INTERNATIONAL

Designation: C1469 − 22

Standard Test Method for Shear Strength of Joints of Advanced Ceramics at Ambient Temperature[1]

This standard is issued under the fixed designation C1469; 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 shear strength of joints in advanced ceramics at ambient temperature using asymmetrical four-point flexure. Test specimen geometries, test specimen fabrication methods, testing modes (that is, force or displacement control), testing rates (that is, force or displacement rate), data collection, and reporting procedures are addressed.

1.2 This test method is used to measure shear strength of ceramic joints in test specimens extracted from larger joined pieces by machining. Test specimens fabricated in this way are not expected to warp due to the relaxation of residual stresses but are expected to be much straighter and more uniform dimensionally than butt-jointed test specimens prepared by joining two halves, which is not recommended. In addition, this test method is intended for joints, which have either low or intermediate strengths with respect to the substrate material to be joined. Joints with high strengths should not be tested by this test method because of the high probability of invalid tests resulting from fractures initiating at the reaction points rather than in the joint. Determination of the shear strength of joints using this test method is appropriate particularly for advanced ceramic matrix composite materials but also may be useful for monolithic advanced ceramic materials.

1.3 Values expressed in this test method are in accordance with the International System of Units (SI) and IEEE/ASTM SI 10.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are noted in 8.1.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1145 Terminology of Advanced Ceramics

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

C1211 Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures

C1275 Test Method for Monotonic Tensile Behavior of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross-Section Test Specimens at Ambient Temperature

C1341 Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramic Composites

D3878 Terminology for Composite Materials

D5379/D5379M Test Method for Shear Properties of Composite Materials by the V-Notched Beam Method

E4 Practices for Force Calibration and Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)

IEEE/ASTM SI 10 American National Standard for Metric Practice

3. Terminology

3.1 *Definitions:*

3.1.1 The definitions of terms relating to shear strength testing appearing in Terminology E6 and to advanced ceramics appearing in Terminologies C1145 and D3878 apply to the

[1] This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.07 on Ceramic Matrix Composites.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

terms used in this test method. Additional terms used in conjunction with this test method are defined as follows.

3.1.2 *advanced ceramic, n*—a highly engineered, high-performance, predominately nonmetallic, inorganic, ceramic material having specific functional attributes. **C1145**

3.1.3 *breaking force [F], n*—force at which fracture occurs.

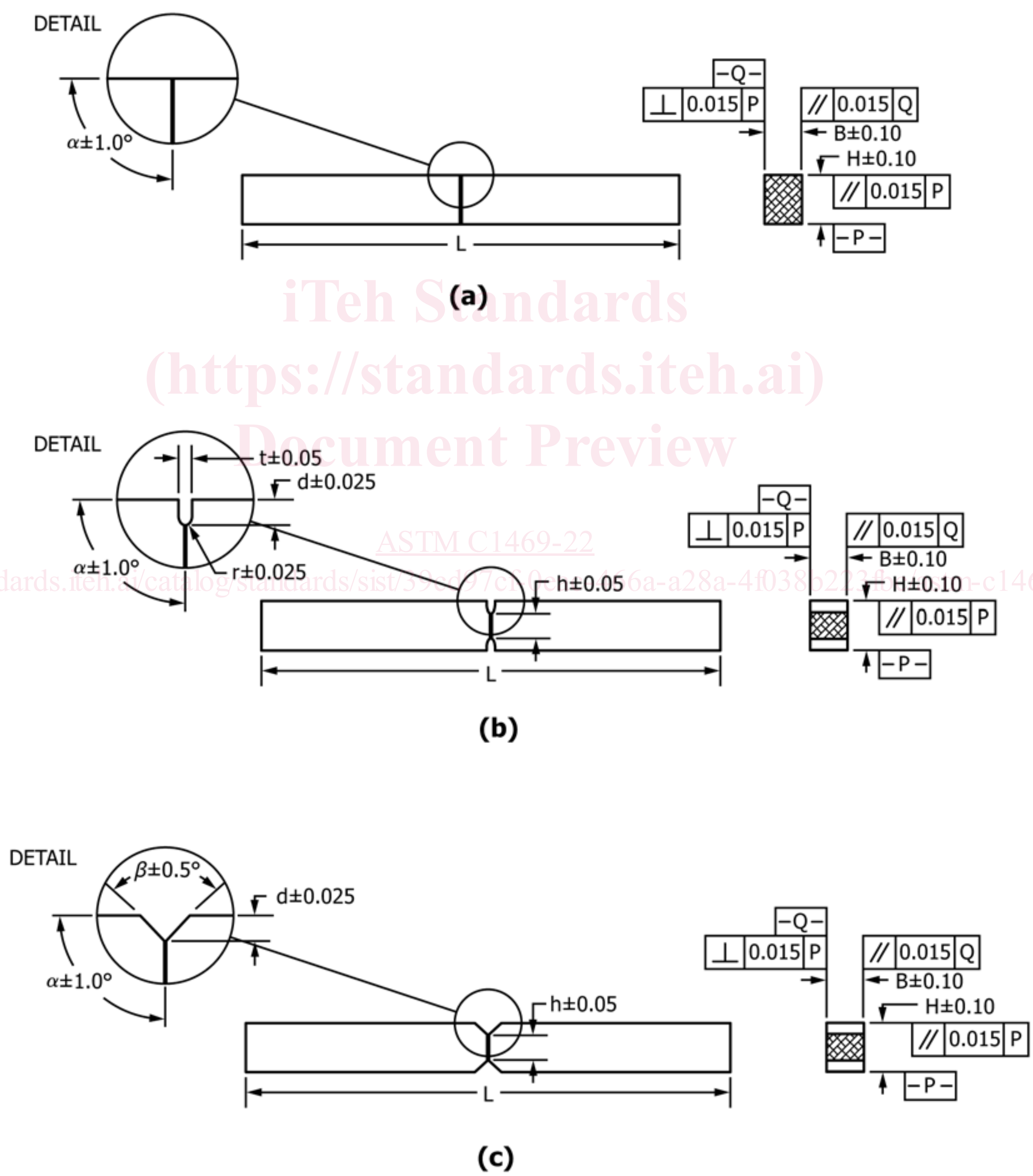
3.1.4 *ceramic matrix composite, n*—material consisting of two or more materials (insoluble in one another) in which the major, continuous component (matrix component) is a ceramic while the secondary component(s) may be ceramic, glass-ceramic, glass, metal, or organic in nature. These components are combined on macroscale to form a useful engineering material possessing certain properties or behavior not possessed by the individual constituents. **C1275**

3.1.5 *joining, n*—controlled formation of chemical or mechanical bond, or both, between similar or dissimilar materials.

3.1.6 *shear strength [F/L^2], n*—maximum shear stress that a material is capable of sustaining. Shear strength is calculated from breaking force in shear and shear area.

4. Summary of Test Method

4.1 This test method describes an asymmetrical four-point flexure test method to determine shear strengths of advanced ceramic joints. Test specimens and test setup are shown



NOTE 1—The width of the joint, which varies between 0.05 and 0.20 mm based on the joining method used, is smaller than that of the notch in (b). All dimensions are given in mm.

FIG. 1 Schematics of Test Specimen Geometries: (a) Uniform, (b) Straight-Notched, and (c) V-Notched

schematically in Fig. 1 and Fig. 2, respectively. Selection of the test specimen geometry depends on the bond strength of the joint, which may be determined by preparing longer test specimens of the same cross section and using a standard four-point flexural strength test, for example, Test Method C1161 for monolithic advanced ceramic base material and Test Method C1341 for composite advanced ceramic base material. If the joint flexural strength is low (that is, <25 % of the flexural strength of the base material), the recommended test specimen geometry for shear strength testing of the joint is the uniform test specimen shown in Fig. 1(a). If the joint flexural strength is moderate (that is, 25 to 50 % of the flexural strength of the base material), the recommended test specimen geometry for shear strength testing of the joint is the straight- or V-notched test specimen shown in Fig. 1(b) and Fig. 1(c), respectively. If the joint flexural strength is high (>50 % of the flexural strength of the base material), this test method should not be used to measure shear strength of advanced ceramic joints because very high contact stresses at the reaction points will provide a high probability of invalid tests (that is, fractures not at the joint).

4.2 The testing arrangement of this test method is asymmetrical flexure, as illustrated by the force, shear, and moment diagrams in Fig. 3(a), Fig. 3(b), and Fig. 3(c), respectively. Note that the greatest shear exists over a region of $\pm S_i/2$ around the centerline of the joint (see Fig. 3(b)). In addition, while the moment is zero at the centerline of the joint, the maximum moments occur at the inner reaction points (see Fig. 3(c)). The points of maximum moments are where the greatest probability of fracture of the base material may occur if the joint flexural strength, and therefore joint shear strength, is too high.

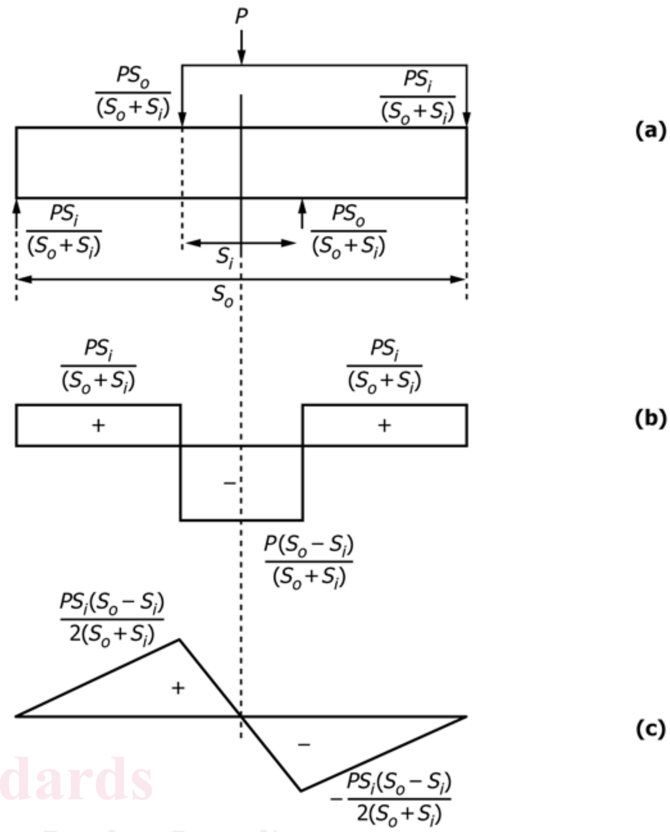


FIG. 3 Idealized (a) Force, (b) Shear, and (c) Moment Diagrams for Asymmetric Four-point Flexure, Where S_o and S_i Are the Outer and Inner Reaction Span Distances, Respectively, and P is the Applied Force

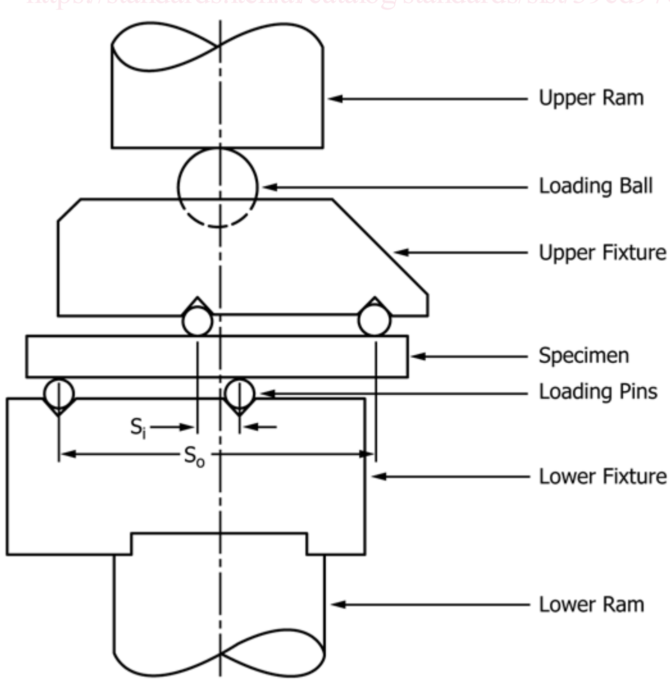


FIG. 2 Schematic of Test Fixture

5. Significance and Use

5.1 Advanced ceramics can be candidate materials for structural applications requiring high degrees of wear and corrosion resistance, often at elevated temperatures.

5.2 Joints are produced to enhance the performance and applicability of materials. While the joints between similar materials are generally made for manufacturing complex parts and repairing components, those involving dissimilar materials usually are produced to exploit the unique properties of each constituent in the new component. Depending on the joining process, the joint region may be the weakest part of the component. Since under mixed-mode and shear loading the load transfer across the joint requires reasonable shear strength, it is important that the quality and integrity of joint under in-plane shear forces be quantified. Shear strength data are also needed to monitor the development of new and improved joining techniques.

5.3 Shear tests provide information on the strength and deformation of materials under shear stresses.

5.4 This test method may be used for material development, material comparison, quality assurance, characterization, and design data generation.

5.5 For quality control purposes, results derived from standardized shear test specimens may be considered indicative of the response of the material from which they were taken for given primary processing conditions and post-processing heat treatments.

6. Interferences

6.1 Fractures that initiate outside of the joint region may be due to factors such as localized stress concentrations, extraneous stresses introduced by improper force transfer. Such fractures will constitute invalid tests.

6.2 Since the joint width is typically small, that is, 0.05 to 0.20 mm, the proper machining of the notches at the joint region is very critical (see Fig. 1). Improper machining of the notches can lead to undesired fracture at the reaction points. Furthermore, nonsymmetrical machining of the notches can be decisive as to how the fracture occurs between the notches.

NOTE 1—Finite element stress analysis of nonsymmetrical notches showed that when there is a misalignment between the notches and the mid-plane of the joint, spurious normal (σ_x) tensile stresses are generated at the notches which tend to "tear" the joint and would artificially affect (reduce) the magnitude of shear strength measured from the joint. The magnitude of these tensile stresses could be significant depending on the material system being investigated. Based on this analysis, it is recommended that the ratio of misalignment between the notch root and mid-plane of the joint, δ, and the distance between the notches, *h*, should be kept to less than 0.0125. (See Fig. 4.)

6.3 In this test method, the shear force required to cause fracture in the joint region depends on the span lengths of S_o and S_i in the fixture[3] (see Fig. 3). These lengths and the strength of the joint relative to that of the base material determine whether fracture takes place at the joint region or at the reaction points. Depending on this relative strength, it may be necessary to conduct preliminary tests to establish the appropriate S_o and S_i distances for the fixture to be used.[4]

6.4 The accuracy of insertion and alignment of the test specimen with respect to the fixture is critical; therefore, preparations for testing should be done carefully to minimize the bending moment at the joint, which strongly depends on the inner and outer reaction spans, as seen in Fig. 3(c). See details in 10.4.

6.5 Test environment (vacuum, inert gas, ambient air, etc.), including moisture content, for example, relative humidity, may have an influence on the measured shear strength. Conversely, testing can be conducted in environments and testing modes and rates representative of service conditions to evaluate material performance under those conditions. When testing is conducted in uncontrolled ambient air with the objective of evaluating maximum strength potential, relative humidity and temperature must be monitored and reported. Testing at humidity levels >65 % RH is not recommended and any deviations from this recommendation shall be reported.

7. Apparatus

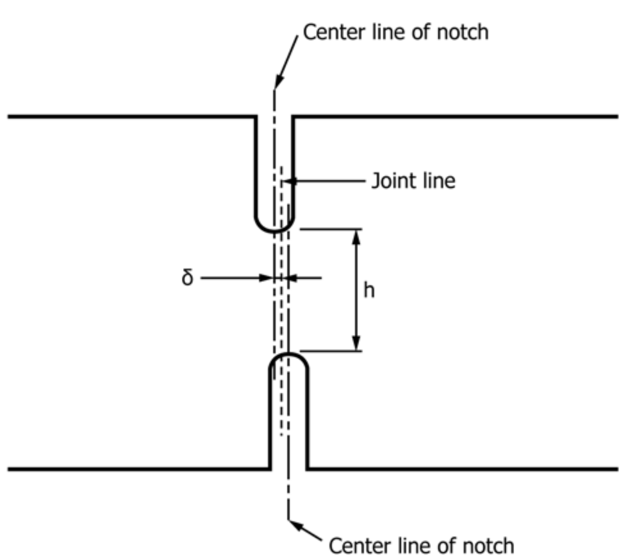
7.1 *Testing Machines*—The testing machine shall be in conformance with Practices E4. The forces used in determining shear strength shall be accurate within ±1 % at any force within the selected force range of the testing machine as defined in Practices E4.

7.2 *Data Acquisition*—Either digital data acquisition systems or analog chart recorders may be used as recording devices, although a digital record is recommended for ease of later data analysis. Recording devices shall be accurate to ±1 % of full scale and shall have a minimum data acquisition rate of 10 Hz, with a response of 50 Hz deemed more than sufficient.

7.3 *Dimension-Measuring Devices*—Micrometers and other devices used for measuring linear dimensions must be accurate and precise to at least 0.01 mm.

7.4 *Combination Square*—Used to draw perpendicular lines to specimen axis at the locations of inner loading points. The tolerance must be within 0.5°.

7.5 *Test Fixture*—The test fixture consists of top and bottom sections, reaction pins, and a force transfer ball, as shown schematically in Fig. 2. The bottom section is placed on a stationary base, for example, a compression platen. The test specimen is positioned between the top and bottom sections of the fixture. The force is transmitted from the test machine to the fixture by the force transfer ball; however, a pin can also be used in place of the force transfer ball. Table 1 contains symbols, nomenclature, and recommended dimensions for the test fixture (Fig. 2), where the tolerances for S_o and S_i after alignment is ±0.2 mm (see 10.4 for details). The tolerances for the diameter of the force transfer ball and reaction pin are ±0.1 mm and ±0.01 mm, respectively.



NOTE 1—It is recommended that the δ/h ratio in both notch types is less than 0.0125.

FIG. 4 Schematic of Misalignment, δ, Between the Joint Line and Notch Root Shown for Straight-Notched Specimen

[3] Slepetz, J. M., Zagaeski, T. F., and Novello, R. F., "In-Plane Shear Test for Composite Materials," AMMRC-TR-78-30, Army Materials and Mechanics Research Center, Watertown, MA, July 1978.

[4] Ünal, Ö., Anderson, I. E., and Maghsoodi, S. I., "A Test Method to Measure Shear Strength of Ceramic Joints at High Temperatures," *Journal of the American Ceramic Society*, Vol 80, No. 5, 1997, pp. 1281–1284.

TABLE 1 Recommended Dimensions for Test Fixture

Dimension	Description	Nominal Value	Tolerance
S_i	Inner span	4.0 mm	±0.2 mm
S_o	Outer span	30.0 mm	±0.2 mm
	Force transfer ball diameter	7.5 mm	±0.1 mm
	Reaction pin diameter	3.00 mm	±0.01 mm

NOTE 2—The reaction pin diameter in this standard is 3 mm, unlike that in Test Method C1161 where it is 4.5 mm. Unpublished finite element analyses have indicated that the smaller pin diameter better approximates the "point loading," thus the stress profile at the joint in Fig. 3.

NOTE 3—It should be indicated that when there are restrictions for pins to rotate freely, as in Fig. 2, the resulting friction may become a factor in the measurements, as indicated in Test Method C1161. So far, however, no systematic study has been conducted in the current test method regarding this issue.

7.5.1 Test fixtures, including the pins and ball, and loading rams shall be stiff and elastic under loading. These pieces may be made of a ceramic with an elastic modulus between 200 and 400 GPa and a flexural strength no less than 275 MPa, as specified in Test Method C1211. Dense high-purity silicon carbide and alumina are the typical candidate materials. Alternatively, the above components may be made of hardened steel which has a hardness no less than HRC 40 or which has a yield strength no less than 1240 MPa, as specified in Test Methods C1161 and C1211.

8. Precautionary Statement

8.1 During the conduct of this test method, the possibility of flying fragments of broken test material 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. Means for containment and retention of these fragments for later fractographic reconstruction and analysis is highly recommended.

9. Test Specimen

9.1 *Test Specimen Geometry*—Depending on the flexural strength of the joint, any one of the three test specimen geometries is suitable for this test method (see 4.1 and Fig. 1(a), Fig. 1(b), and Fig. 1(c)). The opposing notches on the notched test specimens shall be made symmetrically at the centerline of the joint (Fig. 1(b) and Fig. 1(c)). Moreover, the depth of each of the notches shall be one fourth of the overall height of the test specimen (H/4). While the drawings in Fig. 1 show the tolerances for the test specimens, Table 2 shows symbols, nomenclature, and recommended dimensions for the test specimen. If necessary, the test specimen dimensions, that is, length, height, width, and notch depth, if applicable) can be adjusted to meet special requirements. Report any deviation from the recommended values of Table 2.

TABLE 2 Recommended Dimensions for Test Specimens

Dimension	Description	Nominal Value	Tolerance
L	Test specimen length	36.0 mm	±0.5
H	Test specimen height	4.0 mm	±0.1
B	Test specimen width	3.0 mm	±0.1
h	Distance between notches	2.00 mm	±0.05
α	Angle between test specimen axis and joint line	90°	±1°
β	Notch angle (V-notch)	90°	±1°
	Notch root radius (V-notch)	None	—
d	Depth of notch	1.000 mm	±0.025
t	Notch width (straight notch)	0.50 mm	±0.05
r	Notch root radius (straight notch)	0.250 mm	±0.025

9.2 *Test Specimen Preparation*—Any machining procedure may be used that is deemed satisfactory for a class of materials so long as it induces no unwanted surface/subsurface damage or residual stresses. The grinding of uniform test specimen in Fig. 1(a) shall be along the longitudinal axis of the test specimen, according to standard procedures described in Test Methods C1161 and C1211.

9.2.1 Conduct any grinding or cutting with ample supply of appropriate filtered coolant to keep the workpiece and grinding wheel constantly flooded and particles flushed. Grind in at least two stages, ranging from coarse to fine rate of material removal.

9.2.2 Remove stock at a rate on the order of 0.03 mm/pass if using diamond tools that have between 320 and 600 grit. Remove equal stock from each face, where applicable.

9.2.3 Other types of material removal processes may be used if they meet the requirements for dimensional tolerances, surface characteristics, and residual stresses.

9.3 *Handling Precaution*—Exercise care in the storing and handling of finished test specimens to avoid the introduction of severe flaws. In addition, direct attention to pre-test storage of test specimens in controlled environments or desiccators to avoid unquantifiable environmental degradation of test specimens prior to testing.

9.4 *Number of Valid Tests*—Conduct a minimum of ten valid tests per test condition, unless statistically significant results can be obtained from fewer valid tests, such as in the case of a designed experiment. For statistically significant data, the procedures outlined in Practice E122 shall be consulted.

9.5 *Valid Tests*—A valid individual test is one that meets all the following requirements: all the testing requirements of this test method, and fracture occurs in the joint region unless those tests fracturing outside the joint region are interpreted tests for the purpose of censored test analyses.

10. Procedure

10.1 *Test Specimen Dimensions*—Determine the thickness and width of the gage section of each test specimen to within 0.01 mm. Avoid damaging the critical gage section area by performing these measurements either optically, for example, an optical comparator, or mechanically using a flat, anvil-type micrometer. In either case the resolution of the instrument shall be as specified in 7.3. Exercise extreme caution to prevent damaging the test specimen gage section. Record and report the measured dimensions and locations of the measurements for use in the calculation of the shear stress. Use the average of multiple (three or more) measurements in the stress calculations.

10.1.1 Additionally, make post-fracture measurements of the joint region dimensions using instruments described in 10.1. Measure and record only the dimensions at the plane of