INTERNATIONAL

~~Designation:F1538–94~~ Designation: F 1538 – 03 (Reapproved 2009)

Standard Specification for Glass and Glass Ceramic Biomaterials for Implantation[1]

This standard is issued under the fixed designation F 1538; 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 the material requirements and characterization techniques for glass and glass-ceramic biomaterials intended for use as bulk porous or powdered surgical implants, or as coatings on surgical devices, but not including drug delivery systems.

1.2 The biological response to glass and glass-ceramic biomaterials in bone and soft tissue has been demonstrated in clinical use (~~**1-9**~~**1-12**)[2] ~~and laboratory studies **10-14**.~~ and laboratory studies (**13-17**).

1.3 This specification excludes synthetic hydroxylapatite, hydroxylapatite coatings, aluminum oxide ceramics, alpha- and beta-tricalcium phosphate, and whitlockite.

1.4 **Warning—**Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website (http://www.epa.gov/mercury/faq.htm) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C 158 ~~Method for Flexural Testing of Glass (Determination of Modulus of Rupture)~~ Test Methods for Strength of Glass by Flexure (Determination of Modulus of Rupture)

C 169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass

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

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

C 633 Test Method for Adhesion or ~~Cohesive~~Cohesion Strength of ~~Flame-Sprayed~~ Thermal Spray Coatings

C 693 Test Method for Density of Glass by Buoyancy

C 729 Test Method for Density of Glass by the Sink-Float Comparator

C 730 ~~Test Method for Knoop Indentation Hardness of Glass~~[3]

~~C958Method for Determination of Particle Size Distribution of Alumina or Quartz by X-Ray Monitoring of Gravity Sedimentation~~[3] Test Method for Knoop Indentation Hardness of Glass

C 958 Test Method for Particle Size Distribution of Alumina or Quartz by X-Ray Monitoring of Gravity Sedimentation

C 1069 ~~Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption~~[3]

~~C1070Test Method for Determining Particle Size Distribution of Alumina or Quartz by Laser Light Scattering~~[3] Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption

C 1070 Test Method for Determining Particle Size Distribution of Alumina or Quartz by Laser Light Scattering

E 228 Test Method for Linear Thermal Expansion of Solid Materials ~~with~~With a ~~Vitreous Silica~~ Push-Rod Dilatometer

F 748 ~~Practice for Selecting Generic Biological Test Methods for Materials and Devices~~ Practice for Selecting Generic Biological Test Methods for Materials and Devices

F 981 Practice for Assessment of Compatibility of Biomaterials for Surgical Implants with Respect to Effect of Materials on Muscle and Bone

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

~~Current edition approved Dec. 15, 1994. Published February 1995.~~
Current edition approved April 1, 2009. Published April 2009. Originally approved in 1994. Last previous edition approved in 2003 as F 1538 – $03^{\epsilon 1}$.

[2] The boldface numbers in parentheses refer to the list of references at the end of this specification.

[3] 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* ~~, Vol 15.02.~~volume information, refer to the standard's Document Summary page on the ASTM website.

2.2 *Code of Federal Regulations:*[4]
Title 21, Part 820
2.3 *United States Pharmacopoeia:*[5]
Lead <252>
Mercury <261>
Arsenic <211>
~~Heavy Metals <231>Method found in Annual Book of ASTM Standards, vol 13.01.~~
~~2.4 *U.S. Geological Survey Method:*[7]~~
~~(7) Cadmium~~
Heavy Metals <231> Method I
2.4 *U.S. Geological Survey Method:*[6]
Cadmium

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *bioactive glass*—~~an amorphous solid that is not intrinsically adhesive and that is capable of forming a cohesive bond with both hard and soft tissue when exposed to appropriate in vivo or in vitro environments, such as simulated body fluid or tris-hydroxymethylaminomethane buffer, by developing a surface layer of hydroxycarbonate apatite by release of ionic species from the bulk material.~~ —an amorphous silicate-based solid that is not intrinsically adhesive and that is capable of forming a cohesive bond with both hard and soft tissue when implanted, and will develop a hydroxycarbonate apatite layer when exposed to appropriate *in vitro* environments, such as simulated body fluid or tris-hydroxymethylaminomethane buffer.

3.1.2 *bioactive glass-ceramic*—~~an amorphous-derived crystalline solid that is not intrinsically adhesive and that is capable of forming a cohesive bond with bone and soft tissue when exposed to appropriate in vivo or in vitro environments, such as simulated body fluid or tris-hydroxymethylaminomethane buffer, by developing a surface layer of hydroxycarbonate apatite by release of ionic species from the bulk material.~~ —an amorphous-derived crystalline silicate-based solid that is not intrinsically adhesive and that is capable of forming a cohesive bond with bone and soft tissue when implanted, and will develop a hydroxycarbonate apatite layer when exposed to appropriate *in vitro* environments, such as simulated body fluid or tris-hydroxymethylaminomethane buffer.

3.1.3 *bulk material*—intended to describe a unit material used as a load bearing implant.

3.1.4 *coating*—intended to describe a surface layer that is relatively thin compared to the overall dimensions of the prosthetic part that has been coated.

3.1.5 *glass biomaterial*—any one of a number of compositions of amorphous inorganic solids that are used as implant materials for various medical or dental uses, or both.

3.1.6 *glass-ceramic biomaterials*—any one of a number of compositions of an amorphous-derived crystalline solid that is used as an implantable biomaterial for medical or dental use, or both.

3.1.7 *particulate material*—intended to describe several pieces (usually small size) used together within an implant construct.

4. Chemical Requirements

4.1 Bulk compositions shall be tested using Test Method C 169.

4.2 The concentration of ~~heavy metals~~trace element levels in the bioactive glass and glass-ceramics shall be limited as follows:

Element	ppm, max
~~As~~	~~3~~
Arsenic (As)	3
~~Cd~~	~~5~~
Cadmium (Cd)	5
~~Hg~~	~~5~~
Mercury (Hg)	5
~~Pb~~	~~30~~
Lead (Pb)	30
total heavy metals (as lead)	50

~~For referee purposes, the methods listed in 2.2 and~~
Either inductively-coupled plasma/mass spectroscopy (ICP/MS) (**18**), atomic absoprtion (AAS), or the methods listed in 2.3 and 2.4 shall be used.

~~[4] Annual Book of ASTM Standards, Vol 02.05.~~
[4] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.
~~[5] Annual Book of ASTM Standards, Vols 03.01 and 14.02.~~
[5] Available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852-1790, http://www.usp.org.
~~[6] Annual Book of ASTM Standards, Vol 13.01.~~
[6] Crock, J.G., Felichte, F.E., Briggs, P.H., "Determination of Elements in National Bureau of Standards Geological Reference Materials SRM 278 Obsidian and SRM 688 Basalt by Inductively Coupled Plasma-Atomic Emission Spectrometry," *Geostandards Newsletter*, Vol 7, 1983, pp. 335–340.

5. Physical Characterization

5.1 The following physical and mechanical characterizations may be applicable to various bioactive glass and glass-ceramics products and should be used whenever possible to verify the material.

5.1.1 *Density*—The densities of glass and glass ceramic materials are related directly to the processing history and composition of the material. The density of the bulk material shall be measured using Test Methods C 373 or C 729and shall be consistent for the specific materials.

NOTE 1—This test should use a non-aqueous liquid for bioactive glass and glass ceramic materials, which are known to react in an aqueous environment and could thereby affect the measurement.

5.1.2 *Flexural Strength*—When used as bulk materials in load bearing applications, the flexural strength of the bulk material shall be measured using Test Methods C 158.

5.1.3 *Young's Modulus*— When used as a bulk material, Young's Modulus of glass and glass ceramic biomaterials shall be determined following Test Method C 623.

5.1.4 *Hardness*—Where applicable, for characterization of the material, the hardness of bulk samples shall be determined using Test Method C 730. The ~~knoop~~Knoop indentation hardness is one of many properties that is used to characterize glasses. Attempts have been made to relate ~~knoop~~Knoop hardness to tensile strength, but no generally accepted methods are available. Such conversion is limited in scope and should be used with caution, except for special cases in which a reliable basis for conversion has been obtained by conversion tests.

5.1.5 *Surface Area*— The surface area of a particulate may be important in determining the reliability of the bioactivity of the material. Whenever the specific surface area of the material relates to function, the surface area of particulate glass and glass ceramic biomaterials shall be measured using Test Method C 1069.

5.1.6 *Bond Strength of Glass or Glass Ceramic Coating*—When used as a coating on a metallic or ceramic substrate, the bond strength of the coating shall be measured following Test Method C 633.

5.1.7 *Crystallinity*— For glass-ceramic biomaterials, the percent crystallinity and crystal phases present in glass ceramic biomaterials shall be determined by means of X-ray diffraction analysis. While there is no single standard method for determining the crystallinity and crystal phases of glass ceramic materials, techniques such as those detailed in ~~reference 10.16~~Refs (**19**) and ~~10.17~~(**20**) should be followed to standardize methods as much as possible.

5.1.8 *Thermal Expansion*—Thermal expansion shall be measured using Test Method E 228, when materials are to be used for coatings (raw materials are to be measured), or on finished product as a quality control test.

5.1.9 *Particle Size*— When used as a particulate, the particle size shall be measured in accordance with Test Methods C 958 or C 1070.

6. Biocompatibility

6.1 Glass and glass-ceramic biomaterials should be evaluated thoroughly for biocompatibility before human use. Bioactive glass and glass-ceramic materials are unique in their mode of action when implanted in the body due to the released ionic species and the mechanisms by which these materials bond with bony tissue. These materials have been found to exhibit an excellent tissue response in laboratory studies (~~10-14~~13-17) and clinical usage (~~1-7~~1-12). Before any new formulations are used clinically, the tissue response should be characterized by the methods recommended in Practice F 748.~~–~~and F 981 as appropriate.

7. Test Specimen Fabrication

7.1 Test specimens should be prepared concurrent with implant devices, as well as from the same batch of material and by the same processes as those used in fabricating the glass and glass-ceramic implant device.

8. Quality Program Requirement

~~8.1The manufacturer shall conform to good manufacturing practices (2.1) or its equivalent.~~

8.1 The manufacturer shall conform to Quality Systems requirements (2.2) or equivalent.

9. Keywords

9.1 bioactive glass; bioactive glass-ceramics; glass biomaterials; glass-ceramic biomaterial; ~~—surgical~~surgical implants

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 A number of glass-ceramic materials are available commercially. Bioactive glass and glass-ceramic materials are available commercially as synthetic graft materials for maintenance of the alveolar ridge; as devices for spinal ~~fushion;~~fusion; as implants for replacement of the vertebral body, iliac crest, and ossicular chain of the middle ear; as bone filler to substitute for bone defects