Designation: F2393 – 12 (Reapproved 2020)

Standard Specification for High-Purity Dense Magnesia Partially Stabilized Zirconia (Mg-PSZ) for Surgical Implant Applications[1]

This standard is issued under the fixed designation F2393; 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 specification covers material requirements for high-purity, dense zirconium oxide partially stabilized by magnesium oxide (magnesia partially stabilized zirconia (Mg-PSZ)) for surgical implant applications.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

1.4 *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

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

C1198 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Sonic Resonance

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

C1259 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration

C1327 Test Method for Vickers Indentation Hardness of Advanced Ceramics

E112 Test Methods for Determining Average Grain Size

2.2 *American Society for Quality Standard (ASQ):*

C1 Specification of General Requirements for a Quality Program[3]

2.3 *ISO Standard:*

ISO 18754 Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics)—Determination of Density and Apparent Porosity[4]

3. Chemical Requirements

3.1 The chemical composition shall be as follows, measured by ICP-ES, XRF, or mass spectroscopy:

Oxides	Weight percent
$ZrO_2 + HfO_2 + MgO$	≥99.8
MgO	3.1-3.4
HfO_2	≤2.0
Total Other Oxides	<0.20
Other Oxides	
Fe_2O_3	<0.01
SiO_2	<0.05
CaO	<0.02
Al_2O_3	<0.05

NOTE 1—The radioactivity, defined as the sum of the massic activity of U238, Ra226, Th232, and determined by γ-spectroscopy on the ready-to-use powder, should be less than 200 Bq/Kg.

4. Physical Requirements

4.1 The minimum bulk density of magnesia partially stabilized zirconia shall be 5.800 g/cm^3 as determined by Test Method C373 as supplied, with the following modifications or by ISO 18754.

[1] This specification is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.13 on Ceramic Materials.

Current edition approved Aug. 1, 2020. Published August 2020. Originally approved in 2004. Last previous edition approved in 2016 as F2393 – 12 (2016). DOI: 10.1520/F2393-12R20.

[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] Available from American Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI 53203, http://www.asq.org.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

4.1.1 Weight determination per sections 3.1 and 5.1 of Test Method C373 and section 7.1 of ISO 18754 shall be made such that it can be calculated and reported to four significant figures.

4.1.2 The calculation of bulk density in section 12.1 of Test Method C373 or section 7.2 of ISO 18754 shall be as follows, to the second decimal place:

$$\rho_b = \frac{m_1}{m_3 - m_2} \times \rho_1 \quad (1)$$

where:

ρ_b = the bulk density, expressed in kilograms per cubic metre,
m_1 = the mass of the dry test specimen, expressed in kilograms,
m_2 = the apparent mass of the immersed test specimen, expressed in kilograms,
m_3 = the mass of the soaked test specimen, expressed in kilograms, and
ρ_1 = the density of the immersion liquid at the temperature of the test, expressed in kilograms per cubic meter.

4.2 The total porosity shall be no greater than 1.0 vol % and open porosity shall be no greater than 0.1 vol % as determined by Test Method C373.

4.3 The microstructure of Mg-PSZ materials consists primarily of submicron tetragonal precipitates that are coherent within a matrix of cubic grains. The calculation of percent monoclinic phase requires the inclusion of the cubic (111) peak that effectively overlaps the tetragonal peak. The monoclinic phase, as determined by this method, shall be 15.0 % or less on a polished surface with surface finish equivalent to 0.05 µm Ra (0.8 µm cutoff) both before and after autoclaving at 150°C for 24 h. Peak intensity of tetragonal and cubic phase (T(101) + C(111) at 2Θ ~ 30.2°, and monoclinic phase (M(-111) and M(111) at 2Θ ~ 28.3° and 2Θ ~ 31.3°, respectively) shall be identified by X-ray diffraction (Cu K_α radiation) analysis to calculate percent of monoclinic phase by the following equation:[5]

$$\%(M) = \frac{I_{M(-111)} + I_{M(111)}}{I_{M(111)} + I_{M(-111)} + \left(I_{C(111)} + I_{T(101)}\right)} \quad (2)$$

where:

I_{xyz} = intensity of the named X-ray diffraction peak,
M = monoclinic phase,
T = tetragonal phase, and
C = cubic phase.

4.4 Grain size shall be determined and reported using Test Methods E112.

5. Mechanical Properties

5.1 The average room temperature flexural strength shall be 600 MPa (87 000 psi) or greater by 4 point bend testing in accordance with Test Method C1161, test configuration B. A minimum of 10 samples are to be tested.

5.2 Weibull modulus value is not considered mandatory for general acceptance and use of this material. It shall be performed when changing suppliers or when the material is produced via a different process. For certain applications, the manufacturer and end user may agree that Weibull modulus testing is mandatory. If Weibull modulus is determined, test results shall be evaluated in accordance with Practice C1239. The minimum number of test specimens shall be 30 and the minimum acceptable uncensored, unbiased Weibull modulus shall be 10.

5.3 The minimum room temperature elastic modulus shall be 180 GPa (26 200 ksi) in accordance with Test Method C1198. A rectangular specimen with dimensions of 60 by 10 by 3 mm is recommended. An acceptable alternative test method for elastic modulus is Test Method C1259.

5.4 The minimum Vickers hardness value shall be 1000 HV in accordance with Test Method C1327. The load shall be 9.8 N (1kg) and the dwell time shall be 15 s.

6. Test Specimen Fabrication

6.1 Specific test specimens shall be prepared from the same batch of material and by the same processes as those employed in fabricating ceramic implant devices.

7. Quality Program Requirements

7.1 The producer shall maintain a quality program, such as those defined in ASQ C1 to maintain quality consistency of test specimens.

8. Keywords

8.1 advanced ceramics; magnesia partially stabilized zirconia; Mg-PSZ; surgical implant; zirconium oxide

[5] Garvie, Ronald C., and Nicholson, Patrick F., "Phase Analysis in Zirconia Systems," *Journal of the American Ceramic Society*, Vol 55, No. 6, 1972, pp. 303–305.