



Designation: C1674 – 08

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

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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

C373 Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products

C1145 Terminology of Advanced Ceramics

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

C1198 Test Method for Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio for Advanced Ceramics by Sonic Resonance

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

C1259 Test Method for Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio for Advanced Ceramics by

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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.

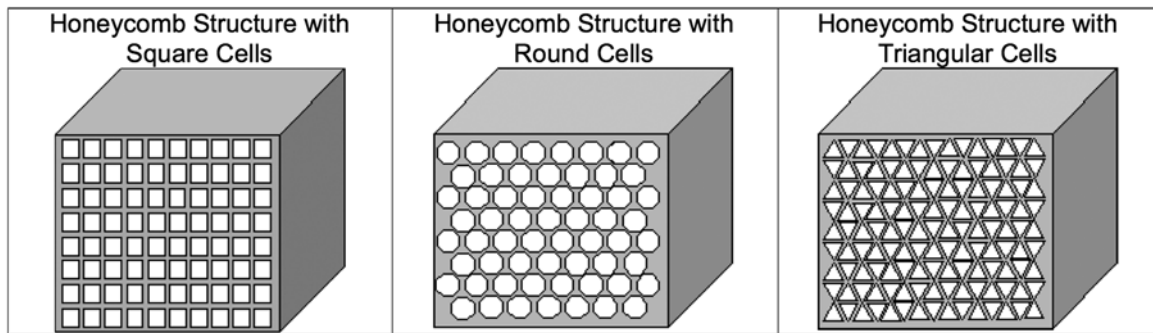
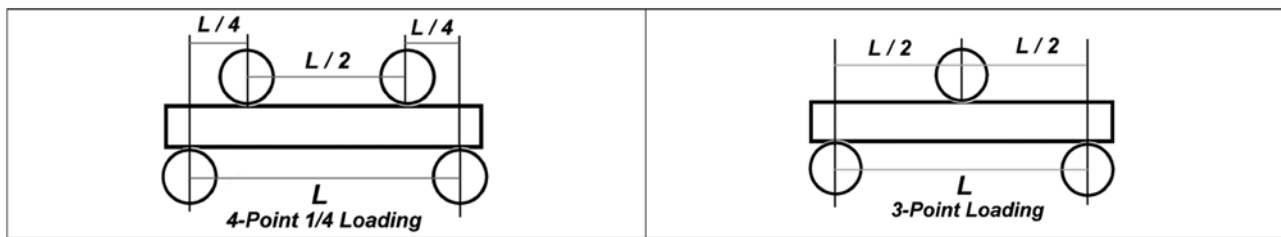


FIG. 1 General Schematics of Typical Honeycomb Ceramic Structures



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

Impulse Excitation of Vibration

[C1292](#) Test Method for Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures

[C1341](#) Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramic Composites

[C1368](#) Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature

[C1525](#) Test Method for Determination of Thermal Shock Resistance for Advanced Ceramics by Water Quenching

[C1576](#) Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Flexural Testing (Stress Rupture) at Ambient Temperature

[D2344/D2344M](#) Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates

[E4](#) Practices for Force Verification of Testing Machines

[E6](#) Terminology Relating to Methods of Mechanical Testing

[E337](#) 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)

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. **C1145**

3.1.2 *breaking force*, [F], *n*—the force at which fracture occurs in a test specimen. **E6**

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.

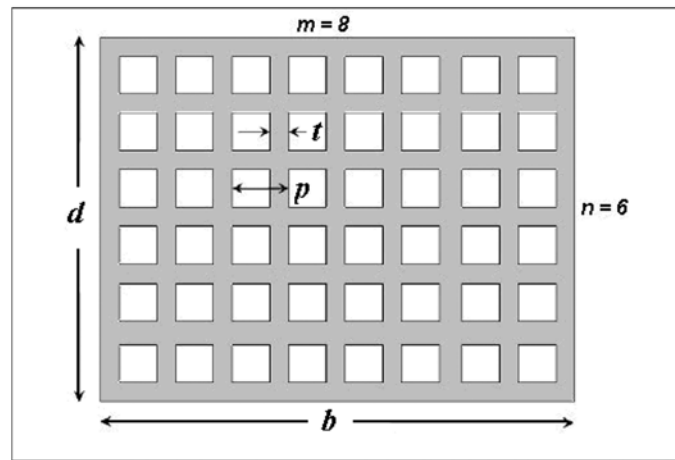
3.1.4 *cell wall thickness*, (*t*), [L], *n*—the nominal thickness of the walls that form the cell channels of the honeycomb structure. (See Fig. 3.)

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.

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. Terminology

3.1 The definitions of terms relating to flexure testing appearing in Terminology **E6** apply to the terms used in this test method. The definitions of terms relating to advanced ceramics appearing in Terminology **C1145** apply to the terms used in this test method. Pertinent definitions, as listed in Terminology **C1145**, Test Method **C1161**, and Terminology **E6**



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

3.1.6.1 *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.)

C1161

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.)

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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. **E6**

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.) **C1161**

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.) **C1161**

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 C1161 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-1/4 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 C1239.

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 C1525.

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 C1198) or by impulse excitation (Test Method C1259) 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 C1239). 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 E4. 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 C1341, 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× the cell pitch, p , giving a linear cell count of 5 or greater. If

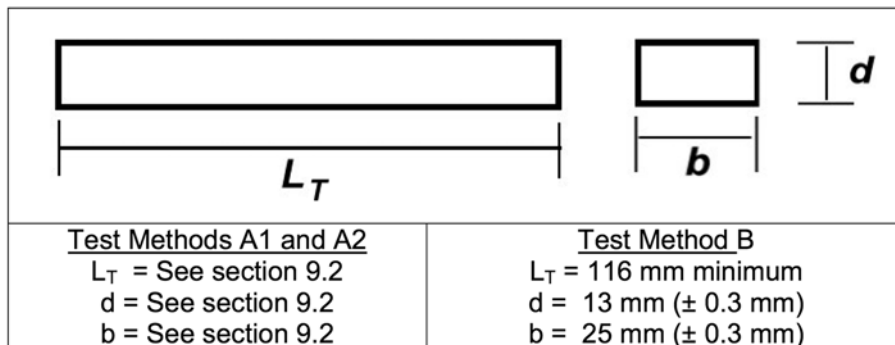


FIG. 4 Test Specimen Geometry (Test Methods A1, A2 and B)

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 surfaces 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

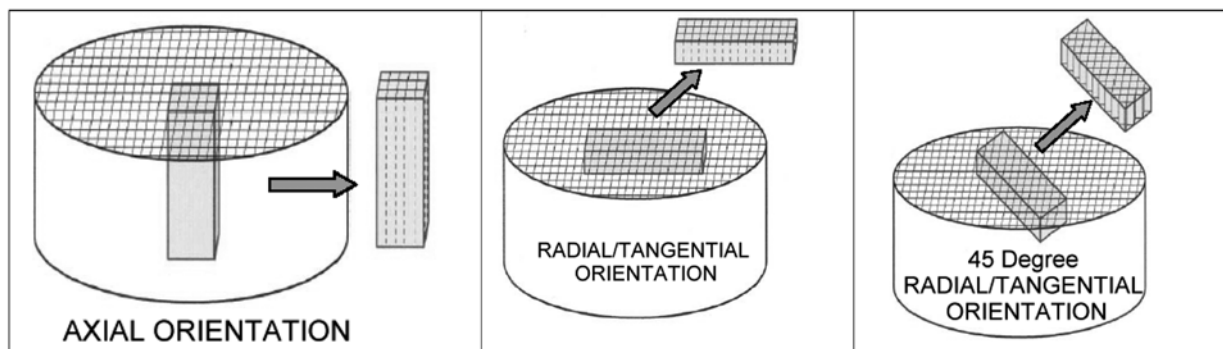


FIG. 5 Axial and Radial/Tangential Test Orientations for Honeycomb Specimens

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

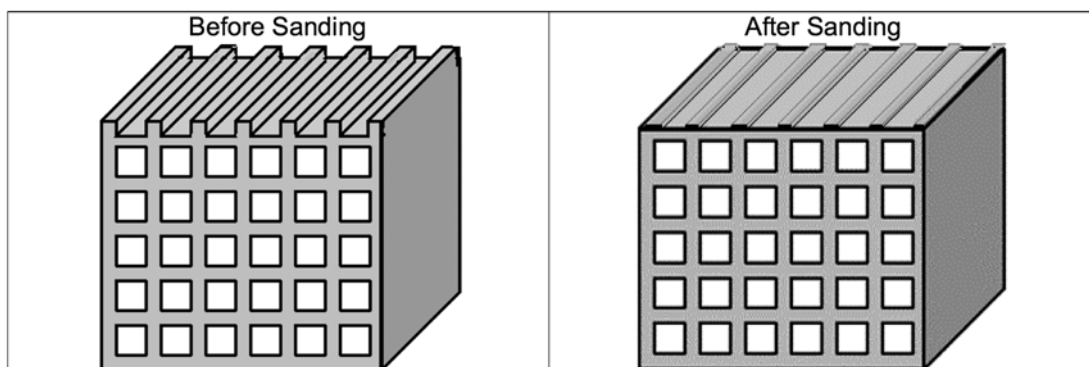


FIG. 6 Wall Rib/Stub Reduction by Gentle Sanding