



Standard Test Method for Flexural Strength of Advanced Ceramics with Engineered Porosity (Honeycomb Cellular Channels) at Ambient Temperatures¹

This standard is issued under the fixed designation C 1674; 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 the determination of the flexural strength (modulus of rupture in bending) at ambient conditions of advanced ceramic structures with 2-dimensional honeycomb channel architectures.

1.2 The test method is focused on engineered ceramic components with longitudinal hollow channels, commonly called “honeycomb” channels. (See Fig. 1.) The components generally have 30 % or more porosity and the cross-sectional dimensions of the honeycomb channels are on the order of 1 millimeter or greater. Ceramics with these honeycomb structures are used in a wide range of applications (catalytic conversion supports (1),² high temperature filters (2, 3), combustion burner plates (4), energy absorption and damping (5), etc.). The honeycomb ceramics can be made in a range of ceramic compositions—alumina, cordierite, zirconia, spinel, mullite, silicon carbide, silicon nitride, graphite, and carbon. The components are produced in a variety of geometries (blocks, plates, cylinders, rods, rings).

1.3 The test method describes two test specimen geometries for determining the flexural strength (modulus of rupture) for a porous honeycomb ceramic test specimen (see Fig. 2):

1.3.1 *Test Method A*—A 4-point or 3-point bending test with user-defined specimen geometries, and

1.3.2 *Test Method B*—A 4-point-1/4 point bending test with a defined rectangular specimen geometry (13 mm × 25 mm × > 116 mm) and a 90 mm outer support span geometry suitable for cordierite and silicon carbide honeycombs with small cell sizes.

1.4 The test specimens are stressed to failure and the breaking force value, specimen and cell dimensions, and loading geometry data are used to calculate a nominal beam strength, a wall fracture strength, and a honeycomb structure strength.

1.5 Test results are used for material and structural development, product characterization, design data, quality control, and engineering/production specifications.

1.6 The test method is meant for ceramic materials that are linear-elastic to failure in tension. The test method is not applicable to polymer or metallic porous structures that fail in an elastomeric or an elastic-ductile manner.

1.7 The test method is defined for ambient testing temperatures. No directions are provided for testing at elevated or cryogenic temperatures.

1.8 The values stated in SI units are to be regarded as standard (IEEE/ASTM SI 10). English units are sparsely used in this standard for product definitions and tool descriptions, per the cited references and common practice in the US automotive industry.

1.9 *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:*³

C 373 Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products

C 1145 Terminology of Advanced Ceramics

C 1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature

C 1198 Test Method for Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio for Advanced Ceramics by Sonic Resonance

C 1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics

C 1259 Test Method for Dynamic Young’s Modulus, Shear

¹ This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.04 on Applications.

Current edition approved June 1, 2008. Published July 2008.

² The boldface numbers in parentheses refer to the list of references at the end of this standard.

³ 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.

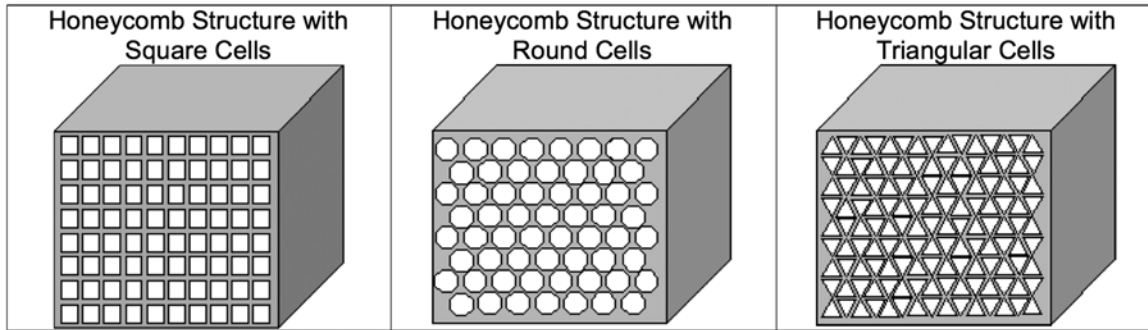
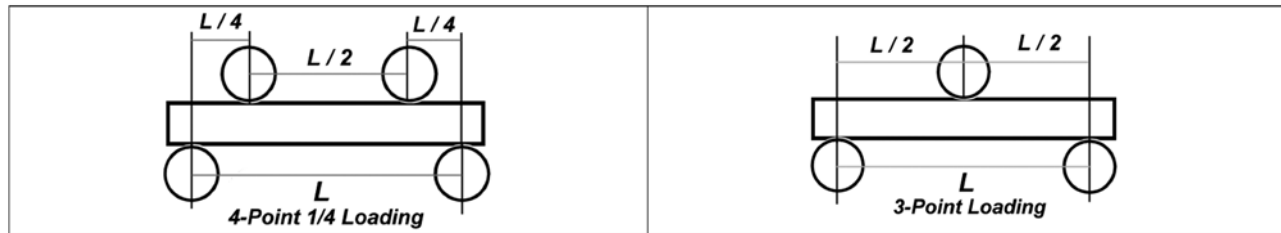


FIG. 1 General Schematics of Typical Honeycomb Ceramic Structures



L = Outer Span Length (for Test Method A, L = User defined; for Test Method B, L = 90 mm)

NOTE 1—4-Point-1/4 Loading for Test Methods A1 and B.

NOTE 2—3-Point Loading for Test Method A2.

FIG. 2 Flexure Loading Configurations

- Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration
- C 1292 Test Method for Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures
- C 1341 Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramic Composites
- C 1368 Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature
- C 1525 Test Method for Determination of Thermal Shock Resistance for Advanced Ceramics by Water Quenching
- C 1576 Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Flexural Testing (Stress Rupture) at Ambient Temperature
- D 2344/D 2344M Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates
- E 4 Practices for Force Verification of Testing Machines
- E 6 Terminology Relating to Methods of Mechanical Testing
- E 337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)
- IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI) (The Modern Metric System)

3. Terminology

3.1 The definitions of terms relating to flexure testing appearing in Terminology E 6 apply to the terms used in this test method. The definitions of terms relating to advanced

ceramics appearing in Terminology C 1145 apply to the terms used in this test method. Pertinent definitions, as listed in Terminology C 1145, Test Method C 1161, and Terminology E 6 are shown in the following section with the appropriate source given in brackets. Additional terms used in conjunction with this test method are also defined.

3.1.1 *advanced ceramic, n*—a highly engineered, high-performance, predominately nonmetallic, inorganic, ceramic material having specific functional attributes. **C 1145**

3.1.2 *breaking force, [F], n*—the force at which fracture occurs in a test specimen. **E 6**

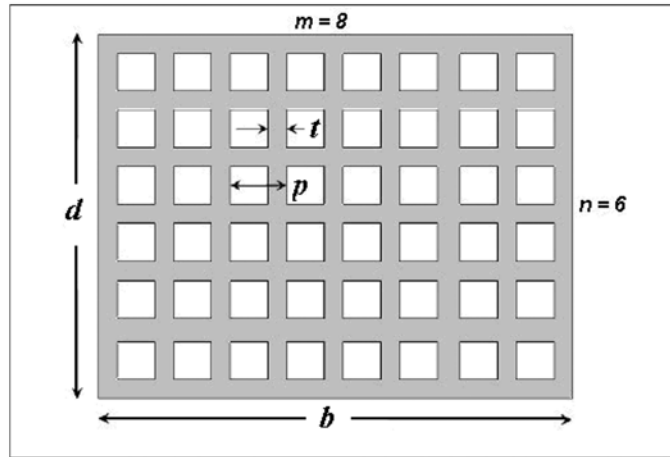
3.1.2.1 *Discussion*—In this test method, fracture consists of breakage of the test bar into two or more pieces or a loss of at least 50 % of the maximum force carrying capacity.

3.1.3 *cell pitch, (p), [L], n*—the unit dimension/s for the cross-section of a cell in the honeycomb component. The cell pitch *p* is calculated by measuring the specimen dimension of interest, the cell count in that dimension, and a cell wall thickness, where $p = (d - t)/n$. (See Fig. 3.)

3.1.3.1 *Discussion*—The cell pitch can be measured for both the height and width of the cell; those two measurements will be equal for a square cell geometry and uniform cell wall thickness and will be unequal for a rectangular cell geometry.

3.1.4 *cell wall thickness, (t), [L], n*—the nominal thickness of the walls that form the cell channels of the honeycomb structure. (See Fig. 3.)

3.1.5 *channel porosity, n*—porosity in the porous ceramic component that is defined by the large, open longitudinal honeycomb channels. Channel porosity generally has cross-sectional dimensions on the order of 1 millimeter or greater.



b = specimen width
 d = specimen thickness
 t = cell wall thickness
 p = cell pitch
 n = linear cell count (height)
 m = linear cell count (width)

FIG. 3 Schematic of Honeycomb Structure with Square Cells Showing Geometric Terms

3.1.6 *complete gage section, n* —the portion of the specimen between the two outer bearings in four-point flexure and three-point flexure fixtures.

3.1.6.1 *Discussion*—In this standard, the complete 4-point flexure gage section is twice the size of the inner gage section. Weibull statistical analysis only includes portions of the specimen volume or surface which experience tensile stresses.

3.1.7 *engineered porosity, n* —porosity in a component that is deliberately produced and controlled for a specific function and engineered performance. The porosity can be microporous (micron and submicron pores in the body of the ceramic) or macroporous (millimeter and larger) cells and channels in the ceramic. The porosity commonly has physical properties (volume fraction, size, shape, structure, architecture, dimensions, etc.) that are produced by a controlled manufacturing process. The porosity in the component has a direct effect on the engineering properties and performance and often has to be measured for quality control and performance verification.

3.1.8 *four-point-1/4 point flexure, n* —a configuration of flexural strength testing where a specimen is symmetrically loaded at two inner span locations that are situated one quarter of the overall span inside the span of the outer two support bearings. (See Fig. 2.)

C 1161

3.1.9 *fractional open frontal area, (OFA), [ND], n* —a fractional ratio of the open frontal area of the honeycomb architecture, calculated by dividing the total frontal area of the open channels by the full frontal area of the full size specimen, as a whole.

3.1.9.1 *Discussion*—The fractional open frontal area of the full size specimen can be calculated from the shape and dimensions of the cells and the wall thickness between cells. (See section 11.4 on Calculations.)

3.1.10 *fully-articulating fixture, n* —a flexure fixture designed to be used both with flat and parallel specimens and with uneven or nonparallel specimens. The fixture allows full independent articulation, or pivoting, of all load and support

rollers about the specimen long axis to match the specimen surface. In addition, the upper or lower roller pairs are free to pivot to distribute force evenly to the bearing cylinders on either side. (See Annex A1 for schematics and discussion.)

C 1161

3.1.11 *honeycomb cell density, n* —a characterization of the honeycomb cell structure that lists the number of cells per unit area and the nominal cell wall thickness. It is common practice in the automotive catalyst industry to use English units for this term, for example:

100/17 density = 100 cells/in.² with a cell wall thickness of 0.017 in.
 200/12 density = 200 cells/in.² with a cell wall thickness of 0.012 in.

3.1.12 *honeycomb cellular architecture, n* —an engineered component architecture in which long cylindrical cells of defined geometric cross-section form a porous structure with open channels in one dimension and a nominal closed-cell architecture in the remaining two dimensions. The cross sectional geometry of the honeycomb cells can have a variety of shapes—square, hexagonal, triangular, circular, etc. (See Fig. 1.)

3.1.12.1 *Discussion*—The cell walls in a honeycomb structure may have controlled wall porosity levels, engineered for filtering, separation effects, and mechanical strength.

3.1.13 *honeycomb structure strength, S_{HS} , [FL⁻²], n* —a measure of the maximum strength in bending of a specified honeycomb test specimen, calculated by considering the complex moment of inertia of the test specimen with its channel pore structure and adjusting for the open frontal area of the cellular specimen. (See Section 11 and Appendix X1.)

3.1.13.1 *Discussion*—The honeycomb structure strength gives a continuum strength that is more representative of the true continuum strength as compared to the nominal beam strength S_{NB} , particularly for specimens where the linear cell count in the smallest cross sectional dimension is less than 15.

3.1.13.2 *Discussion*—The honeycomb structure strength may be used to compare tests for specimens of different cell architectures and sizes and specimen dimensions. However, the calculated honeycomb structure strength is not representative of the failure stress in the outer fiber surface (the wall fracture strength) of the test specimen.

3.1.14 *linear cell count*, [ND], n —the integer number of cells along a given cross-sectional dimension of a test specimen. For the specimen width, the linear cell count is defined as m . For the specimen thickness dimension, the linear cell count is defined as n . (See Fig. 3.)

3.1.15 *modulus of elasticity*, [FL⁻²], n —the ratio of stress to corresponding strain below the proportional limit. **E 6**

3.1.16 *nominal beam strength*, S_{NB} , [FL⁻²], n —In honeycomb test specimens, a measure of the maximum strength in bending, calculated with the simple elastic beam equations using the overall specimen dimensions, disregarding the cellular/channel architecture, and making the simplifying assumption of a solid continuum in the bar. The nominal beam strength is not necessarily representative of the true failure stress in the outer fiber face, because it does not take the effect of channel porosity on the moment of inertia into account. (See Section 11 and Appendix X1.)

3.1.16.1 *Discussion*—The nominal beam strength is calculated without consideration of the dimensions, geometry/shape, cell wall thickness, or linear cell count of the cellular channel architecture in the test specimen. The nominal beam strength can be used for material comparison and quality control for flexure test specimens of equal size, comparable cell geometry, and equivalent loading configuration.

3.1.16.2 *Discussion*—For specimens where the minimum linear cell count is less than 15, the nominal beam strength should not be used for design purposes or material property characterization, because it is not necessarily an accurate approximation of the true failure stress (material strength) in the outer fiber face of the specimen.

3.1.17 *relative density (percent)*, n —a relative measurement of the density of a porous material, defined as the ratio (expressed as a percent) of the bulk density of the specimen to the true/theoretical density of the material composition. The relative density of the specimen is equal to 1 minus the fractional porosity, expressed as a percent. The relative density accounts for both channel porosity and wall porosity.

3.1.18 *semi-articulating fixture*, n —a flexure fixture designed to be used with flat and parallel specimens. The fixture allows some articulation, or pivoting, to ensure the top pair (or bottom pair) of bearing cylinders pivot together about an axis parallel to the specimen long axis, in order to match the specimen surfaces. In addition, the upper or lower pairs are free to pivot to distribute force evenly to the bearing cylinders on either side. (See Annex A1 for schematics.) **C 1161**

3.1.19 *three-point flexure*, n —configuration of flexural strength testing where a specimen is loaded at a location midway between the two outer support bearings. (See Fig. 2.) **C 1161**

3.1.20 *wall fracture strength*, S_{WF} , [FL⁻²], n —In honeycomb test specimens, the calculated failure stress in the outer fiber surface of the specimen, based on the true moment of

inertia of the test specimen, accounting for cell geometry, cell wall thickness, cell architecture, and linear cell count effects in the test specimen. (See Section 11 and Appendix X1.)

3.1.21 *wall porosity*, n —porosity found in the cell walls of the ceramic component, distinct from the open channel porosity. Wall porosity can exist in the ceramic walls in the form of closed and open pores, cracks, and interconnected microchannels, and it can have a wide range of dimensions (from 10 nanometers to 100 micrometers), depending on the ceramic microstructure and fabrication method.

4. Summary of Test Method

4.1 A test specimen with a honeycomb cellular structure and a rectangular cross section is tested as a beam in flexure at ambient temperature in one of the following geometries:

4.1.1 *Test Method A1 (4-Point Loading)*—The test specimen with a user-defined (see 9.2) rectangular geometry rests on two supports and is loaded at two points (by means of two loading rollers), each an equal distance from the adjacent support point. The inner loading points are positioned one quarter of the overall span away from the outer two support bearings. The distance between the loading rollers (the inner gage span) is one half of the complete gage (outer support) span. (See Fig. 2 and section 5.4.)

4.1.2 *Test Method A2 (3-Point Loading)*—The test specimen with a user-defined (see 9.2) rectangular geometry rests on two supports and is loaded by means of a loading roller midway between the two outer supports. (See Fig. 2 and section 5.4.)

4.1.3 *Test Method B (4-Point-1/4 Point Loading)*—The test specimen with a defined rectangular geometry (13 mm × 25 mm × >116 mm) rests on two supports (90 mm apart) and is loaded at two points (by means of two rollers), each an equal distance (22.5 mm) from the adjacent outer support point. (See Fig. 2 and section 5.5.)

4.2 Force is applied to the inner loading point/s and the specimen is deflected until rupture occurs on the outer surface and the specimen fractures and fails

4.3 Three different types of flexural strength (nominal beam strength, wall fracture strength, and honeycomb structure strength) of the specimen are calculated from the breaking force, the specimen dimensions, and the loading geometry, using the elastic beam equations. (See sections 5.7, 11, and Appendix X1 for a detailed description and discussion of the basis, use, and limitations of these three strength calculation formulas.)

5. Significance and Use

5.1 This test method is used to determine the mechanical properties in flexure of engineered ceramic components with multiple longitudinal hollow channels, commonly described as “honeycomb” channel architectures. The components generally have 30 % or more porosity and the cross-sectional dimensions of the honeycomb channels are on the order of 1 millimeter or greater.

5.2 The experimental data and calculated strength values from this test method are used for material and structural development, product characterization, design data, quality control, and engineering/ production specifications.

NOTE 1—Flexure testing is the preferred method for determining the nominal “tensile fracture” strength of these components, as compared to a compression (crushing) test. A nominal tensile strength is required, because these materials commonly fail in tension under thermal gradient stresses. A true tensile test is difficult to perform on these honeycomb specimens because of gripping and alignment challenges.

5.3 The mechanical properties determined by this test method are both material and architecture dependent, because the mechanical response and strength of the porous test specimens are determined by a combination of inherent material properties and microstructure and the architecture of the channel porosity [porosity fraction/relative density, channel geometry (shape, dimensions, cell wall thickness, etc.), anisotropy and uniformity, etc.] in the specimen. Comparison of test data must consider both differences in material/composition properties as well as differences in channel porosity architecture between individual specimens and differences between and within specimen lots.

5.4 Test Method A is a user-defined specimen geometry with a choice of four-point or three-point flexure testing geometries. It is not possible to define a single fixed specimen geometry for flexure testing of honeycombs, because of the wide range of honeycomb architectures and cell sizes and considerations of specimen size, cell shapes, pitch, porosity size, crush strength, and shear strength. As a general rule, the experimenter will have to define a suitable test specimen geometry for the particular honeycomb structure of interest, considering composition, architecture, cell size, mechanical properties, and specimen limitations and using the following guidelines. Details on specimen geometry definition are given in section 9.2.

5.4.1 Four-point flexure (Test Method A1) is strongly preferred and recommended for testing and characterization purposes. (From Test Method C 1161 section 4.5: “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, it is easier to adapt to high temperature and fracture toughness testing, and it is sometimes helpful in Weibull statistical studies. However, four-point flexure is preferred and recommended for most characterization purposes.”)

5.4.2 The three-point flexure test configuration (Test Method A2) may be used for specimens which are not suitable for 4-point testing, with the clear understanding that 3-point loading exposes only a very small portion of the specimen to the maximum stress, as compared to the much larger maximum stress volume in a 4-point loading configuration. Therefore, 3-point flexural strengths are likely to be greater than 4-point flexural strengths, based on statistical flaw distribution factors.

5.5 Test Method B (with a specified specimen size and a 4-point-¼ point flexure loading geometry) is widely used in industry for cordierite and silicon carbide honeycomb structures with small cell size (cell pitch ~2 mm). Test Method B is provided as a standard test geometry that provides a baseline specimen size for honeycomb structures with appropriate properties and cell size with the benefit of experimental repeatability, reproducibility and comparability. (See section 9.3 for details on Test Method B.)

NOTE 2—Specific fixture and specimen configurations were chosen for Test Method B to provide a balance between practical configurations and linear cell count effect limits and to permit ready comparison of data without the need for Weibull-size scaling.

5.6 The calculation of the flexure stress in these porous specimens is based on small deflection elastic beam theory with assumptions that (1) the material properties are isotropic and homogeneous, (2) the moduli of elasticity in tension and compression are identical, and (3) the material is linearly elastic. If the porous material in the walls of the honeycomb is not specifically anisotropic in microstructure, it is also assumed that the microstructure of the wall material is uniform and isotropic. To understand the effects of some of these assumptions, see Baratta et al (8).

NOTE 3—These assumptions may limit the application of the test to comparative type testing such as used for material development, quality control, and flexure specifications. Such comparative testing requires consistent and standardized test conditions both for specimen geometry and porosity architecture, as well as experimental conditions—loading geometries, strain rates, and atmospheric/test conditions.

5.7 Three flexure strength values (defined in Section 3 and calculated in Section 11) may be calculated in this test method. They are the nominal beam strength, the wall fracture strength, and the honeycomb structure strength.

5.7.1 *Nominal Beam Strength*—The first approach to calculating a flexure strength is to make the simplifying assumption that the specimen acts as a uniform homogeneous material that reacts as a continuum. Based on these assumptions, a nominal beam strength S_{NB} can be calculated using the standard flexure strength equations with the specimen dimensions and the breaking force. (See Section 11.)

5.7.1.1 A linear cell count effect (specimen size-cell count effect) has been noted in research on the flexure strength of ceramic honeycomb test specimens (6, 7). If the cell size is too large with respect to the specimen dimensions and if the linear cell count (the integer number of cells along the shortest cross-sectional dimension) is too low (<15), channel porosity has a geometric effect on the moment of inertia that produces an artificially high value for the nominal beam strength. (See Appendix X1.) With the standard elastic beam equations the strength value is overestimated, because the true moment of inertia of the open cell structure is not accounted for in the calculation.

5.7.1.2 This overestimate becomes increasingly larger for specimens with lower linear cell counts. The linear cell count has to be 15 or greater for the calculated nominal beam strength, S_{NB} , to be within a 10 % overestimate of the wall fracture strength S_{WF} .

NOTE 4—The study by Webb, Widjaja, and Helfinstine (6) showed that for cells with a square cross section a minimum linear cell count of 15 should be maintained to minimize linear cell count effects on the calculated nominal beam strength. (This study is summarized in Appendix X1.)

5.7.1.3 For those smaller test specimens (where the linear cell count is between 2 and 15), equations for wall fracture strength and honeycomb structure strength are given in Section 11. These equations are used to calculate a more accurate value for the flexure strength of the honeycomb, as compared to the calculated nominal beam strength.

5.7.2 *Wall Fracture Strength*, S_{WF} , is calculated using the true moment of inertia of the honeycomb architecture, based on the geometry, dimensions, cell wall thickness, and linear count of the channels in the honeycomb structure. The wall fracture strength is a calculation of the true failure stress in the outer fiber surface of the specimen. (Appendix X1 describes the calculation as cited in the Webb, Widjaja, and Helfinstine (6) report). Section 11 on calculations gives the formula for calculating the moment of inertia for test specimens with square honeycomb channels and uniform cell wall thickness.

NOTE 5—The moment of inertia formula given in Section 11 and Appendix X1 is only applicable to square cell geometries. It is not suitable for rectangular, circular, hexagonal, or triangular geometries. Formulas for those geometries have to be developed from geometric analysis and first principles.

5.7.3 *Honeycomb Structure Strength*, S_{HS} , is calculated from the wall fracture strength S_{WF} . This calculation gives a flexure strength value which is independent of specimen-cell size geometry effects. The honeycomb structure strength value can be used for comparison of different specimen geometries with different channel sizes. It also gives a flexure strength value that can be used for stress models that assume continuum strength. (See Appendix X1.) Section 11 on calculations gives the formula for calculating the honeycomb structure strength for test specimens with square honeycomb channels and uniform cell wall thickness.

5.7.4 The following recommendations are made for calculating a flexure strength for the ceramic honeycomb test specimens.

5.7.4.1 For flexure test specimens where the linear cell count is 15 or greater, the nominal beam strength S_{NB} calculation and the honeycomb structure strength S_{HS} are roughly equivalent in value (within 10 %). The nominal beam strength S_{NB} calculation can be used considering this variability.

5.7.4.2 For flexure test specimens where the linear cell count is between 5 and 15, the nominal beam strength S_{NB} calculation may produce a 10 to 20 % overvalue. The S_{NB} value should be used with caution.

5.7.4.3 For flexure test specimens where the linear cell count is less than 5, the nominal beam strength S_{NB} calculation may produce a 20 to 100 % overvalue. It is recommended that the honeycomb structure strength S_{HS} be calculated and used as a more accurate flexure strength number.

5.7.4.4 If specimen availability and test configuration permit, test specimens with a linear cell count of 15 or greater are preferred to reduce the specimen linear cell count effect on nominal beam strength S_{NB} to less than 10 %.

5.8 Flexure test data for porous ceramics will have a statistical distribution, which may be analyzed and described by Weibull statistics, per Practice C 1239.

5.9 This flexure test can be used as a characterization tool to assess the effects of fabrication variables, geometry and microstructure variations, and environmental exposure on the mechanical properties of the honeycombs. The effect of these variables is assessed by flexure testing a specimen set in a baseline condition and then testing a second set of specimens

with defined changes in geometry or fabrication methods or after controlled environmental exposure.

5.9.1 Geometry and microstructure variations would include variations in cell geometry (shape dimensions, cell wall thickness, and count) and wall porosity (percent, size, shape, morphology, etc.).

5.9.2 Fabrication process variations would include forming parameters, drying and binder burn-out conditions, sintering conditions, heat-treatments, variations in coatings, etc.

5.9.3 Environmental conditioning would include extended exposure at different temperatures and different corrosive atmospheres (including steam).

5.10 This flexure test may be used to assess the thermal shock resistance of the honeycomb ceramics, as described in Test Method C 1525.

5.11 The flexure test is not the preferred method for determining the Young's modulus of these porous structures. (For this reason, the deflection of the flexure test bar is not commonly measured in this test.) Young's modulus measurements by sonic resonance (Test Method C 1198) or by impulse excitation (Test Method C 1259) give more reliable and repeatable data.

5.12 It is beyond the scope of this standard to require fractographic analysis at the present time. Fractographic analysis for critical flaws in porous honeycomb ceramics is extremely difficult and of very uncertain value.

6. Interferences and Critical Factors

6.1 *Interferences and Critical Factors*—The critical experimental factors that need to be understood and controlled in this flexure test can be grouped into three categories—material factors, specimen factors, and experimental test factors. The major factors that need to be understood and controlled are:

6.1.1 Microstructure and critical flaw population which affect the material strength,

6.1.2 Specimen size, cell geometry, and cell size considerations,

6.1.3 Machining and surface preparation effects on the flaw population,

6.1.4 Crushing failure under the load points and shear failure in the body of the specimen, and

6.1.5 Environmental effects on the flaw population (slow crack growth and stress corrosion).

6.2 These factors are described in detail in Annex A2, covering the technical background and how the factors have to be controlled and managed.

6.3 One aspect of ceramic failure-flaw dependence that is commonly observed in tests of monolithic ceramics is a test specimen size effect, where larger ceramic specimens have statistically lower strengths than smaller specimens. This is because the probability of finding a larger critical flaw (with a lower fracture strength) increases in specimens with larger stressed volumes, as compared to small test specimens. This size dependence can be analyzed and modeled using Weibull statistical analysis (Practice C 1239). The Weibull specimen size effect may occur in ceramic honeycomb specimens and should be considered as a possible experimental variable. The Weibull specimen size effect is separate and distinct from the linear cell count effect (see 5.5-5.10, and Appendix X1) where

channel porosity has a major effect on the section modulus of specimens with low linear cell counts.

7. Safety

7.1 During specimen cutting, grinding, and preparation, there may be a hazard of dust exposure and inhalation with resulting skin irritation and/or respiratory distress. Appropriate dust elimination, reduction, and protection procedures and equipment should be determined and used.

7.2 During the conduct of this test method, the possibility of flying fragments of broken test specimens may be high. The brittle nature of advanced ceramics and the release of strain energy contribute to the potential release of uncontrolled fragments upon fracture. The containment of these fragments with a suitable safety shield is highly recommended.

7.3 *Waste Disposal*—Hazardous material must be disposed of in accordance with the applicable material safety data sheet and local laws and regulations.

8. Apparatus

8.1 *Testing Machine*—The flexure specimens shall be tested in a properly calibrated mechanical testing machine that can be operated at constant rates of cross-head motion over the range required with a suitable force sensor.

8.1.1 The error in the force measuring system shall not exceed $\pm 1\%$ of the maximum force being measured. Verify the accuracy of the testing machine in accordance with Practice E 4. The force-indicating mechanism shall be essentially free from inertial lag at the cross-head rate used. Equip the system with a means for retaining the readout of the maximum force as well as a record of force versus time.

8.2 Test fixtures are defined for Test Methods A1, A2, and B.

8.2.1 *Test Method A1: 4-Point-1/4 Point Loading*—The specimen rests on two supports and is loaded at two points (by means of two loading bearings), each an equal distance (one quarter of the overall span) from the adjacent outer support point. The distance between the loading bearings (the inner gage span) is one half of the complete gage (outer support) span. (See Fig. 2.) The Method B specimen thickness (d) determines the outer span dimension (L) of the test fixture. (See 9.2.) Test fixtures shall be wide enough to support the entire width of the selected specimen geometry.

8.2.2 *Test Method A2: 3-Point Loading*—The specimen rests on two supports and is loaded at one point (by means of one loading bearing), midway between the two outer support points. (See Fig. 2.) The Method B specimen thickness (d) determines the outer span dimension (L) of the test fixture. (See 9.2.) Test fixtures shall be wide enough to support the entire width of the selected specimen geometry. (Under some cases, e.g. very short specimens, three point loading may be easier to do than the four point loading.)

8.2.3 *Test Method B: 4-Point-1/4 Point Loading*—The outer support span is 90 mm; the inner span is 45 mm. Each inner span point is an equal distance (22.5 mm) from the adjacent outer support point. Test fixtures shall be wide enough to support the entire width of the selected specimen geometry. (See Fig. 2 and section 9.3.)

8.2.4 The test fixture shall be made of a material that is suitably rigid and resistant to permanent deformation at the applied forces and that will give a low system compliance so that most of the crosshead travel is imposed onto the test specimen.

8.2.5 Test fixtures with an articulating geometry shall be used to ensure that the fixtures produce even and uniform loads along the bearing-to-specimen surfaces. An articulated (full or semi) test fixture reduces or eliminates uneven loading caused by geometric variations of the specimen or misalignment of the test fixtures. A rigid test fixture is not permitted, because it cannot accommodate non-uniformity and variations in specimen dimensions. (See Annex A1 for a full description of semi-articulating and articulating fixtures.)

8.2.6 For articulating fixtures, the bearing cylinders shall be free to rotate or rock in order to relieve frictional constraints (with the exception of the center bearing cylinder in three-point flexure, which need not rotate).

8.3 *Support/Load Bearings*—In both the three-point and four-point flexure test fixtures, use contact bearings with rounded edges for support of the test specimen and for force application. The length of the contact bearings shall be at least 10 % greater than the specimen width. The bearing material should be hard enough to minimize abrasion of the bearing surfaces.

NOTE 6—It is recommended that the cylinders be made of a tool steel (case hardened to about HRC 60) or a ceramic with an elastic modulus between 200 and 400 GPa and a flexural strength no less than 275 MPa (40 ksi).

8.3.1 The bearing fixture design shall provide for precise and positive positioning of the bearings with no “slack” or “slop.” Roller bearings positioned against mechanical stops meet this requirement.

8.3.2 Ensure that the bearings have rounded bearing surfaces that are smooth and parallel along their length to an accuracy of ± 0.05 mm.

8.3.3 The diameter of the bearing shall be large enough to avoid point load concentrations that produce localized crushing. Cylindrical bearings commonly have diameters that are 50 to 150 % of the specimen thickness.

NOTE 7—If the specimen has low through-thickness compressive strength such that the failure initiates at the bearing contact surface, the cylinder diameter should be increased to reduce the force concentration and prevent crushing at the contact/load points. Alternately the support span can be increased to reduce the force required for fracture.

8.3.4 Position the outer support bearing cylinders carefully such that the support span distance is accurate to a tolerance of $\pm 1/2\%$.

8.3.5 Position the inner support bearing carefully such that the inner support span distance is accurate to a tolerance of $\pm 1/2\%$.

8.3.6 The inner support bearings for the four-point configurations shall be properly centered and aligned with respect to the outer support bearings to an accuracy of $\pm 1/2\%$ of the outer span length. The center bearing for the three-point configuration shall be centered between the outer support bearings to an accuracy of $\pm 1/2\%$ of the outer span length.

8.3.7 Bearings should be replaced when observable abrasive wear occurs on the bearing surface.

8.4 If failure cracks initiate at the point of contact between the load bearings and wall stubs/asperities on the test specimen, a narrow strip of compliant, cushioning material may be placed between the specimen and the full length of the loading bearings/edges.

NOTE 8—Cushioning materials that have been used are PTFE polymer (Teflon®) gasket material, thick compliant construction paper, or thin polyurethane foam.

8.5 *Deflection Measurement*—Deflection of honeycomb specimens is not commonly measured in flexure tests. If deflection needs to be measured, refer to Test Method C 1341, section 7.4 for guidance and directions.

8.6 *Direct Strain Measurement*—Bonded strain gages are not commonly used for testing porous ceramics because the bonding material can become a significant pore filler, i.e., stiffener, changing the local strain response.

8.7 The test system may include an environmental chamber for testing the specimens under controlled conditions of humidity, temperature, and atmosphere.

8.8 *Data Acquisition*—At the minimum, obtain an autographic record of the applied force as a function of time for the specified cross-head rate. Either analog chart recorders or digital data acquisition systems may be used for this purpose, although a digital record is recommended for ease of subsequent data analysis. Ideally, an analog chart recorder or plotter or an electronic display should be used in conjunction with the digital data acquisition system to provide an immediate display and record of the test as a supplement to the digital record. Ensure that the recording devices have an accuracy of 0.1 % of full scale and that the digital acquisition rate is such to capture changes in force of 0.2 % of full scale.

8.9 *Dimension-Measuring Devices*—Micrometers and other devices used for measuring linear dimensions shall be accurate and precise to at least one half the smallest tolerance to which the individual dimension is required to be measured. For the purposes of this test method, measure the cross-sectional dimensions to within 0.02 mm with a measuring device with an accuracy of 0.01 mm.

8.10 *Calibration*—Calibration of equipment shall be provided by the supplier with traceability maintained to the National Institute of Standards and Technology (NIST). Recalibration shall be performed with a NIST-traceable standard on

all equipment on a six-month interval; with adjustment, replacement or repair of calibrated components; or whenever accuracy is in doubt.

9. Specimen Geometry and Preparation

9.1 *General Guidance*—The test specimen should be large enough so that linear cell count effects on the moment of inertia are minimized in the specimen (as described in Appendix X1). It is recommended that the linear cell count be 15 or greater in the thickness and width dimensions for a honeycomb flexure specimen (see Fig. 3), so that the simpler nominal beam strength equation (S_{NB} , Section 11) can be used to calculate an accurate flexure strength.

NOTE 9—The linear cell count requirement of 15 is based on work and analysis done with cordierite honeycombs with small square cell sizes (Refs (6, 7) and Appendix X1). Different materials and different cell geometries may require different minimum linear cell counts.

NOTE 10—The linear cell count can be measured directly by counting the cells in a given dimension. It can also be calculated by dividing the smallest specimen dimension (width or thickness) for the flexure specimen by the mean cell pitch in that dimension. (See Fig. 4.) (Examples: A 12-mm specimen thickness and a 2.4-mm cell pitch gives a linear cell count of 5. A 36-mm specimen thickness and a 2.4-mm cell pitch give a linear cell count of 15.)

NOTE 11—Test specimens with linear cell counts of less than 15 can be used, but those specimens will require the use of the more complex honeycomb structure strength equation (S_{HS} , Section 11) to calculate an accurate flexure strength.

9.2 *Test Method A*—It is not possible to define a single fixed specimen geometry for flexure testing of all ceramic honeycombs, because of the wide range of honeycomb architectures and considerations of specimen size requirements, cell shapes, cell pitch and size, porosity size, crush strength, and shear strength. As a general rule, the experimenter will have to define a suitable test specimen geometry for the particular honeycomb structure of interest (composition, architecture, cell size, mechanical properties) using the following guidelines.

9.2.1 The user shall define a specimen geometry for Test Method A that gives valid test data (failure in the gage section without major crushing failure or shear failure). Geometry A1 is used for 4-point-1/4 point bending; Geometry A2 is used for 3-point bending. As a guideline, use the following considerations to define a suitable initial test geometry. (See Figs. 3 and 4.)

9.2.1.1 The specimen thickness (d) should be at least $5 \times$ the cell pitch, p , giving a linear cell count of 5 or greater. If

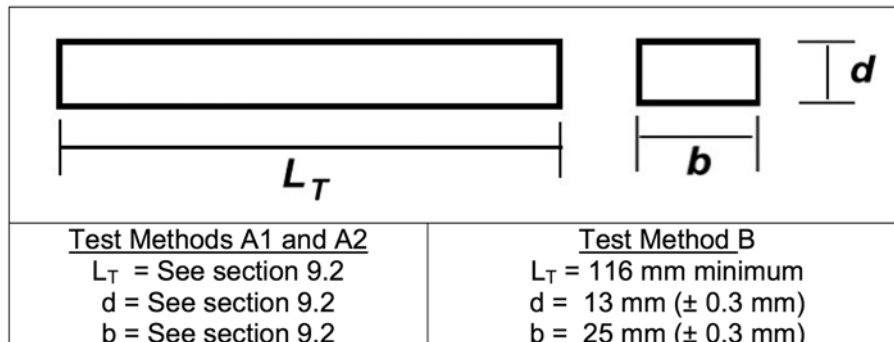


FIG. 4 Test Specimen Geometry (Test Methods A1, A2 and B)

possible, a linear cell count of 15 is recommended. The specimen should be sized to give the maximum linear cell count possible within experimental constraints.

9.2.1.2 The width (b) of the specimen should be $\geq 1 \times$ the defined specimen thickness (d).

9.2.1.3 The outer-span for the flex test should be long enough so that the span-to-depth ratio (L/d , where L is the outer load span and d is the specimen thickness/depth) is at least 6:1 for 4-point testing and 4:1 for 3-point testing.

9.2.1.4 The total length of the specimen (L_T) shall be the length of the defined outer load span plus at least $2 \times$ the thickness of the test specimen. ($L_T = L_{test\ span} + 2d$; this added length reduces the possibility of end chip-off.)

9.2.1.5 *Example*— A honeycomb test specimen has a cell pitch of 5 mm and will be tested in 4-point bend, requiring a span-to-depth ratio of ≥ 6 . Minimum and preferred dimensions for the test specimen are:

Thickness $\geq 5 \times$ mean cell pitch; $15 \times$ preferred
 Minimum Thickness (d) = 5 mm \times 5 = 25 mm.
 Preferred Thickness (d) = 5 mm \times 15 = 75 mm

Width $\geq 1 \times$ defined thickness
 Width (b) for 25 mm thickness (d) \geq 25 mm.
 Width (b) for 75 mm thickness (d) \geq 75 mm

Outer Span $\geq 6 \times$ defined thickness
 Outer Span (L) for 25 mm thickness = 25 mm \times 6 = 150 mm
 Outer Span (L) for 75 mm thickness = 75 mm \times 6 = 450 mm

Specimen Length \geq Outer Span + $2 \times$ specimen thickness
 Specimen length for 25 mm thickness = 150 mm + 50 = 200 mm
 Specimen length for 75 mm thickness = 450 mm + 150 = 600 mm

9.2.2 For Test Method A the cross-sectional dimensional tolerances for the specimen are $\pm 2 \%$ of the width and thickness. Recommended parallelism tolerances on the four longitudinal faces are $\pm 2 \%$ of the width and thickness along the total length. (Specimens that do not meet these parallelism tolerances shall be tested with the fully-articulating loading fixture.)

9.2.3 If the defined specimen geometry does not produce valid results (tension or compression failure in the gage section), adjust the specimen geometry and the fixture geometry (span length, bearing radii, etc.) to produce the desired failure modes.

9.3 Test Method B uses a specifically-defined specimen geometry that is widely used in industry for cordierite and silicon carbide honeycomb structures with small cell size (cell

pitch ~ 2 mm). This geometry is suitable for specimens with moderate crush strength and a mean cell pitch of 2.4 mm or less. Test Method B is provided as a standard test geometry that provides a baseline specimen size for experimental repeatability, reproducibility and comparability for honeycomb structures with appropriate mechanical properties, honeycomb architecture, and cell size.

9.3.1 The Method B test specimen has nominal dimensions of: 13 mm thick (d) by 25 mm wide (b) by a minimum of 116 mm long (L_T), as shown in Fig. 4. The specimen cross section dimensions may be slightly increased or decreased from 13 mm \times 25 mm so that the specimen contains an integer number of cells in each cross sectional dimension and continuous outer surface walls. For Test Method B specimens, the dimensional tolerances for width and thickness along the test bar are ± 0.3 mm. The recommended parallelism tolerances on the four longitudinal faces are ± 0.3 mm along the length of the specimen. (Specimens that do not meet these parallelism tolerances shall be tested with a fully-articulating loading fixture.)

9.4 Specimen Preparation:

9.4.1 The test specimens may be formed directly to the required finished dimensions or they may be cut from sheets, plates, or formed shapes. Test specimens may have to be cut in multiple orientations to evaluate directional anisotropy effects (axial, radial/tangential, 45° , etc.) in the cell architecture of the honeycomb body. (See Fig. 5.)

9.4.2 There may be spatial variations in material properties and honeycomb architecture within a given component. If those variations need to be assessed, a cutting plan should be developed for the test specimens taken from a given component. The cutting plan should be followed and reported, giving the location and orientation of each test specimen cut from a given component.

9.4.3 Test specimens shall be cut to the desired test dimensions using an appropriate method that produces the required nominal dimensional tolerances and parallel faces and minimizes surface damage. The ease of cutting will depend on the material hardness and brittleness, the cell geometry, and the cell wall thickness.

NOTE 12—Large specimens can be cut by hand with a fine, i.e. 14-teeth/inch tooth, hack saw blade handled in a pulling mode. Small specimens, or small channel honeycombs, can be machine cut with a

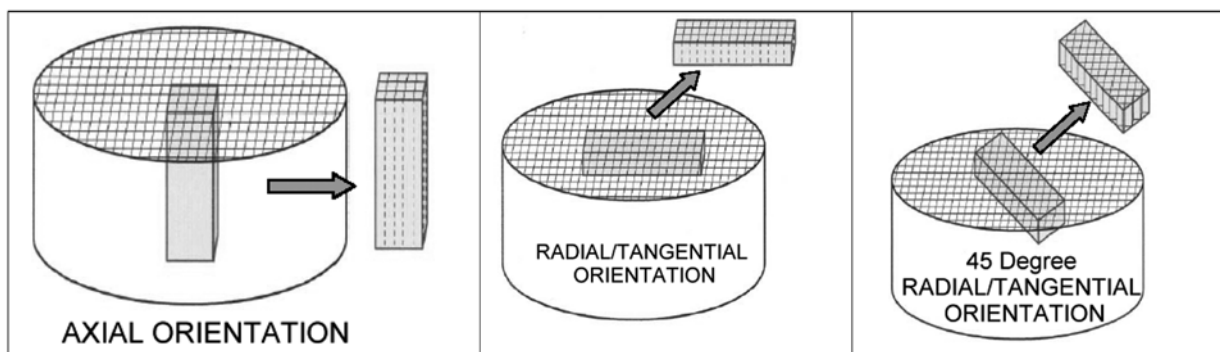


FIG. 5 Axial and Radial/Tangential Test Orientations for Honeycomb Specimens

32-tooth/inch thin blade band saw.

9.4.4 Wet cutting/grinding may have deleterious effects on certain ceramic compositions that are subject to moisture attack. Such specimens require dry cutting or finishing.

9.4.5 Cutting should be done in such a way as to minimize debris which may collect in the open channels. Specimens may be ultrasonically cleaned to remove trapped debris, if water will not degrade or otherwise affect the ceramic composition. All specimens should be thoroughly dried after washing.

9.4.5.1 *Surface Finishing*—Since most honeycomb flexure tests are done to evaluate the strength of the as-prepared wall surface, any surface finishing should be considered as to how it will change the surface condition. Ideally, honeycomb test specimens should be cut and finished so that smooth, undamaged internal walls (with no ribs or wall stubs) act as the bearing surfaces of the test specimen. But it is highly likely that any grinding/sanding/finishing operation that completely removes the wall ribs/stubs will touch the as-prepared wall surface and introduce flaws that will reduce the strength of the specimen.

NOTE 13—These surface finishing guidelines are written for honeycombs configurations with square or rectangular cross-sections, where cutting produces relatively continuous outer surfaces on the test specimen. They are not applicable to specimens cut on a 45° radial orientation or to honeycombs with circular, hexagonal, or triangular cell shapes. Those test configurations will not produce test specimens with continuous outer surface walls. Such specimens will present special challenges in specimen positioning and cushioning materials to produce controlled force application.

9.4.6 To avoid any damage to the pristine wall surfaces of the cut specimens, the specimen should be carefully sanded by hand so that there are short (<25 % of cell wall thickness) residual wall ribs/stubs that will crush at low force levels. (See Fig. 6.) The sand paper shall have a grit of 400 or finer. The small residual stubs at the contact points will not significantly affect the breaking fracture force. (There may be slight incremental force drops during the test, as the stubs crush.)

9.4.6.1 Grind/sand the test specimen surfaces parallel to the induced tensile stresses, that are parallel with the long axis of the test specimen.

9.5 *Specimen Characterization and Documentation*—Depending on the purpose of the test, the available sample and specimen information, and practical limitations of budget and

time, the following characteristics of the test specimens should be considered for report documentation by reference and/or direct testing.

9.5.1 All available pedigree information on the sample and specimens—source information, configuration, manufacturer’s code and lot #, manufacturing date, fabrication methods, history, and other information for traceability and identification

9.5.2 Sample and specimen physical characteristics and architecture—composition, phases, and glass content; relative density (porosity fraction); fractional open frontal area; mean cell dimensions, cell shape/symmetry, cell wall thickness, cell wall condition (high density or microporous), and coatings.

9.5.3 More complete descriptions (anisotropy factors, wall porosity characteristics, statistics on critical dimensions, non-destructive evaluation results, additional density measurements, cutting diagrams, conditioning treatments, etc.) may be available or necessary for interpretation of data. If available from the producer or by independent analysis, such information may be reported as supplementary data.

9.5.4 If the wall fracture strength and honeycomb structure strength will be calculated, a cell wall thickness dimension (t) and cell pitch (p) must be determined or assumed for the test specimens. Cell wall thickness can be measured by caliper measurements for large cell specimens; smaller cell specimens may require microscopic image measurements. It is recommended that an average of multiple (>10) measurements on several bars is used to determine a cell wall thickness (t) value for the strength calculations. Cell pitch can be measured by caliper measurements or by direct count in a measured dimension.

NOTE 14—Examples: A 12-mm specimen thickness and a linear cell count of 5 gives a 2.4-mm cell pitch. A 75-mm specimen thickness and a linear cell count of 15 gives a 5-mm cell pitch.

9.5.5 Cell wall thickness and pitch measurements can be done prior to test specimen fabrication or done directly on the test specimens.

9.5.6 Specimen bulk density can be determined by measurement of mass and overall dimensions and direct calculation. Specimen bulk density, theoretical density and open porosity (water absorption) fraction for a representative test specimen may be measured by Archimedes density measurements (Test

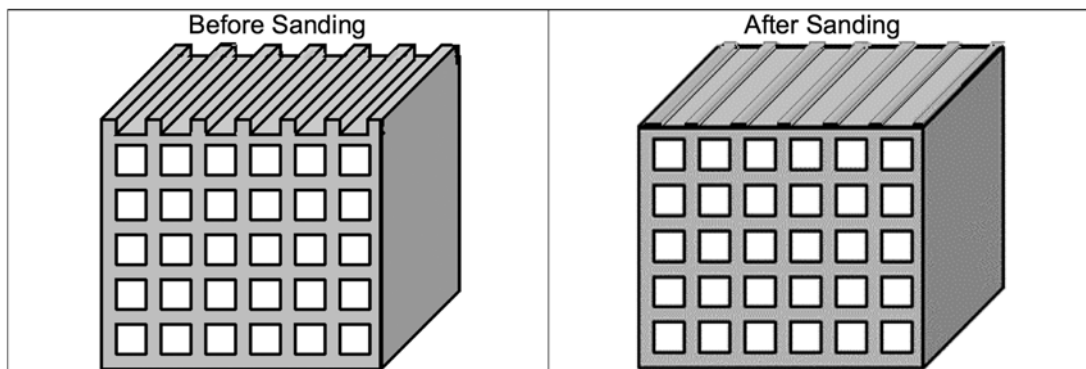


FIG. 6 Wall Rib/Stub Reduction by Gentle Sanding

Method C 373). Wall porosity can also be characterized for size and distribution by mercury porosimetry and gas adsorption measurements.

9.5.7 *Specimen Inspection*—Specimens shall be inspected prior to testing, considering two kinds of anomalies.

9.5.7.1 *Nonuniformity in Dimensions*, marked by warp, twist, or bowing and determined by visual and instrument inspection. If such dimensional variation is detected, fully articulated fixtures may be necessary to avoid point stresses during testing.

9.5.7.2 *Surface Discontinuities and Anomalies*, such as surface preparation damage, large cracks, obvious pores or pits, broken walls and webs, or anomalous surface roughness on the inner and outer gage specimen faces.

9.5.7.3 A 5× to 10× hand loupe or a low power stereo binocular microscope may be used to aid in the examination. If such surface anomalies are detected, the features could be photographed or sketched for the test report, in case the particular specimen produces censored or invalid test results.

9.6 Exercise care in the storage and handling of finished test specimens to avoid the introduction of random scratches and cracks that could be fracture sources.

9.7 In addition, consider pre-test storage of the specimens in controlled environments or desiccators to avoid uncontrolled humidity/environmental effects on the specimens prior to testing.

9.8 Specimens can be marked in pencil or ink on the specimen ends for identification, but not in the middle of the high stress gage section.

9.9 *Number of Specimens*—The number of specimens to be tested depends on the objectives of the test and historical results from similar samples. The following examples are initial guidelines with the understanding that the results of the testing may indicate more tests are needed to obtain appropriate confidence limits.

9.9.1 For spot checking, proof testing, and minimal baseline characterization, three (3) valid test specimens are the minimum for a baseline mean value, understanding that the standard deviation for such low specimen counts will not have a high confidence value.

9.9.2 For material and process development, design work, and baseline statistical purposes, a minimum of 10 specimens shall be required for the purpose of determining a statistical mean with an acceptable standard deviation.

9.9.3 For strength distribution assessment (for example, a Weibull analysis per Practice C 1239), a minimum of 30 specimens shall be recommended. The actual population size needed will depend on whether the resultant statistical analysis generates the desired confidence limits. More than 30 specimens are recommended if multiple-flaw populations are expected or observed.

10. Test Preparation and Procedure

10.1 Verify the load cell identification, capacity, and calibration. Verify the controlled operation of the mechanical test system and the data collection system.

10.2 *Cross-Head Rate*—The cross-head rate shall be chosen so that times-to-failure for typical specimens will range from 30 to 50 seconds. (Be aware that some of these porous

materials will have a large scatter in their strength values and the times-to-failure may vary greatly.) Displacement rate control is the most common control method in this type of test. It is assumed that the fixtures are relatively rigid, except for articulation, and that most of the testing-machine crosshead travel is imposed as strain on the test specimen.

10.2.1 For Test Method A (User-Defined Geometry) the crosshead rate for a $0.1 \times 10^{-3}/s$ strain rate for either the three- or four-point- $1/4$ point mode of loading is calculated as follows:

$$\epsilon = 6ds/L^2 \quad (1)$$

$$s = \epsilon L^2/(6d) \quad (2)$$

where:

ϵ = desired nominal strain rate = $0.1 \times 10^{-3} s^{-1}$,

d = specimen thickness, (mm),

s = crosshead rate, (mm/s), and

L = outer (support) span (mm).

10.2.2 For Test Method A, test one to five of the test specimens at the first selected crosshead rate and check for failure in 30 to 50 seconds. If failure occurs too rapidly or too slowly, adjust the crosshead rate for additional tests to produce the desired average specimen failure in 30 to 50 seconds.

10.2.3 For Test Method B (Fixed Geometry), the initial recommended crosshead displacement rate is 0.01 mm/s (0.5 mm/min), which can be adjusted to meet the test time requirements (and noted on the test report).

10.2.4 The 30 to 50 second time-to-failure is recommended as a baseline experimental parameter to generate consistent results in the test community. It is known that many of these materials may be stress rate sensitive, i.e., exhibit slow crack growth characteristics. Faster stressing rate tests may be necessary, if slow crack growth occurs at these test conditions.

NOTE 15—The sensitivity of flexural strength to stressing rate may be assessed by testing at two or more rates. See Test Method C 1368.

10.3 *Specimen Dimensions and Mass*—Measure the thickness and width of each specimen to an accuracy of 0.1 mm or 1 % whichever is greater. It is recommended that machined surfaces be measured mechanically, using a flat, anvil-type micrometer. Measure the specimens with care to prevent surface damage. In all cases the resolution of the instrument shall meet the requirements specified in section 8.9.

10.3.1 It may be of value to determine the bulk density of each test specimen to check for significant variations in density and porosity between specimens. Measure the length of the specimen to an accuracy of 1 mm and the mass of the specimen to an accuracy of 0.01 g or 1 % whichever is greater. Use the mass and linear dimensions to calculate the bulk density of each test specimen.

10.4 At the start of each test sequence, assemble and align the appropriate flexure test fixture in the required testing configuration. Measure the support point locations so that outer and inner spans are within $\pm 1/2$ % of the required position and alignment values.

10.5 Set and check the cross-head displacement rate on the test machine (Test Method A = user-defined; Test Method B = 0.5 mm/min, unless historical data suggests otherwise).

10.6 Set and check the data collection system for data logging.

10.7 Determine and record the ambient temperature and the relative humidity in accordance with Test Method E 337 or equivalently accurate instrumentation.

10.8 *Specimen Loading*—The specimen should be carefully placed in the test fixture and directly centered below the axis of the applied force with an equal amount of overhang of the specimen beyond the outer bearings.

NOTE 16—Use Teflon® gasket material or compliant paper as a cushioning layer at contact points, if the test specimens develop failures attributed to local asperities and residual wall stubs, as described in section 8.4. Use new interlayer cushion strips for each test specimen.

10.9 If specimen center-point displacement is measured, position, check, and zero the displacement measuring system at the point when the specimen is initially contacted.

10.10 The specimen may be preloaded at an accelerated cross-head rate to remove the slack from the load train. The amount of preload will depend on the material and flexure specimen geometry, and therefore must be determined for each situation. Preload shall not exceed 5 % of the breaking force.

10.11 Check the contact between the bearings and the specimen to ensure even-line loading across the width of the specimen. You may mark the sides of the specimen with a felt tip pen below the support points at the neutral plane to identify the points of force application. The ends of the specimen may be marked to indicate the tensile face of the specimen. The marks can be used as a reference to locate the point of fracture.

10.12 *Conducting the Test*—Initiate the data acquisition. Start the force application. Continue the test until the specimen breaks into two or more pieces or there is a drop of 50 % from the maximum observed force.

NOTE 17—The elastic energy in the ceramic honeycomb flexure test is relatively low and it is often observed that the crack will arrest without generating two separate pieces.

10.13 Measure and record the breaking (maximum) force. After test completion, return the test machine to the original position and stop the data acquisition system. Carefully remove the fractured specimen and any fragments from the test fixture, and retain them for later inspection.

NOTE 18—Check for and brush off any specimen fragments from the contact bearings/rollers in preparation for the next test.

10.14 Check for and record the general location of the fracture initiation (tensile face or compressive face; center, left/right of center, out-of-span) on the test specimen. This may or may not be identifiable, depending on the mode of fracture.

10.15 Fractographic examination of the failed test specimen is not commonly done for porous ceramics, because the critical flaws are not easily identified on the fracture surface.

10.16 *Censored Tests:*

10.16.1 In 4-point- $\frac{1}{4}$ point testing, failure may occur outside the inner gage/span section. Note and record data from such a failure as censored data, i.e., a lower bound to the strength. Censored data shall be reported as such, but not used to calculate average values, unless appropriate censored data statistics are used.

10.16.2 In 3-point tests, failure is likely to occur to the left and right of the point on the tensile face below the center loading point (the point of maximum stress). If failure occurs

more than 10 % of the support span away from the center point, the failure shall be noted and recorded as censored data. Censored data shall be reported as such, but not used to calculate average values, unless appropriate censored data statistics are used.

10.17 *Invalid Tests*—There are two specimen failure mechanisms that invalidate the flexure test.

10.17.1 The first mechanism is by crushing under the bearing/loading points without generating a failure crack within the load span.

10.17.2 The second mechanism is by shear failure near the neutral axis between the outer load point and the inner load point, which are the regions of high shear stress.

10.17.3 Invalid test data shall be reported as such, but not used to calculate average values. However, these must be considered as censored (lower bound) strengths and appropriately analyzed. These invalid tests should suggest how the test setup should be changed (such as spans adjusted or interleaving materials used on the contacts) to produce valid tests.

10.18 To complete a required statistical sample (for example, $n = 10$) for purposes of generating an average strength, test one replacement specimen for each specimen that failed in an invalid or censored manner.

11. Calculation

11.1 The nominal beam strength S_{NB} in four-point- $\frac{1}{4}$ point flexure test is calculated using the standard 4-point- $\frac{1}{4}$ point elastic beam flexure formula as follows:

$$S_{NB} (4 pt) = \frac{Mc}{I} = \frac{3PL}{4bd^2} \quad 4\text{-Point Bend} \quad (3)$$

where:

- S_{NB} = nominal beam strength (MPa),
- M = applied bending moment,
- c = distance between the neutral axis and the outer fiber ($d/2$),
- I = the moment of inertia of the rectangular cross section,
- P = breaking force (N),
- L = outer (support) span (mm),
- b = specimen width (mm), and
- d = specimen thickness (mm).

11.2 The nominal beam strength in a three point flexure test is calculated using the standard 3-point flexure elastic beam formula as follows:

$$S_{NB} (3 pt) = \frac{3PL}{2bd^2} \quad 3\text{-Point Bend} \quad (4)$$

11.3 Eq 3 and 4 shall be used for calculating and reporting the nominal beam strength S_{NB} for the test results.

NOTE 19—The equations for nominal beam strength (4-point and 3-point) can be used with any honeycomb architecture or specimen test orientation.

NOTE 20—It should be recognized however, that Eq 3 and 4 do not necessarily give the stress that was acting directly upon the flaw that caused failure in the outer fiber surface per Appendix X1.

NOTE 21—The conversion between pounds per square inch (psi) and Megapascals (MPa) is included for convenience: (145.04 psi = 1 MPa; therefore, 10 000 psi = 10 ksi = 68.95 MPa).

11.4 The wall fracture strength S_{WF} is calculated using the true moment of inertia which adjusts for the hollow sections of the multiple square section channels in the test specimen tested in an axial orientation.

11.4.1 The true moment of inertia I_T for the test bar with square section channels and uniform cell wall thickness in axial flexure is calculated in Eq 5 (from Webb, Widjaja, and Helfinstine (6)).

NOTE 22—**Caution:** This I_T equation is for test specimens with square section, uniform cell wall thickness honeycomb channels that are tested in the axial orientation. The equation cannot be used for specimens tested in radial/tangential orientations. The equation cannot be used for specimens with rectangular, circular, hexagonal, or triangular cell geometries.

$$I_T = \frac{bd^3}{12} - \left[\frac{mm(p-t)^4}{12} + \frac{mp^2(p-t)^2}{4} \sum_i^n (2i-n-1)^2 \right] \text{ (Square channels)} \quad (5)$$

where:

- I_T = true moment of inertia, considering open channels (mm^4),
- b = specimen width (mm),
- d = specimen thickness (mm),
- m = linear cell count across the specimen width b ,
- n = linear cell count across the specimen thickness d ,
- p = cell pitch for the square cell (mm), and
- t = cell wall thickness for the square cell (mm).

NOTE 23—Fig. 3 illustrates the terms b , d , m , n , p , and t .

NOTE 24—There is an assumption in this equation that the cell squares have sharp square corners. Major rounding of the cell corners (>10 % of cell pitch—estimated) may introduce a calculation error which needs to be considered.

NOTE 25—Alternative equations for the moment of inertia may be derived and used for honeycomb architectures with other section channel geometries (round, hexagonal, or triangular) or for non-uniform cell wall thicknesses, provided that they are consistent with the definitions in section 3.1.10.

11.4.2 The wall fracture strength S_{WF} is calculated for 4-pt- $\frac{1}{4}$ pt and 3-pt bending as:

$$S_{WF} (4 \text{ pt}) = \frac{Mc}{I_T} = \frac{PLd}{16I_T} \quad \text{4-Point} \quad (6)$$

$$S_{WF} (3 \text{ pt}) = \frac{Mc}{I_T} = \frac{PLd}{8I_T} \quad \text{3-Point} \quad (7)$$

where:

- S_{WF} = wall fracture strength (MPa),
- M = applied bending moment,
- c = distance between the neutral axis and the outer fiber ($d/2$),
- I_T = true moment of inertia (mm^4), calculated from Eq 5,
- P = breaking force (N),
- L = outer (support) span (mm), and
- d = specimen thickness (mm).

11.5 The honeycomb structure strength S_{HS} for the honeycomb specimen is calculated by adjusting the wall fracture strength for the open frontal area of the honeycomb specimen.

NOTE 26—This gives a flexure strength value that is a more accurate continuum strength, independent of specimen size and linear cell counts. The calculation has value for comparing data for different specimen sizes and for using finite element stress models when the cell structure detail is

left out of the model for simplicity. (See Appendix X1.)

11.6 The honeycomb structure strength S_{HS} (for 4-point and 3-point geometries) is calculated in Eq 8.

NOTE 27—**Caution:** The term $[(p-t)/p]^2$ is for test specimens with square section, uniform cell wall thickness honeycomb channels that are tested in the axial orientation. The equation cannot be used for specimens tested in radial/tangential orientations. The equation (with the $[(p-t)/p]^2$ term) cannot be used for specimens with rectangular, circular, hexagonal, or triangular cell geometries.

$$S_{HS} = S_{WF}(1 - OFA) = S_{WF} \left(1 - \left(\frac{p-t}{p} \right)^2 \right) \quad (8)$$

where:

- S_{HS} = honeycomb structure strength (MPa),
- S_{WF} = wall fracture strength (MPa),
- OFA = fractional open frontal area of the specimen,
- p = cell pitch for the square cell (mm), and
- t = cell wall thickness for the square cell (mm).

NOTE 28—Alternative equations for the honeycomb structure strength may be derived and used for honeycomb architectures with other section channel geometries (round, hexagonal, or triangular) or for non-uniform cell wall thicknesses, provided that they are consistent with the definitions in section 3.1.10.

11.7 The calculated values for nominal beam strength and the honeycomb structure strength have to be used within the following guidelines. (See Appendix X1.)

11.7.1 For flexure test specimens where the linear cell count is 15 or greater, the nominal beam strength S_{NB} calculation and the honeycomb structure strength S_{HS} are roughly equivalent in value (within 10 %). The nominal beam strength S_{NB} calculation can be used considering this variability.

11.7.2 For flexure test specimens where the linear cell count is between 5 and 15, the nominal beam strength S_{NB} calculation may produce a 10 to 20 % overvalue. The S_{NB} value should be used with caution.

11.7.3 For flexure test specimens where the linear cell count is less than 5, the nominal beam strength S_{NB} calculation may produce a 20 to 100 % overvalue. It is recommended that the honeycomb structure strength S_{HS} be calculated and used as a more accurate flexure strength number.

11.7.4 If specimen availability and test configuration permit, test specimens with a linear cell count of 15 or greater are preferred to reduce the specimen-cell size effect on nominal beam strength S_{NB} to less than 10 %.

11.8 A force versus time graph should be viewed or printed to verify that specimen fracture occurred in an appropriate length of time (30 to 50 seconds). The force-time graph will also show any transient force drops that would indicate minor wall or asperity crushing during force application.

12. Report

12.1 The test report shall include and document the following test information (experimental data, sample data, specimen results data). Mandatory information is listed with an M in parenthesis, (M). Other information is optional, depending on the purpose and use of the test data, the objective of the report, and the availability of the defined information.

12.2 *Test and Experimental Data*—The report shall include the following information for the test set. Any significant

deviations from the procedures and requirements of this test method shall be noted in the report.

12.2.1 Date and location of testing and the name of test operator. (M)

12.2.2 Geometry of the flexure test specimen (include engineering drawing, if necessary) specifying if it is Test Method A with user-defined dimensions or Test Method B with fixed dimensions. (M)

12.2.3 Description of the loading fixture geometry (Test Method A1 or A2 or B) to include inner and outer support span dimensions, the type of articulation and support bearings, and the nominal span-to-depth ratio. If a commercial fixture was used, give the manufacturer and model number for the system. (M)

12.2.4 Test control mode (force, displacement, or strain control) and actual test rate (force rate, displacement rate, or strain rate). Experimental strain rate shall also be reported, if appropriate, in units of s^{-1} . (M)

12.2.5 Test environment including ambient temperature and relative humidity (Test Method E 337 or equivalent), test temperature, and environmental conditions (for example, ambient air, or test chamber with controlled-humidity, dry nitrogen, argon, etc.) (M)

12.2.6 Type and configuration of the test machine (include drawing or sketch, if necessary). If a commercial test machine was used, the manufacturer and model number are sufficient for describing the test machine.

12.2.7 The force capacity and accuracy/resolution of the load cell and manufacturer and model number.

12.2.8 The method of the data collection, specifying the data collection rate, accuracy, and resolution.

12.2.9 Type, configuration, and resolution of displacement measurement equipment, if used (include drawing or sketch if necessary). If commercial displacement devices were used, provide the manufacturer and model number.

12.3 Test Sample Data:

12.3.1 All available and relevant sample pedigree data shall be reported, including sample traceability information. (Sample traceability and other information could be a simple reference code to a database or to another report, or could include such information as vintage data or billet identification data, the lot #, and date the material was manufactured. For commercial materials, include such items as source information, configuration, manufacturer's code, fabrication methods, history, and other information useful for traceability and identification.) (M)

12.3.2 Other useful and available sample information to document the ceramic material—composition and phase/s, amorphous and crystalline content, nominal density, and wall condition (density and porosity values). (M)

12.3.3 Other useful and available sample information to document specimen architecture and the major cell characteristics—cell shape/symmetry, mean cell dimensions (e.g., cell pitch (p)), cell wall thickness (t), and fractional open frontal area. (A drawing or photograph of the cell architecture is recommended.) (M)

12.3.4 Heat treatments, coatings, or pre-test exposures, if any, applied either to the original as-processed material or to the as-prepared flexure specimens. (M)

12.3.5 A description of any anisotropy (architectural and microstructure) in the individual test specimens. (M)

12.3.6 If available, any other information on the sample characteristics (such as wall porosity characteristics, statistics on critical dimensions, additional density measurements, forming/processing/sintering conditions, etc.) which may be available or necessary for interpretation of data.

12.3.7 Description of the method of test specimen preparation including all stages of cutting/machining, grinding, and finishing. Include a cutting diagram showing the location of individual samples as cut from the original as-fabricated specimen.

12.4 Test Results Data:

12.4.1 Number (n) of specimens tested validly (for example, fracture in the gage section) for each test series. In addition, report the total number of specimens tested (n_T) to provide an indication of the test success rate for the particular specimen geometry, material, and test apparatus. (M)

12.4.2 The test orientation (axial, radial/tangential) of the specimens as a group.

12.4.3 Mean, standard deviation, and coefficient of variation for each test series of the following test measurements (reported to three significant figures): (M)

12.4.3.1 The specimen dimensions (length, width, and thickness) and mass, if measured.

12.4.3.2 The breaking force, P (N).

12.4.3.3 Calculated nominal beam strength, S_{NB} (MPa).

12.4.3.4 Wall fracture strength, S_{WF} (MPa), if calculated.

12.4.3.5 Honeycomb structure strength, S_{HS} (MPa), if calculated.

12.4.4 *Individual Specimens*—The report shall include the following information for each specimen tested (reported to three significant figures):

12.4.4.1 *Specimen Dimensions*—Length, width, and thickness in units of mm. For multiple measurements on a single specimen, the averaged value should be recorded and used for strength calculation of that specimen. Report mass of specimens if measured.

12.4.4.2 The test orientation (axial, radial/tangential) of the individual specimens.

12.4.4.3 The breaking force P (N) and the calculated nominal beam strength S_{NB} (MPa).

12.4.4.4 Wall fracture strength S_{WF} (MPa), if calculated.

12.4.4.5 Honeycomb structure strength S_{HS} (MPa), if calculated.

12.4.4.6 Plot of the entire force-time curve for each specimen, if available.

12.4.4.7 *Failure Mode*—If it can be determined, describe if the specimen failed on the tensile or compressive face, outside the gage section, or if it failed by support point crushing or shear.

12.4.4.8 The results of the general examination of each specimen, described in 9.5.7, that is, nonuniformity in major

dimensions—warp, twist, and bowing; surface discontinuities such as large pores, observable cracks, anomalous surface roughness, etc.

12.4.4.9 The results, if available, of any nondestructive evaluations of the test specimens.

12.4.5 Mean (\bar{X}) and standard deviation (SD) values are calculated with the following equations:

$$\text{Mean} = \bar{X} = \left(\sum_{i=1}^n X_i \right) / n \quad (9)$$

$$\text{Standard Deviation} = SD = \sqrt{\left[\sum_{i=1}^n \left(X_i - \bar{X} \right)^2 \right] / \left(n - 1 \right)} \quad (10)$$

where:

X_i = the measured value for a test specimen, and

n = the total number of valid tests.

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

13. Precision and Bias

13.1 The flexure strength of a porous ceramic is not a deterministic quantity, but will vary from one specimen to another, because of variations in the flaw population in the specimens. There will be an inherent statistical scatter in the test results. Weibull statistics can model this variability, as discussed in Practice C 1239. This test method has been devised so that the test precision is high and the bias low compared to the inherent variability of these porous materials.

13.2 *Experimental Errors*—The experimental errors in the flexure test for monolithic ceramic have been thoroughly analyzed and documented in Ref (8). The tolerance specifications in this test method for honeycomb ceramics have been chosen such that the individual variations in dimensions (b and d) for the specimen cross section are on the order of 2 %. Tolerance variations in loading geometry dimensions (L) are ½ % each. An initial propagation of errors calculation under the worst case scenario shows that the total geometric configuration test error is nominally less than 10 % in the nominal beam strength equation. (The most sensitive dimensional measurement is the thickness (d) of the specimen dimension, because it is the smallest dimension and is used in the equation to the second (d^2) power.

NOTE 29—The calculation of the moment of inertia required for I_T and S_{WF} assumes perfect square cells, etc., so that variations in the cell wall thickness and cell pitch of the test specimen will introduce calculation errors. The use of fixtures that do not meet the articulation requirements will also increase the experimental error and deviation of the test values. A more detailed error analysis is being planned and will be conducted to determine sensitivity of the test method to variations in the experimental, material, and specimen parameters.

13.3 Round-robin tests are being planned and will be conducted to determine the repeatability and reproducibility of this test method.

14. Keywords

14.1 advanced ceramics; catalysts; cellular structure; filters; flexural strength; four-point flexure; honeycomb; honeycomb structure strength; nominal beam strength; porosity; three-point flexure; wall fracture strength

ANNEXES

(Mandatory Information)

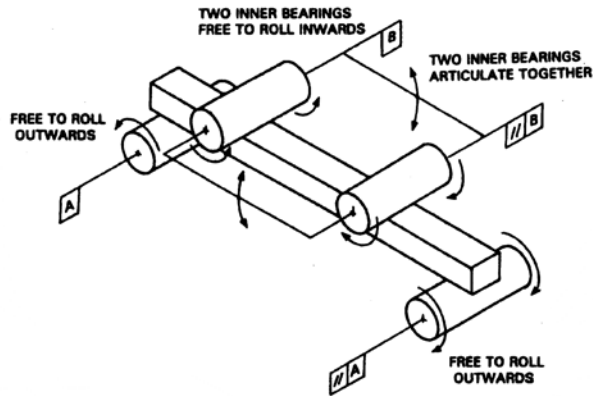
A1. SEMI- AND FULLY-ARTICULATING FOUR-POINT FIXTURES

A1.1 The schematic figures in this annex illustrate semi-articulated and fully-articulated degrees of freedom in the test fixtures. Note that these diagrams are illustrative of what degree of freedom is needed, not necessarily of how to generate those degrees of freedom.

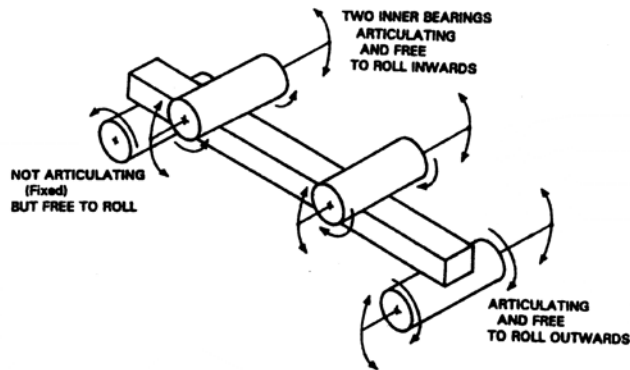
A1.2 Fully-articulated fixtures shall be used for specimens

that are not suitably parallel or flat. (Fully-articulated fixtures may also be used for well-machined specimens.) Semi-articulating fixtures shall only be used with flat and parallel specimens. (See Fig. A1.1.)

A1.3 Examples of designs for semi-articulating and articulating test fixtures are shown in Figs. A1.2 and A1.3.

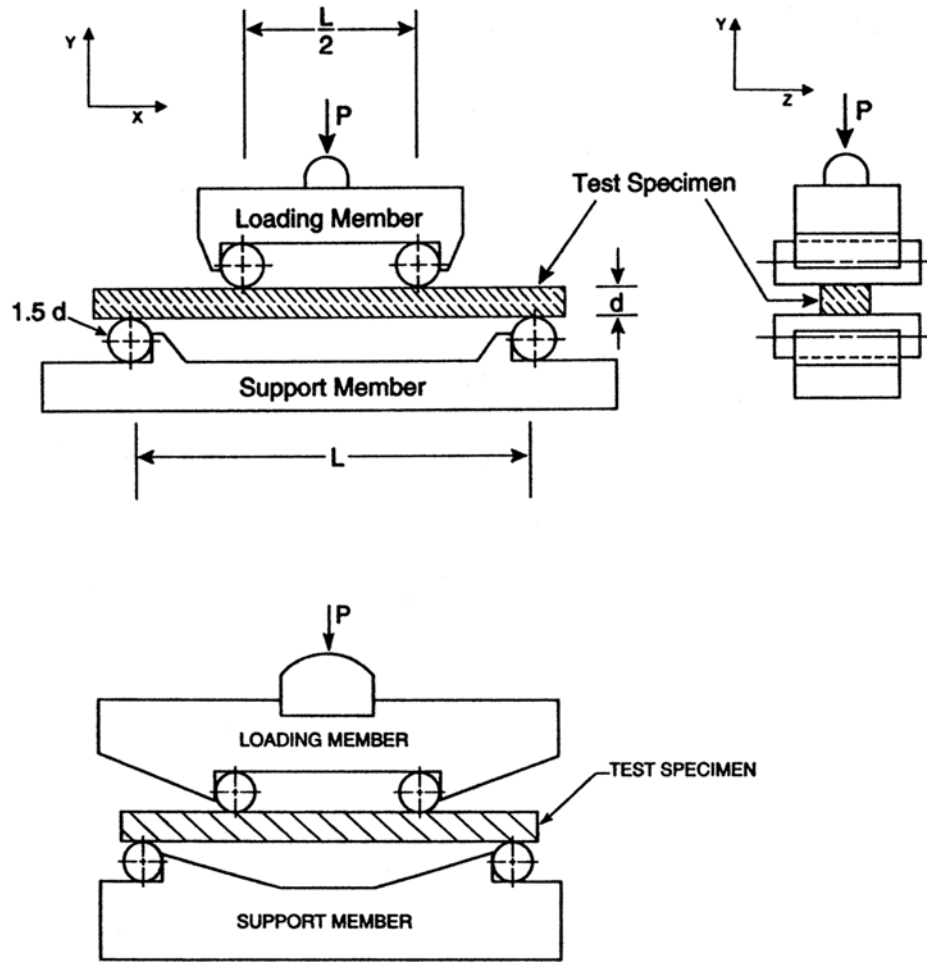


- (a) **SEMI-ARTICULATING.** The two inner bearings are parallel to each other and the bearings are free to roll inwards. The two outer bearings are parallel to each other, and the bearings are free to roll outwards. The two inner bearings can articulate together as a pair to match the specimen top surface. (Alternatively, the two bottom bearings can articulate to match the bottom surface.)



- (b) **FULLY-ARTICULATING.** The two inner bearings are free to roll inwards, and they can independently articulate to match the specimen top surface. The two outer bearings are free to roll, and one bearing can articulate to match the specimen bottom surface.

FIG. A1.1 Four-Point Flexure Fixture



NOTE 1—Bearing cylinders are held in place by low stiffness springs, rubber bands or magnets.

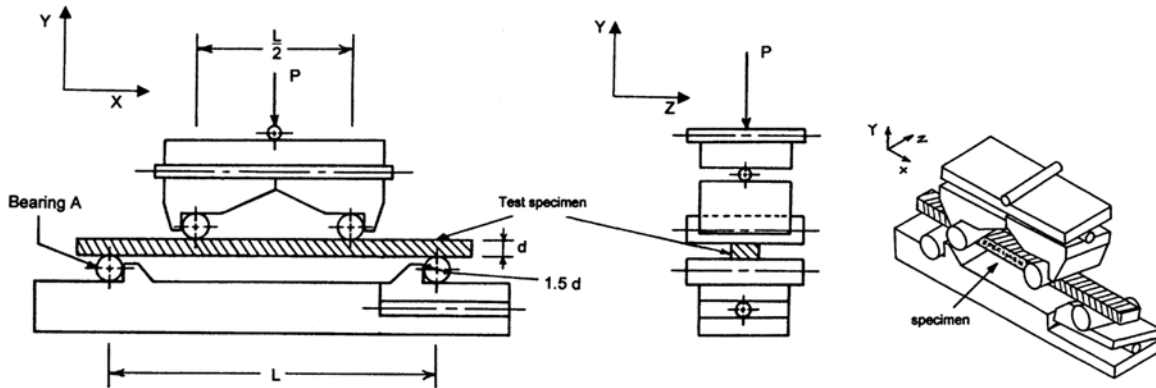
NOTE 2—Configuration:

Specimen Geometry A 4-Point: $d = 13 \text{ mm}$, $L = 90 \text{ mm}$, $L/2 = 45 \text{ mm}$.

Specimen Geometry B 4-Point: Specimen dimensions determined per section 9.3.

NOTE 3—Force is applied through a ball which permits the loading member to tilt as necessary to ensure uniform loading.

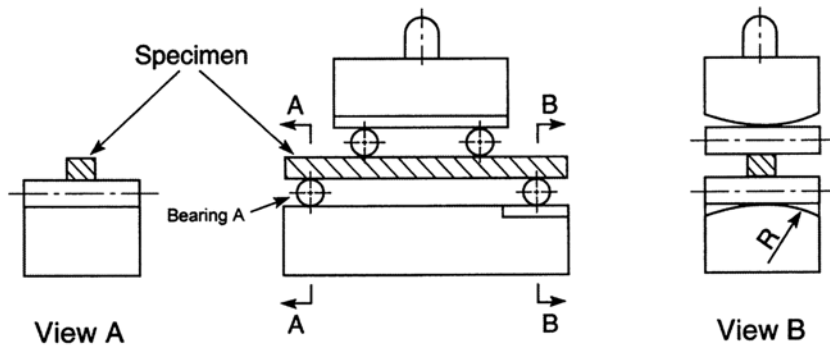
FIG. A1.2 Schematics of Two Semi-Articulating Four-Point Fixtures Suitable for Flat and Parallel Specimens



Note 1:
Configuration

Configuration	L, mm
A	20
B	40
C	80

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.



NOTE 1—Bearing cylinders are held in place by low stiffness springs, rubber bands, or magnets.

NOTE 2—Configuration:

Specimen Geometry A 4-Point: $d = 13 \text{ mm}$, $L = 90 \text{ mm}$, $L/2 = 45 \text{ mm}$.

Specimen Geometry B 4-Point: Specimen dimensions determined per section 9.3.

NOTE 3—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. A1.3 Schematics of Two Fully-Articulating Four-Point Fixtures Suitable Either for Twisted or Uneven Specimens, or for Flat and Parallel Specimens

A2. CRITICAL EXPERIMENTAL AND INTERFERENCE FACTORS IN FLEXURE TESTING OF HONEYCOMB CELLULAR CERAMICS

INTRODUCTION

The critical experimental and interference factors that need to be understood and controlled in this flexure test can be grouped into three categories—material factors, specimen factors, and experimental test factors. All of these factors need to be understood and controlled for successful tests that are accurate, precise, and repeatable.

A2.1 Material Factors:

A2.1.1 *Material Strength and Flaw Population*—The local tensile stress at failure for a ceramic material is dependent on two material factors: (1) the inherent resistance to fracture of

the material in its composition and phase content and (2) the size and severity of the flaws at which cracking and failure initiate in the microstructure. Inherent variations in the size and

geometry of these flaws cause a natural scatter in test results within and between sets of test specimens.

A2.1.1.1 This flaw-dependent strength effect is also applicable to ceramics with engineered porosity, where the porosity features in the ceramic walls and surfaces may be the strength-determining flaws. There is an assumption that the porous ceramic has a linear elastic response within the walls.

A2.1.2 *Material Strength and Microstructure*—Test specimens with identical compositions but with different phases and microstructures (depending on starting materials, forming conditions, and sintering parameters) may very well have different flaw populations and show distinct differences in flexure strength. In particular, the amount and type of porosity in the ceramic walls may be a determining factor for the mechanical strength. Higher density, low porosity walls may be stronger than walls with extensive porosity. It is recommended that the material be well characterized for composition, phase content, microstructure, wall porosity, and flaw population. That information is necessary to interpret and understand the mechanical properties of the test specimens.

A2.2 *Specimen Finishing, Condition, Geometry, and Size Factors:*

A2.2.1 *Machining and Finishing Damage*—Machining and surface preparation of test specimens is a critical factor in flexure testing of porous ceramics. Aggressive or coarse machining practices can produce surface cracks and damage which may have a pronounced effect on flexural strength. This machining damage imposed during specimen preparation can be either a random interfering factor, or an inherent part of the strength characteristic to be measured. Machining damage can be avoided by careful selection of and attention to cutting and finishing methods. Universal or standardized test methods of specimen and surface preparation for porous ceramics do not exist and suitable machining practices will have to be defined and documented for different types of compositions and porosity architectures. It should be understood that final machining steps may or may not negate machining damage introduced during the early course or intermediate machining operations (see section 9.4).

A2.2.2 *Nonuniform Specimens*—If a flexure test specimen is not uniform in dimension and shape, the warping, twisting, and/or bowing may prevent the specimen from properly aligning in the loading fixture. Misaligned specimens will not be uniformly loaded and point forces will develop which may cause premature fracture. Ideally, the test specimens will be uniform in dimension and shape, but this is not always possible. Therefore, the preferred method to ensure uniform loading is to use articulated and semi-articulated fixtures which will conform to specimen variability. (See Section 8 and Annex A1.)

A2.2.3 *Specimen Linear Cell Count Effect* (see Appendix X1)—A specimen linear cell count effect has been noted in research on the flexure strength of ceramic honeycomb test specimens. Flexure test specimens with a low linear cell counts (<15) have calculated nominal beam strength values (using standard elastic beam equations) which are overestimated, because the true moment of inertia of the open cell structure is not accounted for in the calculation. This overestimate be-

comes increasingly larger for smaller specimens with lower linear cell counts. For those smaller specimens (where the linear cell count is between 2 and 15), equations for wall fracture strength and honeycomb structure strength are given in Section 11. These equations are used to calculate a more accurate value for the flexure strength of the honeycomb.

A2.2.4 *Weibull Specimen Size Effects*—One aspect of ceramic failure-flaw dependence that is commonly observed in tests of monolithic ceramics is a test specimen size effect, where larger ceramic specimens have statistically lower strengths than smaller specimens. This is because the probability of finding a larger critical flaw (with a lower fracture strength) increases in specimens with larger stressed volumes, as compared to small test specimens. This size dependence can be analyzed and modeled using Weibull statistical analysis (Practice C 1239). This Weibull specimen size effect may occur in ceramic honeycomb specimens and should be considered as a possible experimental variable. This Weibull specimen size effect is separate and distinct from the linear cell count effect (see A2.2.3 and Appendix X1) where channel porosity has a major effect on the section modulus of specimens with low linear cell counts.

A2.3 *Experimental Test Factors:*

A2.3.1 *Load Point Crushing*—Crushing directly at the load points will occur if local contact stresses exceed the crush strength of the material. If crushing does occur, the contact stresses can be reduced by increasing the outer load span, by increasing the contact radius of the load bearings, or with the addition of a thin sheet of compliant material (see sections 8.3 and 8.4).

A2.3.2 *Shear Failure*—In rare cases shear failure can occur in the body of the ceramic if shear stresses are too high at the neutral axis and the specimen fails in shear, before it fails in flexure. A shear failure is an invalid test. If shear failures do occur for a given specimen geometry, the shear failures can be reduced by increasing the span-to-depth ratio of the test specimens (see section 9.3).

NOTE A2.1—If the shear failure is desired, then an appropriate shear test method should be considered, such as Test Method C 1292 or D 2344/D 2344M.

A2.3.3 *Out of Gage Failures*—Fractures that initiate outside the uniformly stressed region of a flexure specimen (between the inner support points in four-point and directly under the center support point in three-point) may be due to factors such as stress concentrations or strength limiting features in the microstructure of the specimen. In 4-point flexure tests, fractures which occur outside the center gage section are normally considered censored tests; that data may be used with appropriate statistical analyses. In 3-point flexure tests, fractures that occur away from the center point of the bar are considered valid tests.

A2.3.4 *Environmental and Time-Dependent Phenomena*—This method determines the flexural strength at ambient temperature and environmental conditions. Time-dependent phenomena, such as stress corrosion and slow crack growth, may interfere with the determination of the flexural strength at

room temperature and must be considered as possible experimental variables. Slow crack growth and stress corrosion may cause stress relaxation and/or crack growth in flexure specimens during a strength test even for the relatively short times involved during testing, thereby changing the flaw population and the fracture strength. Such influences and effects must be considered as possible experimental variables, if the flexure tests are to be used to generate design data.

A2.3.4.1 Slow crack growth can lead to a rate dependency of flexural strength. Depending on the material composition and its condition, the displacement rate specified in this standard may or may not produce the inert flexural strength whereby negligible slow crack growth occurs. If signs of slow crack growth are observed, retesting with accelerated displacement rates may be necessary, if an inert strength value is required in the test.

NOTE A2.2—Certain oxide ceramics, glasses, and ceramics containing boundary phase glass are susceptible to slow crack growth even at room temperature. Water, either in the form of liquid or as humidity in air, can have a significant effect, even at the rates specified in this standard. On the other hand, many ceramics such as graphite, boron carbide, silicon

carbide, aluminum nitride and many silicon nitrides have little or no sensitivity to slow crack growth at room temperature and the flexural strength in laboratory ambient conditions is the inert flexural strength. If necessary, do a literature search to assess stress corrosion and slow crack growth issues for the material of interest, or use Test Methods C 1368 and C 1576 to establish a time window for slow crack growth effects.

A2.3.4.2 The test environment (vacuum, inert gas, ambient air, etc.), including moisture content (i.e., relative or absolute humidity), may have an accelerating effect on stress corrosion and slow crack growth. If the material is susceptible to such mechanisms, testing to evaluate the maximum strength potential of a material should be conducted in inert environments or at sufficiently rapid testing rates, or both, so as to minimize slow crack growth effects. Conversely, testing can be conducted in environments and testing modes and rates representative of service conditions to evaluate material performance under use conditions.

A2.3.4.3 When testing is conducted in uncontrolled ambient air with the intent of evaluating maximum strength potential, monitor and report the relative humidity and ambient temperature.

APPENDIX

(Nonmandatory Information)

X1. SPECIMEN LINEAR CELL COUNT EFFECTS ON THE FLEXURE STRENGTH OF HONEYCOMB CHANNEL CERAMIC COMPONENTS

X1.1 Webb, Widjaja, and Helfinstine (6) did a study on the effect of specimen size and linear cell count on the flexure strength (4-point- $\frac{1}{4}$ point) of cordierite honeycomb cellular ceramic structures. Test specimens with two different honeycomb cell density configurations were evaluated:

- 100/17 density = 100 cells/in.² [15.5 cells/cm²]
with a cell wall thickness of 0.017 in. [0.432 mm]
- 200/12 density = 200 cells/in.² [31 cells/cm²]
with a cell wall thickness of 0.012 in. [0.305 mm]

NOTE X1.1—The term “cells per square inch” and English units are common practice in the automotive catalyst industry.

X1.1.1 The cells had uniform square cross section in each cell density configuration. Six different specimen sizes were tested for each cell density configuration with the following specimen dimensions and cell counts.

X1.1.2 Fig. X1.1 shows the specimen cross section for Specimen F (2×4) for the 100/17 cell density.

X1.2 Three different flexure strengths (nominal beam strength, wall fracture strength, and honeycomb structure strength) were calculated for each test specimen and then compared.

TABLE X1.1 Specimens Size and Test Geometry in Webb, Widjaja, Helfinstine Study (6)

Size	L_1 mm	L_2 mm	h mm	b mm	Length mm	Height – Linear Cell Count	Width – Linear Cell Count	Number of Specimens
100/17 = 100 cells per square inch and 0.017 in. cell wall thickness								
A	266.7	57.2	38.1	76.2	305	15	30	42
B	177.8	38.1	25.4	50.8	216	10	20	42
C	124.5	26.7	17.8	35.6	152	7	14	47
D	88.9	19.1	12.7	25.4	127	5	10	48
E	53.3	11.4	7.6	15.2	89	3	6	54
F	35.6	7.6	5.1	10.2	76	2	4	54
200/12 = 200 cells per square inch and 0.012 in. cell wall thickness								
A	266.7	57.2	38.1	76.2	305	21	42	40
B	177.8	38.1	25.4	50.8	216	14	28	41
C	125.7	26.9	18.0	36.1	152	10	20	39
D	88.9	19.1	12.7	25.4	127	7	14	40
E	63.5	13.7	9.1	18.3	89	5	10	45
F	38.1	8.1	5.3	10.9	64	3	6	45

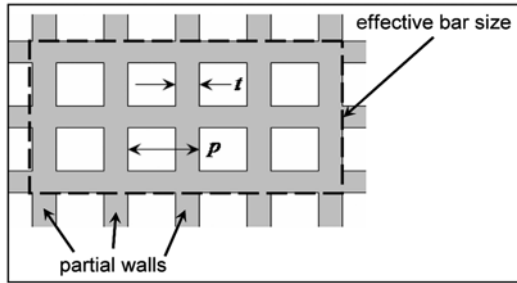


FIG. X1.1 Flexure Bar Cross Section Geometry F (2×4) for 100/17 Cell Density (from Ref 6)

X1.2.1 The nominal beam strength S_{NB} (termed MOR strength in the study) uses the standard elastic beam equations and treats the test specimen bar as a solid continuum, ignoring the cellular structure.

$$S_{NB} (4 pt) = \frac{Mc}{I} = \frac{3PL}{4bd^2} \quad (X1.1)$$

where:

- S_{NB} = nominal beam strength (MPa),
- M = applied bending moment,
- c = distance between the neutral axis and the outer fiber ($d/2$),
- I = the moment of inertia of the rectangular cross section,
- P = breaking force (N),
- L = outer (support) span (mm),
- b = specimen width (mm), and
- d = specimen thickness (mm).

X1.2.2 The wall fracture strength S_{WF} (termed web strength in the study) calculates the flexure strength with a moment of inertia calculation that accounts for the actual cell structure (cell pitch and cell wall thickness).

$$I_T = \frac{bd^3}{12} - \left[\frac{mn(p-t)^4}{12} + \frac{mp^2(p-t)^2}{4} \sum_i^n (2i-n-1)^2 \right] \text{(Square channels)} \quad (X1.2)$$

$$S_{WF} (4 pt) = \frac{Mc}{I_T} = \frac{PLd}{16I_T}$$

where:

- I_T = true moment of inertia, considering open channels (mm^4),
- m = linear cell count across the specimen width b ,
- n = linear cell count across the specimen thickness d ,
- p = cell pitch for the square cell (mm),
- t = cell wall thickness for the square cell (mm),
- S_{WF} = wall fracture strength (MPa), (termed web strength in the Webb study), and
- M = applied bending moment.

X1.2.3 The honeycomb structure strength S_{HS} (termed effective structural strength S_{ES} in the study) calculates the flexure strength with a moment of inertia calculation that accounts for the actual cell structure and adjusts for the open frontal area of the honeycomb.

$$S_{HS} = S_{WF}(1 - OFA) = S_{WF} \left(1 - \left(\frac{p-t}{p} \right)^2 \right) \quad (X1.3)$$

where:

- S_{HS} = Honeycomb structure strength (MPa), (termed effective structural strength in the Webb paper), and
- OFA = fractional open frontal area of the specimen.

X1.2.3.1 This calculation of the honeycomb structure strength essentially translates the wall fracture strength into an effective continuum stress assuming no difference between the walls and channels of the cross section. This honeycomb structure strength can be used as a strength value when a continuum strength is preferred, such as for using finite element stress models when the cell structure detail is left out of the stress model for simplicity.

X1.3 Using the equations for nominal beam strength and honeycomb structure strength, the % overestimate of the nominal beam strength (compared to the honeycomb strength calculation) is plotted for four cordierite honeycomb geometries:

- 200 cells per square inch and 0.012 in. cell wall thickness
- 200 cells per square inch and 0.020 in. cell wall thickness
- 100 cells per square inch and 0.012 in. cell wall thickness
- 100 cells per square inch and 0.020 in. cell wall thickness

X1.3.1 A 2:1 width to height ratio was used, and the calculation was done across a height linear cell count ranging from six (6) to twenty-one (21). The data is plotted in Fig. X1.2. (This figure is taken directly from Webb, Widjaja, and Helfinstine (6) and uses the term “cells per square inch” and English units per industry practice.)

X1.3.2 The figure shows that the percentage overestimate depends not only on the linear cell count of the test specimen, but also on the cell size and cell wall thickness. Thinner cell walls increase the overestimate. As a rule of thumb, a minimum linear cell count of 15 keeps the overestimate of the nominal beam strength within 10 % of the honeycomb structure strength.

X1.4 Using the flexure strength equations and real test data from the Corning data, Figs. X1.3 and X1.4 plot the calculated values for the three calculated flexure strengths for the two test specimen sets (100/17 architecture and 200/12 architecture).

X1.4.1 The calculated data shows the nominal beam strength S_{NB} values tend to increase with smaller specimens that have lower linear cell counts. This is observed in both test sets. Linear cell counts less than 5 have particularly large increases, compared to the specimen cell-size ratios of 10 and 15. This is a test specimen-linear cell count effect which is independent of the material considerations.

X1.4.2 The graphs also show that the honeycomb structure strength (S_{HS}) calculation eliminates the specimen size-linear cell count effect. The S_{HS} calculation gives a consistent value across the different specimen sizes and agrees with the nominal beam strength calculation for the larger high cell count specimens.

X1.5 Based on these calculations and test results, the following recommendations are made for calculating a flexure strength for the ceramic honeycomb test specimens.

X1.5.1 For flexure test specimens where the linear cell count is 15 or greater, the nominal beam strength S_{NB}

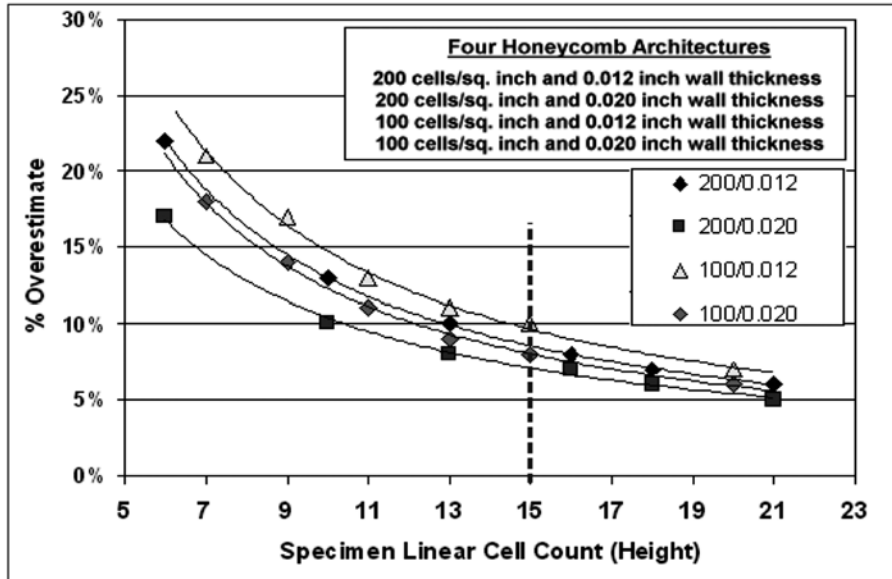


FIG. X1.2 Overestimate of the Calculated Nominal Beam Strength Compared to the Calculated Honeycomb Structure Strength for Four Different Honeycomb Architectures Across a Range of Linear Cell Counts

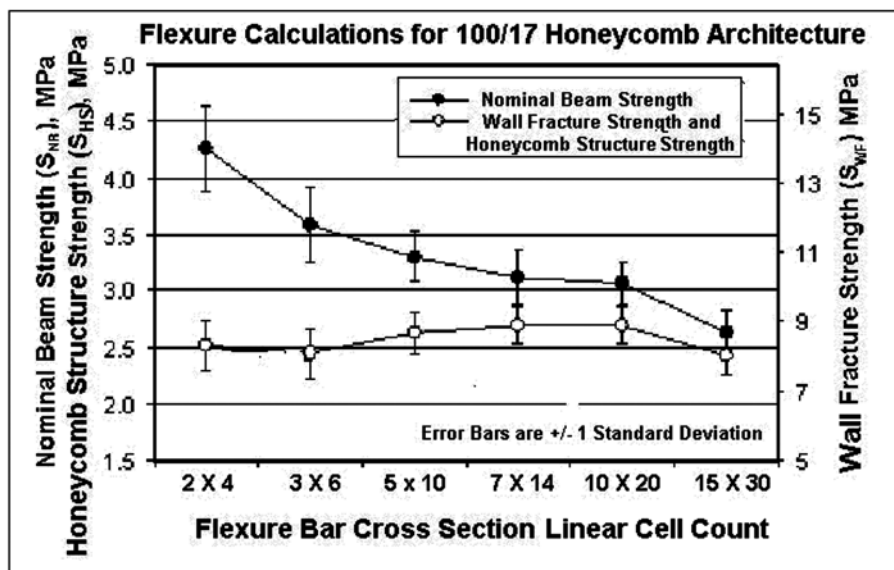


FIG. X1.3 Average S_{NB} , S_{WF} , and S_{HS} Values for Each Size Specimen of the 100/17 Test Set (from Ref 6)

calculation and the honeycomb structure strength S_{HS} are roughly equivalent in value (within 10%). The nominal beam strength S_{NB} calculation can be used considering this variability.

X1.5.2 For flexure test specimens where the linear cell count is between 5 and 15, the nominal beam strength S_{NB} calculation may produce a 10 to 20% overvalue. The S_{NB} value should be used with caution.

X1.5.3 For flexure test specimens where the linear cell count is less than 5, the nominal beam strength S_{NB} calculation

may produce a 20 to 100% overvalue. It is recommended that the honeycomb structure strength S_{HS} be calculated and used as a more accurate flexure strength number.

X1.5.4 If specimen availability and test configuration permit, test specimens with a linear cell count of 15 or greater are preferred to reduce the specimen linear cell count effect on nominal beam strength S_{NB} to less than 10%.

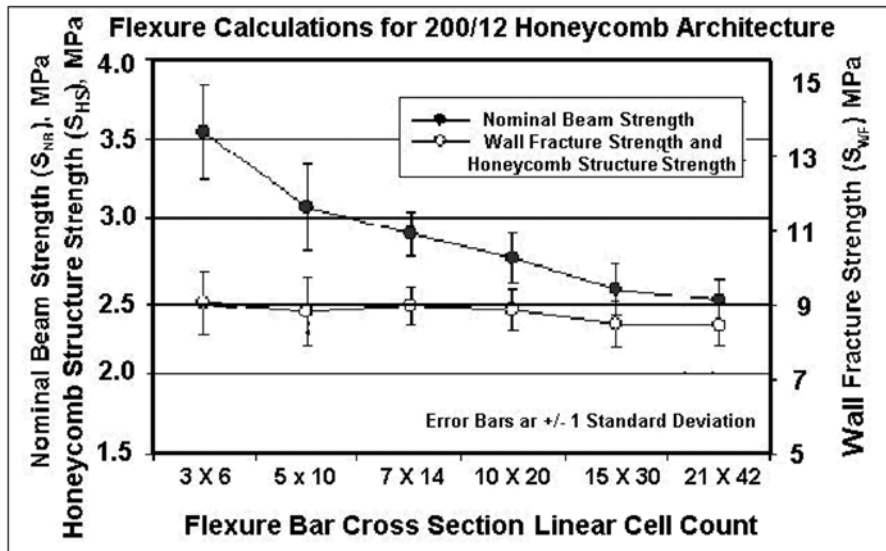


FIG. X1.4 Average S_{NB} , S_{WF} , and S_{HS} Values for Each Size Specimen of the 200/12 Test Set (from Ref 6)

REFERENCES

- (1) Then, P. M., Day, P., "The Catalytic Converter Ceramic Substrate—An Astonishing and Enduring Invention," *Inter-ceram (Germany)*, Vol 49, No. 1, Feb. 2000, pp. 20–23.
- (2) Adler, J., "Ceramic Diesel Particulate Filters," *International Journal of Applied Ceramic Technology*, Vol 2, Issue 6, Nov. 2005, pp. 429–439.
- (3) Narula, C. K., Daw, C. S., Hoard, J. W., and Hammer, T., "Materials Issues Related to Catalysts for Treatment of Diesel Exhaust," *International Journal of Applied Ceramic Technology*, Vol 2, Issue 6, Nov. 2005, pp. 452–466.
- (4) Suzukawa, Y., Sugiyama, S., and Moi, I., "Heat Transfer Improvement and NOx Reduction in an Industrial Furnace by Regenerative Combustion System," *Proceedings of the 31st Intersociety Energy Conversion Engineering Conference, 1996*, IECEC 96, Vol 2, pp. 804–809.
- (5) Jung, W. Y., and Aref, A. J., "A Combined Honeycomb and Solid Viscoelastic Material for Structural Damping Applications," *Mechanics of Materials*, Vol 35, Issue 8, Aug. 2003, pp. 831–844.
- (6) Webb, J. E., Widjaja, S., and Helfinstine, J. D., "Strength Size Effects in Cellular Ceramic Structures," *Ceramic Engineering and Science Proceedings—Mechanical Properties and Performance of Engineering Ceramics II*, Vol 27, Issue 2, Nov. 2006, pp. 521–531.
- (7) Gulati, S. T., Reddy, K. P., "Size Effect on the Strength of Ceramic Catalyst Supports," *SAE Paper 92233*, Oct. 19, 1992.
- (8) 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.

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