



Standard Test Method for Tensile Strength of Monolithic Advanced Ceramics at Elevated Temperatures¹

This standard is issued under the fixed designation C 1366; 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 tensile strength under uniaxial loading of monolithic advanced ceramics at elevated temperatures. This test method addresses, but is not restricted to, various suggested test specimen geometries as listed in the appendix. In addition, test specimen fabrication methods, testing modes (force, displacement, or strain control), testing rates (force rate, stress rate, displacement rate, or strain rate), allowable bending, and data collection and reporting procedures are addressed. Tensile strength as used in this test method refers to the tensile strength obtained under uniaxial loading.

1.2 This test method applies primarily to advanced ceramics which macroscopically exhibit isotropic, homogeneous, continuous behavior. While this test method applies primarily to monolithic advanced ceramics, certain whisker, or particle-reinforced composite ceramics as well as certain discontinuous fiber-reinforced composite ceramics may also meet these macroscopic behavior assumptions. Generally, continuous fiber ceramic composites (CFCCs) do not macroscopically exhibit isotropic, homogeneous, continuous behavior and application of this test method to these materials is not recommended.

1.3 The values stated in SI units are to be regarded as the standard and are in accordance with Practice E 380.

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 and health practices and determine the applicability of regulatory limitations prior to use. Refer to Section 7 for specific precautions.*

2. Referenced Documents

2.1 ASTM Standards:²

C 1145 Terminology of Advanced Ceramics

¹ This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Properties and Performance.

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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.

- C 1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
- C 1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
- C 1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics
- D 3379 Test Method for Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials
- E 4 Practices for Force Verification of Testing Machines
- E 6 Terminology Relating to Methods of Mechanical Testing
- E 21 Practice for Elevated Temperature Tension Tests of Metallic Materials
- E 83 Practice for Verification and Classification of Extensometers
- E 220 Method for Calibration of Thermocouples by Comparison Techniques
- E 337 Test Method for Measure Humidity with a Psychrometer (The Measurement of Wet- and Dry-Bulb Temperatures)
- E 1012 Practice for Verification of Specimen Alignment Under Tensile Loading
- IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI) (The Modern Metric System)

3. Terminology

3.1 Definitions:

3.1.1 Definitions of terms relating to tensile testing and advanced ceramics as they appear in Terminology E 6 and Terminology C 1145, respectively, apply to the terms used in this test method. Pertinent definitions are shown in the following with the appropriate source given in parenthesis. Additional terms used in conjunction with this test method are defined in the following.

3.1.2 *advanced ceramic, n*—a highly engineered, high performance predominately non-metallic, inorganic, ceramic material having specific functional attributes. (See Terminology C 1145.)

3.1.3 *axial strain* [LL^{-1}], n —the average longitudinal strains measured at the surface on opposite sides of the longitudinal axis of symmetry of the specimen by two strain-sensing devices located at the mid length of the reduced section. (See Practice E 1012.)

3.1.4 *bending strain* [LL^{-1}], n —the difference between the strain at the surface and the axial strain. In general, the bending strain varies from point to point around and along the reduced section of the specimen. (See Practice E 1012.)

3.1.5 *breaking load* [F], n —the load at which fracture occurs. (See Terminology E 6.)

3.1.6 *fractography*, n —the means and methods for characterizing a fractured specimen or component. (See Terminology C 1145.)

3.1.7 *fracture origin*, n —the source from which brittle fracture commences. (See Terminology C 1145.)

3.1.8 *percent binding*, n —the bending strain times 100 divided by the axial strain. (See Practice E 1012.)

3.1.9 *slow crack growth*, n —sub critical crack growth (extension) that may result from, but is not restricted to, such mechanisms as environmentally-assisted stress corrosion or diffusive crack growth.

3.1.10 *tensile strength*, S_u [FL^2], n —the maximum tensile stress which a material is capable of sustaining. Tensile strength is calculated from the maximum load during a tension test carried to rupture and the original cross-sectional area of the specimen. (See Terminology E 6.)

4. Significance and Use

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

4.2 High strength, monolithic advanced ceramic materials are generally characterized by small grain sizes ($< 50 \mu\text{m}$) and bulk densities near the theoretical density. These materials are candidates for load-bearing structural applications requiring high degrees of wear and corrosion resistance and elevated-temperature strength. Although flexural test methods are commonly used to evaluate strength of advanced ceramics, the non uniform stress distribution of the flexure specimen limits the volume of material subjected to the maximum applied stress at fracture. Uniaxially-loaded tensile strength tests provide information on strength-limiting flaws from a greater volume of uniformly stressed material.

4.3 Because of the probabilistic strength distributions of brittle materials such as advanced ceramics, a sufficient number of test specimens at each testing condition is required for statistical analysis and eventual design with guidelines for sufficient numbers provided in this test method. Size-scaling effects as discussed in practice C 1239 will affect the strength values. Therefore, strengths obtained using different recommended tensile test specimen geometries with different volumes or surface areas of material in the gage sections will be different due to these size differences. Resulting strength values can, in principle, be scaled to an effective volume or effective surface area of unity as discussed in Practice C 1239.

4.4 Tensile tests provide information on the strength and deformation of materials under uniaxial stresses. Uniform stress states are required to effectively evaluate any non-linear

stress-strain behavior which may develop as the result of testing mode, testing rate, processing or alloying effects, environmental influences, or elevated temperatures. These effects may be consequences of stress corrosion or sub critical (slow) crack growth which can be minimized by testing at appropriately rapid rates as outlined in this test method.

4.5 The results of tensile tests of specimens fabricated to standardized dimensions from a particular material or selected portions of a part, or both, may not totally represent the strength and deformation properties of the entire, full-size end product or its in-service behavior in different environments.

4.6 For quality control purposes, results derived from standardized tensile test specimens can be considered to be indicative of the response of the material from which they were taken for particular primary processing conditions and post-processing heat treatments.

4.7 The tensile strength of a ceramic material is dependent on both its inherent resistance to fracture and the presence of flaws. Analysis of fracture surfaces and fractography as described in Practice C 1322 and MIL-HDBK-790, though beyond the scope of this test method, are recommended for all purposes, especially for design data.

5. Interferences

5.1 Test environment (vacuum, inert gas, ambient air, etc.) including moisture content for example relative humidity) may have an influence on the measured tensile strength. In particular, the behavior of materials susceptible to slow crack growth fracture will be strongly influenced by test environment, testing rate, and elevated temperatures. Testing to evaluate the maximum strength potential of a material should be conducted in inert environments or at sufficiently rapid testing rates, or both, 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. When testing is conducted in uncontrolled ambient air with the intent of evaluating maximum strength potential, monitor and report relative humidity and ambient temperature. Testing at humidity levels $> 65 \%$ relative humidity (RH) is not recommended.

5.2 Surface preparation of test specimens can introduce fabrication flaws that may have pronounced effects on tensile strength. Machining damage introduced during test specimen preparation can be either a random interfering factor in the determination of ultimate strength of pristine material (that is increase frequency of surface initiated fractures compared to volume initiated fractures), or an inherent part of the strength characteristics. Surface preparation can also lead to the introduction of residual stresses. Universal or standardized test methods of surface preparation do not exist. Final machining steps may, or may not negate machining damage introduced during the early coarse or intermediate machining. Thus, report test specimen fabrication history since it may play an important role in the measured strength distributions.

5.3 Bending in uniaxial tensile tests can cause or promote non uniform stress distributions with maximum stresses occurring at the test specimen surface leading to non representative fractures originating at surfaces or near geometrical transitions. Bending may be introduced from several sources including

misaligned load trains, eccentric or mis-shaped test specimens, and non-uniformly heated test specimens or grips. In addition, if strains or deformations are measured at surfaces where maximum or minimum stresses occur, bending may introduce over or under measurement of strains. Similarly, fracture from surface flaws may be accentuated or muted by the presence of the non uniform stresses caused by bending.

6. Apparatus

6.1 *Testing Machines*—Machines used for tensile testing shall conform to the requirements of Practice E 4. The forces used in determining tensile strength shall be accurate within $\pm 1\%$ at any force within the selected force range of the testing machine as defined in Practice E 4. A schematic showing pertinent features of a possible tensile testing apparatus is shown in Fig. 1

6.2 *Gripping Devices:*

6.2.1 *General*—Various types of gripping devices may be used to transmit the measured load applied by the testing machine to the test specimen. The brittle nature of advanced ceramics requires a uniform interface between the grip components and the gripped section of the test specimen. Line or point contacts and non uniform pressure can produce Hertzian-type stress leading to crack initiation and fracture of the test specimen in the gripped section. Gripping devices can be classed generally as those employing active and those employing passive grip interfaces as discussed in the following sections. Uncooled grips located inside the heated zone are termed “hot grips” and generally produce almost no thermal gradient in the test specimen but at the relative expense of grip materials of at least the same temperature capability as the test

material and increased degradation of the grips due to exposure to the elevated-temperature oxidizing environment. Grips located outside the heated zone surrounding the test specimen may or may not employ cooling. Uncooled grips located outside the heated zone are termed “ warm grips” and generally induce a mild thermal gradient in the test specimen but at the relative expense of elevated-temperature alloys in the grips and increased degradation of the grips due to exposure to the elevated-temperature oxidizing environment. Cooled grips located outside the heated zone are termed “ cold grips” and generally induce a steep thermal gradient in the test specimen at a greater relative expense because of grip cooling equipment and allowances, although with the advantage of consistent alignment and little degradation from exposure to elevated temperatures.

NOTE 1—The expense of the cooling system for cold grips is balanced against maintaining alignment which remains consistent from test to test (stable grip temperature) and decreased degradation of the grips due to exposure to the elevated-temperature oxidizing environment. When grip cooling is employed, means should be provided to control the cooling medium to maximum fluctuations of 5 K (less than 1 K preferred) about a setpoint temperature (1)³ over the course of the test to minimize thermally-induced strain changes in the test specimen. In addition, opposing grip temperatures should be maintained at uniform and consistent temperatures within ± 5 K (less than ± 1 K preferred) (1) so as to avoid introducing unequal thermal gradients and subsequent non uniaxial stresses in the test specimen. Generally, the need for control of grip temperature fluctuations or differences may be indicated if test specimen gage-section temperatures cannot be maintained within the limits required in 9.3.2

6.2.1.1 *Active Grip Interfaces*—Active grip interfaces require a continuous application of a mechanical, hydraulic, or pneumatic force to transmit the load applied by the test machine to the test specimen. Generally, these types of grip interfaces cause a force to be applied normal to the surface of the gripped section of the test specimen. Transmission of the uniaxial force applied by the test machine is then accomplished by friction between the test specimen and the grip faces. Thus, important aspects of active grip interfaces are uniform contact between the gripped section of the test specimen and the grip faces and constant coefficient of friction over the grip/test specimen interface.

(a) For cylindrical test specimens, a one-piece split-collet arrangement acts as the grip interface (2, 3) as illustrated by Fig. 2. Close tolerances are required for concentricity of both the grip and test specimen diameters. In addition, the diameter of the gripped section of the test specimen and the unclamped, open diameter of the grip faces must be within similarly close tolerances to promote uniform contact at the test specimen/grip interface. Tolerances will vary depending on the exact configuration as shown in the appropriate specimen drawings.

(b) For, flat test specimens, flat-face, wedge-grip faces act as the grip interface as illustrated in Fig. 3. Close tolerances are required for the flatness and parallelism as well as wedge angle of the grip faces. In addition, the thickness, flatness, and parallelism of the gripped section of the test specimen must be

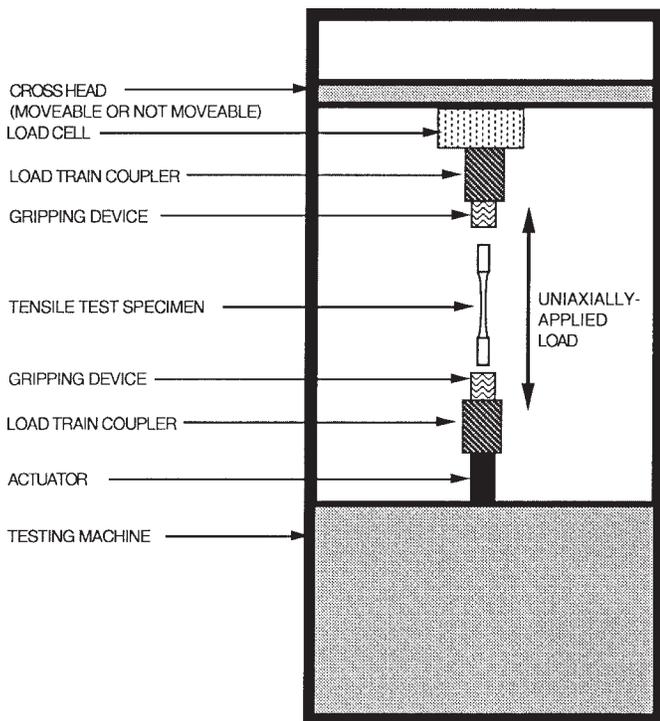


FIG. 1 Schematic Diagram of One Possible Apparatus for Conducting a Uniaxially-Loaded Tensile Test

³ The boldface numbers given in parentheses refer to a list of references at the end of the text.

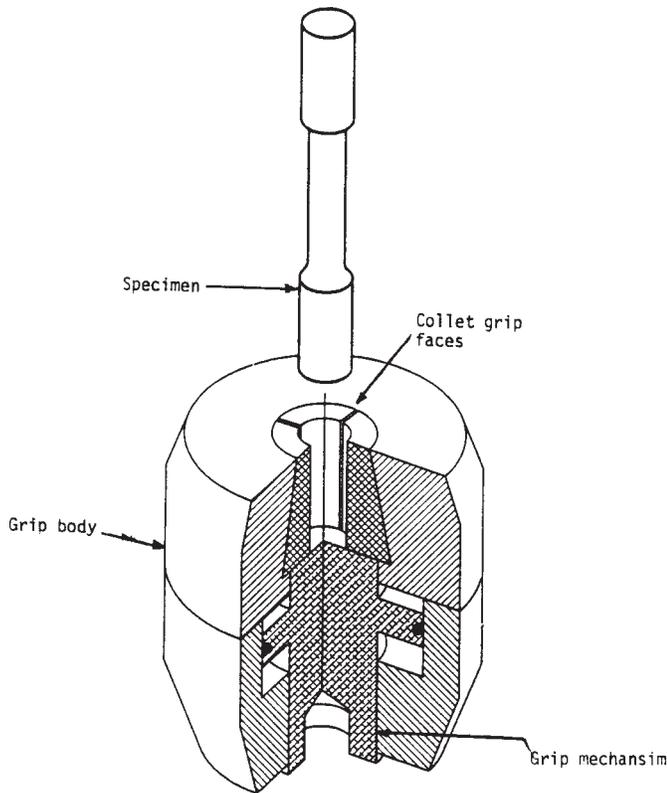


FIG. 2 Example of a Smooth, Split Collet Active Gripping System for Cylindrical Test Specimens

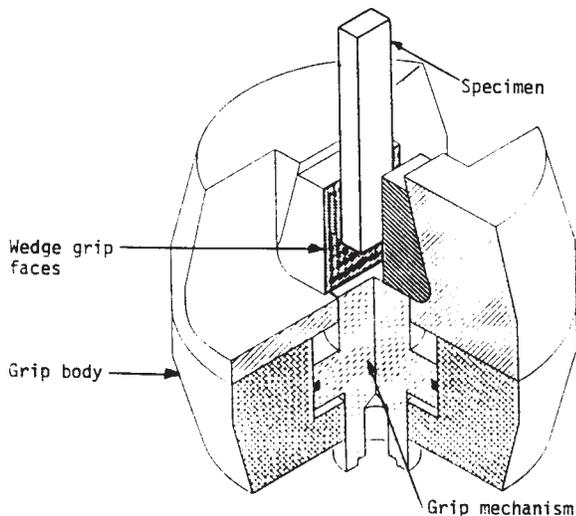


FIG. 3 Example of a Smooth, Wedge Active Gripping System for Flat Test Specimens

within similarly close tolerances to promote uniform contact at the test specimen/grip interface. Tolerances will vary depending on the exact configuration as shown in the appropriate test specimen drawings.

6.2.1.2 *Passive Grip Interfaces*—Passive grip interfaces transmit the force applied by the test machine to the test specimen through a direct mechanical link. Generally, these mechanical links transmit the test forces to the test specimen via geometrical features of the test specimens such as button-

head fillets, shank shoulders, or holes in the gripped head. Thus, the important aspect of passive grip interfaces in uniform contact between the gripped section of the test specimen and the grip faces.

(a) For cylindrical test specimens, a multi-piece split collet arrangement acts as the grip interface at button-head fillets of the test specimen (4) as illustrated in Fig. 4. Because of the limited contact area at the test specimen/grip interface, soft, deformable collet materials may be used to conform to the exact geometry of the test specimen. In some cases tapered collets may be used to transfer the axial force into the shank of the test specimen rather than into the button-head radius (4). Moderately close tolerances are required for concentricity of both the grip and test specimen diameters. In addition, tolerances on the collet height must be maintained to promote uniform axial-loading at the test specimen/grip interface. Tolerances will vary depending on the exact configuration as shown in the appropriate test specimen drawings.

(b) For flat test specimens, pins or pivots act as grip interfaces at either the shoulders of the test specimen shank or at holes in the gripped test specimen head (5,6,7). Close tolerances are required of shoulder radii and grip interfaces to promote uniform contact along the entire test specimen/grip interface as well as to provide for non eccentric loading as shown in Fig. 5. Moderately close tolerances are required for longitudinal coincidence of the pin and hole centerlines as illustrated in Fig. 6.

6.3 Load Train Couplers:

6.3.1 *General*—Various types of devices (load-train couplers) may be used to attach the active or passive grip interface assemblies to the testing machine (for example, Fig. 7). The load-train couplers in conjunction with the type of gripping device play major roles in the alignment of the load train and thus subsequent bending imposed in the test specimen. Load train couplers can be classified, as fixed and non fixed as discussed in the following sections. The use of well-aligned fixed or self-aligning non fixed couplers does not automatically guarantee low bending in the gage section of the tensile test specimen. Well-aligned fixed or self-aligning non fixed couplers provide for well-aligned load trains, but the type and operation of grip interfaces as well as the as-fabricated dimensions of the tensile test specimen can add significantly to the final bending imposed in the test specimen gage section.

6.3.1.1 Regardless of which type of coupler is used, verify alignment of the testing system at a minimum at the beginning and end of a test series unless the conditions for verifying alignment are otherwise met. An additional verification of alignment is recommended, although not required, at the middle of the test series. Use either a dummy or actual test specimen. Allowable bending requirements are discussed in 6.5. See Practice E 1012 for discussions of alignment and Appendix X1 for suggested procedures specific to this test method. A test series is interpreted to mean a discrete group of tests on individual test specimens conducted within a discrete period of time on a particular material configuration, test specimen geometry, test condition, or other uniquely definable qualifier (for example a test series composed of material A

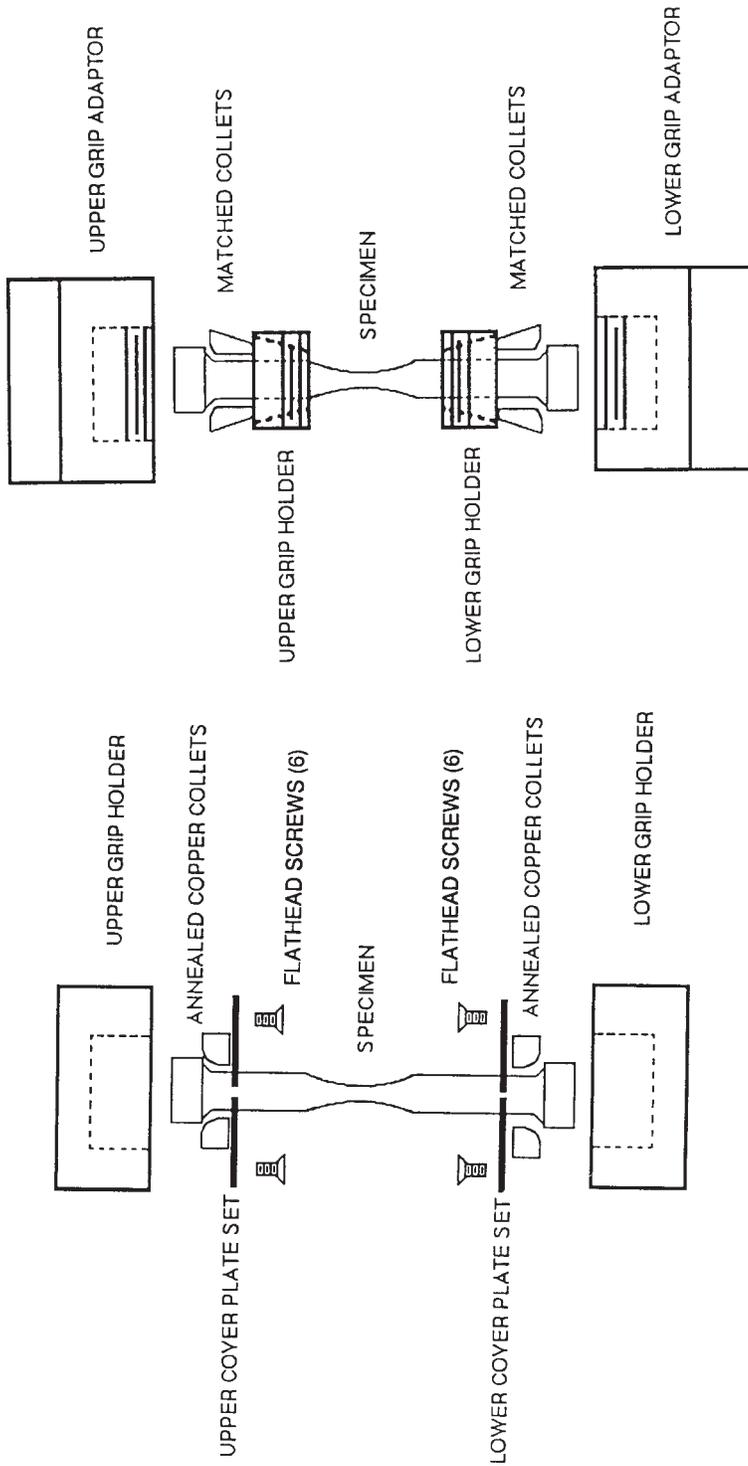


FIG. 4 Examples of Straight- and Tapered-Collet Passive Gripping Systems for Cylindrical Test Specimens (4)

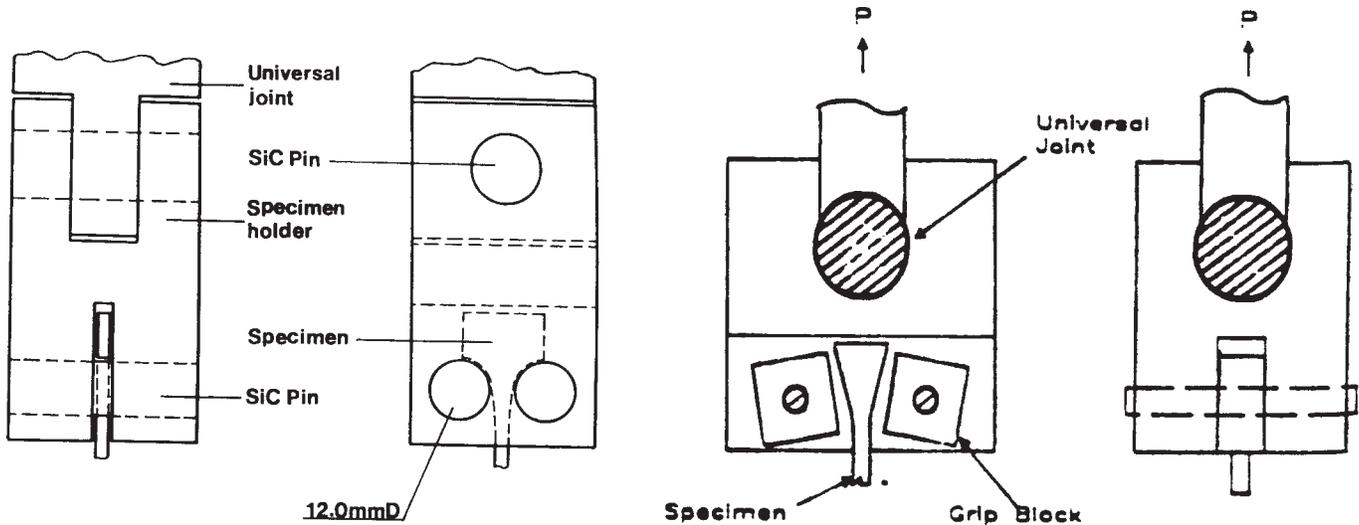


FIG. 5 Examples of Shoulder-Loaded, Passive Gripping Systems for Flat Test Specimens (5,6)

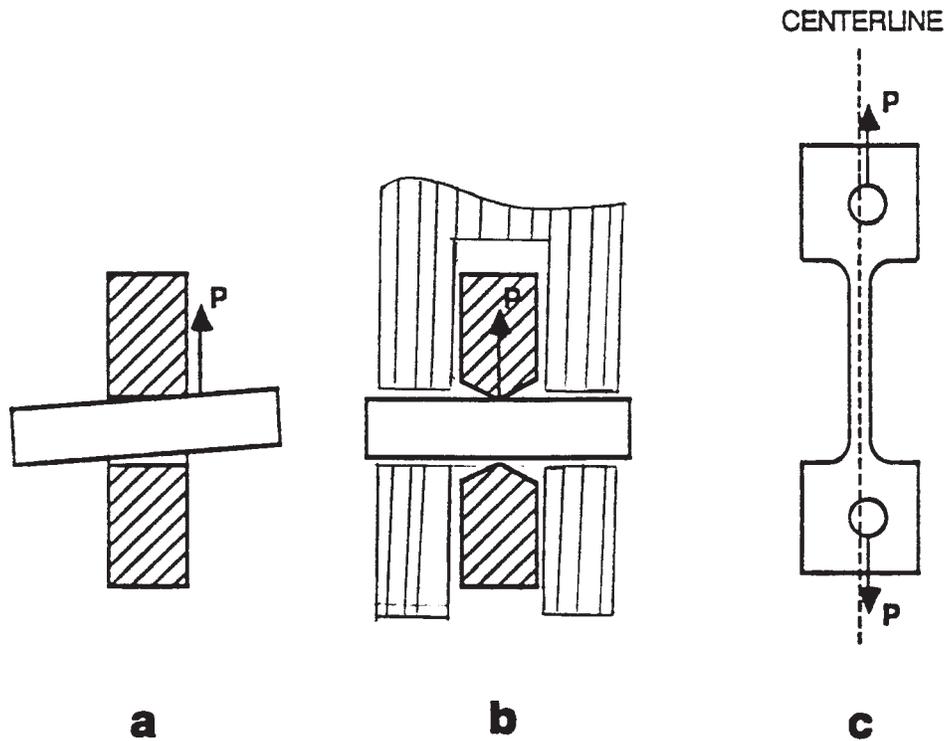


FIG. 6 Example of a Pin-Loaded, Passive Gripping system for Flat Test Specimens (6)

comprising ten test specimens of geometry B tested at a fixed rate in strain control to final fracture in ambient air).

NOTE 2—Tensile test specimens used for alignment verification should be equipped with a recommended eight separate longitudinal strain gages to determine bending contributions from both eccentric and angular misalignment of the grip heads. Although it is possible to use a minimum of six separate longitudinal strain gages for test specimens with circular cross sections, eight strain gages are recommended here for simplicity and consistency in describing the technique for both circular and rectangular cross sections. Dummy test specimens used for alignment verification, should have the same geometry and dimensions of the actual test specimens as well as similar mechanical properties (that is elastic modulus, hardness, etc.) as the test material to ensure similar axial and

bending stiffness characteristics as the actual test specimen and material.

6.3.2 *Fixed Load-Train Couplers*—Fixed couplers may incorporate devices that require either a one-time, pre-test alignment adjustment of the load train which remains constant for all subsequent tests or an in-situ, pre-test alignment of the load train which is conducted separately for each test specimen and each test. Such devices (8, 9) usually employ angularity and concentricity adjusters to accommodate inherent load-train misalignments. Regardless of which method is used, perform an alignment verification as discussed in 6.3.1.1

6.3.3 *Non Fixed Load-Train Couplers*—Non fixed couplers may incorporate devices that promote self-alignment of the

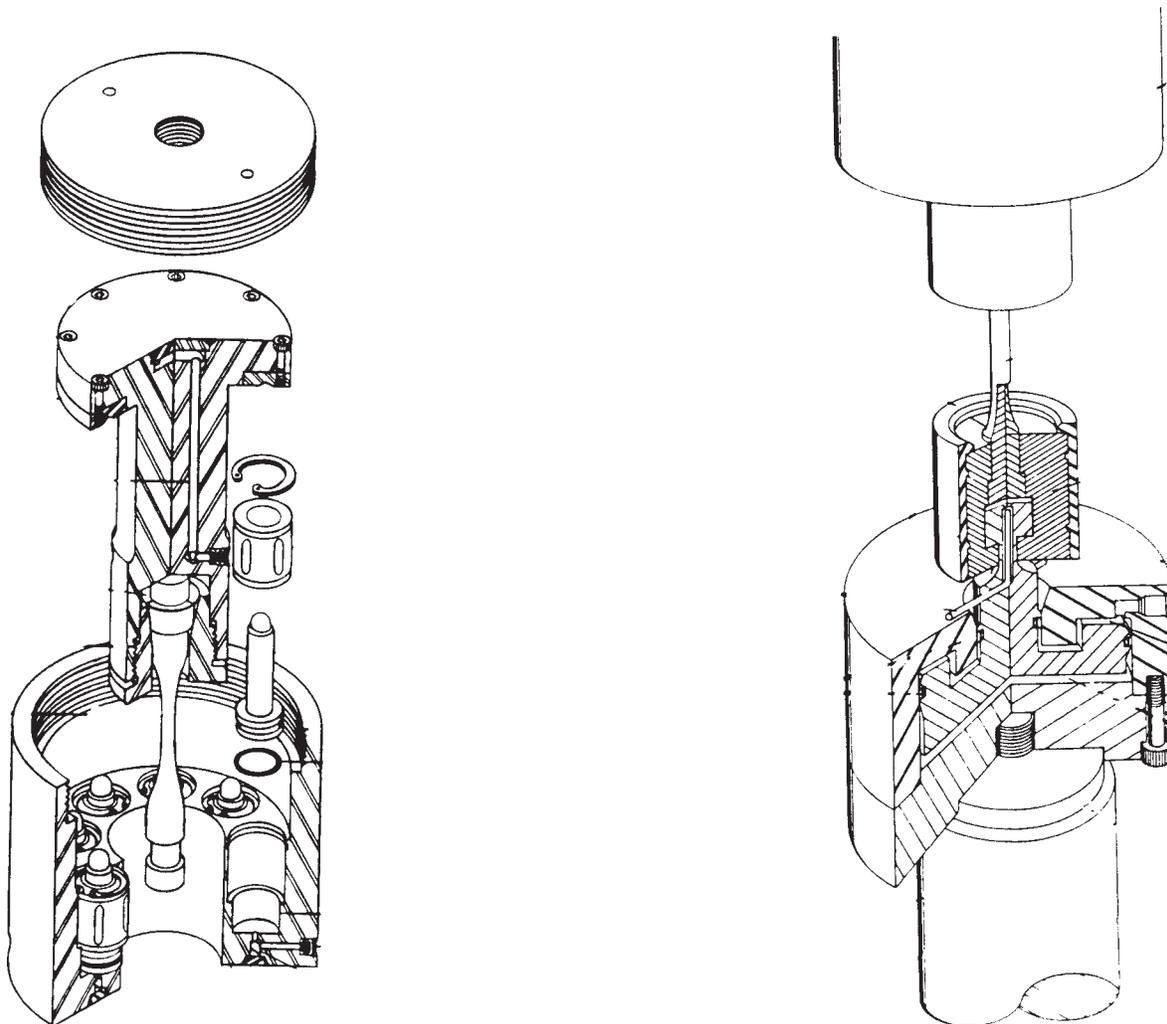


FIG. 7 Examples of Hydraulic, Self-Aligning, Non-Fixed Load Train Couplers (11, 12)

load train during the movement of the crosshead or actuator. Generally such devices rely upon freely moving linkages to eliminate applied moments as the load-train components are loaded. Knife edges, universal joints, hydraulic couplers or air bearings are examples (5, 8, 10, 11, 12) of such devices. Examples of two such devices are shown in Fig. 7. Although non fixed load-train couplers are intended to be self-aligning and thus eliminate the need to evaluate the bending in the test specimen for each test, verify the operation of the couplers and their effect on alignment as discussed in 6.3.1.1.

6.4 *Strain Measurement*—Although strain measurement techniques are not required in this test method, their use is recommended. Strain at elevated temperatures should be determined by means of a suitable extensometer. Appropriate strain measurements can be used to determine elastic constants in the linear region of the stress strain curves and can serve to indicate underlying fracture mechanisms manifested as nonlinear stress-strain behavior.

6.4.1 Extensometers shall satisfy Test Method E 83, Class B-1 requirements. Calibrate extensometers periodically in accordance with Test Method E 83. For extensometers mechanically attached to or in contact with the test specimen, the

attachment should be such so as to cause no mechanical damage to the test specimen surface. Extensometer contact probes must be chosen to be chemically compatible with the test material (for example alumina extensometer extensions and an SiC test specimen are incompatible). In addition, the weight of the extensometer should be supported so as not to introduce bending greater than that allowed in 6.5.

6.5 *Allowable Bending*—Analytical and empirical studies (4) have concluded that for negligible effects on the estimates of the strength distribution parameters (for example Weibull modulus, \hat{m} , and characteristic strength, $\hat{\sigma}_0$), allowable percent bending as defined in Practice E 1012 should not exceed five. These conclusions (4) assume that tensile strength fractures are due to fracture origins in the volume of the material, all tensile test specimens experienced for same level of bending, and that Weibull modulus, \hat{m} , was constant. Thus, the maximum allowable percent bending at fracture for test specimens tested under this test method shall not exceed five. Verify the testing system such that percent bending does not exceed five at a mean strain equal to either one half the anticipated strain at the onset of the cumulative fracture process (for example: matrix cracking stress) or a strain of 0.0005 (500 micro strain) whichever is

greater. Unless all test specimens are properly strain gaged and percent bending monitored until the onset of the cumulative fracture process, there will be no record of percent bending at the onset of fracture for each test specimen. Therefore, verify the alignment of the testing system. See Practice E 1012 for discussions of alignment and Appendix X1 for suggested procedures specific to this test method.

6.6 Heating Apparatus—The apparatus for, and method of, heating the test specimens shall provide the temperature control necessary to satisfy the requirement of 9.3.2.

6.6.1 Heating can be by indirect electrical resistance (heating elements), direct induction, indirect induction through a susceptor, radiant lamp, or direct resistance with the test specimen in ambient air at atmospheric pressure unless other environments are specifically applied and reported.

NOTE 3—While direct resistance heating may be possible in some types of electrically-conductive ceramics, it is not recommended in this test method since the potential exists for uneven heating or arcing, or both, at fracture.

6.7 Temperature-Measuring Apparatus—The method of temperature measurement shall be sufficiently sensitive and reliable to ensure that the temperature of the specimen is within the limits specified in 9.3.2.

6.7.1 For test temperatures less than 2000 K, make primary temperature measurements with noble-metal thermocouples in conjunction with potentiometers, millivoltmeters, or electronic temperature controllers or readout units, or both. Such measurements are subject to two types of error as discussed in MNL 12 (10). Firstly, thermocouple calibration and instrument measuring errors initially produce uncertainty as to the exact temperature. Secondly, both thermocouples and measuring instruments may be subject to variations over time. Common errors encountered in the use of thermocouples to measure temperatures include: calibration error, drift in calibration due to contamination or deterioration with use, lead-wire error, error arising from method of attachment to the test specimen, direct radiation of heat to the bead, heat-conduction along thermocouple wires, etc.

6.7.1.1 Measure temperature with thermocouples of known calibration (calibrated according to Test Method E 220). Calibrate representative thermocouples from each lot of wires used for making noble (for example, Pt or Rh/Pt) metal thermocouples. Except for relatively low temperatures of exposure, noble-metal thermocouples are eventually subject to error upon reuse, unless the depth of immersion and temperature gradients of the initial exposure are reproduced. Consequently, calibrate noble-metal thermocouples using representative thermocouples. Do not reuse degraded noble-metal thermocouples without proper treatment. This treatment includes clipping back the wire exposed to the hot zone, rewelding a thermocouple bead, and properly annealing the rewelded thermocouple bead and wire. Any reuse of noble-metal thermocouples (except after relatively low-temperature use) without this precautionary treatment shall be accompanied by recalibration data demonstrating that calibration of the temperature reading system was not unduly affected by the conditions of exposure.

6.7.1.2 Measurement of the drift in calibration of thermocouples during use is difficult. When drift is a problem during

tests, devise a method to check the readings of the thermocouples on the test specimen during the test. For reliable calibration of thermocouples after use, reproduce the temperature gradient of the test furnace during the recalibration.

6.7.1.3 Thermocouples containing Pt are also subject to degradation in the presence of silicon and silicon-containing compounds. Platinum silicides may form leading to several possible outcomes. One outcome is the embrittlement of the noble-metal thermocouple tips and their eventual degradation and breakage. Another outcome is the degradation of the silicon-containing material (for example, test specimen, furnace heating elements or refractory furnace materials). In all cases, do not allow platinum containing materials to contact silicon containing materials. In particular, do not allow noble-metal thermocouples to contact silicon-based test materials (for example, SiC or Si₃N₄). In some cases (for example, when using SiC heating elements), it is advisable to use ceramic-shielded noble-metal thermocouples to avoid the reaction of the Pt-alloy thermocouples with the SiO gas generated by the volatilization of the SiO₂ protective layers of SiC heating elements.

6.7.1.4 Calibrate temperature-measuring, controlling, and recording instruments versus a secondary standard, such as precision potentiometer, optical pyrometer, or black-body thyristor. Check lead-wire error with the lead wires in place as they normally are used.

6.7.2 For test temperatures greater than 2000 K, less-common temperature measurement devices such as thermocouples of elevated-temperature, non noble-metal alloys (for example W-Re) or optical pyrometry may be used. Since widely-recognized standards do not exist for these less-common devices, report the type of measurement device, its method of calibration, and its accuracy and precision.

6.8 Data Acquisition—At a minimum, obtain an autographic record of applied force versus time. Either analog chart recorders or digital data acquisition systems can be used for this purpose although a digital record is recommended for ease of later data analysis. Ideally, an analog chart recorder or plotter should be used in conjunction with the digital data acquisition system to provide an immediate record of the test as a supplement to the digital record. Recording devices shall be accurate to within $\pm 1\%$ of the selected range for the testing system including readout unit, as specified in Practice E 4, and should have a minimum data acquisition rate of 10 Hz with a response of 50 Hz deemed more than sufficient.

6.8.1 Where strain or elongation of the gage section are also measured, these values should be recorded either similarly to the force or as independent variables of force. Cross-head displacement of the test machine may also be recorded but should not be used to define displacement or strain in the gage section.

6.8.2 At a minimum, record temperature as single points at the initiation and completion of the actual test. However, temperature can also be recorded similarly to force and strain except the record can begin at the start of the heating of the furnace (including ramp-up to test temperature) and ending at the completion of the test.

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

7. Hazards

7.1 **Precaution**—During the conduct of this test method, the possibility of flying fragments of broken test material is quite 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 safety as well as later fractographic reconstruction and analysis is highly recommended.

8. Test Specimen

8.1 Test Specimen Geometry

8.1.1 *General*—The geometry of a tensile test specimen is dependent on the ultimate use of the tensile strength data. For example, if the tensile strength of an as-fabricated component is required, the dimensions of the resulting tensile test specimen may reflect the thickness, width, and length restrictions of the component. If it is desired to evaluate the effects of inherent flaw distributions for a particular material manufactured from a particular processing route then the size of the test specimen and resulting gage section will reflect the desired volume to be sampled. In addition, grip interfaces and load-train couplers as discussed in Section 6 will influence the final design of the test specimen geometry.

8.1.1.1 Fig. 8 illustrates a range of tensile test specimen geometries which have been applied to testing advanced ceramics. Fig. 8 provides only a sampling of possible tensile test specimens for ceramics and by no means purports to represent all possible configurations past or present. The following sections discuss the more common, and thus proven, of these test specimen geometries although any geometry is acceptable if it meets the gripping and bending requirements of this test method. If deviations from the recommended geometries are made, a stress analysis of the test specimen should be conducted to ensure that stress concentrations which could lead to undesired fractures outside the gage sections do not exist. Additionally, the success of an elevated-temperature tensile test will depend on the type of heating system, extent of test specimen heating, and test specimen geometry since these factors are all interrelated. For example, thermal gradients may introduce additional stress gradients in test specimens which may already exhibit stress gradients at ambient temperatures due to geometric transitions. Therefore, untried test configurations should be simultaneously analyzed for both loading-induced stress gradients and thermally-induced temperature gradients to ascertain any adverse interactions.

NOTE 4—An example of such an analysis is shown in Fig. 9 for a monolithic silicon nitride cylindrical button-head tensile test specimen

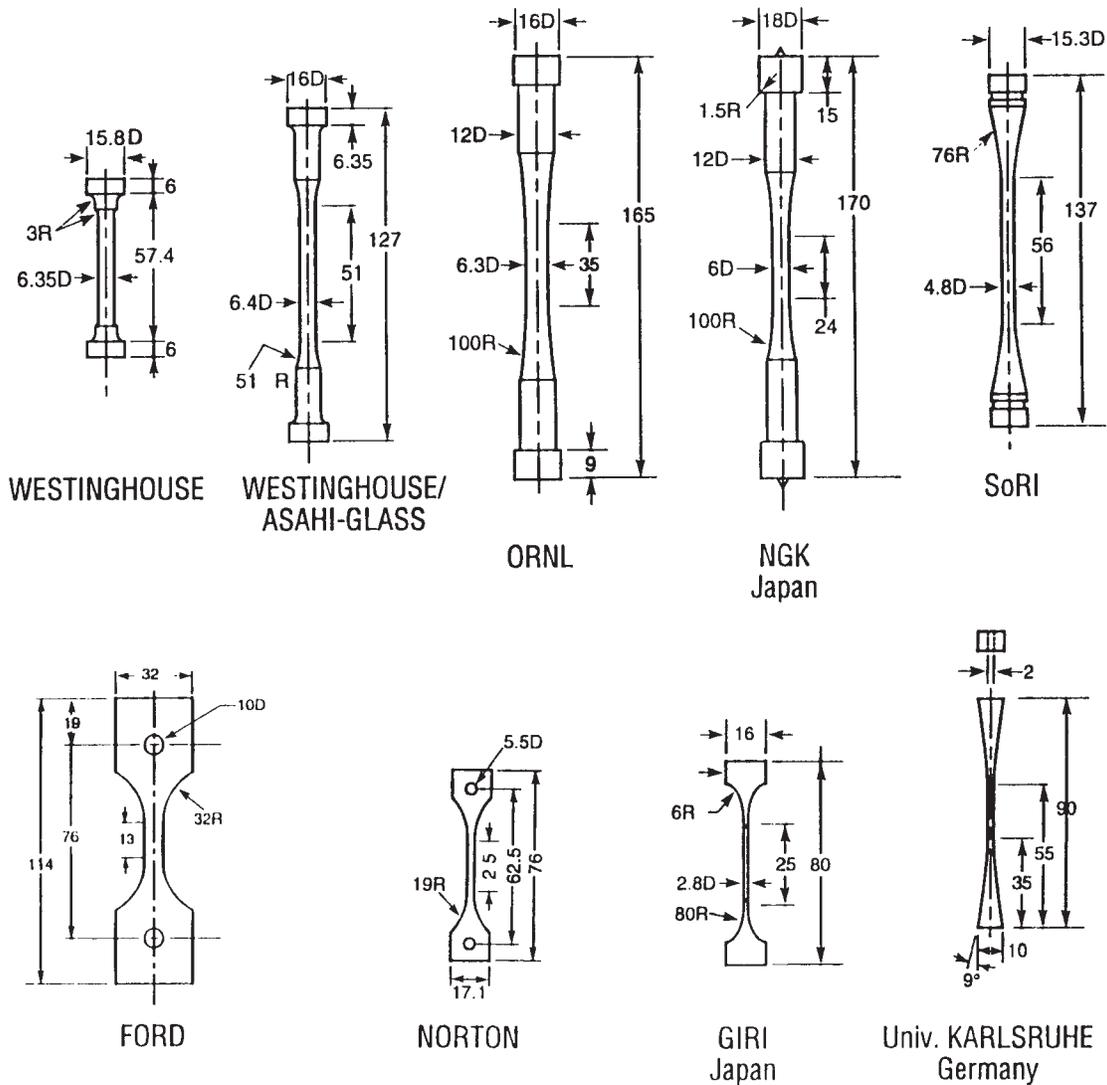
with water-cooled grip heads and a resistance-heated furnace heating only the center 50 mm of the test specimen. This example is a finite element analysis of a specific case for a specific material and test specimen test configuration. Thus, Fig. 9 is intended only as an illustrative example and should not be construed as being representative of all cases with similar test configurations.

8.1.2 *Cylindrical Tensile Test Specimens*—Cylindrical test specimens are generally fabricated from rods of material and offer the potential of testing the largest volume of the various tensile test specimens. In addition, the size of the test specimen lends itself to more readily evaluating the mechanical behavior of a material for engineering purposes. Disadvantages include the relatively large amount of material required for the starting billet, the large amount of material which must be removed during test specimen fabrication, and the need to fabricate the test specimen cylindrically, usually requiring numerically controlled grinding machines, all of which may add substantially to the total cost per test specimen. Gripped ends include various types of button-heads (4, 8, 9, 11, 12, 13) as shown in Fig. 10, Fig. 11, and Fig. 12. In addition, straight shank geometries have been successfully used (2, 3) as shown in Fig. 13 and Fig. 14. Important tolerances for the cylindrical tensile test specimens include concentricity and cylindricity that will vary depending on the exact configuration as shown in the appropriate test specimen drawings.

8.1.3 *Flat Tensile Test Specimens*—Flat test specimens are generally fabricated from plates or blocks of material and offer the potential for ease of material procurement, ease of fabrication, and subsequent lower cost per test specimen. Disadvantages include the relatively small volume of material tested and sensitivity of the test specimen to small dimensional tolerances or disturbances in the load train. Gripped ends include various types of shoulder-loaded shanks (5, 6) as shown in Fig. 15 and Fig. 16. In addition, pin-loaded gripped ends (7) have also been used successfully as shown in Fig. 17. Gage sections of flat tensile test specimens for strength measurements are sometimes cylindrical. While this type of gage section adds to the difficulty of fabrication and therefore cost of the flat tensile test specimen it does not avoid the problem of fractures initiating at corners of non cylindrical gage sections. Corner fractures may be initiated by stress concentrations due to the elastic constraint of the corners but are more generally initiated by damage (chipping, etc.) which can be treated by chamfering the corners similar to that recommended for rectangular cross section bars used for flexure tests (See Text Method C 1161). Important tolerances for the flat tensile test specimens include parallelism of faces and longitudinal alignment of load lines (pin hole centers or shoulder loading points) all of which will vary depending on the exact configuration as shown in the appropriate test specimen drawings.

8.2 Test Specimen Preparation:

8.2.1 Depending upon the intended application of the tensile strength data, use one of the following test specimen preparation procedures. Regardless of the preparation procedure used, report sufficient details regarding the procedure to allow replication.



NOTE 1—All dimensions are in millimetres.

Acronyms: ORNAL = Oak Ridge National Laboratory; NGK = NGK Spark Plug Co.; SoRI = Southern Research Institute; ASEA = ASEA-Ceram; NIST = National Institute of Standards and Technology; GIRI = Government Industrial Research Institute

FIG. 8 Examples of Variety of Tensile Test Specimens Used for Advanced Ceramics

8.2.2 *As-Fabricated*—The tensile test specimen should stimulate the surface/edge conditions and processing route of an application where no machining is used; for example, as-cast, sintered, or injection molded parts. No additional machining specifications are relevant. As-processed test specimens might possess rough surface textures and non-parallel edges and as such may cause excessive misalignment or be prone to non-gage section fractures, or both.

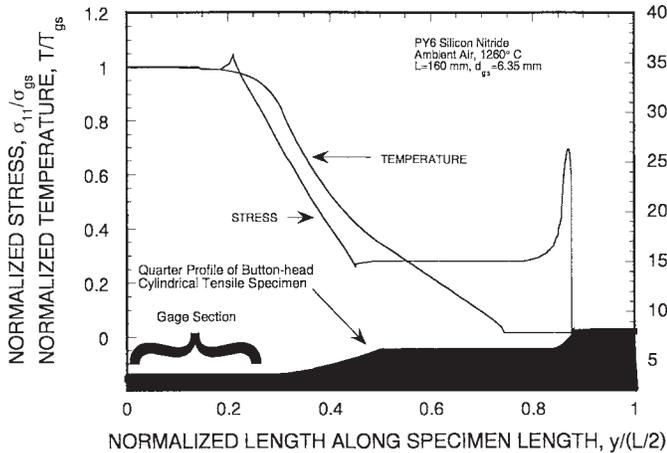
8.2.3 *Application - Matched Machining*—The tensile test specimen should have the same surface/edge preparation as that given to the component. Unless the process is proprietary, the report should be specific about the stages of material removal, diamond grits, diamond-grit bonding, amount of material removed per pass, and type of coolant used.

8.2.4 *Customary Practices*—In instances where a customary machining procedure has been developed that is completely satisfactory for a class of materials (that is, it induces no

unwanted surface/subsurface damage or residual stresses), this procedure should be used.

8.2.5 *Standard Procedure*—In instances where 8.2.2 through 8.2.4 are not appropriate 8.2.5 should apply. This procedure should serve as minimum requirements and a more stringent procedure may be necessary.

8.2.5.1 All grinding or cutting should be done with ample supply of appropriate filtered coolant to keep the workpiece and grinding wheel constantly flooded and particles flushed. Grinding should be done in at least two stages, ranging from coarse to fine rate of material removal. All cutting can be done in one stage appropriate for the depth of cut. The direction of the tangential velocity (due to angular velocity) of the grinding wheel at the point of contact with the test specimen surface should be principally parallel to the longitudinal axis of the test specimen.



NOTE 1—The shaded area at the bottom of the graph represents a section view of one fourth of the tensile specimen cross section from the button headed to the center of the gage section.

FIG. 9 Example of Superposed Stress and Temperature Results from Finite Element Analyses of a Monolithic Silicon Nitride, Button-Head Tensile Test Specimen with Water-Cooled Grips and Resistance-Heated Furnace Heating Only the Center 50 mm of the Test Specimen

8.2.5.2 Material removal rate should not exceed 0.03 mm per pass to the last 0.06 mm. Final finishing should be performed with diamond tools that have between 320 and 600 grit. No less than 0.06 mm per face should be removed during the final finishing phase, and at a rate not more than 0.002 mm per pass. Remove equal stock from each face where applicable.

8.2.5.3 Edge finishing should be comparable to that applied to test specimen surfaces. In particular, the direction of machining should be parallel to the longitudinal axis of the test specimen.

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

8.2.5.5 Generally, surface finishes on the order of average roughness, R_a , of 0.2–0.4 μm are recommended to minimize surface fractures related to surface roughness. However, in some cases the final surface finish may not be as important as the route of material removal due to the generation of subsurface damage during the material removal process.

8.2.5.6 Geometric features such as holes, button-head radiuses, or transition radiuses require just as stringent attention to fabrication detail as that paid to the gage section. Therefore the minimum requirements outlined here should be applied to these geometric features as well as to the gage section.

8.2.6 *Cylindrical Tensile Test Specimen Procedure*—Because of the axial symmetry of the button-head tensile test specimen, fabrication of the test specimens is generally conducted on a lathe-type apparatus. In many instances, the bulk of the material is removed in a circumferential grinding operation with a final, longitudinal grinding operation performed in the gage section to assure that any residual grinding marks are parallel to the applied stress. Beyond those guidelines provided here, Ref. (4) provides more specific details of recommended fabrication methods for cylindrical tensile test specimens.

8.2.6.1 Generally, computer numerical control (CNC) fabrication methods are necessary to obtain consistent test specimens with the proper dimensions within the required tolerances. A necessary condition for this consistency is the complete fabrication of the test specimen without removing it from the grinding apparatus, thereby avoiding the introduction of unacceptable tolerances into the finished test specimen.

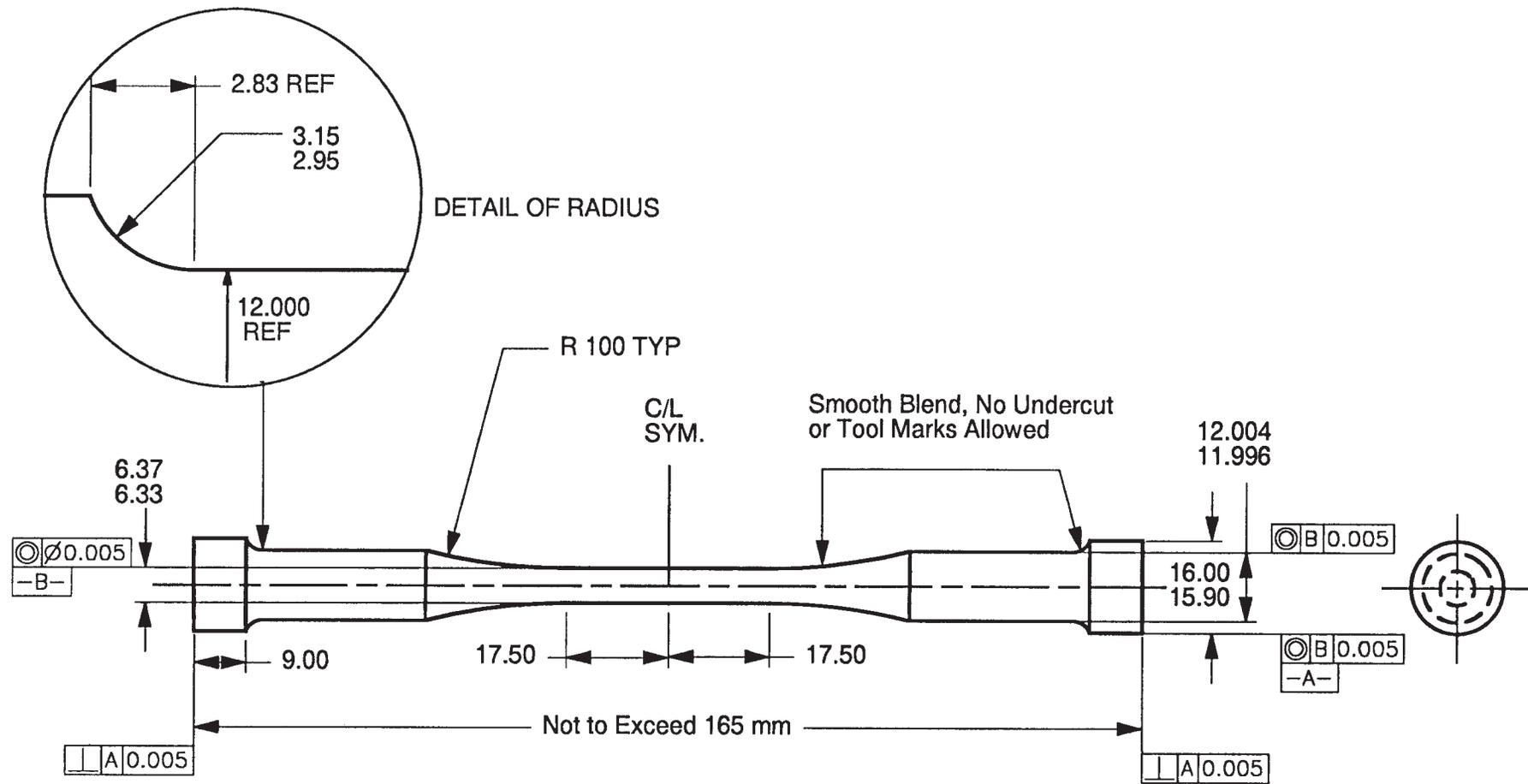
8.2.6.2 Formed, resinoid-bonded, diamond-impregnated wheels (minimum 320 grit in a resinoid bond) are necessary to both fabricate critical shapes (for example, button-head radius) and to minimize grinding vibrations and subsurface damage in the test material. The formed, resin-bonded wheels require periodic dressing and shaping (truing), which can be done dynamically within the test machine, to maintain the cutting and dimensional integrity.

8.2.6.3 The most serious concern is not necessarily the surface finish (on the order of $R_a = 0.2$ to $0.4 \mu\text{m}$) which is a result of the final machining steps. Instead, the subsurface damage is critically important although this damage is not readily observed or measured, and, therefore must be inferred as the result of the grinding history. More details of this aspect have been discussed elsewhere (4). In all cases, the final grinding operation ('spark out') performed in the gage section is to be along the longitudinal axis of the test specimen to assure that any residual grinding marks are parallel to the applied stress.

8.3 *Handling Precaution*—Extreme care should be exercised in storage and handling of finished test specimens to avoid the introduction of random and severe flaws (for example test specimens impact or scratch against each other). Therefore, store each test specimen in separate nonmetallic containers or in a nonmetallic container in which dividers restrict test specimens from contact with each other. In addition, give attention to pre-test storage of test specimens in controlled environments or desiccators to avoid unquantifiable environmental degradation of test specimens prior to testing.

8.4 *Number of Test Specimens*—As discussed in Practice C 1239, the total number of test specimens plays a significant role in the estimates of strength distribution parameters (for example Weibull modulus, \hat{m} , and characteristic strength, $\hat{\sigma}_0$). Initially, the uncertainty associated with parameter estimates decreases significantly as the number of test specimens increases. However a point of diminishing returns is reached when the cost of performing additional tensile strength tests may not be justified. This suggests that a practical number of tensile strength tests should be performed to obtain a desired level of confidence associated with a parameter estimate. Additional details concerning the determination of the strength distribution parameters are provided in Practice C 1239.

8.4.1 It is impossible to state the actual number of test specimens required under this test method since the number of test specimens needed depends on the precision required in the resulting parameter estimate and thus depends on the unique requirements of each application. Practice C 1239 requires the reporting of 90 % confidence bounds for Weibull modulus, \hat{m} , and characteristic strength, $\hat{\sigma}_0$ when a single flaw population is responsible for strength distributions. As an illustrative example, Table 1 shows the upper and lower 90 % confidence

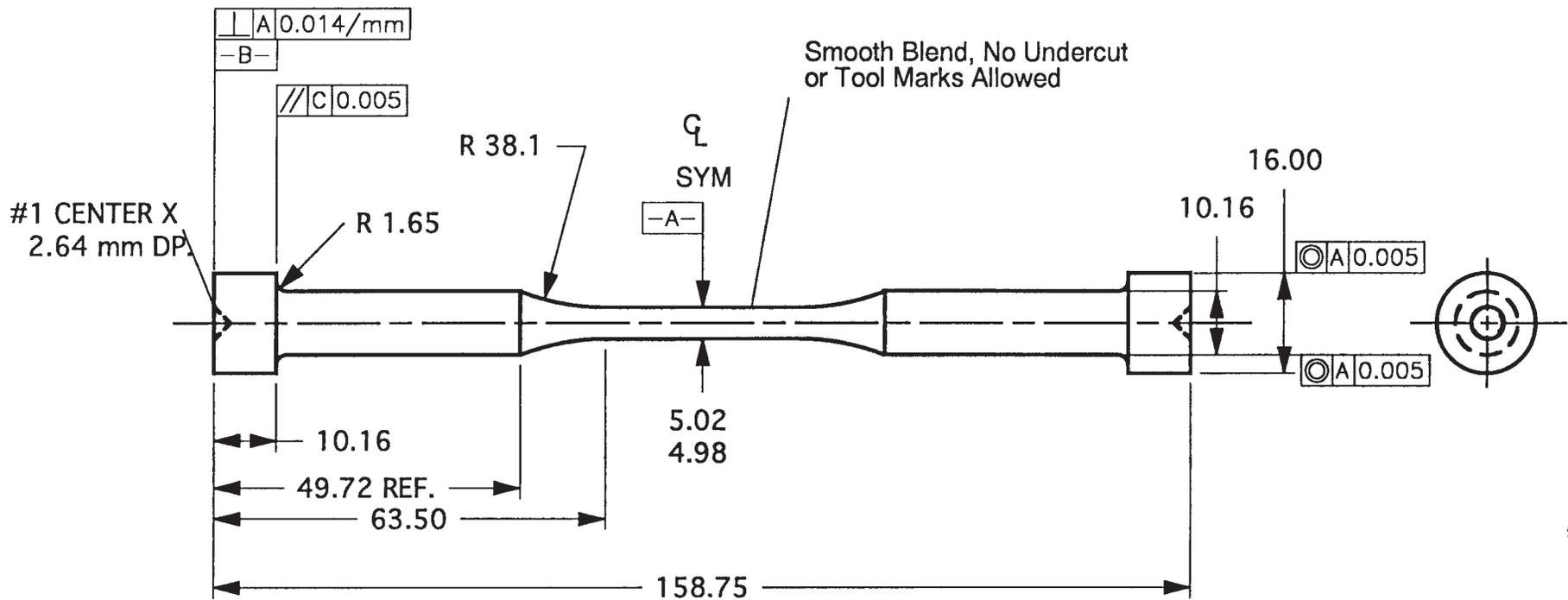


ASTM C 1366 - 04

NOTE: 1) SURFACE FINISH 0.20-0.40 μm ALL OVER EXCEPT BUTTON-HEAD RADIUS WHICH MUST BE $\leq 0.25 \mu\text{m}$ AND END FACES WHICH MAY BE 0.80 μm .
2) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL

Tensile Specimen for Advanced Ceramics
mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001 SCALE: NTS

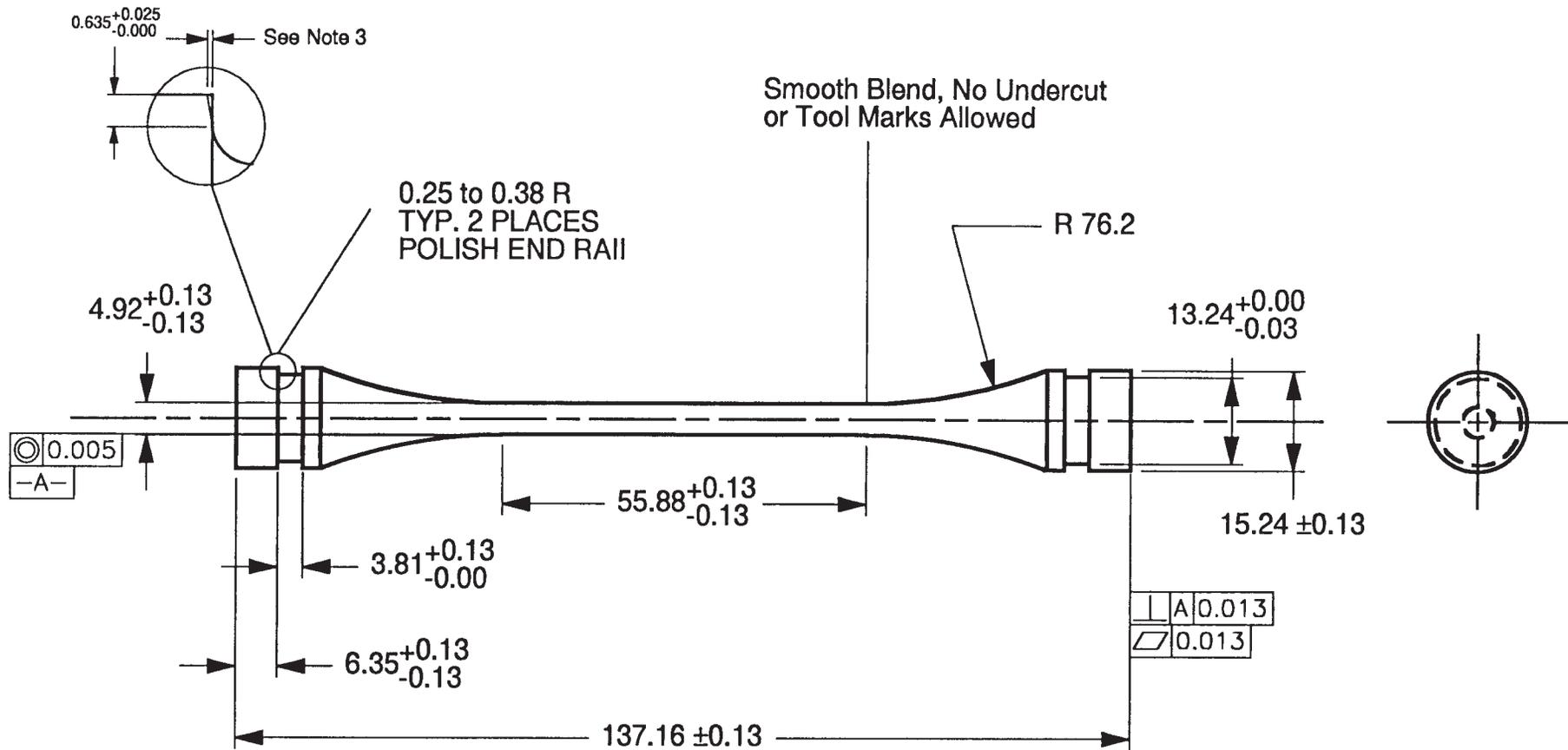
NOTE 1—Illustration not intended to be engineering or production drawing.
FIG. 10 Example of a Cylindrical Button-Head Tensile Test Specimen (4)



NOTE: 1) SURFACE FINISH $0.4 \mu\text{m}$ ALL OVER EXCEPT END FACES WHICH MAY BE $0.8 \mu\text{m}$.
 2) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL

Tensile Specimen for Advanced Ceramics
mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001 SCALE: NTS

NOTE 1—Illustration not intended to be an engineering or production drawing.
 FIG. 11 Example of a Cylindrical, Button-Head Tensile Test Specimen (12)

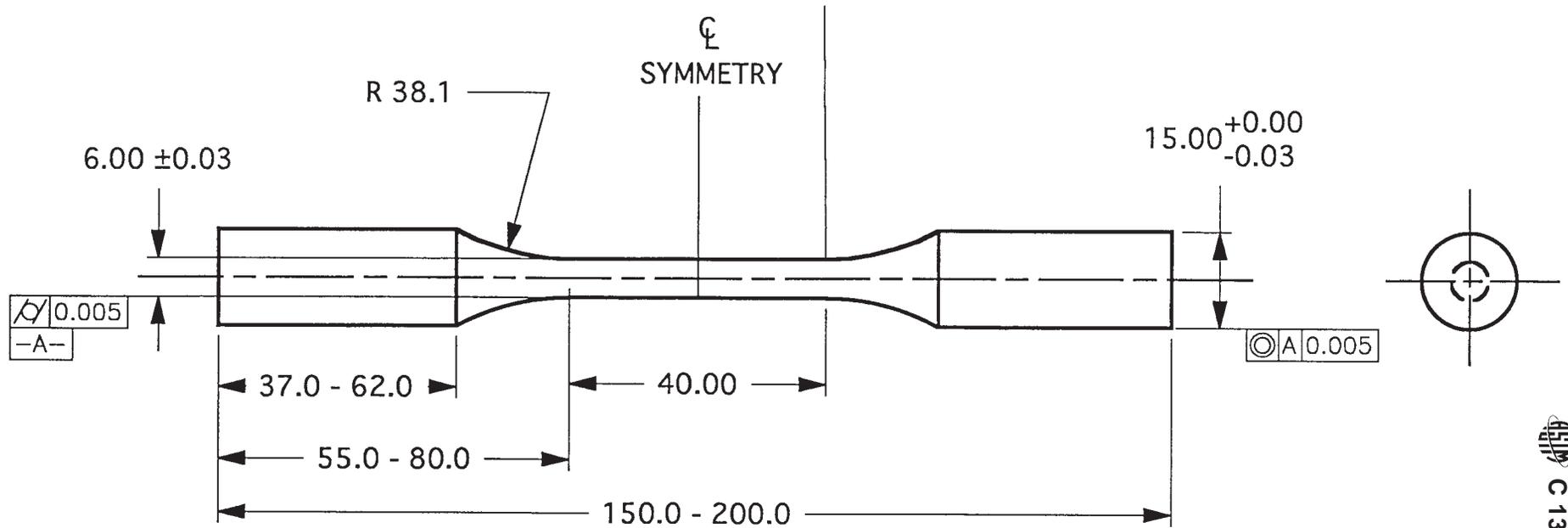


- NOTE: 1) SURFACE FINISH 0.4 μm ALL OVER
 2) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL
 3) EDGE SHOULD BE FLAT AND PERPENDICULAR TO +0.013/-0.000 mm

Tensile Specimen for Advanced Ceramics
mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001 SCALE: NTS

NOTE 1—Illustration not intended to be an engineering or production drawing.
 FIG. 12 Example of a Cylindrical, Button-Head Tensile Test Specimen (14)

Smooth Blend, No Undercut
or Tool Marks Allowed

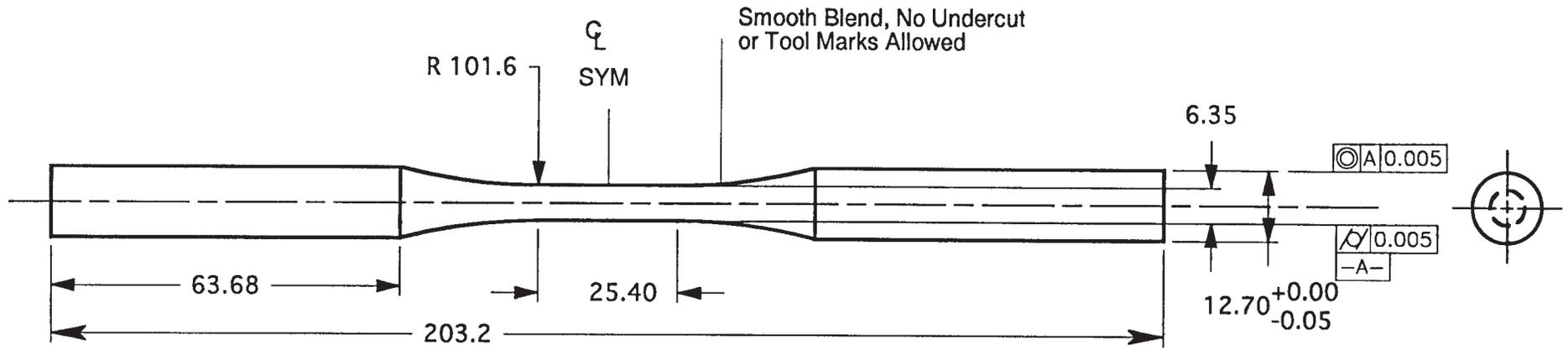


- NOTE: 1) SURFACE FINISH $0.4 \mu\text{m}$ ALL OVER EXCEPT END FACES WHICH MAY BE $0.8 \mu\text{m}$.
 2) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL
 3) TURNING CENTERS PERMITTED BOTH ENDS

Tensile Specimen for Advanced Ceramics
mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001 SCALE: NTS

NOTE 1—Illustration not intended to be an engineering or production drawing.
 FIG. 13 Example of a Flat, Shoulder-Loaded Tensile Test Specimen (9)

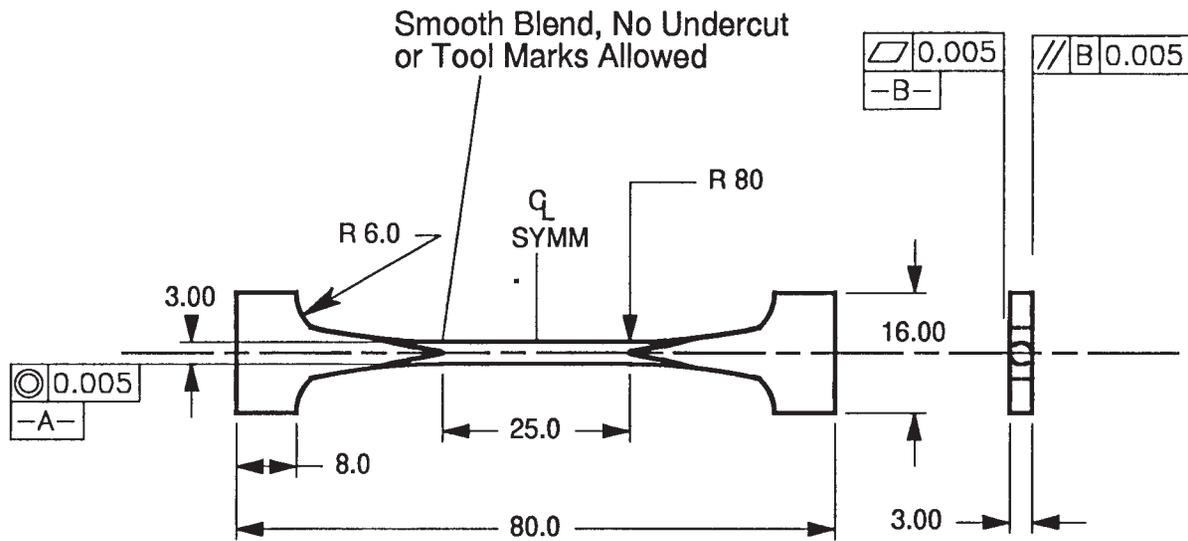
ASTM C 1366 - 04



NOTE: 1) SURFACE FINISH $0.4 \mu\text{m}$ ALL OVER EXCEPT END FACES WHICH MAY BE $0.8 \mu\text{m}$.
 2) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL

Tensile Specimen for Advanced Ceramics
mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001 SCALE: NTS

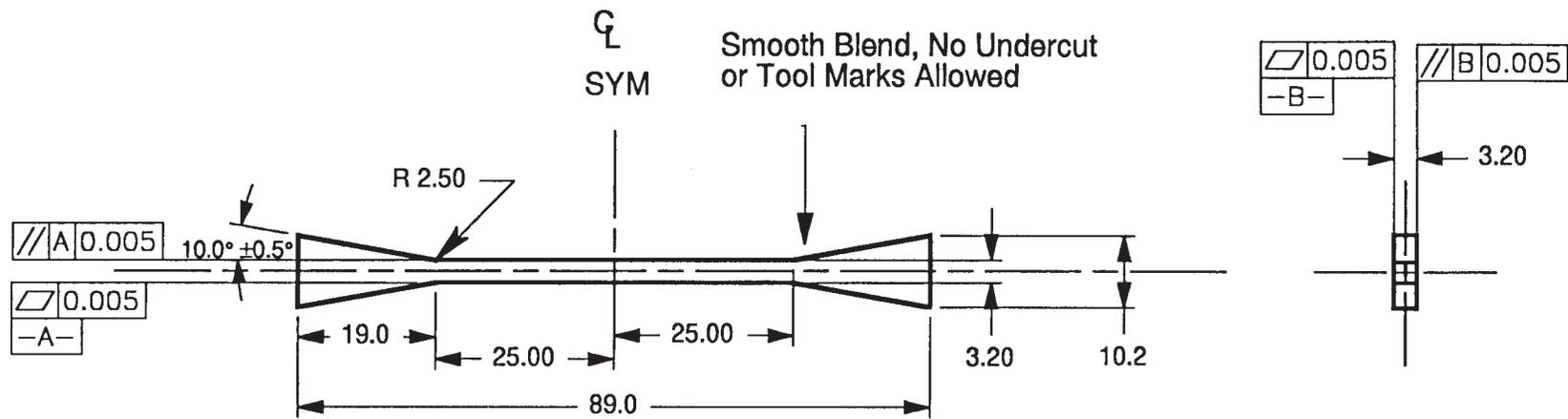
NOTE 1—Illustration not intended to be an engineering or production drawing.
 FIG. 14 Example of a Cylindrical, Straight-Shank Tensile Test Specimen (3)



NOTE: 1) SURFACE FINISH $0.4 \mu\text{m}$ ALL OVER EXCEPT END FACES WHICH MAY BE $0.8 \mu\text{m}$.
 2) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL

Tensile Specimen for Advanced Ceramics
mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001 SCALE: NTS

NOTE 1—Illustration not intended to be an engineering or production drawing.
 FIG. 15 Example of a Flat, Shoulder-Loaded Tensile Test Specimen (5)

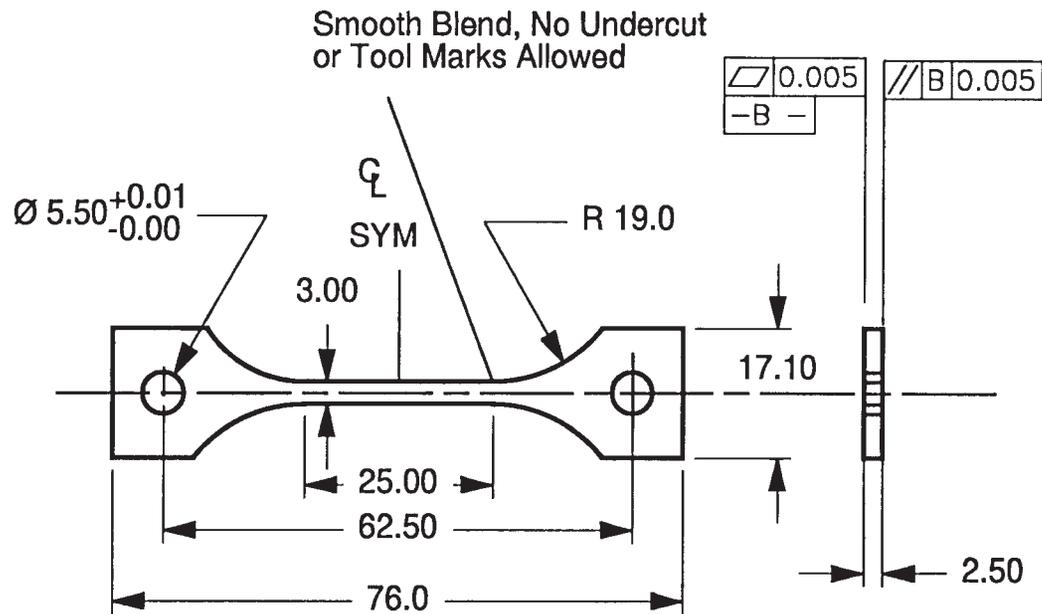


NOTE: 1) SURFACE FINISH $0.4 \mu\text{m}$ ALL OVER EXCEPT END FACES WHICH MAY BE $0.8 \mu\text{m}$.
 2) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL FOR ALL FOUR FACES

Tensile Specimen for Advanced Ceramics

mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001
 SCALE: NTS

NOTE 1—Illustration not intended to be an engineering or production drawing.
 FIG. 16 Example of a Flat, Shoulder-Loaded Tensile Test Specimen (6)



NOTE: 1) SURFACE FINISH 0.4 μm ALL OVER EXCEPT END FACES WHICH MAY BE 0.8 μm .
 2) FINAL GRIND OF GAGE SECTION TO BE LONGITUDINAL FOR ALL FOUR FACES

Tensile Specimen for Advanced Ceramics
mm X.X = 0.1, X.XX = 0.01, X.XXX = 0.001 SCALE: NTS

NOTE 1—Illustration not intended to be an engineering or production drawing.
 FIG. 17 Example of a Flat, Shoulder-Loaded Tensile Test Specimen (7)

TABLE 1 Example of Upper and Lower 90 % Confidence Bounds for Weibull Parameter Estimates Assuming a Single Flaw Population^A

Number of Test Specimens, n	m_{upper}	m_{lower}	$(\sigma_{\theta})_{\text{upper}}$	$(\sigma_{\theta})_{\text{lower}}$
5	14.6	3.6	566	448
10	13.5	5.5	534	469
30	12.2	7.5	517	483

^AFor a biased Weibull modulus, m of 10 characteristic strength, σ_{θ} , of 500 MPa.

bounds for \hat{m} , and $\hat{\sigma}_{\theta}$ for 5, 10, and 30 tests assuming a biased \hat{m} of 10 and $\hat{\sigma}_{\theta}$ of 500 MPa for a single flaw population. As a rule of thumb a minimum of five tests can be conducted to determine an indication of material properties if material cost or test specimen availability limit the number of tests to be conducted. A minimum of ten tests is required for the purposes of estimating a mean.

8.5 *Valid Tests*—A valid individual test is one which meets all the following requirements: (1) all the testing requirements of this test method, and (2) failure occurs in the uniformly-stressed gage section unless those tests failing outside the gage section are interpreted as interrupted tests for the purpose of censored test analysis.

9. Procedure

9.1 *Test Specimens Dimensions*—Determine the diameter or thickness and width of the gage section of each test specimen to within 0.02 mm. Make measurements on at least three different cross sectional planes in the gage section. In the case of the cylindrical test specimens make two measurements (90° apart) on each plane. To avoid damage in the critical gage section area make these measurements either optically (for example, using an optical comparator) or mechanically using a flat, anvil-type micrometer. In either case, the resolution of the instrument shall be as specified in 6.9. Exercise extreme caution to prevent damage to the test specimen gage section. Record and report the measured dimensions and locations of the measurements for use in the calculation of the tensile stress at fracture. Use the average of the multiple measurements in the stress calculations.

NOTE 5—Ball-tipped or sharp-anvil micrometers may damage the test specimen surface by inducing localized cracking and therefore, are not recommended.

9.1.1 Alternatively, to avoid damage to the gage section post-fracture measurements of the gage section dimensions can be made using procedures described in 9.1. In some cases, the fracture process can severely fragment the gage section in the immediate vicinity of the fracture thus making post-fracture measurements of dimensions difficult. In these, cases it is advisable to follow the procedures outlined in 9.1 for pretest measurements to assure reliable measurements.

9.1.2 Conduct periodic, if not 100 %, inspection/measurements of all test specimens and test specimen dimensions to assure compliance with the drawing specifications. Generally, high-resolution optical methods (for example an optical comparator) or high-resolution digital point contact methods (for example coordinate measurement machine) are satisfactory as long as the equipment meets the specifications in 6.9. The frequency of gage section fractures and bending in

the gage section are dependent on proper overall test specimen dimensions within the required tolerances.

9.1.3 In some cases it is desirable, but not required, to measure surface finish to quantify the surface condition. Such methods as contacting profilometry can be used to determine surface roughness of the gage section. When quantified, report the direction(s) of the surface roughness measurement and surface roughness as average surface roughness, R_a , or root-mean-square surface roughness, R_q , at a minimum.

9.2 Test Modes and Rates:

9.2.1 *General*—Test modes and rates can have distinct and strong influences on the fracture behavior of advanced ceramics even at ambient temperatures depending on test environment or condition of the test specimen. Test modes may involve load, displacement, or strain control. Recommended rates of testing are intended to be sufficiently rapid to obtain the maximum possible tensile strength at fracture of the material. However, rates other than those recommended here may be used to evaluate rate effects. In all cases, report the test mode and rate.

9.2.2 *Force Rate*—For most advanced ceramics exhibiting linear elastic behavior, fracture is attributed to a weakest-link fracture mechanism generally attributed to stress-controlled fracture from Griffith-like flaws. Therefore, a load-controlled test, with load generally related directly to tensile stress in brittle, linear elastic advanced ceramics, is the preferred test mode. Force rate can be directly related to stress rate thus simplifying data analysis. Calculate load rate as:

$$\dot{P} = \frac{dP}{dt} = \sigma A \quad (1)$$

where:

\dot{P} = required force rate, N/s,

P = applied force, N,

t = time, s,

σ = recommended (or desired stress rate), MPa/s, and

A = cross sectional area of the specimen gage section, mm².

Calculate the cross sectional area A as:

$$A = wb \text{ for rectangular cross sections} \quad (2)$$

or:

$$A = \frac{\pi d^2}{4} \text{ for circular cross sections} \quad (3)$$

where:

w = width of the gage section, mm,

b = thickness of the gage section, mm, and

d = diameter of the gage section, mm.

NOTE 6—Stress rates > 35 to 50 MPa/s are recommended to reduce the influence of environmental effects and thus obtain the greatest value of ultimate tensile strength. Alternatively, select stress rates to produce final fracture in 5 to 10 s to minimize environmental effects when testing in ambient air. Some materials may not be as sensitive to stress rate, and less rapid stress rates may be employed in these situations.

9.2.3 *Displacement Rate*—The size differences of each test specimen geometry require a different loading rate for any given stress rate. Displacement mode is defined as the control

of, or free-running displacement of, the test machine cross head. Thus the displacement rate can be calculated as follows. Calculate \dot{P} using the required (desired) stress rate as discussed in 9.2.2. Calculate the displacement rate as:

$$\dot{\delta} = \frac{d\delta}{dt} = \left(\frac{1}{k_m} + \frac{1}{k_s} \right) \dot{P} \quad (4)$$

where:

- $\dot{\delta}$ = required (desired) displacement rate of the cross head, mm/s,
- δ = cross-head displacement, mm,
- k_m = stiffness of the test machine and load train (including the test specimen ends and the grip interfaces), N/mm,
- k_s = stiffness of the uniform gage section of the test specimen, N/mm.

NOTE 7—For L as the unrippled length of the test specimen, A as the cross sectional area of the gage section, and E as the elastic modulus of the test material at the elevated test temperature, k_s can be calculated as $k_s = AE/L$. The stiffness k_m can be determined as described in Test Method D 3379 by measuring the force-displacement curves for various test specimen lengths. The plot of k_m (slope of force-displacement curve) versus test specimen gage length is then extrapolated to zero to find the actual machine stiffness. Alternatively, k_m can be estimated using the manufacturer's value for frame stiffness as a starting point and decreasing this value as necessary to account for various links in the load train. If such a method is used, report the assumptions and methods for approximating k_m .

9.2.4 Strain Rate—Strain is the independent variable in non linear analyses such as yielding. As such, strain rate is a method of controlling tests of deformation processes to avoid “runaway” (that is, uncontrolled, rapid failure) conditions. For the linear elastic behavior of most advanced ceramics at ambient temperatures, strain rate can be calculated directly from the required (desired) stress rate such that:

$$\dot{\epsilon} = \frac{d\epsilon}{dt} = \frac{\dot{\sigma}}{E} \quad (5)$$

where:

- $\dot{\epsilon}$ = strain rate in the test specimen gage section, /s, and,
- ϵ = strain in the test specimen gage section.

Strain-controlled tests can be accomplished using an extensometer contacting the gage section of the test specimen as the primary control transducer.

NOTE 8—Strain-controlled tests at elevated temperature using extensometers not tightly attached to tensile test specimens can be problematic. Any slippage can cause erratic behavior of the test machine leading to possible damage to the test machine or test specimen. Conduct strain-controlled tests with caution.

9.2.5 Ramp Segments—Normally, tests are conducted in a single ramp function in a at a single test rate from zero force to the maximum force at fracture. However, in some instances multiple ramp segments might be employed. In these cases a slow test rate is used to ramp from zero force to an intermediate force to allow time for deformation of collet material to critical radii (for example, button-head fillets) in the test specimen. The final ramp segment of the test is conducted from the intermediate force to the maximum force at fracture at the

required (desired) test rate, although hold times are not allowed to avoid environmental effects. Report the type and time duration of the ramp.

9.3 Temperature Control—If thermocouples are used, form the thermocouple bead in accordance with Test Method E 21. Do not attach noble-metal (for example Pt or Rh) thermocouples directly to advanced ceramics to avoid possible chemical incompatibility. The thermocouple junction may be brought close to the test specimen (3 to 6 mm) and shielded from thermal radiation in the furnace. Shielding may be omitted if, for a particular furnace, the difference in indicated temperature from an unshielded bead and a bead inserted in a hole in the test specimen has been shown to be less than one half the variation listed in 9.3.2. Make the bead as small as possible although there should be no shorting of the circuit (such as could occur from twisted wired behind the bead). Use ceramic insulators on the thermocouples in the hot zone. If some other electrical insulation material is used in the hot zone, carefully check it to determine whether the electrical insulating properties are maintained at greater temperatures.

9.3.1 Number of Required Thermocouples—When the length of the test specimen gage section is 25 to 50 mm and thermocouples are used, employ at least two thermocouples, one near each end of the gage section. For lengths of > 50 mm, add a third thermocouple near the center of the gage section length.

9.3.2 Temperature Limits—For the duration of the test do not permit the difference between the indicated temperature and the nominal test temperature exceed the following limits:

$\leq 1273 \text{ K}$	$\pm 3 \text{ K}$
$> 1273 \text{ K}$	$\pm 6 \text{ K}$

In addition, temperature gradient within the uniformly-heated gage section shall not exceed the following per 25 mm of gage section length:

$\leq 773 \text{ K}$	$\pm 5 \text{ K}$
$> 773 \text{ K}$	$\pm 1\% \text{ of the temperature (K)}$

9.3.3 The term “indicated temperature” means the temperature that is indicated by the temperature-measuring device using good quality pyrometric practice. True temperature may vary more than the indicated temperature. The permissible indicated temperature variations of 9.3.2 are not to be construed as minimizing the importance of good pyrometric practice and precise temperature control. All laboratories should keep both indicated and true temperature variations as small as practicable. In view of the extreme dependency of strength of materials on temperature, close temperature measurement is necessary. The limits prescribed represent ranges that are common practice.

9.3.4 Temperature overshoots during heating shall not exceed the following limits:

$\leq 1273 \text{ K}$	3 K
$> 1273 \text{ K}$	6 K

Study the heating characteristics of the furnace and the temperature control system to determine the power input, temperature set point, proportioning control adjustment, and control-thermocouple placement to limit transient temperature overshoots. It may be desirable to stabilize the furnace at a temperature 10 to 25 K less than the nominal test temperature

before making the final adjustments. Report any temperature overshoots with details of magnitude and duration.

9.3.5 Temperature Rates and Hold Time—The rate at which temperature can be increased from ambient to the final test temperature depends on many factors, such as: heating system, temperature controller, test material, and test environment. The hold time at temperature prior to the start of the test should be governed by the time necessary to ensure that the test specimen has reached equilibrium, the time necessary to stabilize the strain-measurement device, and time necessary to ensure that temperature can be maintained within the limits specified in 9.3.2. This hold time should generally not exceed 30 min. Report both the time to attain test temperature and the time at temperature before loading.

NOTE 9—When tensile testing for intrinsic strength, limit time at temperature to that necessary to equilibrate the test specimen at the test temperature. Limiting time at the test temperature will minimize oxidation or time-dependent thermal degradation. In addition, some materials experience degradation due to intermediate-temperature chemical instabilities which occur at temperatures much less than upper-limit elevated temperatures. In these materials, ramp the temperature as rapidly as possible to minimize the exposure time to these intermediate temperatures. Good results have been obtained for heating rates in which the test specimen temperature is ramped from ambient to the test temperature in approximately 30 min.

9.4 Conducting the Tensile Test:

9.4.1 Mounting the Test Specimen—Each grip interface and test specimen geometry described in Sections 6 and 8 will require a unique procedure for mounting the test specimen in the load train. Report any special components (for example, annealed, copper collets) required for each test. Mark the test specimen outside the heated section with an indelible marker as to the top and front (side facing the operator) in relation to the test machine.

9.4.2 Preparations for Testing—Set the test mode and test rate on the test machine. Preload the test specimen to remove the “slack” (that is, loose and non-tensioned) from the load train. For each situation, determine and report the amount of preload which will depend on the material and tensile test specimen geometry. Ready the autograph data acquisition systems for data logging. If desired, begin recording furnace temperature when furnace heating is initiated and continue recording until the completion of the test.

NOTE 10—Thermal expansion of the test specimen during heating may lead to i) changes in alignment if the tensile preload is reduced or ii) build up of axial compressive forces in a fixed actuator system which may damage the test specimen if force control test mode is not employed. The preload should be sufficient to maintain force train alignment while in force control. If load control is not available, the actuator position can be adjusted as necessary during heat up to maintain a preload sufficient to hold the load train alignment.

9.4.2.1 If no extensometry is used enclose the test specimen in the elevated-temperature furnace. Lightly pack refractory insulation to “seal” the test specimen and furnace. Be sure that the insulation is not packed overly tight so as to restrict pullrods or to introduce extraneous lateral or axial forces. Ready the autograph data acquisition systems for data logging. Heat the test specimen to the test temperature at the prescribed rate and hold constant at temperature until the test specimen has reached thermal equilibrium.

9.4.2.2 If extensometry is used, depending on the extensometer, mount it on the test specimen either while the system is cold (ambient-temperature) or after the test specimen has been heated to the test temperature (elevated-temperature) as detailed in the following paragraphs.

9.4.2.3 If the extensometer is mounted to a cold test specimen, mount the extensometer on the specimen gage section at ambient temperature and zero the output. Enclose the test specimen in the elevated-temperature furnace and lightly pack refractory insulation to “seal” the test specimen and furnace. Do not pack insulation overly tight so as to restrict the extensometer arms or pullrods or to introduce extraneous lateral or axial forces. Heat the test specimen to the test temperature at the prescribed rate and hold constant at temperature until the test specimen has reached thermal equilibrium. When the test specimen has reached thermal equilibrium, re-zero the extensometer before conducting the test.

9.4.2.4 If the extensometer is to be mounted to a hot specimen, enclose the test specimen in the elevated-temperature furnace and lightly pack refractory insulation to “seal” the test specimen and furnace. Do not pack insulation overly tight so as to restrict the extensometer arms or pullrods or to introduce extraneous lateral or axial forces. Heat the test specimen to the test temperature at the prescribed rate and hold constant at temperature until the test specimen has reached a desired temperature (usually near or at the test temperature). Mount the extensometer on the test specimen gage section and zero the output. When the test specimen has reached thermal equilibrium, re-zero the extensometer before conducting the test.

9.4.3 Conducting the Test—If test temperature is not recorded continuously, record the test temperature at test initiation. Initiate the data acquisition. Initiate the test mode. After test specimen fracture, disable the action of the test machine and the data collection of the data acquisition system. Record the breaking force with an accuracy of 1.0 % of the force range. Record test temperature at test completion. Cool the test specimen and test apparatus to ambient temperature. Carefully remove the test specimen from the grip interfaces. Take care not to damage the fracture surfaces by preventing them from contacting each other or other objects. Place the test specimen along with any fragments from the gage section into a suitable, non-metallic container for later analysis.

9.4.4 Determine the ambient temperature and relative humidity in accordance with Test Method E 337.

9.4.5 Post-Test Dimensions—If necessary, measure and report gage section cross-sectional dimensions at the fracture location if the gage section has not been overly fragmented by the fracture process. If an exact measure of the cross-sectional dimensions cannot be made due to fragmentation then use the average dimensions measured in 9.1

9.4.5.1 Measure and report the fracture location relative to the midpoint of the gage section. Use the convention that the midpoint of the gage section is 0 mm with positive (+) measurements toward the top of the test specimen as tested (and marked) and negative (–) measurements toward the bottom of the test specimen as tested (and marked). For fracture surfaces which are not normal to the longitudinal axis

the average fracture location may be reported. Record and report the orientation of the fracture and fracture locations.

NOTE 11—Results from test specimens fracturing outside the uniformly stressed gage section are not recommended for use in the direct calculation of a mean tensile strength at fracture for the entire test set. Results from test specimens fracturing outside the uniformly stressed gage section are considered anomalous and can be used only as censored tests (that is, test specimens in which a tensile stress at least equal to that calculated by Eq 6 was sustained in the uniform gage section before the test was prematurely terminated by a non-gage section fracture) as discussed in Practice C 1239 for the determination of estimates of the strength distribution parameters. From a conservative standpoint, in completing a required statistical sample (for example $n = 10$) for purposes of average strength, one replacement test specimen should be tested for each test specimen which fractures outside the uniformly-stressed gage section.

9.5 Fractography—Fractographic examination of each failed test specimen is recommended to characterize the fracture origins. The strength of an advanced ceramic is often limited by discrete fracture origins in the material. Porosity, agglomerates, inclusions, and atypical large grains are examples of fracture origins within the volume of the material. Fracture origins on the surface of the test specimen may be the result of contact stresses, impact events, or adverse environment. When the means are available, fractographic methods should be used to locate, identify, and classify the strength-limiting fracture origin in the advanced ceramic tensile test specimen. Moreover, for the purposes of estimating strength distribution parameters as detailed in Practice C 1239, each classification of fracture origins must be identified as a surface-distributed fracture origin or a volume-distributed fracture origin. Thus, several classifications of fracture origins may exist within the volumes or surface areas of the test specimens in a statistical sample. Fractography can be an interpretative analytical method and the guidelines established in Practice C 1322 and MIL-HDBK-790, should be used to establish objectivity.

10. Calculation

10.1 Tensile Strength—The standard formula for the tensile strength of a uniaxially loaded rod employs the uniaxial breaking force and the cross sectional area of the uniform gage section:

$$S_u = \frac{P_{\max}}{A} \quad (6)$$

where:

- S_u = the tensile strength in units of MPa,
- P_{\max} = the breaking force in units of N, and
- A = the cross sectional area in units of mm^2 such that:

$$A = w b \text{ for rectangular cross sections} \quad (7)$$

or:

$$A = \frac{\pi d^2}{4} \text{ for circular cross sections} \quad (8)$$

where:

- w = the average width of the gage section in units of mm as detailed in 9.1 and 9.1.1,

- b = the average thickness of the gage section in units of mm as detailed in 9.1 and 9.1.1, and
- d = the average diameter of the gage section in units of mm as detailed in 9.1 and 9.1.1.

10.2 Fracture Strain in Tension—If strain is measured in the uniform gage section of the test specimen, the standard formula for the fracture strain in tension of a uniaxially loaded rod can be calculated from the elongation at the breaking force and the original length of the uniform gage section:

$$\epsilon_f = \frac{(l_f - l_o)}{l_o} \quad (9)$$

where:

- ϵ_f = the engineering strain at fracture,
- l_f = the final length of the test specimen gage section in units of mm, and
- l_o = the original gage length of the test specimen in units of mm.

10.3 Modulus of Elasticity—If strain is measured in the uniform gage section of the test specimen the modulus of elasticity (that is, ratio of stress to strain below the proportional limit) can be calculated as the slope of the least squares regression fit of the linear portion of the engineering stress-engineering strain curve. Engineering stress is defined as:

$$\sigma = \frac{P}{A} \quad (10)$$

where:

- σ = the engineering stress in units of MPa,
- P = the applied, uniaxial tensile force in units of N, and
- A = the original cross sectional area in units of mm^2 as defined in Eq 7 and Eq 8.

Engineering strain is defined as:

$$\epsilon = \frac{(l - l_o)}{l_o} \quad (11)$$

where

- ϵ = the engineering strain,
- l = the extensometer gage length at any time in units of mm, and
- l_o = the original extensometer gage length in units of mm.

10.4 Mean, Standard Deviation, and Coefficient of Variation—For each series of tests the mean, standard deviation, and coefficient of variation for each measured value can be calculated as follows:

$$\text{Mean} = \bar{X} = \frac{\sum_{i=1}^n X_i}{n} \quad (12)$$

$$\text{Standard Deviation} = \text{s.d.} = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n-1}} \quad (13)$$

$$\text{Coefficient of variation} = V = \frac{100 (\text{s.d.})}{\bar{X}} \quad (14)$$

where:

X_i = the i th measured value and n is the number of valid tests.

11. Report

11.1 *Test Set*—Include in the report the following information for the test set. Indicate any significant deviations from the procedures and requirements of this test method:

11.1.1 Tensile test specimen geometry used (include engineering drawing),

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

11.1.3 Type, configuration, and resolution of strain measurement equipment if used (include drawing or sketch if necessary). If a commercial extensometer was used, the manufacturer and model number are sufficient for describing the strain measurement equipment,

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

11.1.5 Type and configuration of load-train couplers (include drawing or sketch if necessary). If a commercial load-train coupler was used, the manufacturer and model numbers are sufficient for describing the coupler,

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

11.1.7 Type and configuration of temperature measurement system (include drawing or sketch if necessary). If a commercial temperature measurement system was used, the manufacturer and model number are sufficient for describing the system. However, include the most recent calibration information with the test report.

11.1.8 Number (n) of test specimens tested validly (that is, fracture in the gage section). In addition, report the total number of test specimens tested (n_T) to provide an indication of the expected success rate of the particular test specimen geometry and test apparatus,

11.1.9 Where feasible and possible, all relevant material data including vintage or billet identification. As a minimum, report the date the material was manufactured,

11.1.9.1 For commercial materials, where feasible and possible, report the commercial designation and lot number,

11.1.9.2 For non-commercial materials, where feasible and possible, report the major constituents and proportions as well as the primary processing route including green state and consolidation routes,

11.1.10 Description of the method of test specimen preparation including all stages of machining,

11.1.11 Where feasible and possible, heat treatments, or pre-test exposures, if any, applied either to the as-processed material or to the as-fabricated test specimen,

11.1.12 Test environment including relative humidity (Test Method E 337), ambient temperature and atmosphere (for

example, ambient air, dry nitrogen, silicone oil, etc.), average elevated temperature and average hold time at elevated temperature.

11.1.13 Test mode (load, displacement, or strain control) and test rate (load rate, displacement rate, or strain rate). Report the calculated stress rate, if appropriate, in units of MPa/s.

11.1.14 Percent bending and corresponding average strain in the specimen recorded during the verification of load train alignment as measured at the beginning and end of the test series.

11.1.15 Mean tensile strength (S_u) and standard deviation (s.d.) and coefficient of variation (V).

11.1.16 Estimates of strength distribution parameters (for example, Weibull modulus, m , and characteristic strength, σ_0) as well as appropriate confidence bounds may be calculated and reported in accordance with Practice C 1239.

11.1.17 Mean fracture strain ($\bar{\epsilon}_f$) and standard deviation (s.d.) and coefficient of variation (V), if calculated, and

11.1.18 Mean elastic modulus (\bar{E}) and standard deviation (s.d.) and coefficient of variation (V), if calculated.

11.2 *Individual Test Specimens*—Report the following information for each test specimen tested. Report any significant deviations from the procedures and requirements of this test method:

11.2.1 Temperature of test in K , time to attain test temperature, time at temperature prior to testing, and test environment,

11.2.2 Pertinent overall test specimen dimensions, if measured, such as total length, length of gage section, gripped section dimensions, etc. in units of mm,

11.2.3 Average surface roughness in units of μm , if measured, of gage section and the direction of measurement,

11.2.4 Average cross sectional dimensions, in units of mm,

11.2.5 Pre-load and breaking load in units of N ,

11.2.6 Calculated tensile strength at fracture in units of MPa,

11.2.7 Elastic modulus (if calculated) in units of MPa,

11.2.8 Fracture strain (if calculated),

11.2.9 Percent bending and average strain at fracture (if measured),

11.2.10 Fracture location relative to the gage section midpoint in units of mm (+ is toward the top of the test specimen as marked and - is toward the bottom of the test specimen as marked with 0 being the gage section midpoint); and

11.2.11 Type and location of fracture origin (flaw) relative to the front of the test specimen as marked.

12. Precision and Bias

12.1 The tensile strength of an advanced ceramic is not a deterministic quality but will vary from one test specimen to another as well as from one type of geometry to another depending upon gage section volume or surface area (**15**, **16**). There will be an inherent statistical scatter in the results for finite statistical sample sizes (for example, 30 test specimens). Weibull statistics can model this variability as discussed in Practice C 1239. This test method is intended so that the precision is high and the bias is low compared to the inherent variability of strength of the material.

12.2 Because of the nature of the materials and lack of a wide data base on a variety of applicable advanced ceramics tested in tension at elevated temperatures, no definitive statement can be made at this time concerning precision and bias of the test methods of this test method.

13. Keywords

13.1 advanced ceramic; elevated temperatures; percent bending; tensile strength; tensile testing

APPENDIX

(Nonmandatory Information)

X1. VERIFICATION OF LOAD TRAIN ALIGNMENT AT ROOM TEMPERATURE

X1.1 Purpose of Verification—The purpose of this verification procedure is to demonstrate that the grip interface and load-train couplers can be used by the test operator in such a way as to consistently meet the limit on percent bending as specified in Section 6. Thus, this verification procedure should involve no more care in setup than will be used in the routine testing of the actual tensile test specimen. The bending under tensile force should be measured using verification (or actual) test specimens of exactly the same design as that to be used for the tensile tests. For the verification purposes, strain gages should be applied as shown in Fig. X1.1. Verification measurements should be conducted at the beginning and end of a series of tests with a measurement at the midpoint of the series recommended, whenever the grip interfaces and load-train couplers are installed on a different test machine, whenever a different operator is conducting a series of tests, whenever damage or misalignment is suspected. Since the verification test specimen uses adhesively-bonded strain gages, the verification procedure is to be conducted at room temperature with the implication that the load-train alignment will remain constant at elevated temperatures.

X1.2 Verification Test Specimen—The test specimen used

for verification must be machined very carefully with attention to all tolerances and concentricity requirements. Ideally the verification test specimen should be of identical material to that being tested. However, if this is not possible or desired, an alternate material should be used with elastic modulus, elastic strain capability, and hardness similar to the test material. The test specimen should be carefully inspected with an optical comparator before strain gages are attached to ensure that these dimensional requirements are met. After the strain gages are applied it will no longer be possible to meaningfully inspect the test specimen, so exercise care in handling and using it.

X1.2.1 For simplicity in applying this test method to test specimens with both circular and rectangular cross section gage sections, a minimum of eight foil resistance strain gages should be mounted on the verification test specimen as shown in X1.1. Separate the strain gage planes by at least $\frac{3}{4} l_o$ where l_o is the length of the reduced or designated gage section. Mount four strain gages, equally spaced (90° apart) around the circumference of the gage section on each of two planes located symmetrically about the longitudinal midpoint of the gage section and near either end of the gage section. Insure that the longitudinal centers of all strain gages on the same plane are within 0.5 mm of the same longitudinal distance along the test specimen axis.

NOTE X1.1—These strain gages should be as narrow as possible to minimize strain averaging. Strain gages having active widths of 0.25 to 0.5 mm and active lengths of 1.0-2.5 mm are commercially available and are suitable for this purpose (4). Take care to avoid placing the strain gages closer than one strain gage length from geometric transitions in the gage section which can cause strain concentrations and inaccurate measurements of the strain in the uniform gage section. In addition, to minimize errors due to misalignment of the strain gages, mount strain gages such that the sensing direction is $\pm 2^\circ$ of the longitudinal axis of the test specimen.

X1.3 Verification Procedure—Procedures for verifying alignment are described in detail in Practice E 1012. However, salient points and equations for square and circular cross-sections as currently contained in Practice E 1012 are described here for emphasis. Consult Practice E 1012 for specific details for rectangular cross-sections, especially when the thickness is too thin to strain gage all four sides. The following sections are not intended to replace practice E 1012, but rather are intended to elucidate those aspects which are directly applicable to this particular test method.

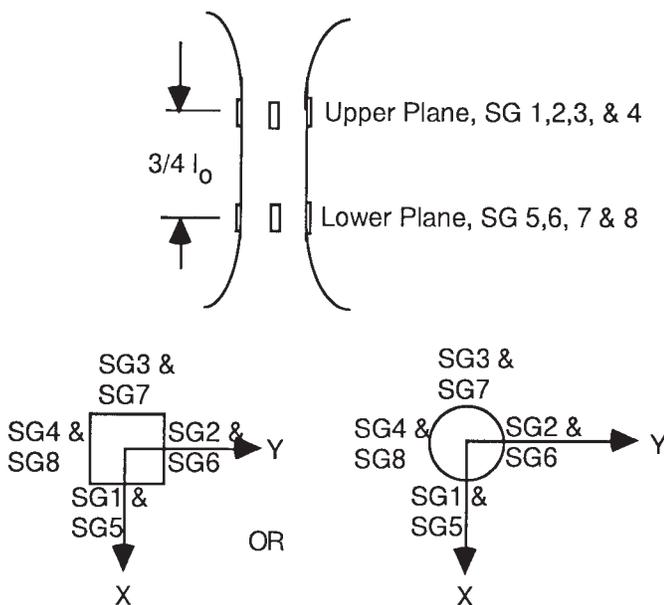


FIG. X1.1 Illustration of Strain Gage Placement on Gage Section Planes and Strain Gage Numbering.

X1.3.1 Mount the top of the test specimen in the grip interface.

X1.3.2 Connect the lead wires of the strain gages to the conditioning equipment and allow the strain gages to equilibrate under power for at least 30 min prior to conducting the verification tests. This will minimize drift during actual conduct of the verifications.

X1.3.3 Zero the strain gages before mounting the bottom of the test specimen in the grip interface. This will allow any bending due to the grips to be recorded.

X1.3.4 Mount the bottom of the test specimen in the grip interface.

X1.3.5 Apply a sufficient force to the test specimen to achieve an average strain of one half the anticipated fracture strain of the test material or a strain of 0.0005 (500 micro strain) whichever is greater. It is desirable to record the strain (and hence bending) as functions of the applied force to monitor any self alignment of the load train.

X1.3.6 Calculate percent bending as follows for *circular or square cross sections* referring to Fig. X1.1 for the strain gage numbers. Calculate percent bending at the upper plane of the gage section as:

$$PB_{upper} = \frac{\epsilon_b}{\epsilon_o} 100 \tag{X1.1}$$

$$\epsilon_b = \left[\left(\frac{\epsilon_1 - \epsilon_3}{2} \right)^2 + \left(\frac{\epsilon_2 - \epsilon_4}{2} \right)^2 \right]^{1/2} \tag{X1.2}$$

$$\epsilon_o = \frac{\epsilon_1 + \epsilon_2 + \epsilon_3 + \epsilon_4}{4} \tag{X1.3}$$

where: $\epsilon_1, \epsilon_2, \epsilon_3$ and ϵ_4 are strain readings for strain gages located at the upper plane of the gage section. Strain gage readings are in units of strain (that is, m/m) and compressive strains are negative.

X1.3.7 Calculate percent bending at the lower plane of the gage section for circular or square cross sections referring to Fig. X1.1 for the strain gage numbers as follows:

$$PB_{lower} = \frac{\epsilon_b}{\epsilon_o} 100 \tag{X1.4}$$

$$\epsilon_b = \left[\left(\frac{\epsilon_5 - \epsilon_7}{2} \right)^2 + \left(\frac{\epsilon_6 - \epsilon_8}{2} \right)^2 \right]^{1/2} \tag{X1.5}$$

$$\epsilon_o = \frac{\epsilon_5 + \epsilon_6 + \epsilon_7 + \epsilon_8}{4} \tag{X1.6}$$

where: $\epsilon_5, \epsilon_6, \epsilon_7$ and ϵ_8 are strain readings for strain gages located at the lower plane of the gage section. Strain gage readings are in units of strain (that is, m/m) and compressive strains are negative.

X1.3.8 For uniform bending across the gage section with the test specimen assuming a C-shape $PB_{upper} \approx PB_{lower}$. C-shape bending reflects angular misalignment of the grips. For non uniform bending across the gage section with the test specimen assuming a S-shape, PB_{upper} may or may not be equal to PB_{lower} . S-shape bending reflects eccentric misalignment of the grip centerlines. These general tendencies are shown in Fig. X1.2. Combinations of C and S shapes may exist. In these cases the S-shape should first be eliminated by adjusting the eccentricity of the grips such that the longitudinally aligned strain gages indicate approximately the same values (for example, $\epsilon_1 \approx \epsilon_5, \epsilon_2 \approx \epsilon_6$, etc.) More detailed discussions regarding bending and alignment are contained in Ref (16)

X1.3.9 Check the effect of the test specimen warpage by rotating the test specimen 180° about its longitudinal axis and performing the bending checks again. If similar results are obtained at each rotation then the degree of alignment can be considered representative of the load train and not indicative of the test specimen. If load-train alignment is within the specifications of 6.5, record the maximum percent bending and conduct the tensile tests. If the load-train alignment is outside the specifications of 6.5 then re-align or re-adjust the load train according to the specific procedures unique to the individual testing setup. Repeat this verification procedure to confirm the achieved alignment.

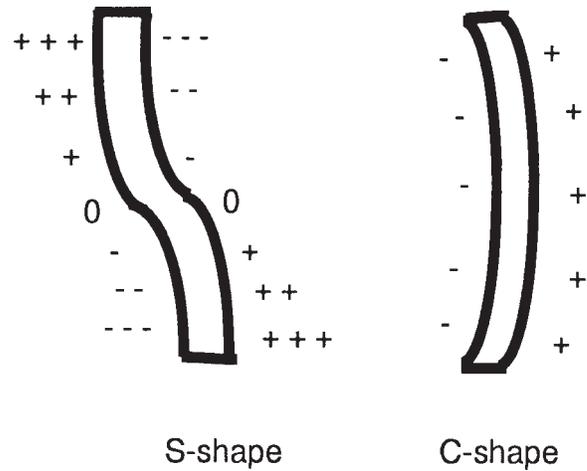


FIG. X1.2 S-Shape and C-Shape Bending of Tensile Test Specimen

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