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Standard Test Methods for Vitrified Ceramic Materials for Electrical Applications[1]

This standard is issued under the fixed designation D116; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods outline procedures for testing samples of vitrified ceramic materials that are to be used as electrical insulation. Where specified limits are mentioned herein, they shall not be interpreted as specification limits for completed insulators.

1.2 These test methods are intended to apply to unglazed specimens, but they may be equally suited for testing glazed specimens. The report section shall indicate whether glazed or unglazed specimens were tested.

1.3 The test methods appear as follows:

Section	Test Method	Related Standard(s)
6	Compressive Strength	C773
13	Dielectric Strength	D618, D149
8	Elastic Properties	C623
15	Electrical Resistivity	D618, D257, D1829
7	Flexural Strength	C674, F417
9	Hardness	C730, E18
5	Porosity	C373
14	Relative Permittivity and Dissipation Factor	D150, D2149, D2520
4	Specific Gravity	C20, C329, F77
10	Thermal Conductivity	C177, C408
12	Thermal Expansion	C539, E288
11	Thermal Shock Resistance	

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in 11.3, 13.5, and 15.3.

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]

C20 Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water

C177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus

C329 Test Method for Specific Gravity of Fired Ceramic Whiteware Materials

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

C408 Test Method for Thermal Conductivity of Whiteware Ceramics

C539 Test Method for Linear Thermal Expansion of Porcelain Enamel and Glaze Frits and Ceramic Whiteware Materials by Interferometric Method

C623 Test Method for Young's Modulus, Shear Modulus, and Poisson's Ratio for Glass and Glass-Ceramics by Resonance

C674 Test Methods for Flexural Properties of Ceramic Whiteware Materials

C730 Test Method for Knoop Indentation Hardness of Glass

C773 Test Method for Compressive (Crushing) Strength of Fired Whiteware Materials

D149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies

D150 Test Methods for AC Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulation

D257 Test Methods for DC Resistance or Conductance of Insulating Materials

D618 Practice for Conditioning Plastics for Testing

D638 Test Method for Tensile Properties of Plastics

[1] These test methods are under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.03 on Methods for Whitewares and Environmental Concerns.

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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.

D1829 Test Method for Electrical Resistance of Ceramic Materials at Elevated Temperatures (Withdrawn 2001)[3]
D2149 Test Method for Permittivity (Dielectric Constant) And Dissipation Factor Of Solid Dielectrics At Frequencies To 10 MHz And Temperatures To 500°C
D2520 Test Methods for Complex Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials at Microwave Frequencies and Temperatures to 1650°C
E18 Test Methods for Rockwell Hardness of Metallic Materials
E288 Specification for Laboratory Glass Volumetric Flasks
F77 Test Method for Apparent Density of Ceramics for Electron Device and Semiconductor Application (Withdrawn 2001)[3]
F417 Test Method for Flexural Strength (Modulus of Rupture) of Electronic-Grade Ceramics (Withdrawn 2001)[3]

3. Significance and Use

3.1 For any given ceramic composition, one or more of the properties covered herein may be of more importance for a given insulating application than the other properties. Thus, it may be appropriate that selected properties be specified for testing these ceramic materials.

3.2 Pertinent statements of the significance of individual properties may be found in the sections pertaining to such properties.

4. Specific Gravity

4.1 *Scope*—Three test methods are given, providing for accuracy, convenience, or testing of small specimens.

4.2 *Significance and Use*—Specific gravity measurements provide data indicating the control of quality of the ceramic material. The thermal maturity of specimens may be estimated from such data. Specific gravity data are related to electrical, thermal, and mechanical properties of ceramics.

4.3 *Procedure:*

4.3.1 When the destruction of the specimen can be tolerated and the highest precision is required, determine the specific gravity in accordance with Test Method C329.

4.3.2 When it is not desirable to destroy the specimen and less precise values are acceptable, determine the specific gravity in accordance with Test Methods C20.

4.3.3 When only a very small specimen is available, determine the specific gravity in accordance with Test Method F77.

5. Porosity

5.1 *Scope*—Three test methods are given based on the relative porosity of the specimens.

5.2 *Significance*—Amount of porosity of a specimen is used as a check on structural reproducibility and integrity.

5.3 *Method A:*

5.3.1 In the case of relatively porous ceramics (water absorption greater than 0.1 %), determine the porosity as water absorption in accordance with Test Method C373.

NOTE 1—Test Method C373 has been found suitable for determining water absorption in the range of 0.1 %, although that test method was derived specifically for absorptions exceeding 3.0 %.

5.3.2 An alternative to Method A, using gas as a fluid, may be found in the literature.[4,5]

5.4 *Method B—Dye Penetration Under Pressure:*

5.4.1 *Apparatus*—The apparatus shall consist of a suitable pressure chamber of such dimensions as to accommodate the test specimen when immersed in the dye solution with arrangements for obtaining and maintaining the required pressure for the required time.

5.4.2 *Reagent*—A fuchsine dye solution consisting of 1 g of basic fuchsine in 1 L of 50 % reagent ethyl alcohol is suitable.

5.4.3 *Specimens*—The specimens shall be freshly broken fragments of the ceramic body, having clean and apparently unshattered surfaces exposed. At least 75 % of the area of such specimens should be free of glaze or other surface treatment. Fragments approximately 5 mm in the smallest dimension up to 20 mm in the largest dimensions are recommended.

5.4.4 *Procedure:*

5.4.4.1 Place the specimen fragments in the pressure chamber and immerse completely in the fuchsine solution.

5.4.4.2 Apply a pressure of 28 MPa (4000 psi) ± 10 % for approximately 15 h. An optional pressure of 70 MPa (10 000 psi) ± 10 % for 6 h may be used.

5.4.4.3 At the conclusion of the application of the test pressure, remove the specimens from the pressure chamber, rinse and dry thoroughly, and break as soon as possible for visual examination.

5.4.4.4 Porosity is indicated by penetration of the dye into the ceramic body to an extent visible to the unaided eye. Disregard any penetration into small fissures formed in preparing the test specimen.

5.4.5 *Report*—The report shall include a statement of the observations recorded in accordance with the examination in 5.4.4.4.

5.4.6 *Precision and Bias*—This test method has been in use for many years, but no statement for precision has been made and no activity is planned to develop such a statement. A statement of bias is unavailable in view of the lack of a standard reference material for this property.

5.5 *Method C—Dye Penetration Under Atmospheric Pressure:*

5.5.1 *Apparatus*—The apparatus shall consist of a suitable open-air chamber of such dimensions as to accommodate the test specimens when immersed in the dye solution.

5.5.2 *Reagent*—The fuchsine solution of 5.4.2 is suitable.

5.5.3 *Specimens*—The specimens of 5.4.3 are suitable.

5.5.4 *Procedure:*

5.5.4.1 Place the test specimens in the chamber and immerse completely in the fuchsine solution.

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

[4] Wasburn, E. W. and Bunting, E. N., "The Determination of the Porosity of Highly Vitrified Bodies," *Journal of the American Ceramic Society*, Vol 5, 1922, pp. 527–535.

[5] Navias, Louis, "Metal Porosimeter for Determining the Pore Volume of Highly Vitrified Ware," *Journal of the American Ceramic Society*, Vol 8, 1925, pp. 816–821.

5.5.4.2 Permit the specimens to remain immersed for 5 min or longer, remove, rinse, dry thoroughly and break as soon as possible for visual examination.

5.5.4.3 Porosity is indicated by penetration into the ceramic body to an extent visible with the unaided eye. Disregard any penetration into small fissure formed in the preparation of the specimens.

5.5.5 *Report*—The report shall include a statement of the observations recorded in accordance with the examination in 5.5.4.3.

5.5.6 *Precision and Bias*—This test method has been in use for many years, but no statement for precision has been made and no activity is planned to develop such a statement. A statement of bias is unavailable in view of the lack of a standard reference material for this property.

6. Compressive Strength

6.1 *Scope*—These test methods provide for the determination of the compressive (crushing) strengths of the full range of ceramics from relatively weak to the very strongest.

6.2 *Significance and Use*—Since many ceramic insulators are subjected to compressive stresses, knowledge of this property is important. The test yields data that are useful for purposes of design, specification, quality control, research, and in the comparison of ceramic materials.

6.3 *Procedure*—Determine compressive strength in accordance with Test Method C773.

7. Flexural Strength

7.1 *Scope:*

7.1.1 This test method includes two procedures: for testing a material for characterization purposes and for testing the material constituting the finished ware.

7.1.2 For the characterization of ceramic compositions, when relatively large specimens may be easily produced, Method A is recommended. Method B is acceptable.

7.1.3 When specimens must be cut from a fired sample Method B is recommended.

7.2 *Significance and Use*—Flexural strength correlates with other mechanical strength properties and is generally the easiest and most economical test procedure available. The values are useful for purposes of design, quality control, research, and the comparison of different ceramic compositions.

7.3 *Procedure:*

7.3.1 *Method A*—Determine the flexural strength in accordance with Test Methods C674.

7.3.2 *Method B—Microbar MOR Test*—Determine the flexural strength in accordance with Test Method F417.

8. Elastic Properties

8.1 *Scope*—This test method obtains, as a function of temperature, Young's modulus of elasticity, the shear modulus (modulus of rigidity), and Poisson's ratio for vitrified ceramic materials.

8.2 *Significance and Use*—The elastic properties of a ceramic are important design parameters for load-bearing applications and give indications of relative rigidity of a material.

8.3 *Procedure*—Determine the elastic properties in accordance with Test Method C623.

9. Hardness

9.1 *Scope*—Two methods are given. Method A requires little in the way of specimen preparation and has a limited capability of differentiating between samples. Method B requires preparation of a polished section of the specimen and has an extended limit of differentiation between samples.

9.2 *Significance and Use*—Hardness can be used as an easily obtained indicator of the thermal maturity of a specimen, particularly when used in conjunction with the specimen specific gravity.

9.3 *Procedure:*

9.3.1 *Method A*—Determine the Rockwell superficial hardness in accordance with Test Methods E18. Use the Type N Scale and a 45 kg major load.

9.3.2 *Method B*—Determine the Knoop hardness in accordance with Test Method C730. Use a polished surface and a 1 kg load.

10. Thermal Conductivity

10.1 *Scope*—The recommended procedures allow the determination of the thermal conductivity of ceramic materials from 40 to 150 °C (100 to 300 °F).

10.2 *Significance*—A ceramic insulator may be subjected frequently to thermal shock or required to dissipate heat energy from electrically energized devices. Thermal conductivity characteristics are useful in designing ceramic insulators for service, research, quality control, and comparison of ceramic compositions.

10.3 *Procedure*—Determine the thermal conductivity in accordance with Test Method C408.

NOTE 2—If thermal conductivity values over a broader temperature range of a lower order of magnitude than those obtainable using Test Method C408 are required, Test Method C177 may be used.

11. Thermal Shock Resistance

11.1 *Scope*—These thermal shock tests may be used for the determination of the resistance of a given ceramic material to simulated environmental heat service conditions.

11.2 *Significance and Use*—These tests serve as an evaluation of the resistance of a particular ceramic composition, shape, and dimension to temperature stress relative to another composition of the same shape and dimensions.

11.3 *Hazards:*

11.3.1 **Warning—**Acetone vapors are flammable and poisonous and should not be breathed. The bath in 11.4.2 shall be operated in a vented hood with no open flames or sparks nearby.

11.3.2 **Warning—**Under certain conditions some ceramic specimens can disintegrate explosively, sending out fragments at damage-producing velocities and causing splashing of bath mediums.

11.3.3 **Warning—**—Face shields, long-sleeve coat, and insulating gloves shall be worn by test personnel to prevent injury.

11.4 *Apparatus:*

11.4.1 *Liquid Cold Bath,* maintained at <1 °C (1.8 °F) and consisting of chopped ice and water.

11.4.2 *Liquid Cold Bath,* maintained at −75 ± 2 °C (−103 ± 3.6 °F) and consisting of acetone and chopped dry ice.

11.4.3 *Dry Cold Bath,* maintained at any (usually simulated service) temperature desired, but controlled to ±5 °C (±9 °F) and consisting of a fluidized sand bath rolled gently with precooled dry air or nitrogen.

11.4.4 *Liquid Hot Bath,* maintained at any prescribed temperature between 65 and 100 °C (149 and 212 °F), but controlled to ±1 °C (±1.8 °F) and consisting of heated water.

11.4.5 *Liquid Hot Bath,* maintained at any prescribed temperature between 90 and 275 °C (194 and 527 °F), but controlled to ±3 °C (±5.4 °F) and consisting of heated glycerin.

11.4.6 *Dry Hot Bath,* maintained at any (usually simulated service) temperature desired, but controlled to ±5 °C (±9 °F) and consisting of a fluidized sand bath with a self-contained heater.

11.4.7 *High-temperature Muffle Furnace,* maintained at any desired temperature above 800 °C (1472 °F), but controlled to ±5 °C (±9 °F).

11.4.8 The volume of any liquid or dry bath medium should be greater than five times the total volume of the test specimens and the immersion device (if used).

11.4.9 Test conditions should be chosen that are sufficiently extreme to cause some structural failures.

11.5 *Specimens*—Test specimens shall be of one or more of the following:

11.5.1 *Type A*—Cylinders 150 mm (6 in.) long and 28.5 mm (1.125 in.) in diameter.

11.5.2 *Type B*—Cylinders 150 mm (6 in.) long and 12.7 mm (0.5 in.) in diameter.

11.5.3 *Type C*—Dumbbells in accordance with Type I of Test Method D638, 6.3 mm (0.25 in.) thick by 114.3 mm (4. in.) in distance between the grips.

11.5.4 *Type D*—Completed parts.

11.5.5 *Type E*—Microbar flexural strength specimens from Section 6.

11.6 *Procedure:*

11.6.1 Immerse the test specimens in the cold bath maintained at the specified temperature. Hold submerged for 5 min, remove, and immediately plunge rapidly into the hot medium held at the prescribed temperature. Hold submerged for 5 min, then repeat the cycles for a total of five times.

11.6.2 If thermal shock resistance to a wider temperature differential is desired, usually due to lack of thermal shock damage at the originally specified differential, increase the temperature differential by 25 to 75 °C (77 to 167 °F) increments and repeat in accordance with 11.6.1 on new specimens at each level.

11.6.3 Immerse the specimens in the fuchsine solution in 5.5.2 for 10 min, remove, rinse, and dry thoroughly. Examine for fracture, crazing, and so forth, under a bright light. If so specified, determine the flexural strength after thermal shock by a method specified in Section 7.

11.7 *Report*—The report shall include the following:

11.7.1 Types and temperatures of baths used,

11.7.2 Number and type (A, B, and so forth) of specimens used,

11.7.3 Visual results on each specimen after each cycle or series of five cycles, and method of observation, and,

11.7.4 If specified, the individual and average flexural or tensile strength of the thermal-shock specimens.

11.8 *Precision and Bias*—This method has been in use for many years, but no statement for precision has been made and no activity is planned to develop such a statement. A statement of bias is unavailable in view of the lack of a standard reference material for this property.

12. Thermal Expansion

12.1 *Scope*—Two methods are recommended: the interferometric method, best suited for examination of physically small specimens, interfaces, or local area, and the dilatometer method, which while not as precise or sensitive as the interferometer method can be used at higher temperatures. Because of these larger specimens, Method B may produce results more representative of massive pieces.

12.2 *Significance and Use*—Thermal expansion is an important design parameter for higher temperature applications and an indicator of relative thermal shock resistance.

12.3 *Procedure:*

12.3.1 *Method A (Interferometer)*—Determine the thermal expansion in accordance with Test Method C539.

12.3.2 *Method B (Dilatometer)*—Determine the thermal expansion in accordance with Test Method E288.

13. Dielectric Strength

13.1 *Scope:*

13.1.1 Methods are given for determining ac dielectric strength under oils.

13.1.2 Two conditioning methods are allowed.

13.2 *Significance*—The dielectric strength of a ceramic is of importance in comparing different materials or controlling quality of different lots. The values obtained usually will have little relation to voltage breakdown realized in service. While mechanical requirements often dictate thickness of dielectrics far greater than needed to withstand the electrical stress, dielectric strength data will serve as a guide in estimating the