



Designation: C 1525 – 03

## Standard Test Method for Determination of Thermal Shock Resistance for Advanced Ceramics by Water Quenching<sup>1</sup>

This standard is issued under the fixed designation C 1525; 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 ( $\epsilon$ ) 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 temperature differences 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 and health practices and determine the applicability of regulatory limitations prior to use.*

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

Current edition approved May 10, 2003. Published July 2003. Originally approved in 2002. Last previous edition approved in 2002 as C 1525 - 02.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- C 1145 Terminology of Advanced Ceramics<sup>2</sup>
- C 1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature<sup>2</sup>
- C 1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Parameters for Advanced Ceramics<sup>2</sup>
- C 1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics<sup>2</sup>
- E 4 Practice for Force Verification of Testing Machines<sup>3</sup>
- E 6 Terminology Relating to Methods of Mechanical Testing<sup>3</sup>
- E 616 Terminology Relating to Fracture Testing<sup>2</sup>
- IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System<sup>4</sup>

#### 2.2 European Standard:

- EN 820-3 Advanced Technical Ceramics—Monolithic Ceramics—Thermomechanical Properties—Part 3: Determination of Resistance to Thermal Shock by Water Quenching<sup>5</sup>

### 3. Terminology

3.1 *Definitions*—The terms described in Terminologies C 1145, E 6, and E 616 are applicable to this standard test method. Specific terms relevant to this test method are as follows:

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

3.1.2 *critical temperature difference,  $\Delta T_c, n$* —temperature difference between the furnace and the ambient temperature water bath that will cause a 30 % drop in the average flexural strength.

<sup>2</sup> Annual Book of ASTM Standards, Vol 15.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 03.01

<sup>4</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>5</sup> Available from CEN, 36, rue de Stassart, B-1050 Brussels, Belgium, www.cenorm.be.

3.1.3 *flexural strength*,  $\sigma_f$ ,  $n$ —a measure of the ultimate strength of a specified beam specimen in bending determined at a given stress rate in a particular environment.

3.1.4 *fracture toughness*,  $n$ —a generic term for measures of resistance to extension of a crack. **E 616**

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

3.1.6 *thermal shock*,  $n$ —a large and rapid temperature change, resulting in large temperature differences within or across a body. **C 1145**

3.1.7 *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 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 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 ( $\Delta T$ ). However, it is important to notice that results may be different for the same  $\Delta T$  but different absolute temperatures. It is therefore specified in this test method to quench to room temperature.

6.4 The formulae presented in this test method apply strictly only to materials that do not exhibit *R*-curve behavior, but have a single-valued fracture toughness. If the test material exhibits a strong *R*-curve behavior, i.e., 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  $\pm 5$  K ( $\pm 5^\circ\text{C}$ ). The temperature shall be measured with suitable thermocouples located no more than 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  $293 \pm 2$  K ( $20^\circ\text{C} \pm 2^\circ\text{C}$ ) is required. The water bath must have sufficient volume to prevent the temperature in the bath from rising more than 5 K ( $5^\circ\text{C}$ ) 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 E 4. Specimens may be loaded in any suitable test machine provided that uniform test rates, either using load-controlled or

displacement-controlled mode, can be maintained. The loads used in determining flexural strength shall be accurate within  $\pm 1.0\%$  at any load within the selected load rate and load range of the test machine as defined in Practice E 4.

7.1.6 The configuration and mechanical properties of the test fixtures shall be in accordance with Test Method C 1161 for use with the standard four-point flexure specimens. If larger test pieces (sizes 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 C 1161.

7.1.7 The method requires a 393 K (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  $10 \pm 0.13$  mm in diameter, 120 mm long.

8.1.1.2 *Type B*—Bars  $3 \pm 0.13$  mm  $\times$   $4 \pm 0.13$  mm in cross section, minimum 45 mm long with chamfered edges, in accordance with type B in Test Method C 1161.

8.1.1.3 *Type C*—Bars  $10 \pm 0.13$  mm  $\times$   $10 \pm 0.13$  mm in cross section, 120 mm long, with chamfered edges.

NOTE 2—The test specimens of 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).<sup>6</sup> Test specimens of 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 C 1161.

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.

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 C 1161 may be used: 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 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 to be determined (for example a Weibull modulus). A minimum of 5 specimens shall be used at each thermal shock temperature. It is recommended that as  $\Delta T_c$  is established, additional 5 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  $\Delta T_c$  and adjoining temperature intervals are desired (for example, Weibull analysis) additional specimens must be tested at these temperature intervals. See Practice C 1239 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) interval.

9.1.3 An efficient “bracketing” search technique for  $\Delta T_c$  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. This search procedure minimizes the number of iterations needed to identify the  $\Delta T_c$ , 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 C 1161.

9.3 Dry the test specimens in an oven at  $393 \pm 10$  K ( $120 \pm 10^\circ\text{C}$ ) 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.

<sup>6</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

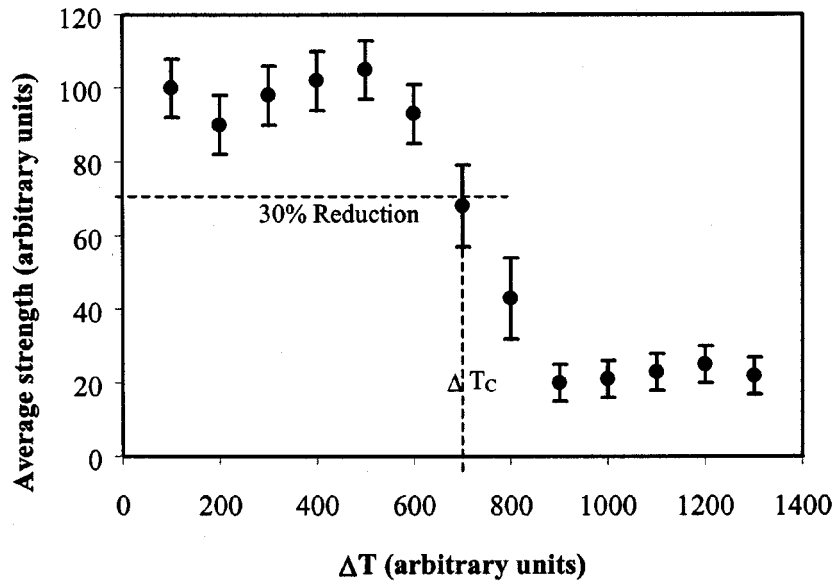


FIG. 1 Typical Plot of Average Strength Versus Quenching Temperature Difference (Not for a Specific Material)

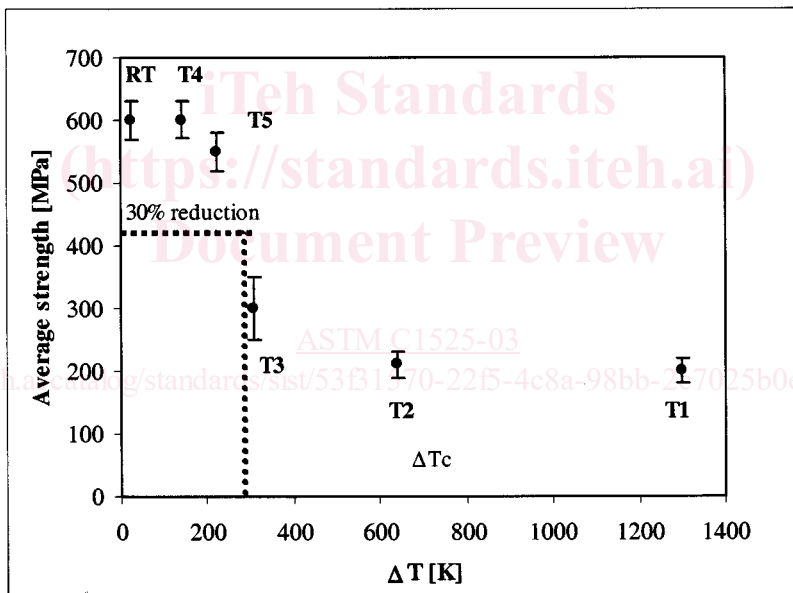


FIG. 2 Example of a Temperature Sequence Using the "Bracketing" Technique for a Material With a Low Thermal Shock Resistance.

9.4 Perform the initial flexural strength test on at least 10 test specimens in accordance with Test Method C 1161 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 30 min to temperatures up to 873 K (600°C); minimum 60 min to temperature greater than 873 K (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. 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 C 1161.

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