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FLEXURAL STRENGTH OF HIGH PERFORMANCE CERAMICS AT AMBIENT TEMPERATURE

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AMSC N/A FSC 9390

DEPARTMENT OF THE ARMY
WASHINGTON, DC 20310

Flexural Strength of High Performance Ceramics at Ambient Temperature

MIL-STD-1942A

1. This Military Standard is approved for use by all Departments and Agencies of the Department of Defense.

2. Beneficial comments (recommendations, additions and deletions) and any pertinent data which may be of use in improving this document should be addressed to the Director, U.S. Army Materials Technology Laboratory, ATTN: SLCMT-MEE, Watertown, MA 02172-0001 by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document, or by letter.

FOREWORD

This Standard was developed to satisfy a need for standardization of a test method for determining the flexural strength of high performance ceramic materials. It is intended to be used for design data generation, for quality control and for material development purposes.

Ceramics are potentially attractive as structural materials because of their high strength-to-weight ratio, high modulus, high corrosion resistance, excellent high-temperature properties, and abundant availability. However, ceramic materials are not only characteristically brittle, but they as yet do not possess the high standards of uniformity, reproducibility, and reliability that are required of conventional structural materials.

One of the major obstacles to the extended use of ceramics as structural entities, devices, or component parts of military end items has been the lack of design data. The reliability of design data for ceramics is generally conceded to be of relatively low order as compared with such materials as metals and plastics. However, use of the previous edition of this Standard has provided a basis for better quality data. This revision represents the best technical consensus available at the time of issuance on a test method for ceramics. It will yield accurate and reproducible results upon which design decisions can be based and will serve as the basis for acceptance/rejection inspection for compliance with specification requirements.

The use of this standard method since 1983 has brought consistency to the flexural strength testing of structural ceramics. Comparative analysis of data is more meaningful. Significant experimental errors have been minimized. This Standard will continue to permit the generation of high quality and reproducible design data.

CONTENTS

1. SCOPE

1.1 Purpose. This Standard describes a test method for determining the flexural strength, or modulus of rupture (MOR) of brittle beam test specimens at ambient temperature.

The method provides for both four-point loading and three-point loading. This standard is suitable for high performance ceramics where flexural strength is in excess of 100 MPa. The grain or particulate size of the material should be no greater than 1/50th of the smallest dimension of the specimen.

1.2 General comments. The four-point test is the preferred mode of testing. This is primarily due to the larger amount of material that experiences the maximum stress in four point loading. Several reference standard specimen sizes and reference standard fixture spans have been selected. A 1/4 point configuration has been chosen for the four-point flexural fixture. Rectangular cross-section test specimens of beam thickness to width ratios of 3:4 is the standard. Reference AMMRC TR 85-21 provides comprehensive details and considerations that led to the development of this Standard.

2. REFERENCED DOCUMENTS

2.1 Government documents. The following documents form a part of this Standard to the extent specified herein.

PUBLICATIONS

AMMRC TR-82-20 - Requirements for Flexure Testing of Brittle Materials, Francis I. Baratta, April, 1980. AD A113 937

AMMRC TR-85-21 - Commentary on US Army Standard Test Method for Flexural Strength of High Performance Ceramics at Ambient Temperature, George D. Quinn, Francis I. Baratta and James A. Conway, August 1985. AD A160 873

MTL TR-87-35 - Errors associated with Flexure Testing of Brittle Materials, Francis I. Baratta, George D. Quinn, and William T. Matthews, July 1987. AD A187 470

MTL TR-89-62 - Flexure strength of advanced ceramics/a round robin exercise. George E. Quinn, July 1989 AD A212 010

(Applications for copies of this report should be addressed to NTIS, US Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161.)

2.2 Non-Government publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM E4 - Load Verification of Testing Machines
ASTM E337 - Relative Humidity by Wet-and-Dry Bulb Psychrometer

(Applications for copies of ASTM publications should be addressed to ASTM, 1916 Race Street, Philadelphia, PA 19103)

3. DEFINITIONS

3.1 Flexural strength. A measure of the ultimate strength of a specified beam in bending. This is also referred to as the modulus of rupture. The flexural strength is the maximum outer fiber stress at the time of failure, regardless of where fracture originates.

3.2 Maximum outer fiber stress. The maximum stress acting upon the flexural test specimen. It occurs at the specimen surface opposite the central load bearing in the three-point configuration method and between the two inner load bearings in the four-point configuration.

3.3 Three-point loading. A configuration of flexural strength testing such that a specimen is loaded by a bearing which is located midway between two support bearings (figure 1).

3.4 Four-point - 1/4 point flexure. A configuration of flexural strength testing such that a specimen is loaded by two bearings which are located one-quarter of the overall span, away from the outer two support bearings (figure 1).

4. GENERAL REQUIREMENTS

4.1 Assumptions. The formulation for outer fiber stress is based on simple beam theory wherein it is assumed that the ceramics to be evaluated are linearly elastic: the moduli of elasticity in tension and compression are identical; the material is homogeneous and isotropic; and shear strains are negligible.

4.2 General comments.

4.2.1 It is recognized that flexural strength for a group of test specimens is influenced by variables associated with the test procedures. These include the rate of loading, test environment, specimen and fixture sizes and specimen preparation. Such factors are specified in the test procedure or are required in the report.

4.2.2 This Standard can be expanded to include elevated temperature testing provided that all of the conditions of Paragraph 4.1 are applicable. Creep can invalidate these assumptions and can compromise the strength measurements. Strain rate effects upon strength may become more important at higher temperatures.

4.3 Testing machine. Any suitable testing machine may be used provided that uniform rates of direct loading can be maintained. The load measuring system shall be essentially free of initial lag at the loading rates used and shall be equipped with a means for retaining an indication of the maximum load applied to the specimen.

The accuracy of the testing machine shall conform to the requirements of ASTM E4, except that the breakload shall be measured to ± 0.5%.

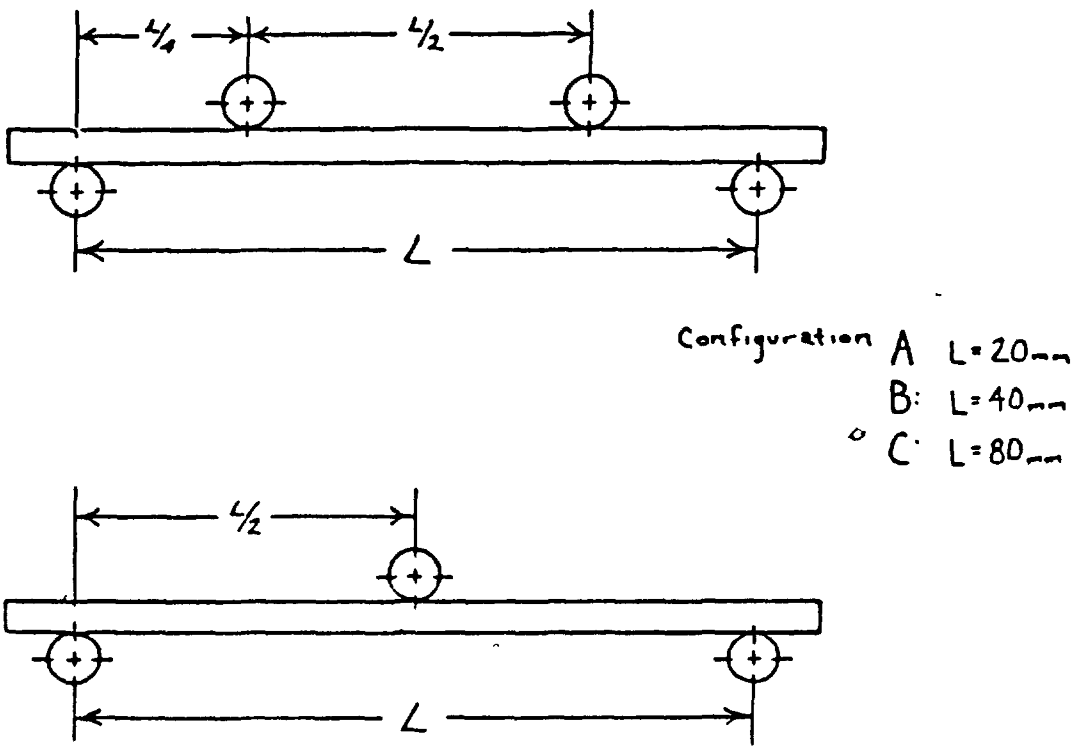

FIGURE 1. The four point-1/4-point and three-point fixture configuration.

5. DETAILED REQUIREMENTS

5.1 Specimens. Three specimen sizes are specified. They are to be used in conjunction with three fixtures defined later. Size A specimens are not preferred except for instances when larger specimens are not feasible.

5.1.1 Dimensions. For specimen thickness to width configuration of 3:4:

5.1.1.1 Specimen Size A. 1.50 ± .03 mm X 2.00 ± .03 X 25 mm or longer as shown in figure 2. The four long faces shall be flat and parallel within .015 mm. This specimen is to be used with fixture configuration A.

5.1.1.2 Specimen Size B. 3.00 ± .03 mm X 4.00 ± .03 mm X 45 mm or longer as shown in figure 2. All other specifications are the same as specimen size A. This specimen is to be used with fixture configuration B.

5.1.1.3 Specimen Size C. 6.00 ± .03 mm X 8.00 ± .03 mm X 85 mm or longer as shown in figure 2. The four long faces shall be flat and parallel within .03 mm. This specimen is to be used with fixture configuration C.

5.1.2 Specimen preparation. Depending upon the intended application of the flexural strength data, one of the following specimen preparation procedures can be used:

5.1.2.1 As fabricated. The flexural specimen is to 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. It is common for such specimens to have twists, warpage, nonuniform cross sections and larger chamfers than those specified in this Standard. If the specimens cannot be fabricated according to the requirements of figure 2, then the variances shall be stated in the report.

5.1.2.2 Application matched machining. The specimen shall have the same surface preparation as that given to the intended component. The machining procedure shall supersede the requirements stated in paragraph 5.1.2.3. Unless the process is proprietary, the report shall be specific about the stages of material removal, wheel grits, wheel bonding and material removed per pass.

5.1.2.3 Standard procedure. All grinding shall be done with an ample supply of water-based coolant to keep workpiece and wheel constantly flooded and particles flushed and filtered. 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, parallel to the specimen long axis shown in figure 3. No Blanchard or rotary grinding shall be used. If water is believed to be deleterious to the material, then an alternative suitable grinding media shall be permitted, providing it is described in the report. It should be recognized, however, that adoption of such standard machining recommendations will not guarantee identical results, but it should significantly enhance the consistency of results.

The stock removal rate shall not exceed .03 mm per-pass to the last .06 mm per face. Final and intermediate finishing shall be performed with a diamond wheel that is between 320 and 500 grit. No less than .06 mm per face shall be removed during the final finishing phase, and at a rate of not more than 0.002 mm per pass. Remove approximately equal stock from opposite faces.

If a specimen can be prepared with an edge that is free of machining damage, then a chamfer is not required. Otherwise, the four long edges of each specimen shall be uniformly chamfered at 45°, a distance of 0.12 ± 0.03 mm. They can be rounded with a radius of 0.15 ± 05 mm. Edge finishing must be comparable in all respects to that applied to the specimen surfaces. In particular, the direction of machining must be parallel to the specimen long axis. The specimen end faces need not be precision machined.

5.1.2.4 Handling precautions. Care should be exercised in 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. Specimens shall be individually wrapped or separated by tissue or some similar protective material.

5.2 Test fixtures.

5.2.1 Four point-1/4 point flexure - recommended procedure. Quarter point fixtures with one of the following support loading span combinations are to be used
(see figure 1):

Fixture Configuration A. 20/10mm
Fixture Configuration B. 40/20mm } (Recommended over configuration A.)
Fixture Configuration C. 80/40mm }

Configuration A is not preferred since it is somewhat more error prone than the larger configurations. It is to be used only when a larger configuration is not obtainable.

5.2.2 Three-point flexure - alternative procedure If circumstances prevent the usage of the four-point mode of loading, then three-point flexure can be employed. The same (outer) spans as specified in para 5.2.1 are to be used.

5.3 Load bearings.

5.3.1 Bearing cylinder hardness. 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 that 1200 MPa. The cylinders can alternatively be made of a ceramic with an elastic modulus between 2.0 and 4.0 X 10^5 MPa and a flexural strength no less than 275 MPa. The supported cylinder length shall be at least 3 times the specimen width. The portions of the test jig that support the bearing cylinders must also be hardened to prevent permanent deformation. The above requirements are intended to ensure that ceramics with strengths up to 1400 MPa and elastic moduli as high as 480 GPa can be safely tested.

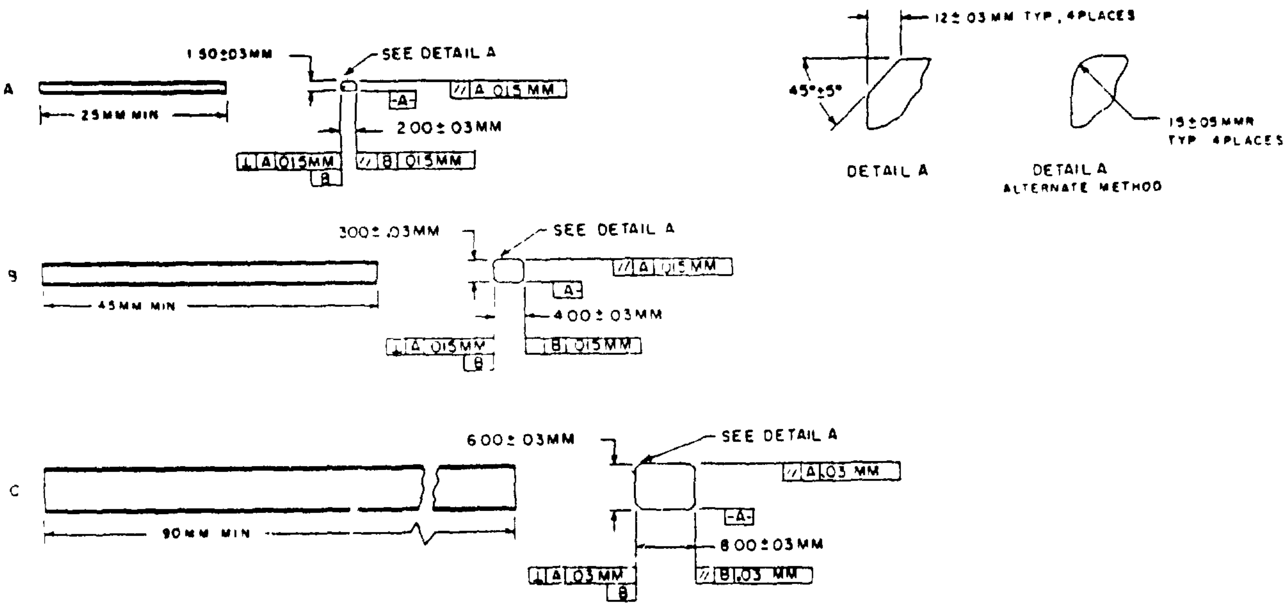

FIGURE 2. The standard test specimens.

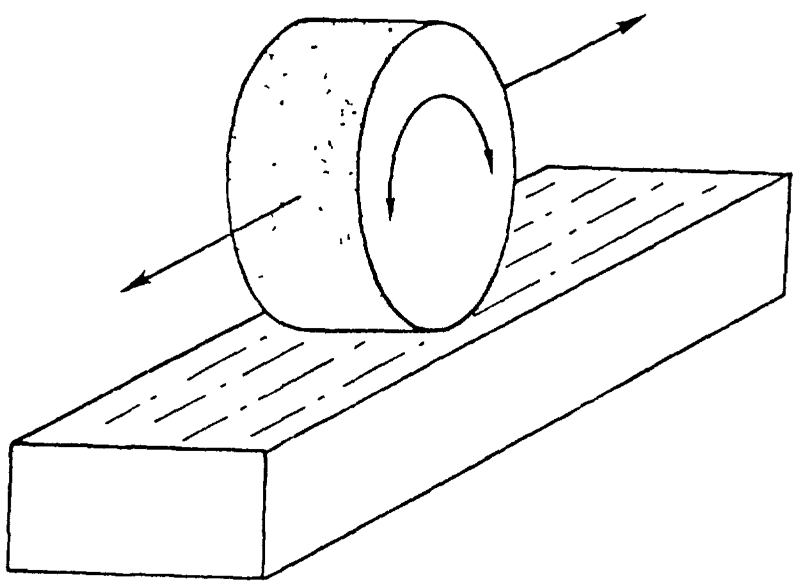

FIGURE 3. Required preparation procedure for surface grinding.

Higher strength specimens will require higher hardness bearings. Rollers of HRC 60 are readily available and permit a ceramic specimen of up to 4300 MPa strength and 480 GPa stiffness to be tested without yielding. The hardness requirements of the bearings support and other fixture parts (especially articulating points) will vary depending upon the fixture design. But, in most instances, concentrated line loadings will not cause yielding of pieces which are at least three times wider than the specimen and are made of steel with an HRC of 25 or greater.

5.3.2 Bearing cylinder rotation. The bearing cylinders must be free to rotate in order to eliminate any frictional restraints. This can be accomplished by the use of a needle bearing assembly, or the design scheme shown in figure 4. In the latter scheme, the inner bearings shall be free to roll inward, and the other bearings, outward.

The middle bearing cylinder in three point loading fixtures does not have to rotate, whereas the outer bearings do.

5.3.3 Positioning of bearings. The bearing cylinders shall be carefully positioned such that the spans are accurate to ± .10 mm. The load application bearing for the three-point configurations shall be positioned midway between the support bearing to within ± .10 mm. The load application (inner) bearings for the four point-1/4 point configurations shall be centered with respect to the support (outer) bearings to within ± .10 mm.

5.3.4 Bearing cylinder diameter. The bearing cylinder diameter shall be approximately 1.5 times the cross sectional thickness of the test specimen size employed.

Configuration A	2 - 2.5 mm
Configuration B	4.5 mm
Configuration C	9.0 mm

5.3.5 Pivoting. For the four-point configurations, the loading member (inner span) or support member (outer span) shall be pivoted about a central transverse axis (z), to ensure equal distribution of load between the two bearing cylinders (see figure 4). A design which allows the lower bearing to pivot is also acceptable.

Independent pivoting of the bearing cylinders, about the specimen long axis (x), is not required provided that the specimens have been prepared according to the requirements of para 5.1.2 and providing that the bearing cylinders themselves are all parallel to each other within .015 mm. A suitable four point-1/4 point fixture is shown in figure 4. Figure 4 is only intended as a general schematic and is not a detailed design drawing.

Independent pivoting of the bearing cylinders is required if specimens are as-fired, heat treated, or oxidized in such a manner that the specimens are warped or an irregular surface precludes even load application. A suitable four point-1/4 point fixture for these types of specimens is shown in figure 5.

5.4 Procedure

5.4.1 General. Carefully place each specimen into the test fixture to preclude possible damage and to ensure alignment of the specimen in the fixture. There should be an equal amount of overhang of the specimen beyond the outer bearing and the specimen should be directly centered (in the z-direction) below the axis of the applied load. It is recommended that the contact between the load bearing and the specimen be inspected to ensure there is line and not point loading.

Mark the specimen so that the points of load application are shown, and also so that the tensile and compression faces can be distinguished. Carefully drawn pencil marks can suffice.

Put cushioning material around specimen to prevent pieces from flying out of fixtures upon fracture. Cotton or crumbled tissue paper are adequate for this purpose. This step will ensure operator safety and preserve primary fracture pieces.

Slowly apply the load at right angles to the fixture. The axis of load application (y) shall be centered over the specimen width (as shown in the right-hand views of figures 4 and 5) to within 0.10 mm. A small initial load is permissible to maintain alignment.

5.4.2 Loading rates. The crosshead rates will be chosen so that the strain rate upon the specimen shall of the order of 1.0×10^{-4} per second.

The fixture and test machine shall be relatively stiff compared to the specimen, such that most of the crosshead travel is imposed onto the specimen.

The strain rate for either the three- or the four point-1/4 point mode of loading is:

$$\dot{\varepsilon} = 6ds/L^2$$

Where $\dot{\varepsilon}$ is the strain rate, d is the specimen thickness, s is the crosshead speed, and L is the outer (support) span. This assumes all crosshead motion is transmitted to the load bearings.

Suggested crosshead speeds for the different testing configurations are given below:

Fixture A	0.2 mm/min
Fixture B	0.5 mm/min
Fixture C	1.0 mm/min

5.4.3 Breakload. Measure the breakload with an accuracy of ± 0.5%

5.4.4 Specimen dimension. Determine the thickness and width of each specimen to within .0025 mm. In order to avoid damage from gaging in the critical area make the measurement after the specimen has broken at a point near the fracture origin. Retain and preserve all primary fracture fragments for fractographic analysis if appropriate.

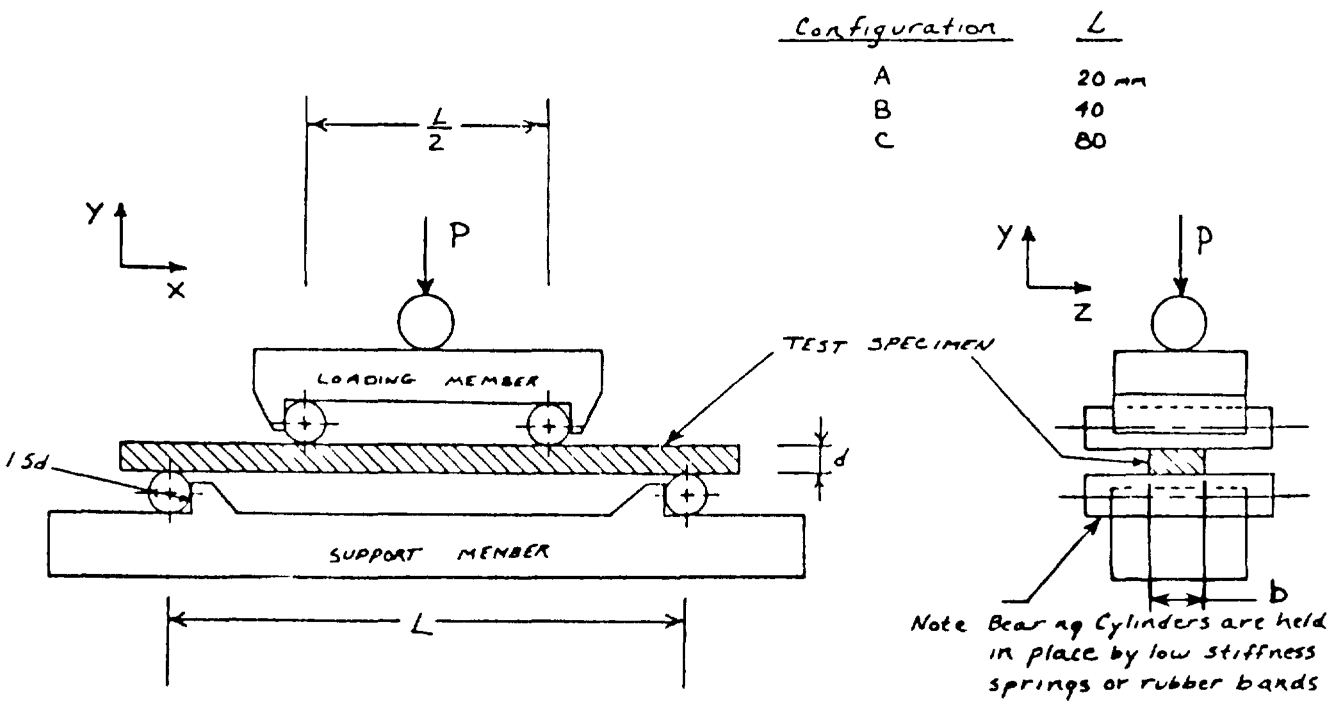

FIGURE 4. General schematic of a four-point 1/4 point fixture suitable for machined specimens.

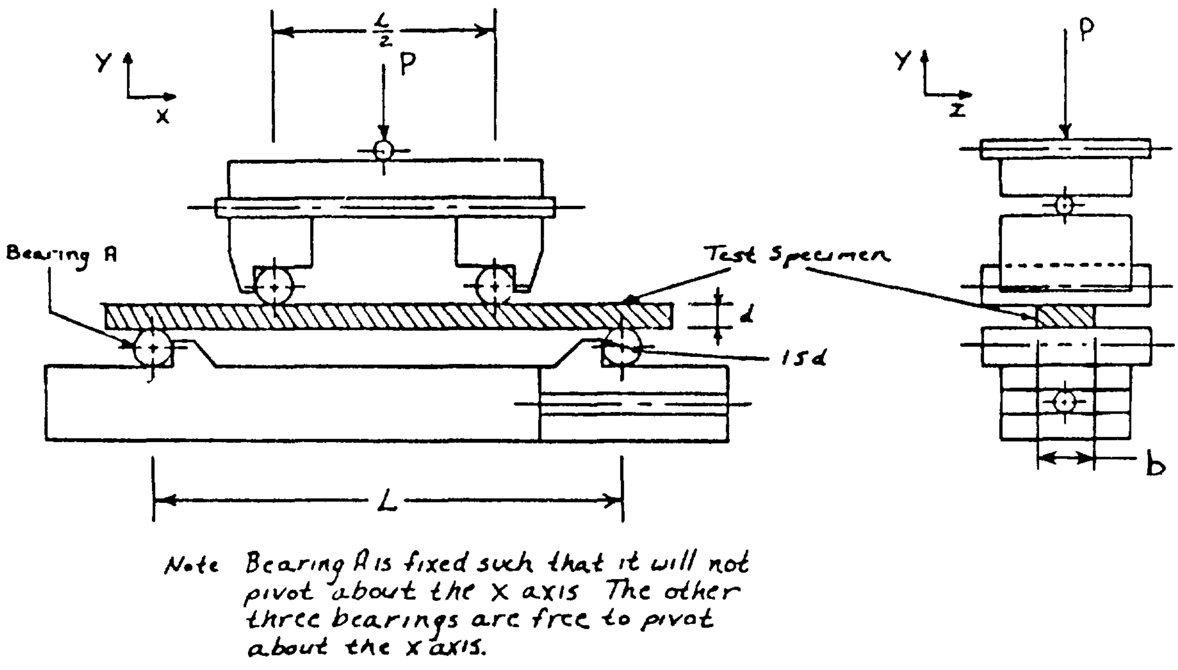

FIGURE 5. General schematic of a four-point 1/4 point fixture suitable for sintered, heat treated or oxidized specimens.

5.4.5 Number of specimens. A minimum of ten (10) specimens shall be required for the purpose of estimating the mean. A minimum of thirty (30) shall be necessary if estimates regarding the form of the strength distribution are to be reported (i.e., a Weibull modulus).

5.5 Calculations

The standard formula for the strength of a beam in four-point-1/4 point flexure is:

(1)
$$S = \frac{3\ PL}{4\ bd^2}$$

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

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

Where:

P = breakload
L = support span
b = specimen width
d = specimen thickness
(see figures 1, 2, and 5).

5.5.2 Comment: Formulas (1) and (2) are the common equations used when reporting the strength of a specimen. (It should be recognized however, that they do not necessarily give the stress that was acting directly upon the flaw which caused failure.) It is worth noting here that conventional Weibull analyses are based upon the maximum stress in the specimen at failure, Formulas (1) and (2).

5.6 Report - Test reports shall include:

(a) Test configuration and specimen size used

(b) All relevant material data including chemical composition, processing procedure, bulk density, vintage data or billet identification data. (Did all specimens cone from one billet?) As a minimum, the date of manufacture must be reported.

(c) Exact method of specimen preparation, including all stages of machining procedure.

(d) Heat treatment or exposure, if any.

(e) Strength of every specimen in MPa to three significant figures, mean and standard deviation of these strengths, machine crosshead rate, test environment (relative humidity, temperature, etc.), and individual specimen density (optional)

(f) Deviations from prescribed procedure.

(g) Number of specimens tested and number of specimens that were meant to be tested/machined but weren't due to failure during machining, handling or rejection of obvious defects, as well as the reason.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Uses of flexure data. Flexural testing is used for quality control, material development or material characterization purposes. In these instances, the issues of interest are consistency within a component, consistency between different components, consistency with time and identification of strength limiting defects. Adoption of standard test methods will help in these instances to insure that experimental error does not obscure the results. Controversy currently exists as to whether flexure data can be used for component design. The shortcomings of flexure data in this regard are well known and need not be elucidated here.Nevertheless, it is generally agreed that flexure data can only be used for design if all of the following conditions are met:

a. The defects that limit strength in the component are of a nature identical to those in the flexural specimens, and the defects are distributed in an identical manner.

b. The flexural specimen is substantially larger than the largest flaws.

c. Four point-1/4 point flexure is used to maximize the amount of material under stress.

d. An appropriate size effect analysis can be confirmed to be applicable.

e. A sufficiently large number of specimens are tested to allow a reasonable statistical interpretation of the results.

f. The experimental error is small in the flexure testing.

g. Flexure specimen preparation does not induce significant material alterations or residual stresses.

Conditions (a) and (b) indicate that comprehensive fractographic analysis is mandatory if flexure data is to be used for design. It is beyond the scope of this standard to prescribe fractographic procedure, however. The adoption of this standard should enhance the value of flexure data for design, but it will not guarantee success in this regard.

Time dependent phenomena such as stress corrosion or slow crack growth can influence strength tests. The effect can be meaningful even at room temperature for the relatively short times involved in testing. This must be considered if flexure tests are to be used for design purposes.

6.2 Test specimen sizes and crosshead rates. It is recognized that the strength of a ceramic can be dependent upon test specimen size. In general, the larger the specimen, the weaker it is likely to be. Such size influence can be analyzed via statistical theories of strength. (see AMMRC TR 82-20 and MTL TR 87-35) In the interests of permitting greater compatability of data, specific specimen and fixture sizes are required by the Standard. All data should be reported in a manner conducive to statistical analysis with respect to size effect analysis. It is beyond the scope of this standard to prescribe a size effect analysis however.

Crosshead rates have been selected such that the strain rates are approximately the same for the specimen - fixture configurations. The rates are relatively fast to minimize time dependent phenomena. Times to failure for typical ceramics will range from 3 to 30 seconds.

6.3 Specimen surface preparation. Test specimen surface preparation can have a pronounced effect upon flexural strength due to the introduction of machining flaws which can be strength limiting. Surface preparation can also lead to surface residual stresses. Machining damage incurred during specimen preparation can either be a random interfering factor, or an inherent part of the strength characteristic to be measured.

It is recognized that the final steps of surface preparation may not negate prior damage from intermediate machining stages. Therefore the entire process of specimen preparation is relevant and must be reported. Universal, optimum or standardized methods of surface preparation do not exist. Nevertheless, some minimum requirements are specified in the Standard.

Specimens with fired, oxidized, heat treated or specially machined surfaces may also be evaluated. Specimens with machined surfaces intended to simulate a specific component finish may also be used. If the required parallelism and overall specimen tolerances cannot be met, then sophisticated test fixtures may be required to minimize errors due to non-uniform load application along the bearing edges. This is particularly important for fired or sintered specimens.

6.4 Defect distribution in specimens. The flexure strength of brittle ceramic specimen is determined by the materials inherent resistance to fracture (fracture toughness) and the largest, most unfavorably located defect in the specimen. It is often assumed that there is only one type of flaw that limits strength in a group of specimens. Multiple defect distributions may, however, exist within specimens. Within a limited number of specimens, a genuine defect distribution may manifest itself as strength limiting in only a few specimens. Reporting so called extraneous data is required since such data may in fact be valid. The number of specimens required by this Standard has been established with the intent of determining not only reasonable confidence limits on strength distribution parameters, but also to discern multiple distributions. Appropriate fractography can provided added, independent information to enhance the confidence level in the determination or Weibull parameters.

6.5 Minimizing experimental errors. The specimen sizes, fixture sizes and testing requirements in this Standard have been carefully chosen so as to minimize experimental errors identified in AMMRC TR 82-20 and MTL TR 87-35.

6.6 Subject term (key word) listing.

Design, ceramics
Loading, three-point, ceramics
Loading, four-point, ceramics
Structural ceramics, testing
Surface preparation, specimens, ceramics

Custodians:
- Army - MR
- Navy - AS
- Air Force - 11

Review activities
- Army - GL
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3a. NAME OF SUBMITTING ORGANIZATION	4. TYPE OF ORGANIZATION (Mark one) ☐ VENDOR ☐ USER ☐ MANUFACTURER ☐ OTHER (Specify) ______
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5. PROBLEM AREAS

a. Paragraph Number and Wording

b. Recommended Wording

c. Reason/Rationale for Recommendation

6. REMARKS

7a. NAME OF SUBMITTER (Last, First, MI) – Optional	b. WORK TELEPHONE NUMBER (Include Area Code) – Optional
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