Designation: F3333 – 20

Standard Specification for Chopped Carbon Fiber Reinforced (CFR) Polyetheretherketone (PEEK) Polymers for Surgical Implant Applications[1]

This standard is issued under the fixed designation F3333; 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 chopped carbon-fiber reinforced (CFR) polyetheretherketone (PEEK) polymer in pellets, filaments used in additive manufacturing, or fabricated forms. It provides requirements and associated test methods for these thermoplastic composites 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 The properties included in this specification are those applicable for chopped CFR-PEEK compounds and fabricated forms only. Materials or forms containing colorants, fillers other than carbon fibers, processing aids, or other additives, as well as polymer blends which contain PEEK, or reclaimed materials, are not covered by this specification.

1.2.1 This standard does not include continuous carbon-fiber reinforced PEEK composites, which are fabricated using a different process than chopped CFR-PEEK.

1.2.2 This standard can include CFR-PEEK compounds that are fabricated with the use of coupling (sizing) agents. However, when coupling agents are used to improve wetting of the carbon fibers, the biological risk assessments and biocompatibility testing should consider these coupling agents.

1.3 This specification is designed to recommend physical, chemical, and biological test methods to establish a reasonable level of confidence concerning the performance of CFR-PEEK polymers for use in medical implant devices.

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

1.5 When evaluating material in accordance with this specification, hazardous materials, operations, and equipment may be involved.

1.6 *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.*

1.7 *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]

D256 Test Methods for Determining the Izod Pendulum Impact Resistance 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

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

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

D4000 Classification System for Specifying Plastic Materials

E1994 Practice for Use of Process Oriented AOQL and LTPD Sampling Plans

F748 Practice for Selecting Generic Biological Test Methods for Materials and Devices

F2026 Specification for Polyetheretherketone (PEEK) Polymers for Surgical Implant Applications

[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.

2.2 *ISO Standards:*[3]

ISO 178 Plastics—Determination of Flexural Properties

ISO 180 Plastics—Determination of Izod Impact Strength

ISO 527 Plastics—Determination of Tensile Properties—Part 1: General Principles

ISO 1183-1 Plastics—Methods for Determining the Density of Non-Cellular Plastics—Part 1: Immersion Method, Liquid Pycnometer Method and Titration Method

ISO 10993 Biological Evaluation of Medical Devices, Parts 1–12

ISO 13485 Medical Devices—Quality Management Systems—Requirements for Regulatory Purposes

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *compound, n*—material, part, or device fabricated from a virgin form in such a way as to contain intentional or unintentional adjuvant substances.

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

3.1.3 *virgin form, 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 CFR-PEEK polymer in the scope of this specification is combination of a pure semicrystalline homopolymer consisting of phenylene rings connected by ether (E) and carbonyl (or ketone, K) groups along the polymer chain (see Appendix X1), specified by Specification F2026, and by chopped carbon fibers (see below). The structure of the PEEK polymer is defined by the repeating unit EEK.

4.2 The structure of the chopped carbon fiber is characterized by its route of manufacture (for example, polyacrylonitrile- (PAN-) or Pitch-based), as well as by the median length and diameter of the fibers. The use of sizing agents is permissible provided the resulting CFR-PEEK grade satisfies the biocompatibility requirements of the application (see 7.2).

4.3 The structure of the CFR-PEEK compound is characterized by its volume fraction of fibers.

4.4 Types of PEEK plastics, molding, and extrusion grades are described in Classification D4000.

5. Properties

5.1 The properties listed below shall be considered in selecting material(s) in accordance with the specific end-use requirements.

5.2 The infrared spectrum[4] of the medical-grade PEEK polymer used in the manufacture of these materials is characteristic of their molecular repeating units. A representative spectrum is listed in Appendix X3 of Specification F2026. The PEEK polymer, specified in Specification F2026, shall yield an infrared spectrum which exhibits major bands only at the wavelengths listed for a standard reference spectrum of that material.

5.3 The physical and chemical property requirements for the virgin polymer are listed in Table 1 of Specification F2026. If additional characteristics are necessary because of a specific application, the procedures referenced in Section 2 of Specification F2026 are recommended, or as agreed upon between the vendor and the purchaser.

5.4 The viscosity requirements will vary, depending upon the grade and test method. The method and requirements shall be agreed upon between the vendor and the purchaser.

5.5 The chemical, physical, and mechanical properties of fabricated forms are related to the processes utilized in producing the fabricated form (for example, molding, machining, sterilization, and so forth). Additionally, the properties necessary for a particular device to perform properly will

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

[4] Silverstein, R. M., Bassler, G. C., and Morrill, T. C., *Spectrometric Identification of Organic Compounds, 5th Edition*, John Wiley & Sons, New York, 1991.

TABLE 1 Requirements for Injection-Molded CFR-PEEK Fabricated Forms

Property	Test Method	Requirement	
		Resin Type 1[B] (PAN)	Resin Type 2[B] (Pitch)
Density, kg/m^3	ASTM D792 or D1505; or ISO 1183	1380–1440	1360–1460
Tensile Strength at Break, 23 °C, MPa, min	ASTM D638, Type 1, 5 mm/min or ISO 527, Type 1A, 5 mm/min	180	120
Elongation at Break, %, min[A]	ASTM D638, Type 1, 5 mm/min or ISO 527, Type 1A, 5 mm/min	1.2	1.2
Flexural strength, min, MPa	ASTM D790 or ISO 178	280	180
Flexural modulus, min, GPa	ASTM D790 or ISO 178	16	8
Izod Impact Strength, Notched Izod, min	ISO 180, kJ/m^2 (or ASTM D256 0.254 cm depth, 0.025 cm radius, J/m)	6 (59)	4 ([C])

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

[B] The properties given in Table 1 are for specific grades of CFR-PEEK having approximately 30 % by weight carbon fiber content.

[C] Izod requirements for Type 2 material per Test Method D256 in Table 1 are not available (only the ISO 180 requirements apply).

vary from one device type to another. Table 1 lists the required properties of nonsterilized, injection-molded fabricated forms from commercially available CFR-PEEK compounds.

5.6 Test specimens shall be injection molded in order to meet the requirements listed in Table 1.

5.6.1 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.

5.6.2 Sub-optimal processing could result in porosity which would potentially affect the mechanical performance. A technique such as X-ray or micro-CT with the ability to detect internal pores of 100 µm or smaller should be used.

5.6.3 Processing techniques can result in anisotropy which could potentially affect the mechanical performance, and should be evaluated through mechanical testing appropriate to the application

5.7 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 in Test Methods D648 and D695 may be appropriate.

5.7.1 With reduced crystallinity, certain polymers have been shown to be more susceptible to environmental stress cracking.[5,6] Depending upon the implant application, the end user should evaluate the material for environmental stress cracking resistance.[5,6]

5.8 Extraneous matter and inclusions should be evaluated in fabricated forms using a validated test method agreed upon by the vendor and purchaser in accordance with their respective quality system requirements. Because consolidated PEEK is opaque, optical methods of inspection are limited and cumbersome. X-ray inspection of the entire consolidated form or an equivalent nondestructive inspection technique with the ability to detect >100 µm inclusions or particles of extraneous matter is preferred. The acceptance criteria of the extraneous matter and inclusion inspection shall be agreed upon by the purchaser and vendor, and the results of the inspection shall be documented in the lot inspection report.

6. Sampling

6.1 Where applicable, the requirements of this specification shall be determined for each lot of polymer utilizing sampling sizes and procedures described in Practice E1994, or an equivalent standard.

7. General Requirements

7.1 *Quality System Requirements*—The CFR-PEEK compounds and fabricated forms as described in the scope of this specification should be produced in accordance with an ISO 13485-certified quality management system.

7.2 *Biocompatibility*—CFR-PEEK has been shown to produce a well-characterized level of biological response following long-term clinical use.[7] The results of these studies and the clinical history indicate an acceptable level of biological response in the applications in which the material has been utilized.[8] When new applications or modification to the material or physical forms of the materials are being contemplated, biocompatibility 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. A biological risk assessment shall be determined on a final finished device in accordance with ISO 10993-1. A review article[8] includes an extensive bibliography regarding the biocompatibility of CFR-PEEK biomaterials.

8. Keywords

8.1 CFR; CFR-PEEK; chopped carbon fiber reinforcement; PEEK; polyetheretherketone

[5] Hay, J. N. and Kemmish, D. J., "Environmental Stress Crack Resistance and Absorption of Low-Molecular-Weight Penetrants by Poly(Aryl Ether Ether Ketone)," *Polymer*, Vol 29, 1988, pp. 613–618.

[6] Srivastava, A. P., Depke, N., and Wolf, C. J., "Environmental Stress Deformation of Poly(ether ether ketone)," *J. Applied Polymer Science*, Vol 66, 1997, pp. 725–731.

[7] Kurtz, S. M. and Devine, J. N., "PEEK Biomaterials in Trauma, Orthopedic, and Spinal Implants," *Biomaterials*, Vol 28, No. 32, 2007, pp. 4845–4869.

[8] Green, S., "Compounds and Composite Materials," in: *The PEEK Biomaterials Handbook, 2nd Edition*, S. M. Kurtz (Ed.), William Andrew, Elsevier, Burlington, MA, 2019, pp. 23–48.

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 Medical grade CFR-PEEK compounds may be processed by most techniques available for thermoplastic polymers. Medical devices and components of medical devices made of CFR-PEEK compounds 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 the design, fabrication, intended function, and method of sterilization of the device. Therefore, it is suggested that the manufacturer test the device in order to determine the maximum number of sterilization cycles to which it can be safely subjected.