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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Specification for High-Purity Dense Yttria Tetragonal Zirconium Oxide Polycrystal (Y-TZP) for Surgical Implant Applications[1]

This standard is issued under the fixed designation F 1873; 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 yttria tetragonal zirconium oxide polycrystal (Y-TZP) for surgical implant applications.

1.2 The values stated in SI units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

C 373 Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products[2]

C 1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature[3]

C 1198 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Sonic Resonance[3]

C 1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics[3]

C 1259 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramic by Impulse Excitation of Vibration[3]

C 1327 Test Method for Vickers Identation Hardness of Advanced Ceramics[4]

E 112 Test Method for Determining Average Grain Size[4]

F 748 Practice for Selecting Generic Biological Test Methods for Materials and Devices[5]

F 981 Practice for Assessment of Compatibility of Biomaterials for Surgical Implants with respect to Effects of Materials on Muscle and Bone[5]

2.2 *American Society for Quality Control Document:*

C 1 Specification of General Requirements for a Quality Program[6]

3. Chemical Composition

3.1 The chemical composition shall be as follows, measured by X-ray fluorescence (XRF) or mass spectroscopy:

	Weight %
ZrO_2 HfO_2+ Y_2O_3	≥ 99
Y_2O_3	4.5 to 5.4
HfO_2	≤ 5
Al_2O_3	≤ 0.5
Other Total Oxides	≤ 0.5
TiO_2	
SiO_2	
CaO	
Fe_2O_3	
Na_2O	
K_2O	

NOTE 1—The radioactivity, defined as the sum of the massic activity of U^{238}, Ra^{226}, Th^{232} and determined by γ-spectroscopy on the ready to use powder, should be less than 200 Bq/kg. This value will be reviewed at the next revision of this standard and will be based upon the radioactivity data from ceramic implant manufacturers.

4. Physical Properties

4.1 The minimum bulk density of yttrium oxide stabilized zirconium oxide shall be 6.00 g/cm^3 or greater as determined by Test Method C 373 as applied with the following modifications:

4.1.1 Weight determination in 3.1 and 5.1 of Test Method C 373 shall be made to the nearest 0.5 mg.

4.1.2 The calculation of bulk density in 12.1 of Test Method C 373 shall be calculated as follows:

$$B=(D\cdot d)/M-S) \quad (1)$$

where:

B = bulk density, g/cm^3,
D = dry weight, g,
M = saturated weight, g,
S = suspended weight, g, and
d = density of water at the temperature when measurement is taken.

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 C 373.

4.3 *Grain Size*—The mean linear intercept distance shall be 0.6 µm or less, in accordance with Section 10 of Test Method E 112.

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[1] This specification is under the jurisdiction of ASTM Committee F-4 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.13 on Ceramic Materials.

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

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

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

[5] *Annual Book of ASTM Standards*, Vol 13.01.

[6] Available from the American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

4.4 The monoclinic phase shall be 5 % or less on a polished surface with surface finish equivalent to 0.05 µm Ra (0.8 µm cutoff). Peak height of tetragonal phase, T(111) at $2\theta = 30.2°$, and monoclinic phase, M($\bar{1}$11) at $2\theta = 28.2°$ and M(111) at $2\theta = 31.3°$ shall be identified by X-ray diffraction (Copper K Alpha radiation) analysis to calculate percent of monoclinic phase by the following equation (**1**)[7]:

$$\text{monoclinic phase } \% = M(\bar{1}11) + M(111)/M(\bar{1}11) + T(111) + M(111) \quad (2)$$

5. Mechanical Properties

5.1 The average room temperature flexural strength shall be 800 MPa (116 ksi) or greater by four point bend in accordance with Test Method C 1161. A minimum of ten samples (sample size "B") are to be tested.

5.2 A Weibull modulus value is not considered mandatory for general acceptance and use of this material. For certain applications, the manufacturer and end user may agree that Weibull modulus testing is mandatory. If Weibull modulus is tested it shall be tested in accordance with Practice C 1239. The minimum number of specimens tested shall be 30 and the minimum acceptable Weibull modulus shall be 10.

5.3 The minimum room temperature elastic modulus shall be 200 GPa (29×10^6 psi) in accordance with Test Method C 1198. A rectangular specimen with dimensions of 60 mm by 10 by 3 mm is recommended. An acceptable alternative test method for elastic modulus is Test Method C 1259.

5.4 The minimum Vickers hardness value shall be 1200 HV in accordance with Test Method C 1327. The load shall be 1 kg 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 the program defined in ASQC C1 to maintain quality consistency of test specimens.

8. Keywords

8.1 advanced ceramics; surgical implant; Y-TZP; yttria stabilized zirconia; zirconium oxide

[7] The boldface numbers given in parentheses refer to a list of references at the end of the text.

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 The purpose of this specification is to characterize the composition and properties of high purity dense yyttria tetragonal zirconium oxide polycrystal (Y-TZP) to assure consistency in the starting material used in the manufacture of medical devices, in particular surgical implants.

X2. BIOCOMPATIBILITY

X2.1 This specification is needed to ensure a high-quality material for use in biological applications. Yttria stabilized tetragonal zirconia polycrystal (Y-TZP) has been demonstrated **(2, 3)** to exhibit a well-characterized biological response that is equivalent to or better than that exhibited by the reference materials cited and tested in Practice F 981 and Practice F 748 or equivalent. The chemical, physical and mechanical requirements contained in this specification serve as criteria for a high-purity, consistent product that can be implanted in the body. The suitability of this material from a human implant perspective is dependent on the specific application. The biological test appropriate for the specific site, such as recommended in method Practice F 748 should be used as a guideline. Further testing (roughness, static/fatigue strength, aging effects, etc.) may be required for specific applications.

X3. STERILIZATION OF Y-TZP

X3.1 Immersion of Y-TZP in water at elevated temperature and pressure has been shown to cause changes in the material structure and properties **(4)**. These property changes are affected by the processing of the basic material.

X3.2 Post processing, including any type of sterilization, should be cautiously considered only after consultation with the manufacturer of the material or device.

X4. RADIOACTIVITY OF Y-TZP

X4.1 Biomedical materials are known to contain radioactivity. This does not, however mean that these materials pose an unacceptable risk to human health or even that they pose any net risk. Zirconia materials rank well below the dose limit of 1mSv/Year defined for the general public **(5)**. This limit recommended by the International Commission on Radiological Protection (ICRP) is oriented to the average natural dose (1.5 to 3.5 mSv/Year) caused by naturally occurring radionuclides and cosmic radiation.

REFERENCES

(**1**) Garvie, Ronald C. and Nicholson, Patrick S. "Phase Analysis in Zirconia Systems," *J. Amer. Ceram. Soc*., 55 [6], 1972, pp. 303–305.

(**2**) Ohashi, T. et al, "Studies on Mechanical Strength, Tissue Affinity and Cytoxicity of UTZ-30 Zirconia," *The World Biomaterials Congress*, 1988.

(**3**) Christel, P. et al, "Mechanical Properties and Short Term in Vivo Evaluation of Yttrium-Oxide-Partially-Stabilized Zirconia," *J. Biomed. Mater. Res*., Vol 23, Jan. 1989, pp.45-61.

(**4**) Lilley, E., "Review of Low Temperature Degradation in Y-TZPs," in *Corrosion and Corrosive Degradation of Ceramics, Ceramics Transactions*, Vol 10 Ed. R. Tressler and N. McNallan, 1990.

(**5**) Porstendorfer, J., Reineking, A., and Willert, H.G., "Radiation Risk Estimation Based on Activity Measurements of Zirconium Oxide Implants," *Journal of Biomedical Materials Research*, Vol 32, 1996, pp. 663-667.