Designation: C1862 – 17

Standard Test Method for the Nominal Joint Strength of End-Plug Joints in Advanced Ceramic Tubes at Ambient and Elevated Temperatures[1]

This standard is issued under the fixed designation C1862; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the push-out force, nominal joint strength, and nominal burst pressure of bonded ceramic end-plugs in advanced ceramic cylindrical tubes (monolithic and composite) at ambient and elevated temperatures (see 4.2). The test method is broad in scope and end-plugs may have a variety of different configurations, joint types, and geometries. It is expected that the most common type of joints tested are adhesively bonded end-plugs that use organic adhesives, metals, glass sealants, and ceramic adhesives (sintered powders, sol-gel, polymer-derived ceramics) as the bonding material between the end-plug and the tube. This test method describes the test capabilities and limitations, the test apparatus, test specimen geometries and preparation methods, test procedures (modes, rates, mounting, alignment, testing methods, data collection, and fracture analysis), calculation methods, and reporting procedures.

1.2 In this end-plug push-out (EPPO) test method, test specimens are prepared by bonding a fitted ceramic plug into one end of a ceramic tube. The test specimen tube is secured into a gripping fixture and test apparatus, and an axial compressive force is applied to the interior face of the plug to push it out of the tube. (See 4.2.) The axial force required to fracture (or permanently deform) the joined test specimen is measured and used to calculate a nominal joint strength and a nominal burst pressure. Tests are performed at ambient or elevated temperatures, or both, based on the temperature capabilities of the test furnace and the test apparatus.

1.3 This test method is applicable to end-plug test specimens with a wide range of configurations and sizes. The test method does not define a standardized test specimen geometry, because the purpose of the test is to determine the nominal joint strength and nominal burst pressure of an application-specific plug-tube design. The test specimen should be similar in size and configuration with the intended application and product design.

1.4 Calculations in this test method include a nominal joint strength which is specific to the adhesives, adherends, configuration, size, and geometry of the test specimen. The nominal joint strength has value as a comparative test for different adhesives and plug configurations in the intended application geometry. When using nominal joint strength for comparison purposes, only values obtained using identical geometries should be compared due to potential differences in induced stress states (shear versus tensile versus mixed mode). The joint strength calculated in this test may differ widely from the true shear or tensile strength (or both) of the adhesive due to mixed-mode stress states and stress concentration effects. (True adhesive shear and tensile strengths are material properties independent of the joint geometry.)

1.5 In this test, a longitudinal failure stress is being calculated and reported. This longitudinal failure stress acts as an engineering corollary to the burst pressure value measured from a hydrostatic pressure test, which is a more difficult and complex test procedure. Thus this longitudinal failure stress is recorded as a nominal burst pressure. As a general rule, the absolute magnitude of the nominal burst pressure measured in this EPPO test is different than the absolute magnitude of a burst pressure from a hydrostatic burst pressure test, because the EPPO test does not induce the hoop stresses commonly observed in a hydrostatic pressure test.

1.6 The use of this test method at elevated temperatures is limited by the temperature capabilities of the loading fixtures, the gripping method (adhesive, mechanical clamping, etc.), and the furnace temperature limitations.

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

1.8 *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.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the*

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Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1145 Terminology of Advanced Ceramics
C1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics
C1469 Test Method for Shear Strength of Joints of Advanced Ceramics at Ambient Temperature
D907 Terminology of Adhesives
D3878 Terminology for Composite Materials
D4896 Guide for Use of Adhesive-Bonded Single Lap-Joint Specimen Test Results
E4 Practices for Force Verification of Testing Machines
E6 Terminology Relating to Methods of Mechanical Testing
E105 Practice for Probability Sampling of Materials
E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process
E220 Test Method for Calibration of Thermocouples By Comparison Techniques
E230/E230M Specification for Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples
E251 Test Methods for Performance Characteristics of Metallic Bonded Resistance Strain Gages
E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)
E1012 Practice for Verification of Testing Frame and Specimen Alignment Under Tensile and Compressive Axial Force Application
IEEE/ASTM SI 10 American National Standard for Metric Practice

3. Terminology

3.1 *Definitions:*

3.1.1 The definitions of terms relating to strength testing appearing in Terminology E6 apply to the terms used in this test method. The definitions of terms relating to advanced ceramics appearing in Terminology C1145 apply to the terms used in this test method. The definitions of terms relating to fiber-reinforced composites appearing in Terminology D3878 apply to the terms used in this test method. The definitions of terms relating to adhesives in Terminology D907 apply to the terms used in this test method. Pertinent definitions as listed in Practice E1012, Terminology C1145, Terminology D3878, Terminology D907, and Terminology E6 are shown in the following with the appropriate source given in parentheses. Key terms are given below.

3.1.2 *adherend, n*—a body held to another body by an adhesive. **(D907)**

3.1.3 *adhesion failure, n*—rupture of an adhesive bond in which the separation appears visually to be at the adhesive/adherend interface. **(D907)**

3.1.4 *adhesive, n*—a substance capable of holding materials together by surface attachment. **(D907)**

3.1.4.1 *Discussion*—'Adhesive' is a general term and includes among others cement, glue, mucilage, and paste. All of these terms are loosely used interchangeably. Various descriptive adjectives are applied to the term 'adhesive' to indicate certain characteristics as follows: (*1*) physical form, that is, liquid adhesive, tape adhesive, etc.; (*2*) chemical type, that is, silicate adhesive, resin adhesive, etc.; (*3*) materials bonded, that is, paper adhesive, metal-plastic adhesive, can label adhesive, etc.; (*4*) condition of use, that is, hot setting adhesive, room temperature setting adhesive, etc.

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

3.1.6 *ceramic matrix composite, n*—material consisting of two or more materials (insoluble in one another), in which the major, continuous component (matrix component) is a ceramic while the secondary component(s) may be ceramic, glass/ceramic, glass, metal, or organic in nature. These components are combined on macroscale to form a useful engineering material possessing certain properties or behavior not possessed by the individual constituents. **(C1145)**

3.1.7 *cohesive failure, n*—rupture of a bonded assembly in which the separation appears visually to be in the adhesive or the adherend. **(D907)**

3.1.8 *elastic stress limit, [FL^{-2}], n*—the greatest stress which a material is capable of sustaining without any permanent strain remaining upon complete release of the stress, in units of MPa. **(E6)**

3.1.9 *joining, n*—controlled formation of chemical or mechanical bond, or both, between similar or dissimilar materials. **(C1469)**

3.1.10 *shear stress, [FL^{-2}], n*—the stress component tangential to the plane on which the forces act. **(E6)**

3.1.11 *true shear strength, [FL^{-2}], n*—the maximum uniform shear stress which a material is capable of sustaining in the absence of all normal stresses. **(D4896)**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *collet(s), n*—a sleeve placed on a shaft or tube and tightened so as to grip the shaft or tube.

3.2.1.1 *Discussion*—Collets may come in a variety of forms. A common example is a split conical collet which features a cone-shaped segmented sleeve that is tightened with a tapered collar.

3.2.2 *failure, n*—an arbitrary point beyond which a material or system ceases to be functional for its intended use.

3.2.2.1 *Discussion*—Failure strength is commonly defined by the force parameter (force, moment, torque, stress, etc.) applied to a test specimen that produces brittle fracture and loss of load-carrying capability or permanent deformation beyond a specified limit such as the elastic stress limit. Due to the

[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.

ceramic nature of the ceramic components being tested, failure will typically be catastrophic.

3.2.3 *nominal burst pressure, P_{NB} [FL^{-2}], n*—a burst pressure value calculated from the push-out force at failure and the face area of the end-plug in units of MPa.

3.2.4 *nominal joint strength, S_{NJ} [FL^{-2}], n*—the calculated strength at failure in units of MPa, calculated from the push-out force and the calculated adhesive bond area of the defined test specimen.

3.2.5 *push-out force, F_{PO} [F], n*—in a push-out test with a specific test specimen geometry and size, the force level at which failure occurs in units of N.

3.2.5.1 *Discussion*—Push-out force is defined at failure, however reductions in force during testing due to microcracking or other means that do not meet failure criteria may be tracked and reported.

4. Summary of Test Method

4.1 This test method is used to determine the push-out force, the nominal joint strength, and the nominal burst pressure of bonded ceramic end-plugs, typically using adhesives, in advanced ceramic cylindrical tubes (monolithics and composites) at ambient and elevated temperatures. Test specimens are prepared by bonding a fitted ceramic plug into one end of a ceramic tube. The test specimen tube is secured into a loading fixture and an axial compressive force is applied to the interior face of the end-plug until failure occurs. The axial force required to fracture (or yield) the test specimen joint is measured and used to calculate a nominal joint strength and a nominal burst pressure. Tests are done at ambient temperatures and at elevated temperatures, based on test furnace and test fixture temperature capabilities.

4.2 Typical end-joint test specimens and a typical test system are shown schematically in Figs. 1 and 2, respectively. Selection of the test specimen geometry and size depends on the functional design of the application-specific tube and the size limitations of the available test material.

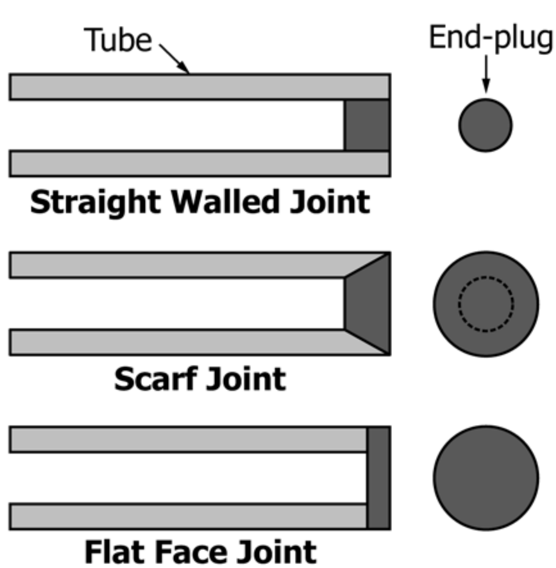


FIG. 1 Ceramic Test Specimens with Different End-Plug Configurations

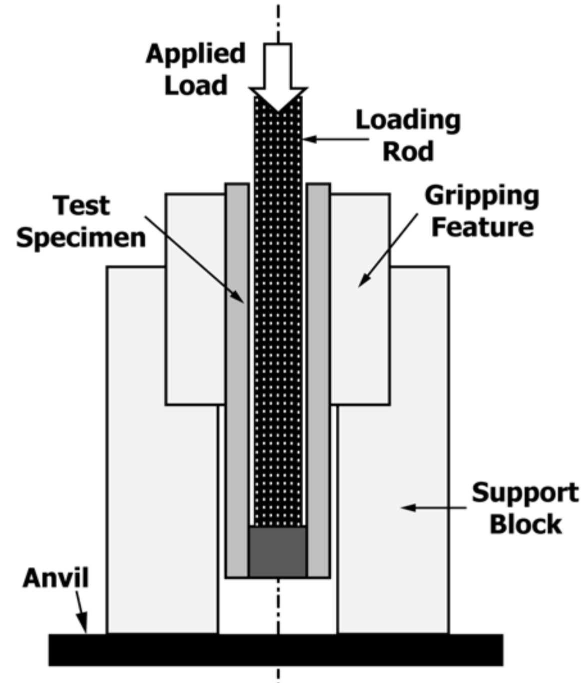


FIG. 2 Example EPPO Test Method Schematic

4.3 The force application arrangement of this test method is direct axial compression on the end face of the plug, where the predominant forces (shear, tensile, and mixed mode) occur in the circumferential adhesive bond section between the plug and the tube.

5. Significance and Use

5.1 Advanced ceramics are candidate materials for high-temperature structural applications requiring high strength along with wear and corrosion resistance. In particular, ceramic tubes are being considered and evaluated as hermetically tight fuel containment tubes for nuclear reactors. These ceramic tubes require end-plugs for containment and structural integrity. The end-plugs are commonly bonded with high-temperature adhesives into the tubes. The strength and durability of the test specimen joint are critical engineering factors, and the joint strength has to be determined across the full range of operating temperatures and conditions. The test method has to determine the breaking force, the nominal joint strength, the nominal burst pressure, and the failure mode for a given tube/plug/adhesive configuration.

5.2 The EPPO test provides information on the strength and the deformation of test specimen joints under applied shear, tensile, and mixed-mode stresses (with different plug geometries) at various temperatures and after environmental conditioning.

5.3 The end-plug test specimen geometry is a direct analog of the functional plug-tube application and is the most direct way of testing the tubular joint for the purposes of development, evaluation, and comparative studies involving adhesives and bonded products, including manufacturing quality control. This test method is a more realistic test for the intended geometry than the current shear test of ceramic joints (Test Method C1469), which uses an asymmetric four-point shear test on a flat adhesive face joint.

5.4 The EPPO test method may be used for joining method development and selection, adhesive comparison and screening, and quality assurance. This test method is not recommended for adhesive property determination, design data generation, material model verification/validation, or combinations thereof.

6. Interferences

6.1 The EPPO test in its basic form is a variation of the common single-lap joint shear test geometry, based on the rotation of a single-plane lap joint to form a cylindrical lap joint. So the complexities of the single-lap joint (as described in Guide D4896) are carried over to the EPPO test.

6.2 As described in Guide D4896, many factors (geometric, adhesive properties, adherend properties, force levels) affect the stress levels in the adhesive bond section and the failure strength values in a given experimental adhesive bond lap-type test. All of these factors interact to determine the actual stress levels at different points in the test specimen joint section. For full engineering analysis of the joint system and the test results, all of these factors should be carefully controlled and measured during testing.

6.2.1 The strain and stress conditions in the bond section may vary spatially, based on variations in the bond morphology and properties and the stress-strain interaction with the adherends. Critical factors are adhesive bond length and thickness, adhesive shear and tensile moduli and Poisson's ratio, adherend thickness, adherend shear and tensile moduli and Poisson's ratio, and interface surface conditions.

6.2.2 Depending on the type of adhesive and the process conditions, the adhesive bond may contain residual stresses and critical flaws that may affect the experimental strength. This is a particular concern with many of the high-temperature adhesives commonly used to bond advanced ceramics. In many cases, the residual stresses and critical flaw populations increase with larger bond section sizes and bond thicknesses.

6.3 Misalignment in the load system produces bending stresses in the joint that give erroneous test results. Bending stresses develop as a result of misaligned end-plugs in the tube specimens, out-of-tolerance test specimens (straightness and concentricity), out-of-tolerance test specimens and misfit of end-plugs, misalignment of the test specimen in the grip fixture, and misalignment load train components.

6.4 A common variable in adhesives is the different modes of joint failure: elastic-brittle versus ductile-plastic that occur for different types of adhesives and at different temperatures for a given adhesive. For each adhesive system and test condition, the failure criteria have to be appropriately defined to determine the point at which the adhesive functionally fails under stress.

6.5 The gripping mechanism shall be sufficiently strong at the test conditions so that the test specimen is securely held in the grip section and failure occurs in the end-plug section, not in the grip section of the test specimen. Grip failure is more likely at elevated temperatures, because of degradation of the grip adhesive at elevated temperatures and because of differential thermal expansion stresses between the grip fixture and the test specimen.

6.6 The adhesive properties may change with temperature and with time, either under test specimen conditioning or in aggressive test environments. In particular, ceramic and glass adhesives often fail by slow crack growth under moisture or elevated temperature conditions (or both), which may produce a different flaw population and microstructure, a change in failure mechanisms, or a combination thereof.

6.7 At elevated testing temperatures, differential thermal stresses caused by different thermal expansion coefficients between the end-plug, the adhesive, and the adherend often introduce additional stresses that may produce premature adhesive failure.

7. Apparatus

7.1 *Testing Machine*—Test specimens shall be tested in compressive loading with any suitable testing machine provided that uniform rates of direct loading are maintained. The force-measuring system shall be free of initial lag at the loading rates used, and shall be equipped with a means for retaining readout of the maximum force as well as a force-time or force-displacement record. Machines used for axial compression testing shall conform with and have an accuracy in accordance with Practices E4.

7.1.1 *Cross-Head Displacement Measurement*—The cross-head displacement should be measured as a record of the force-time response of the test specimen. Cross-head displacement of the test machine shall not be used to define displacement or strain in the end-plug test section.

7.1.2 *Force-Measurement Devices*—The measurement devices used in determining the force shall be accurate within ±1 % at any force within the selected load range of the testing machine as defined in Practices E4. Force calibration shall be performed in compression for universal machines.

7.2 *Test Apparatus Fixture:*

7.2.1 *General*—The test apparatus shall be designed, fabricated, and assembled so that the compressive force is applied to the test specimen axially, uniformly, and with negligible friction. The test apparatus shall apply an axial compressive force to the interior face of the end-plug without inducing excessive bending stresses or transverse shear stresses in the test specimen. Force application should be accomplished with a universal testing machine with appropriate gripping and loading fixtures. A typical test apparatus consists of a base plate, a support block, a gripping fixture, and a loading rod. A schematic of a test apparatus is shown in Fig. 2.

NOTE 1—It is not the intent of this test method to require specific loading and alignment fixtures for testing. Different test apparatus configurations can be designed and used for testing. The primary requirement is that the test fixture (as designed and fabricated) securely grips the test specimen and that the force is applied axially and uniformly. An example of an axial test apparatus for small ceramic tube specimens (10-mm diameter and 50 to 70 mm long) is described in Appendix X1.

7.2.2 The test apparatus shall be built with adequate materials and sized large enough to contain the test specimen and to support the applied forces without deformation or damage to the apparatus at the test temperatures. Flat bearing surfaces on the base plate, the support block, and the grip fixture shall have flat and parallel surfaces to within 0.002 m/m.

NOTE 2—At ambient temperatures, the fixture materials are commonly high-strength, high-hardness steels. At elevated temperatures (>500 °C), high-nickel alloys or high-strength ceramics (aluminum oxide, silicon carbide) are necessary for strength, hardness, and stability at the test temperature. Selected materials need to be compatible with materials being tested to avoid chemical interactions at high temperatures.

7.2.3 *Gripping Fixture*—A gripping fixture is necessary to secure the test specimen in the test apparatus without slipping or breakage while force is applied. The gripping fixture also aligns the test specimen in the load train. Gripping fixtures for tube specimens are grouped into two classes: mechanical grip fixtures (mechanical clamps, collets, and collars) and adhesive bonding into grips. The gripping mechanism should be designed to apply as uniform a pressure as possible across the test specimen in order to reduce induced stresses in the test specimen. Additional information on gripping methods can be found in Appendix X2.

NOTE 3—The brittle nature of advanced ceramics requires a uniform force application between the grip fixture and the gripped section of the test specimen. Line or point contacts and nonuniform forces can produce stress concentrations and Hertzian stresses, leading to crack initiation and fracture of the test specimen in the gripped section. The selection of a gripping method depends on the strength, rigidity, and brittleness of the ceramic tubes. Mechanical grips are an option if they secure the test specimen without slipping or breakage in the grips at the test conditions. If the tubes are small, thin-walled, brittle, and rigid, adhesive gripping methods are typically more successful than mechanical gripping.

7.2.4 *Loading Rod*—The loading rod shall be straight, rigid, and strong enough to apply force directly to the plug face without bending, deformation, or damage. The loading rod shall be long enough to reach the bottom of the test specimen with direct contact to the upper loading anvil. Adequate precautions shall be taken to avoid/minimize friction between the loading rod and the interior of the test specimen tube. The loading rod diameter should be 90 % of the inside diameter of the tube.

7.2.5 *Support Block*—The purpose of the support block is to align and hold the gripping fixture in place. Alignment features in the support block may use conical or spherical seats to maintain axial and lateral alignment of the gripping fixture.

7.2.6 *Alignment*—The test apparatus shall be designed and constructed to keep extraneous bending stresses and strains around the circumference of the test specimen at less than ±10 % difference from the mean stress around the circumference.

NOTE 4—Misalignment bending stresses can develop with nonuniform test specimens (variations in tube diameter, concentricity, and straightness; non-parallel end-plug faces; see 10.2) and from misalignments in the load train.

7.2.6.1 A compliant layer such as copper or graphite sheet may be used between the face/tip of the loading rod and the interior face of the end-plug to reduce or eliminate stress concentrations and misalignments.

7.2.6.2 The loading rod may use hemispherical or rounded features/fixtures or other alignment aides at the top and bottom to maintain axial alignment of the applied force. The flat face of the hemispherical load plate should sit on the end-plug to avoid point contact stresses on the end-plug (see Fig. 3). This alignment correction may not require a compliant layer.

7.3 *Strain Gauges*—Strain gages are not used in this test method to measure adhesive strain in the end-plug bond section during testing. Strain gages on the test specimen tube may be used to assess bending stresses and strains produced by misalignment (12.3.5). If used, strain gages shall be selected and used per Test Methods E251.

7.4 *Data Acquisition*—Applied force and cross-head displacement as a function of time shall be recorded. Use either digital data acquisition systems or analog chart recorders for this purpose, although a digital record is recommended for ease of later data analysis. Recording devices shall be accurate to 1.0 % of full scale. A minimum data acquisition rate of 10 Hz shall be used, and the acquisition rate shall be fast enough to capture the maximum force within 1 %.

7.5 *Dimension-Measuring Devices*—Micrometers, calipers, and optical microscopy used for measuring linear dimensions shall be accurate and precise to at least one-half the smallest unit to which the individual dimension is required to be measured. For testing small diameter (<20 mm) test specimens, the measuring devices should have an accuracy of 0.01 mm.

7.6 *Elevated Temperature Testing:*

7.6.1 *General*—This test method is applicable to elevated temperature testing with the use of suitable furnace equipment and temperature control and measurement. The test temperature shall be selected based on the functional temperature requirements of the ceramic application. The furnace may have

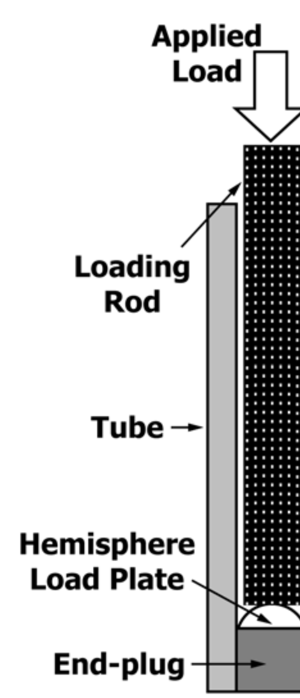


FIG. 3 Loading Rod Schematic Using a Hemispherical Load Plate

an air, inert, or vacuum environment, as required. If an inert or vacuum chamber is used, and it is necessary to direct the force through a bellows, fittings, or seal, it shall be verified that losses or errors in force measurement do not exceed 1 % of the expected failure forces.

7.6.2 *Furnace Configuration*—The furnace system shall be constructed and have a temperature-control system to maintain a constant temperature in the end-plug test section during each testing period. The variation in temperature with time during the test shall be no greater than ±5 °C or ±1 % of the test temperature, whichever is larger. The furnace system shall be configured so that spatial thermal differences along the length of the end-plug test section of the test specimen are no greater than ±5 °C or ±1 % of the test temperature (whichever is larger).

NOTE 5—Furnace systems can be configured in a variety of ways to accommodate test specimens, including traditional box furnace designs or small resistance heating elements in close proximity to the end-plug section. Heating can be done with any suitable heating method (indirect electrical resistance heating elements, direct induction, indirect induction through a susceptor, radiant lamp, or direct resistance in the test specimen) that maintains proper temperature conditions.

7.6.3 *Temperature Measurement*—The temperature-measurement device for the test specimen shall have a resolution of 2 °C or better. If temperature is measured with a thermocouple, the test specimen temperature shall be monitored with the thermocouple tip located no more than 1 mm from the end-joint section of the test specimen. Either a fully sheathed or exposed bead junction may be used. If a sheathed tip is used, it shall be verified that negligible error is associated with the sheath.

7.6.3.1 A separate thermocouple may be used to control the furnace chamber if necessary, but the test specimen temperature shall be the reported temperature of the test.

7.6.3.2 The thermocouple(s) shall be calibrated and used in accordance with Test Method E220 and Specification E230/E230M.

7.6.3.3 The temperature measurement shall be accurate to within ±5 °C. The accuracy shall include the error inherent to the thermocouple as well as any errors in the measuring instruments.

7.6.4 *System Equilibrium*—The time for the system to reach thermal equilibrium at test temperature shall be determined for the test temperature to be used. This shall be performed for both hot-furnace loading or cold-furnace loading, to support test specimen heat-up per 12.4.2.

7.6.5 *Temperature Data Acquisition*—At a minimum for elevated temperature tests, record temperature as single measurements at the initiation and completion of the actual test. However, temperature may also be recorded continuously, similar to force and strain except the record begins at the start of the heating of the furnace (including ramp-up to test temperature).

8. Calibration and Standardization

8.1 Calibration of equipment (force measurement, strain measurement, thermocouples, etc.) shall be provided by the supplier against standards traced to a national measurement institute, such as the National Institute of Standards and Technology (NIST). Recalibration shall be performed with traceable standards on all equipment on a yearly interval or whenever accuracy is in doubt.

8.2 *Reference Materials*—There are currently no standard reference materials for this type of test.

9. Hazards

9.1 *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. Caution should be used during collection of fragments as they may be sharp.

9.2 *Precaution*—Elevated temperature testing often produces the possibility of fire, burns, and electrical shorts. Furnaces shall be properly designed, assembled, and operated to minimize those hazards.

9.3 *Precaution*—Exposed fibers at the edges of fiber-reinforced composite test specimens present a hazard due to the sharpness and brittleness of the ceramic fiber. Inform all persons required to handle these materials of such conditions and the proper handling techniques.

10. Test Specimens, Preparation, and Sampling

10.1 *Test Specimen Geometry*—While EPPO test specimens are defined as a joined tube and end-plug, a variety of test specimen geometry is acceptable if it meets the gripping, fracture location, bending limits, and temperature profile requirements of this test method. A minimum length between the bottom of the grips and the inner surface of the end-plug shall be 25 mm to ensure that fracture is not influenced by grip-induced stresses on the test specimen.

NOTE 6—The exact geometry is dependent on the purpose of the test and the design configuration and geometry of the end-use component. Generally, the dimensions (length, diameter, wall thickness, end-plug geometry, etc.) of the end-plug test specimen will reflect the size and dimensions of the end-use component, although it might not be possible to test exceedingly large tube-joints due to limits of test equipment. If it is desired to evaluate the effects of geometry and the adhesive processing, then the size of the test specimen and resulting bond geometry will be selected to accurately assess the test variables. In addition, grip methods will influence the final length and design of the test specimen geometry. These different test objectives will produce a wide range of test specimen diameters and length and preclude the use of a single, standardized test specimen geometry. An example of a test specimen geometry and test apparatus developed in 2015 for silicon carbide composite tubes for the nuclear industry is shown in Appendix X1.

10.1.1 A major factor in the design of the test specimen is the configurational fit between the end-plug and the tube. Critical factors are the bond geometry (for example, straight-wall plug, scarf-joint plug, flat-face plug; see Fig. 1), the bond length and area, and the adhesive bond thickness between the tube inside diameter (ID) and the plug outside diameter (OD). The test specimen bond geometry may match the bond configuration of the end-use component.

10.1.2 *Elevated Temperature Geometry*—The geometry of the test specimen for elevated temperature testing shall be dependent on the type and configuration of the furnace, the requirements of temperature uniformity, and ambient temperature test specimen geometry.

NOTE 7—Thermal gradients can introduce additional stress gradients in test specimens which might already exhibit stress gradients at ambient temperatures due to geometric transitions. Therefore, analyze untried test configurations simultaneously for both loading-induced stress gradients and thermally induced temperature gradients to ascertain any adverse interactions.

10.2 *Dimensional Tolerances*—Dimensional tolerances shall be defined by the test designer. Dimensional tolerance requirements of the end-use component may act as a guideline.

10.2.1 *Tube Geometry*—The test designer should define dimensional tolerances in consideration of misalignment stresses based on variations in OD, ID, straightness, and concentricity of the test specimen tube geometry.

10.2.2 For the test specimen OD, the grip section of the test specimen may be finished, smoothed, or turned to produce a uniform diameter and suitable surface finish for the gripping fixture to effectively secure the test specimen. Machining of the ceramic should be performed with appropriate media and fluid cooling to minimize surface damage and machining stresses. Grip section machining may be done before or after end-plug insertion.

10.2.3 *Tube/End-Plug Fit*—The test designer should define dimensional tolerances for the end-plug and the joint section of the tube that ensure uniform and consistent fit of the end-plug in the tube. The parallelism tolerance of the two end-plug faces may be defined so that the inner end-plug face is perpendicular to the loading rod axis in the test apparatus to within ±2°.

10.2.4 The test designer shall define and document a quality evaluation procedure for the test specimen, defining the critical test specimen parameters (dimensions, tolerances, fit, surface condition, etc.), the methods of measurement, and the pass-fail requirements for the selected parameters.

10.3 *Joint Processing*—Bond material and processing method shall be selected to support the test objectives or to match the end-use application, or both. A joint processing preparation procedure shall be defined and documented, describing the bond material, specimen and bond geometry, specimen and end-plug preparation steps, assembly/alignment steps, processing methods and conditions, and machining/finishing steps.

NOTE 8—The presence of excess joint material on the interior face of the end-plug can interfere with the alignment of the loading rod and introduce bending stresses. Steps to minimize joint material on the interior face during bonding can be useful for avoiding this issue.

10.3.1 Prior to bonding, measure, record, and ensure that the dimensions of the specimen tubes and the end-plugs meet defined dimensional requirements (OD, ID, straightness, parallelism, etc.).

10.3.2 Prepare all test specimens with bonded end-plugs per the defined test specimen preparation and adhesive bonding procedure.

10.3.3 Nondestructive evaluation (radiography, computerized tomography, ultrasonics, thermal imaging, etc.) may be used to assess the alignment and fit of the end-plug and the condition of the adhesive joint (gaps, porosity, thickness uniformity, etc.).

10.3.4 Perform and record all quality evaluation tests and the results of any nondestructive evaluations, and include them in the final report.

10.4 The test specimens shall be properly packaged and stored to minimize environmental exposure and bond degradation caused by moisture, heat, or cold.

10.5 Test specimens may be selected from as-fabricated components for the purposes of quality control, if the test is designed for the as-fabricated geometry. Specimen sampling from a production lot shall be done per Practice E105.

10.6 *Definitions of Valid and Invalid Tests:*

10.6.1 *Failure Location*—A valid test is a test in which failure (fracture or permanent deformation) occurs by adhesive or cohesive failure within the end-plug length of the test specimen. Breakage may occur in or run into the tube section and include partial fracture in the tube section if it is within the bond/end-plug length or within 10 % of the OD past the end-plug (Fig. 4). Fracture in the tube away from the end-plug is considered an invalid test. Slippage, breakage, or both, in the grip section produces an invalid test.

NOTE 9—Although considered an invalid test, fracture in the tube that is both away from the end-plug and away from the grips provides information on the strength of the tube relative to the strength of the joint and may be of interest to the end user.

10.6.2 *Alignment Criteria*—A valid test occurs when maximum percent misalignment bending is under 10 % of the average strain (see 12.3.5 and X3.1). Percent bending over 10 % is considered a non-valid test.

10.7 *Required Number of Valid Tests*—Five (5) tests shall be a minimum number of valid tests for determining an average. Ten (10) tests is recommended as a minimum number of valid tests for determining an average with a standard deviation calculation. For full statistically significant data, the procedures outlined in Practice E122 should be consulted.

11. Conditioning

11.1 Finished test specimens may be conditioned at different temperatures and environments for defined periods of time to assess temperature and environmental effects on the joint strength and durability. Any conditioning treatments should be fully defined for time, temperature, and environmental conditions. Test specimens should be weighed, measured for

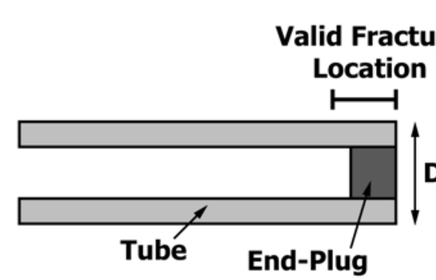


FIG. 4 Valid Test Section Encompassing the Length of the End-Plug Plus 0.1× the Tube OD

dimensions, and visually examined before and after conditioning to assess physical changes in the test specimens.

12. Procedure

12.1 *Test Specimen Measurement and Examination:*

12.1.1 Measure and record required dimensional specifications as outlined in 10.2 to within 0.02 mm or 0.05°.

12.1.2 Visually examine the outside surface of the end-plug and the outside surface of the test specimen for surface variations and anomalies.

12.1.3 Visually examine the interior face of the end-plug for excess adhesive that interferes with the loading rod. Record the type and location of any observed anomalies.

12.2 *Preparation of the Test Apparatus System:*

12.2.1 Based on the expected failure mode of the end-plug adhesive at the test temperature, define a failure criteria for the test; elastic-brittle fracture for brittle adhesives or the limit of permanent deformation for ductile-plastic adhesives, or both. (See Fig. 5.)

12.2.2 The EPPO test is commonly done in displacement control. Set a constant cross-head speed so that test specimen failure occurs within 10 to 60 s. While the required cross-head speed will depend on the test specimen size, the bond geometry, and the nominal joint, typical rates for testing are 1 to 10 mm/min. Preliminary tests may be necessary to determine the appropriate cross-head speed.

12.2.2.1 Slower or faster test rates may be used to evaluate strain rate effects on the joint strength.

12.2.3 Set up and align the testing apparatus in the testing machine.

12.2.4 Set up, turn on, and check the universal test machine control system, the force and strain measurement systems, and the data acquisition system. Set the defined test mode and test rate on the test machine.

12.2.5 Measure and record the ambient temperature and relative humidity (Test Method E337) in the laboratory at the start and end of the test sequence.

12.2.6 For elevated temperature testing, install, set up, and check the operation and control of the furnace system with the test apparatus installed.

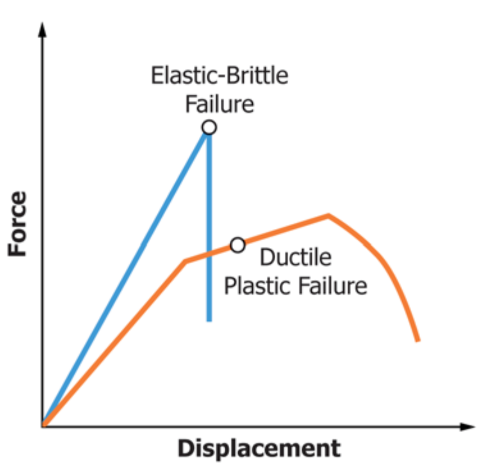


FIG. 5 Force Displacement Curves for Elastic-Brittle and Ductile-Plastic Failure

12.3 *Test Specimen Mounting and Alignment:*

12.3.1 *Adhesive Gripping of the Test Specimen*—If adhesive grip bonding is used, the end-plug test specimen(s) should be fitted, aligned, and bonded into the gripping fixture per the grip adhesive manufacturer's process instructions. The defined bonding process (curing, heating, sintering, melting, heat treating) shall be followed and controlled to produce a test specimen securely fitted and aligned in the gripping fixture. (See Appendix X2.)

12.3.2 *Mechanical Gripping of the Test Specimen*—If mechanical gripping is used, align, fit, and secure the test specimen into the gripping fixture.

12.3.3 The angular alignment of the fitted test specimen in the gripping fixture may be measured to ensure that the test specimen is aligned and concentric with the gripping fixture to within ±2°.

12.3.4 Fit and align the gripping fixture (with the test specimen) and the loading rod into the test apparatus (in the test furnace for elevated temperature testing).

12.3.5 Alignment 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 fixtures and load train fixtures are changed or installed on a different test machine, whenever a different operator is conducting a series of tests, or when damage or misalignment is suspected.

12.3.5.1 The alignment of the test specimen in the test apparatus should be experimentally checked by using a strain-gaged test specimen and checking the strain at four points around the circumference of the specimen tube. (See Appendix X3.) The maximum allowable percent misalignment bending among the four strain gages is 10 %, as measured and calculated in Appendix X3.

12.3.5.2 If experimental conditions, time, and cost permit, all test specimens may be strain gaged for alignment check (see Appendix X3) and checked for misalignment in the initial stages of each test. Test specimens that exceed the misalignment limit shall be realigned or discarded.

12.3.6 Safety shields should be placed around the test specimen and test fixture to contain and collect fracture fragments.

12.4 *Test Initiation:*

12.4.1 The test load train should be preloaded to approximately 5 % of the expected push-out force to seat the components of the test apparatus, remove slack, and check for grip slippage.

12.4.2 For elevated temperature testing, begin furnace heat-up and recording of test specimen temperature. The test specimen shall be heated at a defined heating rate to the designated test temperature and held at the designated constant test temperature until the test specimen reaches thermal equilibrium. During heat-up, the preload shall be adjusted to maintain constant force on the test specimen and to compensate for thermal expansion stresses.

12.4.3 Start data acquisition and start force application at the defined displacement rate. Load the test specimen to the defined failure criteria, as shown by either brittle fracture failure or by a predetermined permanent deformation beyond the elastic stress limit with increasing compliance or maximum force (or both). See Fig. 5.

12.5 *Test Completion:*

12.5.1 Record the force at failure (push-out force) and the force-time/displacement data, if available.

12.5.2 Collect and remove any test specimen fragments from the test apparatus. Remove the test specimen from the gripping fixture. Save the test specimen and the fragments for failure analysis.

12.5.3 A valid test is a test in which the test specimen failure occurs in the end-plug section, as described in 10.7.

12.5.4 To complete a required sample set for a statistical average, one replacement test specimen shall be tested for each invalid test specimen.

12.5.4.1 The test specimen fracture surface should be visually examined to determine if the location of failure is in the end-plug section, necessary for a valid test. Visual examination may show if the failure mode is adhesive at the bond line or is cohesive in the adhesive bond material, the end-plug, or the ceramic tube. Microscopic examination may be necessary to determine adhesive failure or cohesive failure in the adhesive. Any evidence of cracking emanating from the gripping region is cause for an invalid test. For failure in advanced ceramic tubes and ceramic-based joints, post-fracture analysis should be performed using the guidelines in Practice C1322.

13. Calculation or Interpretation of Results

13.1 The push-out force (F_{PO}) is the measured force (N) at the defined failure criteria.

13.2 The adhesive bond area (A_A) is the calculated area of the adhesive bond surface specific to the end-plug configuration. For each different configuration the adhesive bond area is calculated from the bond geometry. Surface area equations for bond areas of straight wall, scarf joint, and flat-face bonds are given in Annex A1.

13.3 The nominal joint strength (S_{NJ}) in MPa is calculated for valid tests as:

$$S_{NJ} = F_{PO}/A_A \tag{1}$$

where:

F_{PO} = the measured push-out force at failure (N), and
A_A = the adhesive bond area (mm^2) specific to the end-plug bond geometry.

13.4 The nominal burst pressure (P_{NB}) in MPa is calculated as:

$$P_{NB} = F_{PO}/A_F = F_{PO}/\left(\pi\,(d/2)^2\right) \tag{2}$$

where:

F_{PO} = the measured push-out force at failure (N),
A_F = the interior face area of the end-plug (mm^2), and
d = the diameter of the interior face area of the end-plug (mm) or the inner diameter of the ceramic tube (mm).

13.5 *Statistics*—For each test specimen set, calculate the average, standard deviation, and the coefficient of variation for the selected test data: F_{PO}, S_{NJ}, and P_{NB}. Only valid tests shall be used in this calculation.

$$\text{mean} = \bar{X} = \frac{\left(\sum_{i}^{n} = {}_1 x_i\right)}{n} \tag{3}$$

$$\text{standard deviation} = \text{s.d.} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_i - \bar{X}\right)^2}{n-1}} \tag{4}$$

$$\text{percent coefficient of variation} = \text{CV} = \frac{100(\text{s.d.})}{\bar{X}} \tag{5}$$

where:

X_i = the measured value, and
n = the number of valid tests.

13.6 The force-time or force displacement curve should be plotted and analyzed for the mode of failure as illustrated in Fig. 5. The force-time/displacement curves may also show signs of system alignment compliance and test specimen slippage in the grips.

14. Report

14.1 The report shall include the following information for the test set. Any significant deviations from the procedures and requirements of this test method shall be noted in the report.

14.1.1 Name of laboratory, location, date of test, and test operators.

14.2 *Test Materials and Test Specimens:*

14.2.1 Description of the end-plug material and the ceramic tube material: source, material description, method of fabrication, material specifications and designations, lot number, date of fabrication, and any other necessary information.

14.2.2 Description of the end-plug geometry and ceramic tube geometry (include engineering drawing, if available) with dimensions and tolerances.

14.2.3 A calculation of the adhesive bond area (A_A) for the test specimens.

14.2.4 Preparation, machining, and conditioning of the joining surfaces for bonding.

14.2.5 Description of the end-plug adhesive material and bonding process: source, composition, chemistry, method of fabrication, tooling, and conditioning.

14.2.5.1 Machining and finishing of the grip section of the test specimen.

14.2.5.2 Test specimen conditioning parameters, if any, and test specimen storage conditions.

14.3 *Equipment and Test Parameters:*

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

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

14.3.3 Description of all the test apparatus components (include an engineering drawing, if available) in terms of geometry and material.

14.3.4 Detailed description of the gripping fixture, with geometry and dimensions.

14.3.5 Detailed description of the grip adhesive and bonding procedure for test specimen gripping, if used.

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

14.3.7 Method and date of calibration of the force and strain measurement systems.

14.3.8 Description of the alignment check procedure and the results of the alignment check.

14.3.9 Test parameters: mode of control, cross-head displacement rate, pre-load force, selected test temperatures.

14.3.10 The definition of the failure criteria for this adhesive system under these test conditions.

14.3.11 Ambient humidity and temperature in the laboratory during the tests.

14.3.12 If used, a description of the furnace system (configuration, heating method, temperature control and measurement) and the heating protocol.

14.3.13 Any variations in the test procedure, compared to the specification.

14.4 *Test Results:*

14.4.1 Mean, standard deviation, CV, and valid test count, n, for the push-out force (F_{PO}) for all valid tests at each reported test temperature.

14.4.2 Mean, standard deviation, CV, and valid test count, n, for the calculated nominal joint strength (S_{NJ}), for all valid tests at each reported test temperature.

14.4.2.1 When reporting nominal joint strength, the geometry of the test specimens and induced stress (shear, tension, mixed) in the joint shall be reported. Nominal joint strength shall only be used for comparison of data when test specimens share a common test geometry and stress state. Stress state shall be included as a subscript or in parentheses for reporting purposes, that is, $S_{NJ,shear}$ or nominal joint strength (shear).

14.4.3 Mean, standard deviation, CV, and valid test count, n, for the calculated nominal burst pressure (P_{NB}), for all valid tests at each reported test temperature.

14.4.4 Number of valid and invalid tests at each reported test temperature.

14.4.5 Test specimen test temperatures and ambient temperature and humidity.

14.5 *Data for Individual Test Specimens:*

14.5.1 Measured end-plug dimensions (diameter and length) and cylinder dimensions (OD, ID, and length) for each test specimen.

14.5.2 Measured dimensional variations in the end-plug and cylinder for each test specimen.

14.5.3 Any observed joint or surface anomalies and NDE results prior to testing.

14.5.4 Test specimen test temperature.

14.5.5 Push-out force (F_{PO}), calculated nominal joint strength (S_{NJ}) and calculated nominal burst pressure (P_{NB}), and classification as a valid or invalid test.

14.5.6 Force-displacement/time curves, if available.

14.5.7 Mode of fracture at the joint, and appearance of each part of the test specimen (tube/joint/endplug) after failure.

14.5.8 Any fractographic observations and the method of fractographic analysis.

15. Precision and Bias

15.1 An "analysis of errors" study shows that the measurement of the end-plug diameter is the dominant source of measurement error, because of the use of diameter squared in the calculation of surface area. All other measurement errors produce linear errors in the calculations.

15.2 Repeatability studies are strongly affected by material variations in the adhesive bonding of test specimens. Single-laboratory repeatability studies have been performed and are found in the literature.

15.3 A full inter-laboratory round-robin study is planned for ambient temperature testing, based on the availability of funding, sufficient test specimens, and test apparatus sets.

16. Keywords

16.1 adhesive bonding; ceramic composite; ceramic joint; end-plug push-out test; nominal burst pressure; nominal joint strength

ANNEX

(Mandatory Information)

A1. CALCULATION OF ADHESIVE BOND SURFACE AREA IN THE EPPO TEST SPECIMEN

A1.1 The calculation of the nominal joint strength requires a measurement of the surface area of the adhesive bond of the end-plug. The adhesive bond area is calculated for the specific end-plug configuration, assuming basic symmetrical geometry. The equations for the adhesive bonding areas of three basic end-plug geometries are given in Fig. A1.1, where:

D = outer diameter of the specimen tube (mm),
d = inner diameter of the specimen tube (mm), and
L = length of the end-plug.

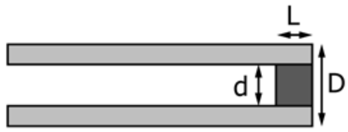
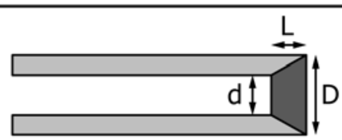
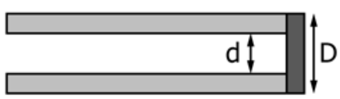
Geometry and Bond Surface Area	Surface Area Equation
 **Straight Walled Joint**	$A_A = L \times \pi d$
 **Scarf Joint**	$A_A = (\pi(D + d)/2) \times [((D - d)/2)^2 + L^2]^{1/2}$
 **Flat Face Joint**	$A_A = \pi[(D/2)^2 - (d/2)^2]$

FIG. A1.1 Adhesive Bond Surface Area Calculations for Three Types of End-Plug Geometries

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLE OF A TEST SPECIMEN GEOMETRY AND A TEST APPARATUS

X1.1 In 2013 – 2015 the Department of Energy funded a research project (DE-NE0000612) with General Atomics (GA) to develop an end-plug push-out test for ceramic tubes (both monolithic ceramics and ceramic matrix composite (CMC) materials). The ceramic tubes are being developed as cladding materials for nuclear fuel, replacing zirconium alloys. The objective of the project was to develop, validate, and document an experimental test method for determining the mechanical strength of different end-plug geometries and different adhesives in ceramic tubes at ambient and elevated temperatures.

X1.2 Two types of ceramic materials were used as specimen tubes:

(1) Silicon carbide fiber-silicon carbide matrix composite tubes, consisting of Tyranno SA3 SiC fibers in a chemical-vapor-infiltrated silicon carbide matrix, and

(2) Sintered monolithic silicon carbide tubes. End plugs were monolithic silicon carbide produced by sintering or hot pressing.

Two types of end-plug adhesives were evaluated: a high-strength epoxy for room temperature test development and a proprietary high-temperature ceramic adhesive for functional testing.

X1.3 Test Specimen Geometry

X1.3.1 Specimen tubes were approximately 10 mm in outer diameter with an interior diameter of ~7 mm, a wall thickness of ~1.5 mm, and a tube length of 45 to 60 mm. Scarf (see Fig. X1.1) and butted end-plug geometries were explored.

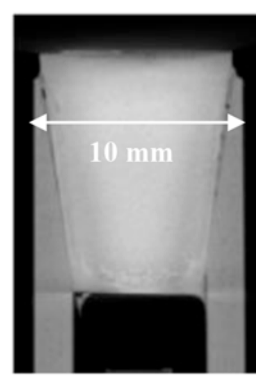


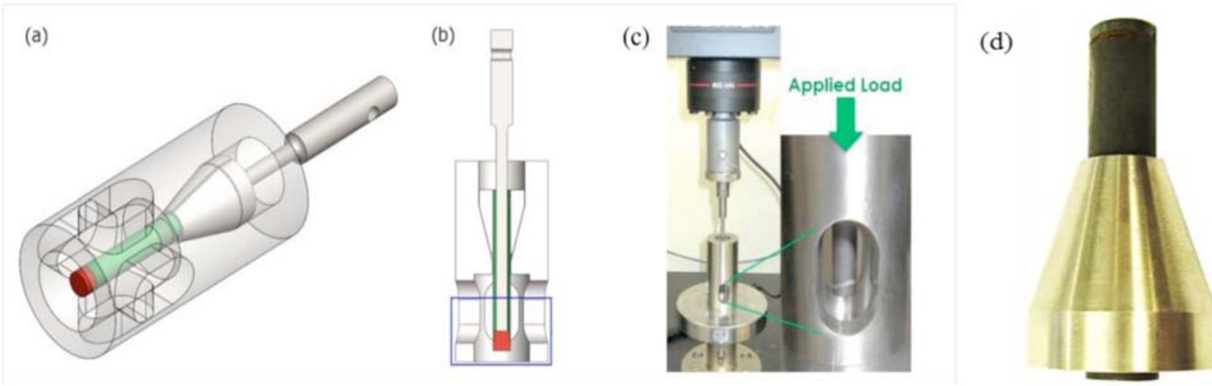
FIG. X1.1 XCT Image of Scarf Joint Test Specimen

X1.4 Test Apparatus

X1.4.1 The test apparatus consisted of a support block, a loading rod, and a split conical collet with the test specimen as shown in Fig. X1.2. The test specimen tube is adhesively bonded in the split conical collet with a 25-mm grip bond length. The conical collet fits into the support block, where the taper of the collet serves to self-align the test specimen as well as to apply a small amount of normal force to the tube to aid in grip strength. U-joints are included in the load train to minimize misalignment.

X1.5 Test Method

X1.5.1 A compressive force is applied through the loading rod to the interior face of the end-plug until failure occurs. A crosshead speed of 1.5 mm/min is used during testing.



(a) isometric view
(b) section view showing load rod inserted into specimen tube
(c) assembled fixture in use
(d) test specimen bonded into conical steel collet

FIG. X1.2 EPPO Test Fixturing

X1.6 Gripping Method

X1.6.1 Mechanical action grips (wedge grips and collet grips) were initially evaluated for these ceramic test specimens, but they commonly failed by test specimen slippage and failure in the grips. Adhesive grip bonding was more successful. For room temperature testing, a high-strength epoxy was used to bond the specimen tube into the split collet. For high-temperature testing, metal brazes, sealing glasses, and ceramic adhesives were evaluated.

X1.7 Alignment Issues and Bending Stresses

X1.7.1 In the test development effort, significant effort was put forth in identifying potential causes of misalignment and minimizing the resulting stresses. Strain gages around the outer circumference of the end-plug section were used to measure percent bending during testing. The principal source of these misalignment strains were: off-angle and off-center loading rod, off-axis misaligned interior face of the end-plug, residual adhesive on the face of the end plug, and out-of-tolerances and poor fit of the test specimen in the grip fixture. The tolerances for misalignment cited in the test method were derived from this testing, and recommendations on compliant layers, U-joints, and hemispherical alignment devices were developed.

X1.8 Nominal joint strengths and nominal burst pressure were successfully obtained for all geometries and material types at room and high temperature (300 °C, 600 °C, and 750 °C in air) with a test efficacy of approximately 85 %. A typical example of the force-versus-extension plots obtained during room temperature testing is seen in Fig. X1.3. A complete analysis of the test methodology, the resulting data, and a discussion of the test results is found in the reference literature.[3,4,5]

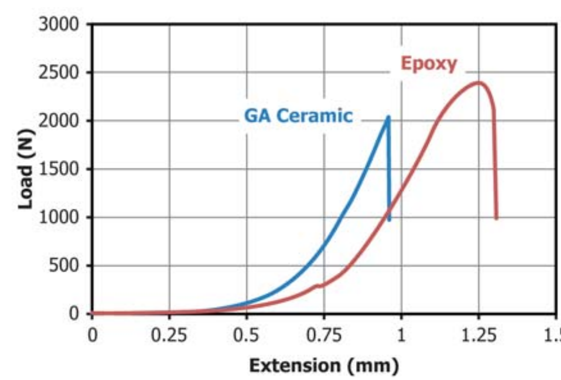


FIG. X1.3 Force-Extension Plots for GA EPPO Tests

[3] Khalifa, H. E., Deck, C. P., Gutierrez, O., Jacobsen, G. M., Back, C. A., "Fabrication and Characterization of Joined Silicon Carbide Cylindrical Components for Nuclear Applications," *Journal of Nuclear Materials*, Vol 457, 2015, pp. 227–240.

[4] Khalifa, H. E., Jacobsen, G. M., Gutierrez, O., Back, C. A., "Out-Of-Pile Characterization and Testing of Joined Cylindrical Components for SiC-Based Nuclear Fuel Cladding," *Transactions of the American Nuclear Society*, Vol 109, 2013, pp. 419–422.

[5] Jacobsen, G. M., Khalifa, H. E., Kearns, Y., Gutierrez, O., Deck, C. P.,"High Temperature Testing of Geometrically Relevant, Nuclear Grade Silicon Carbide Joints," *Transactions of the American Nuclear Society*, Vol 113, 2015, pp. 535–538.

X2. TYPES OF GRIPPING SYSTEMS

X2.1 In the EPPO test, the test specimen tube is secured in a gripping fixture and the gripping fixture is seated on or into the support block. Two types of grip fixtures are commonly used for gripping cylindrical test specimens: mechanical grip fixtures and adhesive-bonded grip fixtures.

X2.2 Mechanical Grip Fixtures

X2.2.1 Mechanical grips use the direct application of a force normal to the grip surface of the tube test specimen. These mechanical grips commonly use three types of devices to clamp onto the test specimen: jaw chuck clamps, collet chucks, and clamping shaft collars (see Fig. X2.1). All these gripping devices use radial mechanical force to produce static friction forces that keep the test specimen from slipping out of the grip. The gripping devices also act as mechanical stop to hold the test specimen on/in the support block.

X2.2.2 Jaw chuck clamps use three, four, or six jaws in a self-centering adjustable plate or body to grip the test specimen at three, four, or six points around the circumference.

X2.2.3 A collet chuck consists of a flexible segmented collet with a cylindrical inner surface and tapered outer surface that is radially compressed in a matching tapered receiving sleeve by the collet cap that pulls or pushes the collet into the sleeve. The compression of the collet segments clamps onto the test specimen positioned in the center of the collet. A collet chuck applies clamping force more uniformly around the entire outer surface of the test specimen, as compared to jaw chucks, which commonly apply line contacts.

NOTE X2.1—For collet chucks, close tolerances are required for the diameter of the grip section of the test specimen, because of low diametrical tolerance in the collet. Actual specimen diameter tolerances will depend on the exact configuration and acceptance dimensions of the collet. A uniform diameter of the tube specimen can be produced by direct machining/turning of the grip section.

X2.2.4 Clamping shaft collars are one- or two-piece sleeves where screws compress the collar around the test specimen. Two-part shaft collars provide more even pressure distribution around the circumference of the test specimen, compared to one-piece split collars. Longer collars provide more grip surface.

X2.2.5 Three important aspects of mechanical grip interfaces are: uniform contact force around the test specimen (minimizing point and line stresses), sufficient grip length, and an effective coefficient of friction over the grip/specimen mating surface.

X2.3 Adhesive-Bonded Grip Fixtures

X2.3.1 Adhesive-bonded grip fixtures are fitted sleeves, collars, or blocks with a precisely machined internal central bore in which the tubular test specimen is adhesively bonded to secure the test specimen into the grip fixture (see Fig. X2.2). The bonded grip fixture can be a single piece or a split fixture. Two-part split fixtures (along the length of the fixture) are more easily assembled with uniform adhesive distribution. Test specimen removal is also easier for split fixtures.

X2.3.2 Both the inner bore of the grip fixture and the OD of the grip section of the test specimen shall be precisely machined to provide enough space for the adhesive layer and to keep the test specimen centered and aligned.

X2.3.3 A suitable adhesive should be selected based on the fixture material, the test specimen material, and the selected test temperature. For adhesive gripping at ambient temperatures for the EPPO test, high-strength epoxy adhesives are generally suitable for securing the test specimen in the grip fixture. A commonly used adhesive is a two-part room temperature curing, tough, high-strength [20 to 35 MPa (3 to 5 ksi)] epoxy. High-temperature epoxies may be used at temperatures below 250 °C, provided enough grip length is provided. At elevated test temperatures (typically greater than 250 °C for ceramics), appropriate high-temperature bonding materials (metal solder, metal braze compounds, ceramic cements, sealing glasses) should be selected for gripping that have sufficient bond strength at the desired test temperature.

X2.3.4 The specimen tube should fit snugly in the grip with a thin (~0.1 to 0.2 mm) space for the adhesive, providing uniform bonding contact between the gripped section of the test specimen and the surface of the grip fixture. Bonding is done on the OD of the test specimen for a sleeve grip configuration. Grips with center cores have bonding on both the OD and the ID of the test specimen.

X2.3.5 The length of the bonded grip section of the test specimen shall be long enough to distribute the shear forces across a large bonding surface and to keep the shear stress

FIG. X2.1 Mechanical Grip Devices for the EPPO Test

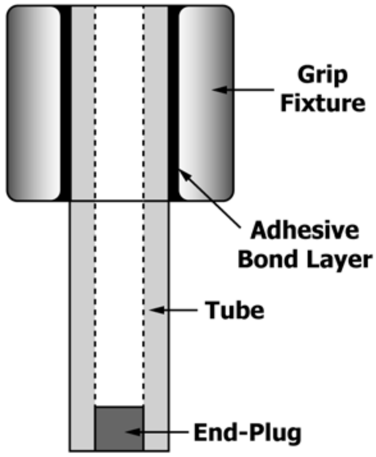


FIG. X2.2 Schematic of Adhesively Bonded Grip Fixture and Test Specimen

below the failure strength of the adhesive. Insufficient bonding surface in the grips causes adhesive failure in the grip before test specimen failure. As a rule of thumb, the bond shear forces that develop from the maximum tensile force in the test specimen should produce shear stresses <50 % of the nominal shear strength of the grip adhesive. The required length of the bonding grip surface length for tube is estimated with the following equation:

$$L_{bond} = (K \times F_{PO}) / [S_{GA} \times (\Pi \times D_{bond})] \quad \text{(X2.1)}$$
$$S_{GA} = (K \times F_{PO}) / (A_{GB})$$
$$A_{GB}(\text{OD Bond}) = L_{bond} \times (\Pi \times D_{bond})$$

where:

L_{bond} = required length of the bonding grip section (mm),
K = selected safety factor (2 for 50 % reduction),
F_{PO} = expected push-out force of the end-plug bond (N),
S_{GA} = shear strength of the grip adhesive (MPa),
A_{GB} = area of grip bonding, and
D_{bond} = effective diameter of the bonding zone (mm) (tube OD).

X2.3.6 Elevated temperature adhesives (brazes, sealing glasses, ceramic cements) commonly require a high-temperature process step, which potentially produces residual stresses between the test specimen and the grip fixture after cool-down from the process step. These residual stresses are a cause of premature failure of the adhesive or the test specimen in the grip section. The residual stresses develop from differences in the thermal expansion coefficients between the specimen material and the grip material. This is a particular issue when ceramic test specimens are bonded into metal fixtures.

X2.3.7 Removal of the test specimen from the grips commonly requires the use of thermal or chemical attack of the adhesive bonding the test specimen into the grips.

X3. VERIFICATION OF SPECIMEN AND LOAD TRAIN ALIGNMENT

X3.1 Purpose of Verification

X3.1.1 The purpose of this verification procedure is to demonstrate that the grip system and load train couplers are used by the test operator in such a way as to consistently meet the limit on percent bending as specified in 10.6.2. 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 compressive force should be measured using verification (or actual) test specimens of exactly the same design as that to be used for the EPPO tests. For the verification purposes, strain gages should be applied as shown in Fig. X3.1. 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 fixtures and load train fixtures are changed or installed on a different test machine, whenever a different operator is conducting a series of tests, or when damage or misalignment is suspected.

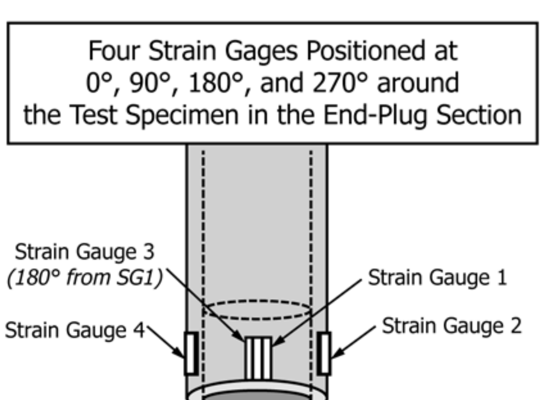


FIG. X3.1 Strain Gage Placement on EPPO Test Specimen

X3.2 Practice E1012 should be used as a basic guideline for assessing load train and test specimen alignment in the test system.

X3.3 When test conditions, time, and budget permit, each test specimen may be strain gaged and tested per the following instructions to check for misalignment bending stresses as a part of each individual test.

X3.4 Verification Specimen

X3.4.1 The verification specimen should be of identical materials and geometry to that being tested with attention to all tolerances and concentricity requirements. However, in the case of CFCCs the type of reinforcement or degree of residual porosity may complicate the consistent and accurate measurement of strain. Therefore, it is recommended that an alternate material (isotropic, homogeneous, and continuous) should be used with elastic modulus, elastic strain capability, and hardness similar to the test material. The verification specimen should be carefully inspected with an optical comparator before strain gages are attached to ensure that these requirements are met. After the strain gages are applied it will no longer be possible to meaningfully inspect the verification specimen, so care should be exercised in handling and using it.

X3.5 For simplicity, four foil resistance strain gages should be mounted around the circumference (0°, 90°, 180°, and 270°) of the verification specimen within 5 mm of the end-plug section, as shown in Fig. X3.1. High-temperature strain gages may be used for high-temperature testing.

X3.6 Verification Procedure

X3.6.1 Procedures for verifying alignment are described in detail in Practice E1012. However, salient points for EPPO verification/test specimens are described here for emphasis.

X3.6.2 Assemble and align all the load train fixtures and components in the test system frame.

X3.6.3 Connect the lead wires of the four 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.

X3.6.4 Mount the verification specimen in the grip fixture and position and align the grip fixture (with the test specimen) in the load train.

X3.6.5 Zero the strain gages of the verification specimen in the load train, without the loading rod in position.

X3.6.6 Insert and align the loading rod in the test specimen. Apply a sufficient force to the verification specimen to achieve a mean strain (among the four strain gages) equal to either one-half the anticipated strain at the onset of the cumulative fracture process (for example, matrix cracking stress) in the test material or a strain of 0.0005 (that is, 500 microstrain), whichever is greater. Note that it is desirable to record the strain (and hence percent bending) as a function of the applied force to monitor any self alignment of the load train.

X3.6.7 Calculate the maximum percent bending as follows, referring to Fig. X3.1 for the strain gage numbers. Percent bending in the verification plane of the end-plug section is calculated as follows (per Practice E1012).

Average axial strain:

$$a = (e_1 + e_2 + e_3 + e_4)/4 \tag{X3.1}$$

where:

e_1, e_2, e_3, and e_4 = the measured strains at the four strain gage locations; the subscript indicates the order of the strain gages around the specimen.

Local bending strain:

$$b_1 = e_1 - a \tag{X3.2}$$

$$b_2 = e_2 - a$$

$$b_3 = e_3 - a$$

$$b_4 = e_4 - a$$

Maximum bending strain:

$$B = \frac{1}{2}\left[(b_1 - b_3)^2 + (b_2 - b_4)^2\right]^{\frac{1}{2}} \tag{X3.3}$$

Maximum percent bending strain:

$$PB = (B/a) \times 100 \tag{X3.4}$$

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