Designation: D 2442 – 75 (Reapproved 2001)

Standard Specification for Alumina Ceramics for Electrical and Electronic Applications[1]

1. Scope

1.1 This specification covers the requirements for fabricated alumina parts suitable for electronic and electrical applications and ceramic-to-metal seals as used in electron devices. This standard specifies limits and methods of test for electrical, mechanical, thermal, and general properties of the bodies used for these fabricated parts, regardless of part geometry.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 *ASTM Standards:*

C 20 Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water[2]

C 108 Symbols for Heat Transmission[2]

C 242 Terminology of Ceramic Whitewares and Related Products[3]

C 408 Test Method for Thermal Conductivity of Whiteware Ceramics[3]

C 573 Methods for Chemical Analysis of Fireclay and High-Alumina Refractories[4]

C 623 Test Method for Young's Modulus, Shear Modulus, and Poisson's Ratio for Glass and Glass-Ceramics by Resonance[3]

D 116 Methods of Testing Vitrified Ceramic Materials for Electrical Applications[5]

D 149 Test Methods for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies[5]

D 150 Test Methods for AC Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulation [5]

D 257 Test Methods for DC Resistance or Conductance of Insulating Materials[5]

D 618 Practice for Conditioning Plastics for Testing[6]

D 1711 Terminology Relating to Electrical Insulation[5]

D 1829 Test Method for Electrical Resistance of Ceramic Materials at Elevated Temperatures[5]

D 2149 Test Method for Permittivity (Dielectric Constant) and Dissipation Factor of Solid Ceramic Dielectrics at Frequencies to 10 MHz and Temperatures to 500°C[5]

D 2520 Test Method for Complex Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials at Microwave Frequencies and Temperatures to 1650°C[7]

E 6 Terminology Relating to Methods of Mechanical Testing[8]

E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[9]

E 122 Practice for Calculating Sample Size to Estimate, with a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process[10]

E 165 Practice for Liquid Penetrant Inspection Method[11]

E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer[10]

F 19 Test Method for Tension and Vacuum Testing Metalized Ceramic Seals[12]

F 77 Test Method for Apparent Density of Ceramics for Electron Device and Semiconductor Application[3]

F 109 Terminology Relating to Surface Imperfections on Ceramics[3]

F 134 Test Methods for Determining Hermeticity of Electron Devices with a Helium Mass Spectrometer Leak Detector[12]

F 417 Test Method for Flexural Strength (Modulus of Rupture) of Electronic-Grade Ceramics[3]

2.2 *Other Standards:*

[1] This specification is under the jurisdiction of Committee C-21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.03 on Fundamental Properties.

This specification also includes material and suggestions provided by ASTM Committee D-9 on Electrical and Electronic Insulating Materials.

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[2] *Annual Book of ASTM Standards*, Vol 15.01.

[3] *Annual Book of ASTM Standards*, Vol 15.02.

[4] *Annual Book of ASTM Standards*, Vol 03.05.

[5] *Annual Book of ASTM Standards*, Vol 10.01.

[6] *Annual Book of ASTM Standards*, Vol 08.01.

[7] *Annual Book of ASTM Standards*, Vol 10.02.

[8] *Annual Book of ASTM Standards*, Vol 03.01.

[9] *Annual Book of ASTM Standards*, Vol 15.05.

[10] *Annual Book of ASTM Standards*, Vol 14.02.

[11] *Annual Book of ASTM Standards*, Vol 03.03.

[12] *Annual Book of ASTM Standards*, Vol 10.04.

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes[13]
MIL-STD-883 Test Methods and Procedures for Microelectronics[14]
ANSI B46.1 Surface Texture[15]

3. Terminology

3.1 *Definitions:*

3.1.1 The applicable definitions of terms in the following documents shall apply to this specification: Symbols C108, and Definitions C 242, D 1711, E 6, E 12, and F 109.

4. Classification

4.1 Ceramics covered by this specification shall be classified by alumina content as follows:

Type	Alumina Content Weight percent, min
I	82
II	93
III	97
IV	99

5. Basis of Purchase

5.1 Purchase orders for ceramic parts furnished to this specification shall include the following information:

5.1.1 Type designation (see 3.1),

5.1.2 Surface finish and allowable defect limits (if required) (Definitions F 109, ANSI B46.1, and Appendix X1),

5.1.3 Part drawing with dimensional tolerances (Appendix X1),

5.1.4 Specific tests (if required),

5.1.5 Certification (if required), and

5.1.6 Packing and marking.

6. Requirements

6.1 This material shall conform to the electrical, mechanical, thermal, and general property requirements specified in Table 1, Table 2, Table 3, and Table 4.

6.2 Dimensional and surface finish requirements of the parts shall be as agreed between the supplier and the purchaser; however, guidance for establishing such an agreement is provided in Appendix X1.

6.3 *Visual Requirements*:

6.3.1 Parts shall be uniform in color and texture. Cracks, blisters, holes, porous areas, inclusions, and adherent foreign particles shall not be permitted. The limits of surface imperfections such as pits, pocks, chips (open or closed), surface marks, fins, ridges, and flow lines shall be set by mutual agreement between the supplier and the purchaser. Limiting dimensions for these defects, when required for clarification, will be listed in the parts drawing or purchase description. For definitions of the surface imperfections enumerated above, see Definitions F 109.

TABLE 1 Electrical Requirements

Property	Type I	Type II	Type III	Type IV
Dielectric constant, max 25°C:				
at 1 MHz	8.8	9.6	9.8	10.1
at 10 GHz	8.7	9.6	9.8	10.1
Dissipation factor, max 25°C:				
at 1 MHz	0.002	0.001	0.0005	0.0002
at 10 GHz	0.002	0.001	0.0005	0.0002
Volume resistivity, min Ω·cm:				
at 25°C	10^{14}	10^{14}	10^{14}	10^{14}
at 300°C	1×10^{10}	1×10^{10}	1×10^{10}	7×10^{10}
at 500°C	4×10^{7}	2×10^{7}	8×10^{7}	1×10^{8}
at 700°C	4×10^{6}	2×10^{6}	6×10^{6}	1×10^{7}
at 900°C	4×10^{5}	2×10^{5}	8×10^{5}	1×10^{6}
Dielectric strength:				
3.175 mm (0.125 in.) min kV/mm	9.85 (250 V/mil)	9.85 (250 V/mil)	9.85 (250 V/mil)	9.85 (250 V/mil)

TABLE 2 Mechanical Requirements

Property	Type I	Type II	Type III	Type IV
Flexural strength, min avg,[A] MPa (psi)	240 (35 000)	275 (40 000)	275 (40 000)	275 (40 000)
Modulus of elasticity, min, GPa (psi)	215 (31×10^{6})	275 (40×10^{6})	310 (45×10^{6})	345 (50×10^{6})
Poisson's ratio, average	0.20 to 0.25	0.20 to 0.25	0.20 to 0.25	0.20 to 0.25

[A] Maximum permissible coefficient of variation is 10 percent.

6.3.2 For hermetic seal applications at least ¾ of the width of the seal surface shall remain intact at the location of any defect.

6.3.3 On other surfaces the limits for defects are such that the dimensional tolerances of the part are not affected at the location of the defect.

7. Test Specimens

7.1 The preferred specimens for test are, where possible, the actual part. When necessary, however, specific test specimens shall be prepared from the same batch of material and by the same processes as those employed in fabricating the ceramic part insofar as possible.

8. Specimen Preparation

8.1 The specimens for tests described in 9.1-9.3 shall be preconditioned in accordance with Procedure A of Test Methods D 618.

9. Test Methods

9.1 *Dielectric Constant and Dissipation Factor*—Determine in accordance with Test Methods D 150. Determine values at higher frequencies in accordance with Test Methods D 2520.[16] Determine values at higher temperatures in accordance with Test Method D 2149.

[13] Available from the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.

[14] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

[15] Available from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

[16] For another suitable method see *Dielectric Materials and Applications*, edited by Von Hippel, A., John Wiley and Sons, Inc., New York, N.Y., 1954.

TABLE 3 Thermal Requirements

Property	Type I		Type II		Type III		Type IV	
	min	max	min	max	min	max	min	max
Mean coefficient of linear thermal expansion,µ m/m·°C:								
25–200°C	5.4	6.2	5.2	6.7	5.2	6.5	5.5	6.7
25–500°C	6.5	7.0	6.6	7.4	6.7	7.5	6.8	7.6
25–800°C	7.0	7.7	7.3	8.1	7.4	8.1	7.3	8.1
25–1000°C	7.4	8.2	7.5	8.3	7.6	8.3	7.5	8.4
Thermal conductivity, cal/s·cm·°C:								
at 100°C	0.023	0.049	0.031	0.077	0.048	0.073	0.052	0.090
at 400°C	0.015	0.022	0.014	0.036	0.022	0.033	0.023	0.047
at 800°C	0.009	0.018	0.009	0.021	0.014	0.021	0.014	0.025
Thermal shock resistance	pass		pass		pass		pass	
Maximal deformation at 1500°C	...		...		0.51 mm (0.02 in.)		0.51 mm (0.02 in.)	

TABLE 4 General Requirements[A]

Property	Type I	II	III	IV
Density, apparent min[B] g/cm^3	3.37	3.57	3.72	3.78
Composition, min weight percent	82	93	97	99
Gas Impermeability	gas tight			
Liquid Impermeability	pass			
Metallizability[A]	[C]			

[A] Vendors shall, upon request, provide information on these properties as well as a visual standard of a typical microstructure of their specific ceramic body depicting its grain size and pore volume. Changes in microstructure of the ceramic are not acceptable as they can affect the behavior of the ceramic toward a metallizing process.

[B] The apparent density of a ceramic body is a function of the amount and the density of the primary Al_2O_3 phase and the secondary phase plus the amount of pores inherent to that body. The acceptable density limits for a specific alumina body must be consistent with the composition and the pore volume of the ceramic supplied by supplier and shall be agreed upon between the purchaser and the supplier. Variation in the apparent density of a specific ceramic body shall be within ±1 percent of the nominal value.

[C] Generally, very high alumina content results in increased difficulty of metallizing; however, variations in metallizing compositions and techniques can produce excellent seals in all four types of alumina ceramics. Because of a wide variation in materials and techniques, no specific test is recommended. A referee test for seal strength is Method F 19.

9.2 *Volume Resistivity*—Determine in accordance with Test Methods D 257. For elevated temperature measurements use Procedure A of Test Method D 1829.

9.3 *Dielectric Strength*—Run this test under oil in accordance with 6.1.1 of Test Methods D 149, with a rise rate of 1000 V/s on a 3.175-mm (0.125-in.) thick test specimen.

9.4 *Flexural Strength*—Determine in accordance with Test Method F 417 or Methods D 116. Somewhat lower values will result if Methods D 116 are used. The method to be used shall be agreed upon between the supplier and the purchaser.

9.5 *Modulus of Elasticity and Poisson's Ratio*—Determine in accordance with Test Method C 623.

9.6 *Thermal Expansion*—Determine in accordance with Test Method E 228.

9.7 *Thermal Conductivity*—Determine in accordance with Test Method C 408. For temperatures in excess of 149 C (300 F), use a suitable method.[17]

9.8 *Thermal Shock Resistance*—This test is to be agreed upon between supplier and purchaser. It is suggested that the cold end of the cycle be ice water at 0°C. Methods of heating and conditions at elevated temperatures shall be negotiated. The transfer from one temperature extreme to another shall be immediate.

9.9 *Temperature Deformation*—Determine deformation at 1500°C in accordance with Appendix X2.

9.10 *Apparent Density*—Determine in accordance with Test Method F 77. For large ceramic parts not covered by this method, determine in accordance with Test Methods C 20.

9.11 *Compositional Analysis*—Use either quantitative emission spectrographic analysis of the fired ceramic with alumina content determined by difference or Methods C 573 after assuming that all determined metallic and reactive elements originally are present as their highest form of oxide.

9.12 *Gas Impermeability*—When air fired at 900°C for 30 min and handled with tweezers only, then tested on a helium mass spectrometer leak detector capable of detecting a leak of 10^{-9} atm·cm^3/s, the ceramic is considered impermeable if a specimen 0.254 mm (0.010 in.) thick shows no indication of helium leakage when an area of 322.6 mm^2 (0.5 in.^{2}) is tested for 15 s at room temperature (Method 1014, Seal, of MIL-STD-883 and Test Methods F 134).

9.13 *Liquid Impermeability*—Determine in accordance with Methods D 116.

9.14 *Surface Imperfections*—Examine visually for surface imperfections with or without the aid of a dye penetrant as in Practice E 165. Agreement by purchaser and supplier regarding specific techniques is strongly recommended.

9.15 *Surface Finish*—If surface finish is specified, it shall be determined by any appropriate method agreed upon by purchaser and supplier.

10. Inspection

10.1 When agreed upon between the manufacturer and the purchaser, the purchaser may inspect the ceramic parts and verify the test results at the manufacturer's facility. Otherwise

[17] For a suitable method see Francl, J., and Kingery, W. D., "An Apparatus for Determining Conductivity by a Comparative Method," *Journal of the American Ceramic Society*, JACTA Vol 37, 1954, p. 80.

the purchaser shall inspect and test the ceramic parts within one month of the date of receipt by the purchaser or at such other times as may be agreed upon between the purchaser and the manufacturer.

10.2 When agreed upon between the manufacturer and the purchaser, the manufacturer shall supply, prior to fabrication, duplicate test specimens to the purchaser for his testing purposes. These specimens shall be identical with those tested by the manufacturer, insofar as it is possible.

11. Lot Acceptance Procedure

11.1 Unless otherwise specified Practice E 122 shall be used. When so specified, appropriate sample sizes shall be selected from each lot according to MIL-STD-105. Each quality characteristic shall be assigned an AQL value in accordance with MIL-STD-105 definition for critical, major, and minor classifications. Inspection levels shall be agreed upon between the supplier and the purchaser.

12. Certification

12.1 Any test results requiring certification shall be explicitly agreed upon, in writing, between the purchaser and the manufacturer.

13. Packing and Marking

13.1 Special packing techniques shall be subject to agreement between the purchaser and the manufacturer. Otherwise all parts shall be handled, inspected, and packed in such a manner as to avoid chipping, scratches, and contamination, and in accordance with the best practices to provide ample protection against damage during shipment.

13.2 The ceramics furnished under this specification shall be identified by the name or symbol for the ceramic body and, if necessary, by an identification number. This identification number shall provide ready access to information concerning the fabrication history of the particular ceramic part and shall be retained on file at the manufacturer's facility for one month after that particular lot or batch has been accepted by the purchaser.

APPENDIXES

(Nonmandatory Information)

X1. DIMENSIONAL TOLERANCES AND SURFACE FINISH

X1.1 Scope

X1.1.1 The general dimensional tolerances listed below are to be considered typical for most high alumina ceramics, particularly those of simple geometry and good symmetry. Specific unique designs are always considered as individual cases, since it may be necessary to apply broader tolerance to them, and are, therefore, subject to agreement between the purchaser and the supplier.

X1.1.2 Grinding and other finishing operations permit closer dimensional tolerances which are comparable to those obtainable on metal. Since grinding generally is done with diamond tools and is an expensive operation, careful consideration should be given to the actual need for close tolerances.

X1.2 Tolerances

X1.2.1 *Unglazed Surfaces*—The tolerance is ±1 % but not less than ±0.127 mm (0.005 in.).

X1.2.2 *Glazed Surfaces*—The tolerance is ±2 % but not less than ±0.305 mm (0.012 in.).

X1.2.3 *Angular Dimensions*—The tolerance is ±2°.

X1.2.4 *Parallelism*—Parallelism will be considered satisfactory if the thickness measured at any point is within the dimensional tolerance.

X1.2.5 *Ellipticity*—Ellipticity (or deviation from a true circle) shall be determined by dividing the maximum outside diameter by the minimum outside diameter measured in the same planes, perpendicular to the axis. The maximum value is 1.02 when the wall thickness is 12 % or more, of outside diameter and is 1.03 when less than 12 % of outside diameter.

X1.2.6 *Concentricity*—This shall be expressed as a deviation of centers. A total indicator reading of 1 % of the outside diameter or 0.254 mm (0.010 in.), whichever is larger, is considered typical where all diameters are either all ground or all unground.

X1.2.7 *Camber*—Camber shall be expressed as the ratio between arch height and the maximum length of the part. A maximum camber of 0.006 cm/cm (0.006 in./in.) of length is considered typical.

X1.2.8 *Surface Finish*—Surface finish is the deviation of the heights and depths of surface irregularities from a central reference line. The value obtained is the arithmetic average deviation of the magnitude of surface irregularities taken at equally spaced intervals and is expressed in microinches. A roughness-width cut-off of 0.76 mm (0.03 in.) is generally recommended. Ranges of surface finish generally available are listed in Table X1.1. Individual surface finish values shall be specified with required tolerances.

TABLE X1.1 Surface Finish Ranges

Type Surface	Finish, µin.
Polished or glazed	0 to 30
Ground	10 to 50
As fired	5 to 65

X2. METHOD FOR MEASURING DEFORMATION OF A CERAMIC BAR AT 1500°C

X2.1 Scope

X2.1.1 This appendix described the method of conducting a thermal deformation test for high alumina ceramics.

X2.2 Apparatus

X2.2.1 *Plane reference fixture* comprising three steel or alumina balls ¼ in. or 6 mm in diameter seated such that their centers form the vertices of an isoceles triangle with base 19 ± 1 mm or 0.75± 0.05 in. and altitude 127.00 ± 0.76 mm (5.00 ± 0.03 in.) and with a dial gage or depth micrometer mounted at a point within 0.5 mm (0.02 in.) of the center of the triangle.

X2.2.2 *Test fixture* comprising two high alumina end pieces at least 27.9 mm (1.1 in.) wide and 15 mm (0.6 in.) long spaced 127.00 ± 0.76 mm (5.00 ± 0.03 in.) apart.

X2.2.3 *Furnace* sufficient to maintain one or more test fixtures at 1500 ± 10°C in a wet hydrogen atmosphere for 30 min. The cooling rate shall be controlled and must not exceed 100 C/h.

X2.3 Test Specimen

X2.3.1 Three or more specimens are required for this test.

X2.3.2 Each specimen used in this test shall be a bar of the ceramic type being investigated, 4.57 ± 0.13 mm (0.180 ± 0.005 in.) in depth, 25.4 ± 2.5 mm (1.0 ± 0.1 in.) in width, and 152.4 ± 2.5 mm (6.0 ± 0.1 in.) in length.

X2.4 Procedure

X2.4.1 Test at least three bars.

X2.4.2 Center a test bar on a test fixture and place the plane reference fixture over the test bar so that the centers of the balls are over the edges of the end pieces of the test fixture.

X2.4.3 Determine and record to the nearest 0.02 mm or 0.001 in. the dial or micrometer reading of the midpoint of the upper surface of the test specimen.

X2.4.4 Remove the plane reference fixture and place the test bar and test fixture in the furnace. Heat in a wet hydrogen atmosphere (dew point range − 34 C to + 38°C) to 1500 ± 10°C and hold for 30 min. Cool to room temperature at a rate not to exceed 100°C/h.

X2.4.5 Remove the test bar and test fixture from the furnace and place the plane reference fixture over the test bar in the same position as in X2.4.2.

X2.4.6 Determine and record to the nearest 0.001 in. or 0.02 mm the dial or micrometer reading of the midpoint of the upper surface of the test specimen.

X2.4.7 Repeat X2.4.2-X2.4.6 for the remaining test bars.

X2.5 Calculations

X2.5.1 For each bar determine the deformation by taking the difference between the two readings (X2.4.3 and X2.4.6).

X2.6 Report

X2.6.1 Report the following information:

X2.6.1.1 Identification of specimens, and

X2.6.1.2 Deformation of each specimen.