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Standard Guide for Extensively Irradiation-Crosslinked Ultra-High Molecular Weight Polyethylene Fabricated Forms for Surgical Implant Applications[1]

This standard is issued under the fixed designation F2565; 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 guide covers extensively crosslinked ultra-high molecular weight polyethylene (UHMWPE) materials (fabricated forms) that are produced starting with virgin resin powders and consolidated forms meeting all the requirements of Test Method F648.

1.2 This guide does not cover fabricated forms of ultra-high molecular weight polyethylene which have received only gas plasma, ethylene oxide, or less than 40 kGy ionizing radiation treatments, that is, materials treated only by historical sterilization methods.

1.3 This guide pertains only to UHMWPE materials extensively crosslinked by gamma and electron beam sources of ionizing radiation.

1.4 The specific relationships between these mechanical properties and the *in vivo* performance of a fabricated form have not been determined. While trends are apparent, specific property-polymer structure and polymer-design relationships are not well understood. These mechanical tests are frequently used to evaluate the reproducibility of a fabrication procedure and are applicable for comparative studies of different materials.

1.5 The following precautionary caveat pertains only to the test method portion, Section 5, of this guide. *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]

D638 Test Method for Tensile Properties of Plastics

D695 Test Method for Compressive Properties of Rigid Plastics

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

D2765 Test Methods for Determination of Gel Content and Swell Ratio of Crosslinked Ethylene Plastics

E647 Test Method for Measurement of Fatigue Crack Growth Rates

F619 Practice for Extraction of Medical Plastics

F648 Specification for Ultra-High-Molecular-Weight Polyethylene Powder and Fabricated Form for Surgical Implants

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

F749 Practice for Evaluating Material Extracts by Intracutaneous Injection in the Rabbit

F756 Practice for Assessment of Hemolytic Properties of Materials

F763 Practice for Short-Term Screening of Implant Materials

F813 Practice for Direct Contact Cell Culture Evaluation of Materials for Medical Devices

F895 Test Method for Agar Diffusion Cell Culture Screening for Cytotoxicity

F981 Practice for Assessment of Compatibility of Biomaterials for Surgical Implants with Respect to Effect of Materials on Muscle and Bone

F2003 Practice for Accelerated Aging of Ultra-High Molecular Weight Polyethylene after Gamma Irradiation in Air

[1] This guide 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.

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F2102 Guide for Evaluating the Extent of Oxidation in Ultra-High-Molecular-Weight Polyethylene Fabricated Forms Intended for Surgical Implants
F2183 Test Method for Small Punch Testing of Ultra-High Molecular Weight Polyethylene Used in Surgical Implants
F2214 Test Method for *In Situ* Determination of Network Parameters of Crosslinked Ultra High Molecular Weight Polyethylene (UHMWPE)
F2381 Test Method for Evaluating Trans-Vinylene Yield in Irradiated Ultra-High Molecular Weight Polyethylene Fabricated Forms Intended for Surgical Implants by Infrared Spectroscopy
F2625 Test Method for Measurement of Enthalpy of Fusion, Percent Crystallinity, and Melting Point of Ultra-High-Molecular Weight Polyethylene by Means of Differential Scanning Calorimetry
F2759 Guide for Assessment of the Ultra High Molecular Weight Polyethylene (UHMWPE) Used in Orthopedic and Spinal Devices

2.2 *ISO Standards:*[4]
ISO 10993 Biological Evaluation of Medical Devices, Parts 1-12
ISO 527 Plastics—Determination of Tensile Properties—Part 1: General Principles

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *fabricated form*—any bulk shape of UHMWPE, fabricated from the virgin polymer powder and used during the process of fabricating surgical implants prior to crosslinking, packaging, and sterilization.

3.1.2 *extensively crosslinked UHMWPE*—UHMWPE material that has been subjected to total doses of gamma and/or electron beam ionizing irradiation greater than 40 kGy for the purpose of generating crosslinks within the material.

3.1.3 *ionizing radiation*—gamma or high energy electron radiation.

3.1.4 *crosslinking*—the process by which ionizing radiation produces chemical bonds between two UHMWPE molecules.

4. Sampling

4.1 Where applicable, the requirements of this guide shall be determined for each lot of powder and fabricated form by sampling sizes and procedures according to Practice D1898, or as agreed upon between the purchaser and seller.

5. Extensively Crosslinked UHMWPE Fabricated Form Requirements

5.1 *Compositional Requirements:*

5.1.1 The virgin powder and fabricated forms from which the extensively crosslinked material is manufactured shall meet all the requirements of Practice F648.

5.2 *Physical Requirements:*

TABLE 1 UHMWPE Mechanical and Physical Assessments, Part 1

Test Description	Method
Tensile Strength	D638 or ISO 527
Ultimate	
Yield	
Elongation, %	D638
Izod impact strength, kJ/m^2	F648, Annex A1
Elastic modulus	D638
Compression modulus, MPa	D695
Thermal properties	F2625
Percent crystallinity	
Melting temperature	

5.2.1 The manufacture of an extensively crosslinked UHMWPE material may be accomplished many different ways. Therefore, each manufacturer of such material(s) has developed its own proprietary method(s) for doing so. The end result of this variation is that some of the mechanical properties of extensively crosslinked materials currently used for orthopaedic implant applications exhibit a wide range of values. When this is coupled with the fact that the limiting value for any specific mechanical property necessary for clinical success is yet unknown, a listing of such data for these materials is currently impractical. It is more useful and practical to describe standard methods suitable for characterizing these materials.

5.2.2 *UHMWPE Mechanical and Physical Assessments—Part 1*—The tests shown in Table 1 should be conducted on the extensively crosslinked UHMWPE. Alternative tests may be considered with documented analysis and rationale.

5.2.3 *Mechanical and Physical Assessment—Part 2*—The tests shown in Table 2 should be conducted on the extensively crosslinked UHMWPE. Alternative tests may be considered, such as electron spin resonance (see Appendix X1) with documented analysis and rationale.

5.2.4 *Preclinical Simulation*—Functional testing on the finished UHMWPE component that simulates clinical functions and known failure modes should be considered. Testing that should be considered include creep, accelerated aging, or shelf-life testing, or combinations thereof, functional fatigue loading, and wear as described in Guide F2759. Practice F2003 should be considered for determining relative oxidative stability.

6. Biocompatibility

6.1 This material has been shown to produce a well characterized level of biological response following long term clinical use in humans. 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

TABLE 2 Mechanical and Physical Assessment, Part 2

Test Description	Method
Small punch ultimate load, N	F2183
Fatigue crack propagation	E647
Swell ratio	D2765 or F2214
Oxidation index (OI), surface oxidation index (SOI) and OI Maximum	F2102
t-Vinylene content, trans-vinylene index (TVI)	F2381

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

utilized. When new applications of the material, or modification to the material or physical forms of the materials are being contemplated, the applicable parts of ISO 10993 and Practice F748 should be considered and testing considered as described in Practices F619, F749, F756, F763, F813, and F981 as well as Test Method F895.

7. Keywords

7.1 fabricated forms; powdered form; ultra-high molecular weight polyethylene

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 This guide is intended to describe the minimum set of required test methods that are necessary to fully characterize the physical, chemical, and mechanical behavior of an irradiated, extensively crosslinked UHMWPE material that is intended for use in orthopedic or spine implants.

X1.2 In 1995, the Food and Drug Administration published a guidance document for the characterization of UHMWPE materials (**1**).[5] Since that time, extensively crosslinked materials have been developed, and the 1995 FDA guidance document has been withdrawn, but is available upon request. Therefore, one of the expected uses for the current ASTM guide is to provide guidance to regulatory bodies and orthopedic manufacturers by identifying a standardized set of test methods for characterizing extensively crosslinked UHMWPE materials.

X1.3 While it is currently possible to identify which test methods are necessary for characterizing extensively crosslinked UHMWPE, it remains impractical to assign minimum acceptable values for each test method. For many of these methods, the association between the properties measured and clinical performance is currently unknown. Therefore, it is the responsibility of the manufacturer to develop its own minimum dataset for its process of producing extensively crosslinked UHMWPE, using the test methods in this guide for process validation.

X1.4 Although the test methods listed in Table 1 are intended to be the minimum data set necessary for process validation with an extensively crosslinked UHMWPE material, they are not all intended to be performed routinely during quality control. It is the responsibility of the manufacturer to develop a specification for its material, and to identify which of the test methods listed in Table 1 will be performed routinely for quality control purposes.

X1.5 Fatigue resistance is a desirable property for extensively crosslinked UHMWPE materials. Table 1 lists fatigue crack propagation tests, in accordance with Test Method E647, as the reference test method for this attribute. However, certain linear elastic fracture mechanics principles (for example, the assumption of the plane strain conditions), which underlie the methods of fatigue crack propagation assessment outlined in Test Method E647, are not strictly applicable to ductile polymers, such as UHMWPE, regardless of whether or not the material has been extensively crosslinked. Therefore, the interpretation of fatigue crack propagation test data for UHMWPE is currently limited, because the results of such tests are specimen-geometry specific. Furthermore, there is some debate in the literature about which are the most useful properties to measure for highly crosslinked UHMWPE during fatigue crack propagation testing, such as the exponent during the Paris regime and/or ΔK inception value (**2-4**). If, in the future, a more relevant fatigue characterization test can be identified for UHMWPE, it may be incorporated into Table 1.

X1.6 Similarly, analysis of free radicals is also considered to be important for extensively crosslinked materials. Electron spin resonance spectroscopy is currently considered to be the method of choice for quantifying the concentration and type of free radicals in UHMWPE, however a standard has not yet been developed for this purpose. Until such time as a standard method for measuring free radicals in UHMWPE has been created (at which point Table 1 will be updated), the reader is referred to the literature for details related to this procedure (**5**).

X1.7 Although this guide lists a minimum number of characterization tests for an extensively crosslinked UHMWPE material in Table 1, this guide does not purport to address all of the functional testing that a manufacturer should perform to evaluate the fatigue and wear performance of a particular device. Device testing is recognized to be crucial for UHMWPE implants, but is beyond the scope of this guide.

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

REFERENCES

(1) Food and Drug Administration, "Data Requirements for Ultra-High Molecular Weight Polyethylene (UHMWPE) Used in Orthopedic Devices," Orthopedic Devices Branch, Rockville, MD, March 28, 1995.

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