analysis and the Army Flexure Test Method," *Fractography of Glass and Ceramics*, Vol 22 of *Advances in Ceramics*, American Ceramic Society, 1988, pp. 314–334.
- (7) Quinn, G. D., "Flexure Strength of Advanced Ceramics—A Round Robin Exercise," U.S. Army MTL TR 89-62, July 1989.
- (8) Davies, D. G. S., "The Statistical Approach to Engineering Design in Ceramics," *Proceedings of the British Ceramic Society*, Vol 22, 1979, pp. 429–452.
- (9) Ritter, J. Jr., Bandyopadhyay, N., and Jakus, K., "Statistical Reproducibility of the Dynamic and Static Fatigue Experiments," *Ceramic Bulletin*, Vol 60, No. 8, 1981, pp. 798–806.
- (10) Weibull, W., "Statistical Distribution Function of Wide Applicability," *Journal of Applied Mechanics*, Vol 18, 1951, p. 293.
- (11) Tennery, V., "International Energy Agency Annex II," Ceramic Technology Newsletter, Number 23, April–June 1989.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.