Designation: ~~C1525 – 04 (Reapproved 2013)~~ C1525 – 18

Standard Test Method for Determination of Thermal Shock Resistance for Advanced Ceramics by Water Quenching[1]

This standard is issued under the fixed designation C1525; 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 describes the determination of the resistance of advanced ceramics to thermal shock by water quenching. The method builds on the experimental principle of rapid quenching of a test specimen at an elevated temperature in a water bath at room temperature. The effect of the thermal shock is assessed by measuring the reduction in flexural strength produced by rapid quenching of test specimens heated across a range of temperatures. For a quantitative measurement of thermal shock resistance, a critical temperature interval is determined by a reduction in the mean flexural strength of at least 30 %. The test method does not determine thermal stresses developed as a result of a ~~steady-state~~ steady-state temperature ~~differences~~difference within a ceramic body or of thermal expansion mismatch between joined bodies. The test method is not intended to determine the resistance of a ceramic material to repeated shocks. Since the determination of the thermal shock resistance is performed by evaluating retained strength, the method is not suitable for ceramic components; however, test specimens cut from components may be used.

1.2 The test method is intended primarily for dense monolithic ceramics, but may also be applicable to certain composites such as whisker- or particulate-reinforced ceramic matrix composites that are macroscopically homogeneous.

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

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C373 Test Methods for Determination of Water Absorption and Associated Properties by Vacuum Method for Pressed Ceramic Tiles and Glass Tiles and Boil Method for Extruded Ceramic Tiles and Non-tile Fired Ceramic Whiteware Products

C1145 Terminology of Advanced Ceramics

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

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

C1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics

E4 Practices for Force Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

E616 Terminology Relating to Fracture Testing (Discontinued 1996) (Withdrawn 1996)[3]

IEEE/ASTM SI 10 ~~Standard for Use of the International System of Units (SI): The Modern Metric System~~American National Standard for Metric Practice

[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 Mechanical 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

2.2 *European Standard:*[4]

EN 820-3 Advanced Technical Ceramics—Monolithic Ceramics—Thermomechanical Properties—Part 3: Determination of Resistance to Thermal Shock by Water Quenching

3. Terminology

3.1 *Definitions:*

3.1.1 The terms described in Terminologies C1145, E6, and E616 are applicable to this standard test method. Specific terms relevant to this test method are as follows:

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

3.1.3 *critical temperature difference, ΔT_c, [θ], n*—temperature difference between the furnace and the ambient temperature water bath that will cause a 30 % drop in the average flexural strength.

3.1.4 *flexural strength, σ_f, [FL^{-2}], n*—a measure of the ultimate strength of a specified beam specimen in bending, determined at a given stress rate in a particular environment.

3.1.5 *fracture toughness, n*—a generic term for measures of resistance to extension of a crack. **E616**

3.1.6 *slow crack growth (SCG), n*—subcritical crack growth (extension) which may result from, but is not restricted to, such mechanisms as ~~environmentally-assisted~~ environmentally assisted stress corrosion or diffusive crack growth. **C1145**

3.1.7 *thermal shock, n*—a large and rapid temperature change, resulting in large temperature differences within or across a body. **C1145**

3.1.8 *thermal shock resistance, n*—the capability of material to retain its mechanical properties after exposure to one or more thermal shocks.

4. Summary of Test Method

4.1 This test method indicates the ability of an advanced ceramic product to withstand the stress generated by sudden changes in temperature (thermal shock). The thermal shock resistance is measured by determining the loss of strength (as compared to as-received specimens) for ceramic test specimens quickly cooled after a thermal exposure. A series of rectangular or cylindrical test specimen sets ~~are~~is heated across a range of different temperatures and then quenched rapidly in a water bath. After quenching, the test specimens are tested in flexure, and the average retained flexural strength is determined for each set of specimens quenched from a given temperature. The "critical temperature difference" for thermal shock is established from the temperature difference (exposure temperature minus the water quench temperature) that produces a 30 % reduction in flexural strength compared to the average flexural strength of the as-received test specimens.

5. Significance and Use

5.1 The high temperature capabilities of advanced ceramics are a key performance benefit for many demanding engineering applications. In many of those applications, advanced ceramics will have to perform across a broad temperature range with exposure to sudden changes in temperature and heat flux. Thermal shock resistance of the ceramic material is a critical factor in determining the durability of the component under transient thermal conditions.

5.2 This test method is useful for material development, quality assurance, characterization, and assessment of durability. It has limited value for design data generation, because of the limitations of the flexural test geometry in determining fundamental tensile properties.

5.3 Appendix X1 (following EN 820-3) provides an introduction to thermal stresses, thermal shock, and critical material/geometry factors. The appendix also contains a mathematical analysis of the stresses developed by thermal expansion under ~~steady state~~ steady-state and transient conditions, as determined by mechanical properties, thermal characteristics, and heat transfer effects.

6. Interferences

6.1 Time-dependent phenomena such as stress corrosion or slow crack growth may influence the strength tests. This might especially be a problem if the test specimens are not properly dried before strength testing.

6.2 Surface preparation of test specimens can introduce machining flaws, which may have a pronounced effect on the measured flexural strength. The surface preparation may also influence the cracking process due to the thermal shock procedure. It is especially important to consider surface conditions in comparing test specimens and components.

6.3 The results are given in terms of a temperature difference between furnace and quenching bath (ΔT). However, it is important to notice that results may be different for the same ΔT but different absolute temperatures. It is therefore specified in this test method to quench to room temperature.

[4] Available from European Committee for Standardization (CEN), 36 rue de Stassart, B-1050, Brussels, Belgium, http://www.cenorm.be.

6.4 The formulae presented in this test method apply strictly only to materials that do not exhibit ~~R~~R-curve~~-curve~~ behavior, but have a single-valued fracture toughness. If the test material exhibits a strong R-curve behavior, that is, increase in fracture toughness with increasing crack length, caution must be taken in interpreting the results.

6.5 Test data for specimens of different geometries are not directly comparable because of the effect of geometry on heat transfer and stress gradients. Quantitative comparisons of thermal shock resistance for different ceramic compositions should be done with equivalent test specimen geometries.

7. Apparatus

7.1 *Test Apparatus:*

7.1.1 The test method requires a thermal exposure/quenching system (consisting of a furnace, specimen handling equipment, and a quench bath) and a testing apparatus suitable for measuring the flexural strength of the test specimens.

7.1.2 The test method requires a furnace capable of heating and maintaining a set of test specimens at the required temperature to ~~± 5 K (± 5°C).~~ ±5 K (±5 °C). The temperature shall be measured with suitable thermocouples located no more than ~~2 mm~~ 2 mm from the midpoint of the specimen(s) in the furnace. Furnaces will usually have an open atmosphere, because air exposure is common during the transfer to the quench bath.

NOTE 1—If air exposure is detrimental, a special furnace-quench system can be set up in which both the furnace and the quench unit are contained within an inert atmosphere container. A common design for such a system consists of a tube furnace positioned vertically above the quench tank, so that the test specimen drops directly into the tank from the furnace.

7.1.3 The method requires ~~a~~ test specimen handling equipment designed so that the test specimen can be transferred from the furnace to the quenching bath within 5 s.

7.1.4 A water bath controlled to 293 ± 2 K ~~(20°C~~(20 ± ~~2°C)~~2 °C) is required. The water bath must have sufficient volume to prevent the temperature in the bath from rising more than 5 K ~~(5°C)~~(5 °C) after test specimen quenching. It is recommended that the bath be large enough for the test specimens to have cooled sufficiently before reaching the bottom of the bath, or contain a screen near the bottom to prevent the test specimens from resting directly on the bottom of the bath.

7.1.5 The universal test machine used for strength testing in this test method shall conform to the requirements of ~~Practice~~Practices E4. Specimens may be loaded in any suitable test machine, provided that uniform test ~~rates, either~~ rates using either load-controlled or displacement-controlled ~~mode,~~mode can be maintained. The loads used in determining flexural strength shall be accurate to within ±1.0 % at any load within the selected load rate and load range of the test machine as defined in ~~Practice~~Practices E4.

7.1.6 The configuration and mechanical properties of the test fixtures shall be in accordance with Test Method C1161 for use with the standard four-point flexure specimens. If larger test pieces ~~(sizes~~(size A or C below) are employed, the test fixture shall be scaled accordingly. There are currently no standard fixtures for testing cylindrical rods in flexure; however, the fixtures to be used shall have the appropriate articulation. Test fixtures without appropriate articulation shall not be permitted; the articulation of the fixture shall meet the requirements specified in Test Method C1161.

7.1.7 The method requires a 393 K ~~(120°C)~~(120 °C) drying oven to remove moisture from test specimens before (if needed) and after quench testing.

7.1.8 A micrometer with a resolution of 0.002 mm (or 0.0001 in.) or smaller should be used to measure the test piece dimensions. The micrometer shall have flat anvil faces. The micrometer shall not have a ball tip or sharp tip, since these might damage the test piece if the specimen dimensions are measured prior to fracture. Alternative dimension measuring instruments may be used, provided that they have a resolution of 0.002 mm (or 0.0001 in.) or finer and do no harm to the specimen.

8. Test Specimens

8.1 The ceramic test specimens shall be pieces specifically prepared for this purpose from bulk material or cut from components.

8.1.1 *Specimen Size*—Three specimen geometries are defined for use in this test method:

8.1.1.1 *Type A*—Rods 10 ± 0.13 mm in diameter, 120 mm long.

8.1.1.2 *Type B*—Bars 3 ± 0.13 mm × 4 ± 0.13 mm in cross section, minimum 45 mm long with chamfered edges, in accordance with ~~type~~Type B in Test Method C1161.

8.1.1.3 *Type C*—Bars 10 ± 0.13 mm × 10 ± 0.13 mm in cross section, 120 mm long, with chamfered edges.

NOTE 2—The test specimens of Types A and C ~~type~~ are intended to be large enough to produce a materials ranking that is basically independent of specimen size and appropriate for larger test specimens (**1, 2**~~)~~).[5] Test specimens of Type B ~~type~~ may require greater quenching temperature differences in order to produce strength reduction. These test specimens may not correctly rank the relative behavior of larger components. Only Type B coincides with Type B in Test Method C1161.

NOTE 3—Under some circumstances, the edges of prismatic test specimens or the ends of cylindrical test specimens may be damaged by spallation during the quench test. These specimens should be discarded from the batch used for strength testing if the damage will interfere with the strength test. In any case, such spallation must be noted in the report. Spallation problems can be alleviated by chamfering sharp edges.

NOTE 4—The parallelism tolerances on the four longitudinal faces are 0.015 mm for B and C and the cylindricity for A is 0.015 mm.

[5] The boldface numbers in parentheses refer to a list of references at the end of this standard.

8.2 *Test Specimen Preparation*—Depending on the intended application of the thermal shock data, one of the four test specimen preparation methods described in Test Method C1161 may be used: ~~As-Fabricated, Application-Matched Machining, Customary Procedures, or Standard Procedures.~~as-fabricated, application-matched machining, customary procedures, or standard procedures.

8.3 *Handling Precautions*—Care shall be exercised in storing and handling of test specimens to avoid the introduction of random and severe flaws, such as might occur if test specimens were allowed to impact or scratch each other.

8.4 *Number of Test Specimens*—A minimum of ~~10~~ten specimens shall be used to determine as-received strength at room temperature. A minimum of 30 is required if estimates regarding the form of the strength distribution ~~is~~are to be determined (for example, a Weibull modulus). A minimum of ~~5~~five specimens shall be used at each thermal shock temperature. It is recommended that as ΔT_c is established, an additional ~~5~~five specimens be tested at this as well as the adjacent temperature intervals. This will allow for determination of the mean and standard deviation. If estimates regarding the form of the strength distribution at the ΔT_c and adjoining temperature intervals are desired (for example, Weibull analysis) additional specimens must be tested at these temperature intervals. See Practice C1239 for guidance on estimating Weibull parameters.

9. Procedure

9.1 *Test Exposure Temperatures:*

9.1.1 The maximum exposure target temperature of the furnace for the thermal shock test of a given advanced ceramic will be determined from the maximum performance temperature required for a specific application, specified in a comparative thermal shock test, or cited in test literature.

9.1.2 The initial exposure temperature can be determined from literature values, prior test experience, or from a 50 % value of the maximum exposure temperature. Follow-on exposure/quench tests shall be performed such that the critical temperature difference is determined within a 50 K ~~(50°C)~~(50 °C) interval.

9.1.3 An efficient "bracketing" search technique for ΔT_c can be employed wherein the initial exposure temperature is chosen high enough that a definitive strength drop (>30 % of as-received strength, see Fig. 1) is expected and observed. (If the strength drop is not observed, repeat the test with a higher initial temperature.) The second exposure temperature is chosen at the midpoint of the first exposure temperature and room temperature. Each subsequent exposure temperature is selected at the midpoint between the lowest temperature producing a >30 % strength drop and the highest temperature to produce a <30 % strength drop. (See Figs. 2 and 3.) Continue the iteration until the temperature interval ~~is~~ between iterations is less than ~~100°C.~~100 °C. This search procedure minimizes the number of iterations needed to identify the ΔT_c, as compared to a stepwise fixed increment search procedure.

9.2 Clean the test specimens in water or alternate fluid to remove any cutting solutions or other contaminants. A final rinse in a quickly evaporating solvent such as acetone or ethanol is recommended. Determine the thickness and width of each test specimen in accordance with Test Method C1161. Determine the mass of each test specimen to an accuracy of ~~0.1%~~0.1 % or better. Calculate the bulk density for each specimen (Bulk density = mass / (length ~~x~~× width ~~x~~× thickness)

NOTE 5—If the calculated bulk density varies significantly ~~(<3%)~~(<3 %) between specimens or if the mean of the density for all the specimens is 95 % or less of the theoretical density of the test material, specimen porosity may be a critical experimental factor in thermal shock. If the porosity in specimens is of concern, the apparent porosity and apparent specific gravity of selected specimens may be measured prior to thermal shock testing using

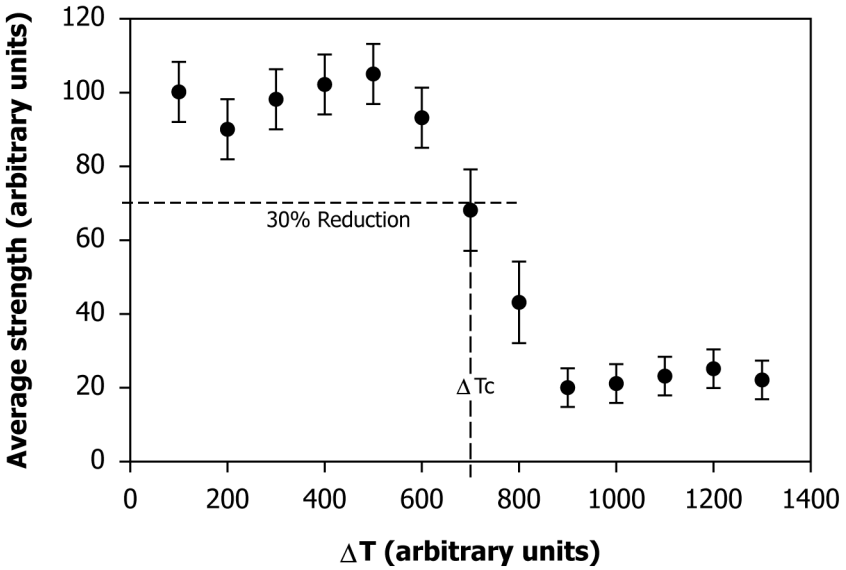

FIG. 1 Typical Plot of Average Strength ~~Versus~~versus Quenching Temperature Difference (Not for a Specific Material)

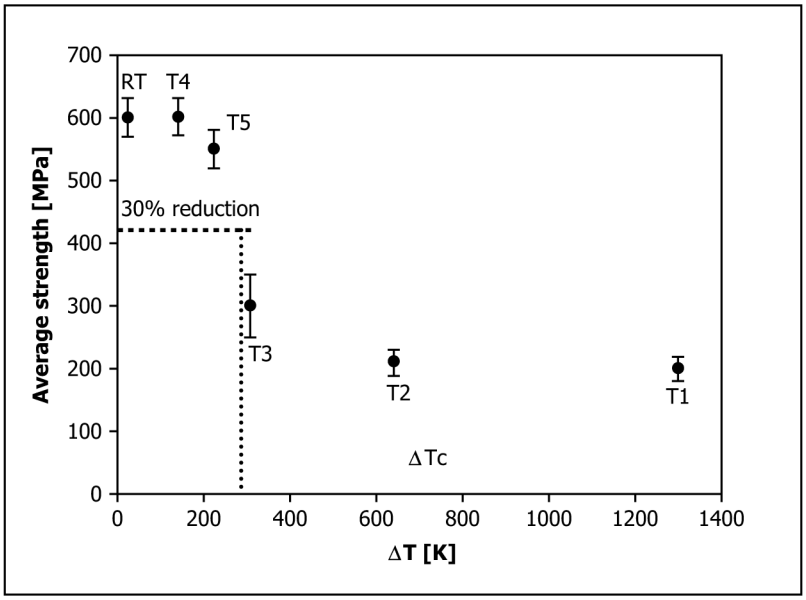

FIG. 2 Example of a Temperature Sequence Using the ″Bracketing″ Technique for a Material ~~With~~with a Low Thermal Shock ~~Resistance.~~Resistance

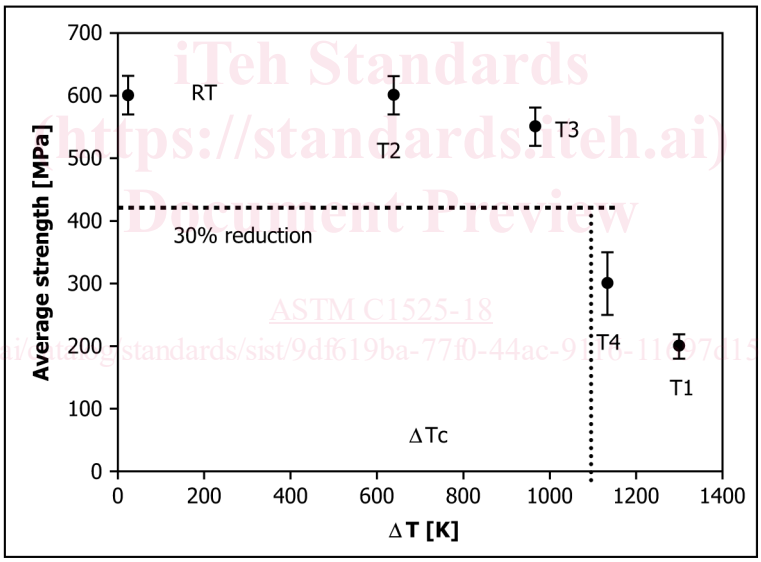

FIG. 3 Example of a Temperature Sequence Using the ″Bracketing″ Technique for a Material ~~With~~with a High Thermal Shock ~~Resistance.~~Resistance

an Archimedes density measurement (Test ~~Method~~Methods C373) and reported. Specimens that have been measured by Test ~~Method~~Methods C373 should be thoroughly dried out per ~~paragraph~~9.3.

9.3 Dry the test specimens in an oven at 393 ± 10 K (120 ± ~~10°C)~~10 °C) for 2 h. Allow the specimens to cool to room temperature in a dessicator. Select the specimens for quench testing and store in the dessicator until furnace exposure.

9.4 Perform the initial flexural strength test on at least ~~10~~ten test specimens in accordance with Test Method C1161, using the appropriate test machine and fixture.

9.5 Determine the mean and standard deviation of the strength of the as-received specimens.

9.6 Place the first set (minimum five test specimens) of quench test specimens in the cold furnace and heat slowly ~~[minimum~~(minimum 30 min to temperatures up to 873 K ~~(600°C);~~(600 °C); minimum 60 min to temperature greater than 873 K ~~(600°C)]~~(600 °C)) to the initial exposure temperature. Equilibrate at the exposure temperature for a period of 15 min and check/record the exposure temperature. After equilibration, remove the test specimens singly from the furnace, and transfer each of them to the quench bath as quickly as possible, but in no more than ~~5 s.~~ 5 s. A specific orientation of the specimens during this operation is not required.

9.7 After quenching, dry the test specimens in the drying oven and store, if necessary, in a dessicator per 9.3, before strength testing at room temperature.

9.8 Conduct strength tests on the quenched and dried test specimens in flexure at room temperature in accordance with Test Method C1161.

9.9 Calculate flexural strength according to Section 10, and compare the average flexural strength for the quenched test specimens to the strength of the as-received test specimens. A 30 % decrease in flexural strength for a given ΔT will meet the critical ΔT ~~requirement,~~requirement; see Figs. 2 and 3.

9.10 Once the exposure temperature for the ΔT is determined, repeat the test exposure/quench/strength test for the critical temperature as well as for one 50 K ~~(50°C)~~(50 °C) temperature interval above and one below this ΔT. Calculate average flexural strength and standard deviation for the three sets of test specimens and compare those values with those obtained for the as-received test specimens. Often an increase in the standard deviation is observed for the sets tested around ΔT_c (**43**), and this may help in determining the critical temperature interval. An example of a typical graph of average strength versus temperature interval is given in Fig. 1.

9.11 If desired, expose and test additional test sets to determine the strength reduction across the entire temperature regime of interest.

9.12 Performing fractographic analysis according to Practice C1322 is recommended for the as-received test specimens as well as for the test specimens tested at ΔT_c. Fractography could be helpful in determining the location and source of critical fracture flaws in the as-received test specimens and assessing if thermal shock produces a change in the critical flaw population with a corresponding strength drop.

10. Calculation

10.1 Evaluate flexural strength of the prismatic test specimens according to the formula for four-point flexure (see Test Method C1161):

$$S = \frac{3P(L_o - L_i)}{2b\,d^2} \tag{1}$$

where:

S = flexural strength, Pa,
P = measured fracture load, N,
L_o and L_i = outer and inner spans, respectively, m,
b = test specimen width, m, and
d = test specimen height, m.

Evaluate the strength of cylindrical test specimens as follows:

$$S = \frac{P(L_o - L_i)}{\pi r^3} \tag{2}$$

where:

r = radius of the test specimen cylinder.

10.2 Evaluate the mean, $\bar{S}$, and standard deviation, SD, according ~~to~~to:

$$\bar{S} = \frac{\sum_1^n S}{n} \tag{3}$$

$$SD = \sqrt{\frac{\sum_1^n (S - \bar{S})^2}{(n-1)}} \tag{4}$$

10.3 Calculate the ΔT for each exposure/quench test, where:

$$\Delta T = T_x - T_0 \tag{5}$$

where:

T_x = exposure test temperature, K or °C, and
T_0 = quench bath temperature, K or °C.

10.4 Plot the mean flexural strength and the standard deviations for each test set versus the ΔT, as shown in Fig. 1.