Designation: F2820 – 12

Standard Specification for Polyetherketoneketone (PEKK) Polymers for Surgical Implant Applications[1]

This standard is issued under the fixed designation F2820; 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 virgin polyetherketoneketone (PEKK) polymer resin as supplied by a vendor (for example, in pellets, powder, and fabricated forms). It provides requirements and associated test methods for these thermoplastics when they are to be used in the manufacture of intracorporeal devices such as surgical implants or components of surgical or dental devices.

1.2 As with any material, some characteristics may be altered by the processing techniques (for example, molding, extrusion, machining, assembly, and sterilization) required for the production of a specific part or device. Therefore, properties of fabricated forms of these polymers should be evaluated using test methods which are appropriate to ensure safety and efficacy as agreed upon by the vendor, purchaser, and regulating bodies. With reduced crystallinity, certain polymers have been shown to be more susceptible to environmental stress cracking. Depending upon the implant application, the end user should characterize the material for environmental stress cracking resistance.

1.3 The properties included in this specification are those applicable for PEKK polymers only. Indicated properties are for fabricated forms. Fabricated forms and materials containing colorants, fillers, processing aids, or other additives, as well as polymer blends which contain PEKK, or reclaimed materials are not covered by this specification.

1.4 This specification is designed to recommend physical, chemical, and biological test methods to establish a reasonable level of confidence concerning the performance of virgin PEKK polymers for use in medical implant devices. The properties listed should be considered in selecting material(s) in accordance with the specific end-use requirements.

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

1.6 When evaluating material in accordance with this specification, hazardous materials, operations, and equipment may be involved. *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

D256 Test Methods for Determining the Izod Pendulum Impact Resistance of Plastics

D570 Test Method for Water Absorption of Plastics

D638 Test Method for Tensile Properties of Plastics

D648 Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position

D695 Test Method for Compressive Properties of Rigid Plastics

D696 Test Method for Coefficient of Linear Thermal Expansion of Plastics Between −30°C and 30°C with a Vitreous Silica Dilatometer

D790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D955 Test Method of Measuring Shrinkage from Mold Dimensions of Thermoplastics

D1238 Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer

D1505 Test Method for Density of Plastics by the Density-Gradient Technique

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

[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 F04.11 on Polymeric Materials.

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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.

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

D3417 Test Method for Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry (DSC) (Withdrawn 2004)[3]
D3418 Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry
D4000 Classification System for Specifying Plastic Materials
F748 Practice for Selecting Generic Biological Test Methods for Materials and Devices

2.2 *ISO Standards:*[4]

ISO 178 Plastics—Determination of Flexural Properties
ISO 180 Plastics—Determination of Izod Impact Strength
ISO 527 Plastics—Determination of Tensile Properties
ISO 604 Plastics—Determination of Compressive Properties
ISO 1183 Plastics—Methods for Determining the Density of Non-cellular Plastics—Part 2: Density Gradient Column Method
ISO 10993 Biological Evaluation of Medical Devices, Parts 1–124
ISO 9001 Quality Systems Management
ISO 13485 Medical devices—Quality Management Systems—Requirements for Regulatory Purposes

2.3 *Other Documents:*[5]

United States Pharmacopeia Vol XXI, or latest edition

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *fabricated forms, n*—those items into which the virgin polymer resin may be converted. These include shapes and forms produced by means of machining, extruding, and compression molding virgin polymer resin into a subsequent entity (for example, fibers, tubes, rods, slabs, sheets, film, or complex shaped parts and devices).

3.1.2 *formulated compound, n*—materials, parts, or devices fabricated from virgin polymer resin in such a way as to contain intentional or unintentional adjuvant substances.

3.1.3 *virgin polymer, n*—the initially-delivered form of the polymer as synthesized from its monomers prior to any processing or fabrication into a medical device. The provided resin is typically in the form of pellets, granules, or powder and is the material from which fibers, tubes, rods, slabs, sheets, films, or specific parts and devices are fabricated.

4. Classification

4.1 The PEKK polymer in the scope of this specification is a pure semi-crystalline copolymer consisting of phenylene rings connected by ether (E) and carbonyl (or ketone, K) groups along the polymer chain (see Appendix X2). Its polymeric structure is defined by the repeating unit EKK. This repeat unit may be of two isomers—one where the K-K linkages are either of 1,4 arrangement (so-called 'Para' or 'T') or of 1,3 arrangement (so-called 'Meta' or 'I'). The ratio of these isomers defines the types of PEKK. The T/I ratio is determined at synthesis and is currently of two types.

4.1.1 *Type I PEKK*—This EKK polymer is made with a T/I ratio of 60/40. The resulting polymer system has a crystal kinetic behavior that makes processing in either amorphous or semi-crystalline forms practical.

4.1.2 *Type II PEKK*—This EKK polymer is made with a T/I ratio of 80/20. The resulting polymer system is used in a semi-crystalline state.

4.2 Types of PEKK plastics, molding and extrusion grades are described in Classification D4000.

5. Properties

5.1 The infrared spectrum[6] of these materials is characteristic of their molecular repeating units. A representative spectrum is listed in Appendix X3. The PEKK polymer shall yield an infrared spectrum, which exhibits major bands only at the wavelengths listed for a standard reference spectrum of that material.

5.1.1 The infrared spectrum, as used in this specification, is to identify the material as polyetherketoneketone (PEKK) but does not necessarily indicate an acceptable degree of material purity.

5.1.2 The presence of additional bands in the sample's infrared spectrum compared to that of the reference material may indicate a different polyaryletherketone (PAEK) material (for example, PEEK, PEKEKK, PEK) or impurities, or both.

5.2 The physical and chemical property requirements for the virgin polymer are listed in Table 1. If additional characteristics are necessary because of a specific application, the procedures

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

[5] Available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852-1790, http://www.usp.org.

[6] Silverstein, R. M., Bassler, G. C., and Morrill, T. C., "Spectroscopic Identification of Organic Compounds," 5th ed., John Wiley & Sons, New York, NY.

TABLE 1 Required Properties[A] of Virgin Polymer Resin

Parameter	Method	Type I	Type II
Glass Transition Temperature, Tg (°C)	DSC[B], 20°K/min, sealed sample, Tg taken on second reheat, D3418	145 – 175	145 – 175
Melt Temperature, Tm (°C)	DSC[B], 20°K/min, sealed sample, Tm taken as max point on reheat endotherm, D3418	285 – 315	345 – 375
Viscosity	As agreed per 5.3	As agreed per 5.3	As agreed per 5.3
Total heavy metals as load, %	US Pharmacopeia, Test 231	<0.1	<0.1

[A] Properties provided by Oxford Performance Materials, Inc.
[B] Differential Scanning Calorimetry

referenced in Section 2 are recommended, or as agreed upon between the vendor and the purchaser.

5.3 The viscosity requirements shall be agreed upon between the vendor and the purchaser

5.4 The chemical, physical, and mechanical properties of fabricated forms are related to the processes utilized in producing the fabricated form (for example, molding, machining, and sterilization). Additionally, the properties necessary for a particular device to perform properly will vary from one device type to another. Table 2 lists some typical properties of non-sterilized fabricated forms.

5.5 Test specimens shall be fabricated (for example, machined, injection molded) from the virgin polymer, or finished part, in such a way as to effectively represent the material characteristics of the non-sterilized finished part.

5.6 Tests and test procedures shall be such as to ensure a high level of control and characterization of the virgin polymer as received from the supplier. The test methods referenced in Section 2 may be appropriate.

5.7 *Quality System Requirements:*

5.7.1 The PEKK virgin polymer resin as described in the scope of this specification should be produced in accordance with an ISO 9001 or ISO 13485 certified Quality Management System.

5.7.2 The PEKK fabricated forms as described in the scope of this standard should be produced in accordance with an ISO 13485 certified Quality Management System.

6. Sampling

6.1 The material should be sampled in accordance with the standard sampling procedures, such as those described in Practice D1898, or other sampling techniques unless otherwise agreed upon between the consumer and the supplier.

7. Biocompatibility

7.1 Biocompatibility of PEKK polymers and implant devices made using these materials shall be determined in accordance with Practice F748 or the ISO 10993 series, unless otherwise agreed upon between the packager and the consumer and regulating bodies.

8. Keywords

8.1 PEKK; polyetherketoneketone

TABLE 2 Typical Properties[A] of Fabricated Forms

PEKK Type I				
Parameter	ISO Methods and Values		ASTM Methods and Values	
Density, kg/m^3	ISO 1183	1270	D1505	1270
Tensile Strength: At Break, MPa	ISO 527, Type 1B,50 mm/min	90	D638, Type I, 5.08 cm/min	90
Tensile Modulus: GPa	ISO 527, Type 1B,50 mm/min	3.4	D638, Type I, 5.08 cm/min	3.4
Percent elongation: at break[B] ,%	ISO 527, Type 1B,50 mm/min	80	D638, Type I, 5.08 cm/min	80
Flexural Strength: MPa	ISO 178	138	D790	138
Flexural Modulus: GPa	ISO 178	3.3	D790	3.3
Compressive Strength: MPa	ISO 604	103	D695	103
Impact Strength: Notched Izod, J/m	ISO 180	6 (kJ/m^2)	D256, 0.254 cm depth, 0.025 cm radius	50 (J/m)

[A] Properties provided by Oxford Performance Materials, Inc
[B] Use an extensometer for measuring strain and calculating percent elongation.

PEKK Type II				
Parameter	ISO Methods and Values		ASTM Methods and Values	
Density, kg/m^3	ISO 1183	1310	D1505	1310
Tensile Strength: At Break, MPa	ISO 527, Type 1B,50 mm/min	110	D638, Type I, 5.08 cm/min	110
Tensile Modulus: GPa	ISO 527, Type 1B,50 mm/min	4.5	D638, Type I, 5.08 cm/min	4.5
Percent elongation: at break[A] ,%	ISO 527, Type 1B,50 mm/min	10	D638, Type I, 5.08 cm/min	10
Flexural Strength: MPa	ISO 178	193	D790	193
Flexural Modulus: GPa	ISO 178	4.6	D790	4.6
Compressive Strength: MPa	ISO 604	207	D695	207
Impact Strength: Notched Izod, J/m	ISO 180	3 (kJK/m^2)	D256, 0.254 cm depth, 0.025 cm radius	50 (Jm)

[A] Use an extensometer for measuring strain and calculating percent elongation.

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 The PEKK polymers may be processed by most techniques available for thermoplastic polymers. Medical devices and components of medical devices made of PEKK polymers may be sterilized. Sterilization methods successfully used include steam, ethylene oxide, and irradiation. Repeated sterilization may weaken parts fabricated of any plastic material. The number of times a given part may be sterilized safely without fear of subsequent failure depends on a number of factors including the molecular weight of the polymer and design, fabrication, intended function, and method of sterilization of the device. Therefore, it is imperative that the manufacturer test the device in order to determine the maximum number of sterilization cycles to which it can be safely subjected.

X1.2 The potential to develop a significant level of crystallinity is an important characteristic of these materials. Performance characteristics are related to the percent crystallinity. Certain additives and processes (for example, excessive cross linking) can limit these materials' ability to crystallize. Therefore, this feature of the polymer and its fabricated form should be evaluated, using appropriate test methods, to ensure efficacy.

X1.3 A formulated compound or fabricated part or device may contain optional adjuvant substances required for the fabrication or intended use of the end product. The biocompatibility of these adjuvant substances, and subsequent formulated compounds, parts, and devices shall be established in accordance with Practice F748 or the ISO 10993 series.

X2. CHEMICAL STRUCTURE OF PEKK

X3. REPRESENTATIVE INFRARED SPECTRA OF PEKK

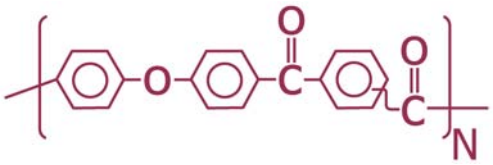

FIG. X2.1 Chemical Structure of PEKK

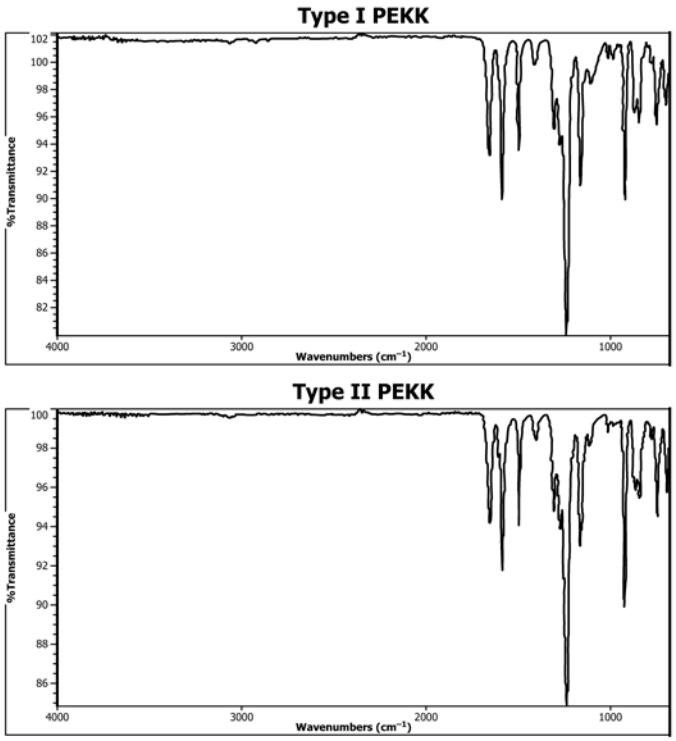

FIG. X3.1 Representative Infrared Spectra of PEKK

RELATED MATERIAL

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