



Designation: F3160 – 16

Standard Guide for Metallurgical Characterization of Absorbable Metallic Materials for Medical Implants¹

This standard is issued under the fixed designation F3160; 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 guidance document provides metallurgical characterization information that may be beneficial in the evaluation of absorbable metallic materials intended for medical implant applications. This guide is primarily intended for absorbable metallic materials that undergo further processing into a fabricated final device. Therefore, this standard does not require assessments that are more appropriately conducted on the final device, such as biological evaluation. However, a few relevant standards for finished implant devices are included for information purposes.

1.2 The purpose of this guide is to identify appropriate test methods and relevant medical product standards that can be used to develop future standards for new or modified absorbable metallic materials.

1.3 This guide is not intended to cover other major classes of absorbable materials such as polymers, ceramics, composites, and tissue-engineered materials.

1.4 This standard guide is focused on the chemical, physical, microstructural, and mechanical properties plus inspection guidelines for metallic materials that are used for medical implants designed to be absorbed in the body over a period of time. This guide focuses on material characterizations and does not address device specific mechanical testing that may be necessary to determine safety and functionality of the implant.

1.5 Compliance with materials specifications developed in accordance with this standard may not necessarily result in a material suitable for its intended purpose. Additional testing specific to the intended use may be required.

1.6 Since surface modifications of medical implants are generally applied in the latter stages of manufacturing, this standard guide does not cover the characterization of either absorbable or non-absorbable surface coatings that are metallic in origin such as oxides or from the addition of other materials

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 F04.12 on Metallurgical Materials.

Current edition approved April 1, 2016. Published May 2016. DOI: 10.1520/F3160-16.

such as ceramics or polymers. However, this standard does apply to absorbable metallic materials, regardless of the presence or absence of a coating.

1.7 *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

- A262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels
- A342/A342M Test Methods for Permeability of Weakly Magnetic Materials
- A480/A480M Specification for General Requirements for Flat-Rolled Stainless and Heat-Resisting Steel Plate, Sheet, and Strip
- A484/A484M Specification for General Requirements for Stainless Steel Bars, Billets, and Forgings
- A555/A555M Specification for General Requirements for Stainless Steel Wire and Wire Rods
- A751 Test Methods, Practices, and Terminology for Chemical Analysis of Steel Products
- A957 Specification for Investment Castings, Steel and Alloy, Common Requirements, for General Industrial Use
- B69 Specification for Rolled Zinc
- B80 Specification for Magnesium-Alloy Sand Castings
- B86 Specification for Zinc and Zinc-Aluminum (ZA) Alloy Foundry and Die Castings
- B90/B90M Specification for Magnesium-Alloy Sheet and Plate
- B107/B107M Specification for Magnesium-Alloy Extruded Bars, Rods, Profiles, Tubes, and Wire
- B199 Specification for Magnesium-Alloy Permanent Mold Castings
- B403 Specification for Magnesium-Alloy Investment Castings

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.

- B557M** Test Methods for Tension Testing Wrought and Cast Aluminum- and Magnesium-Alloy Products (Metric)
- B661** Practice for Heat Treatment of Magnesium Alloys
- B949** Specification for General Requirements for Zinc and Zinc Alloy Products
- B953** Practice for Sampling Magnesium and Magnesium Alloys for Spectrochemical Analysis
- B954** Test Method for Analysis of Magnesium and Magnesium Alloys by Atomic Emission Spectrometry
- D3648** Practices for the Measurement of Radioactivity
- E8/E8M** Test Methods for Tension Testing of Metallic Materials
- E18** Test Methods for Rockwell Hardness of Metallic Materials
- E29** Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E45** Test Methods for Determining the Inclusion Content of Steel
- E112** Test Methods for Determining Average Grain Size
- E290** Test Methods for Bend Testing of Material for Ductility
- E340** Practice for Macroetching Metals and Alloys
- E354** Test Methods for Chemical Analysis of High-Temperature, Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt Alloys
- E384** Test Method for Microindentation Hardness of Materials
- E407** Practice for Microetching Metals and Alloys
- E536** Test Methods for Chemical Analysis of Zinc and Zinc Alloys
- E1245** Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis
- E1382** Test Methods for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis
- E1441** Guide for Computed Tomography (CT) Imaging
- E2627** Practice for Determining Average Grain Size Using Electron Backscatter Diffraction (EBSD) in Fully Recrystallized Polycrystalline Materials
- F601** Practice for Fluorescent Penetrant Inspection of Metallic Surgical Implants
- F629** Practice for Radiography of Cast Metallic Surgical Implants
- F2052** Test Method for Measurement of Magnetically Induced Displacement Force on Medical Devices in the Magnetic Resonance Environment
- F2119** Test Method for Evaluation of MR Image Artifacts from Passive Implants
- F2182** Test Method for Measurement of Radio Frequency Induced Heating On or Near Passive Implants During Magnetic Resonance Imaging
- F2213** Test Method for Measurement of Magnetically Induced Torque on Medical Devices in the Magnetic Resonance Environment
- F2229** Specification for Wrought, Nitrogen Strengthened 23Manganese-21Chromium-1Molybdenum Low-Nickel Stainless Steel Alloy Bar and Wire for Surgical Implants (UNS S29108)
- F2393** Specification for High-Purity Dense Magnesia Partially Stabilized Zirconia (Mg-PSZ) for Surgical Implant Applications
- F2581** Specification for Wrought Nitrogen Strengthened 11Manganese-17Chromium-3Molybdenum Low-Nickel Stainless Steel Alloy Bar and Wire for Surgical Implants (UNS S29225)
- F2902** Guide for Assessment of Absorbable Polymeric Implants
- IEEE/ASTM SI 10** American National Standard for Metric Practice
- 2.2 *Aerospace Material Standards (AMS)*:³
- AMS 2248** Chemical Check Analysis Limits Corrosion and Heat Resistant Steels and Alloys, Maraging and Other Highly-Alloyed Steels, and Iron Alloys
- AMS 2630** Inspection, Ultrasonic Product Over 0.5 inch (12.7 mm) Thick
- AMS 2632** Ultrasonic Inspection of Thin Materials
- 2.3 *ISO Standards*:⁴
- ISO 404** Steel and Steel Products—General Technical Delivery Requirements
- ISO 643** Steels—Micrographic Determination of the Apparent Grain Size
- ISO 3116** Magnesium and Magnesium Alloys—Wrought Magnesium Alloys—Fourth edition
- ISO 3815-1** Zinc and Zinc Alloys—Part 1: Analysis of Solid Samples by Optical Emission Spectrometry
- ISO 3815-2** Zinc and Zinc Alloys—Part 2: Analysis by Inductively Coupled Optical Emission Spectrometry
- ISO 4967** Steel—Determination of Content of Nonmetallic Inclusions—Micrographic Method Using Standard Diagrams
- ISO 6892-1** Metallic Materials—Tensile Testing—Part 1: Method of Test at Room Temperature
- ISO 10993-15** Biological Evaluation of Medical Devices—Part 15: Identification and Quantification of Degradation Products from Metals and Alloys
- ISO 10993-17** Biological Evaluation of Medical Devices—Part 17: Establishment of Allowable Limits for Leachable Substances
- ISO 10993-18** Biological Evaluation of Medical Devices—Part 18: Chemical Characterization of Materials
- ISO/TS 10993-19-Part 19** Physico-chemical, Morphological and Topographical Characterization of Materials
- ISO 13067** Microbeam Analysis—Electron Backscatter Diffraction—Measurement of Average Grain Size
- ISO 13485** Medical Devices Quality Management Systems—Requirements for Regulatory Purposes—Second edition
- ISO 16220** Magnesium and Magnesium Alloys—Magnesium Alloy Ingots and Castings
- ISO/TS 17137** Cardiovascular Implants and Extracorporeal Systems—Cardiovascular Absorbable Implants

³ Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096, <http://www.sae.org>.

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

ISO 24173 Microbeam Analysis—Guidelines for Orientation Measurement Using Electron Backscatter Diffraction
 ISO/TR 37137 Biological Evaluation of Medical Devices—Guidance for Absorbable Implants
 ISO 9001 Quality Management Systems—Requirements

3. Terminology

3.1 Definitions:

3.1.1 *absorbable, adj—in the body*, an initially distinct foreign material or substance that either directly or through intended degradation can pass through or be metabolized or assimilated by cells and/or tissue. **F2902**

4. Summary of Guide

4.1 Novel absorbable metallic implant materials that do not yet have industry standards are being developed for use in applications such as cardiovascular, orthopedic, general surgical, and diagnostic. The lack of a standard guide for the metallurgical characterization of these new materials may contribute to material property non-uniformity which could create variation in the observed performance of the final product. It is the intent of this guide to provide material property information, corresponding test methods, and relevant medical product standards to serve as a guide for the metallurgical characterization of new or modified materials. These guidelines are tabulated to assist with the development of specifications for new or modified absorbable metallic materials.

4.2 This guide is focused on providing a characterization scheme for materials prior to their fabrication into a finished medical implant. The characterization and testing of absorbable metallic materials after they have been manufactured into finished and sterilized medical implants will depend on the design, mass, shape, anatomic location, and end-use application. <https://standards.iteh.ai/catalog/standards/sist/441bf145-4>

5. Significance and Use

5.1 The metallurgical properties of materials used to manufacture absorbable metallic implants can influence biological reactions and mechanical interaction with soft and hard tissue in the body. This standard guide describes a suggested material characterization scheme for absorbable metallic materials to ensure reproducibility of properties prior to their manufacture into medical implants.

6. Characterization

6.1 Overview:

6.1.1 The metallurgical characterization of absorbable metallic materials includes the influence of chemical, microstructural, physical, and mechanical properties in addition to material inspection methods that are utilized to document initial material quality and uniformity.

6.1.2 This metallurgical characterization guide will focus on magnesium (Mg)-base, iron (Fe)-base, and zinc (Zn)-base alloys since these are the main classes of metallic materials that have been documented in the literature for potential absorbable metallic implant applications. This guide may be applicable to other absorbable alloy systems.

6.1.3 The below described evaluations are general in their scope, with additional unlisted material assessments potentially needed to address the performance requirements of a specific implant application.

6.2 General:

6.2.1 General requirements are specified in **B90/B90M** and **B107/B107M** for wrought Mg-base product forms; **A480/A480M**, **A484/A484M**, and **A555/A555M** for wrought Fe-base product forms; and **B949** for Zn-base product forms.

6.2.2 Requirements are specified in **B403** for Mg-base castings; **A957** for Fe-base castings; and **B86** for Zn-base castings.

6.3 Chemical:

6.3.1 Some examples of absorbable Mg compositions that have been investigated to date include pure Mg, conventional Mg alloys, and experimental Mg alloys containing combinations of aluminum (Al), calcium (Ca), lithium (Li), manganese (Mn), neodymium (Nd), rare earth (RE), silver (Ag), yttrium (Y), zinc (Zn), or zirconium (Zr).

6.3.2 Some examples of absorbable Fe compositions that have been investigated to date include pure Fe, a range of Fe-Mn alloys with and without silicon (Si) or palladium (Pd), and novel binary Fe compositions containing either aluminum (Al), boron (B), carbon (C), cobalt (Co), sulfur (S), or tungsten (W).

6.3.3 Some examples of absorbable Zn compositions that have been investigated to date include pure Zn, commercial Zn alloys, and binary Zn-Mg combinations.

6.3.4 Relevant technical information⁵ has been published for the majority of the absorbable alloy compositions documented in 6.3.1 and 6.3.2.

6.3.5 Elemental concentrations present at specified major, minor, residual, or trace levels are defined in **A751** and ISO 3116. Within this context, a trace classification does not include elements that are a specified component of the alloy, regardless of concentration.

NOTE 1—For the materials covered by this standard, no biocompatibility assessment is required since this standard focuses on characterization of materials that are subject to further fabrication into a final device. However, since the overall composition of any absorbable implant can be expected to have a significant impact on its biocompatibility, the entire composition of materials covered by this standard (including the intended composition, metallic degradation species, and any residual or trace metallic or non-metallic impurities) should be preliminarily assessed for their potential impact on any later ISO 10993 risk assessment of the final device. In addition to the ISO 10993 series, additional supplemental guidance specific for the biological evaluation of absorbable implants can be found in ISO TR37137.

6.3.6 Chemical analysis may be performed according to **E354** for Fe-base materials, **B954** for Mg-base materials, and **E536** for Zn-base materials.

6.3.7 Chemical check analysis tolerance limits specified in AMS 2248 may be used to verify heat analysis of Fe-base materials.

⁵ Hendra Hermawan, Biodegradable Metals from Concept to Applications, Springer, 2012, ISSN 2192-1091; ISSN 2192- 1105 (electronic).

6.3.8 Rare earth (RE) elements⁶ include the fifteen lanthanide elements (atomic number 57 to 71) plus the transition metals scandium (Sc) and yttrium (Y).

6.3.9 Processing aids and binders that may be used with powder metallurgy materials shall be documented in order to evaluate the residual composition of additives that are present after final powder consolidation and heat treatment processes.

6.4 Physical:

6.4.1 Magnetic permeability of metallic implant materials should be assessed to determine the potential for a magnetic response. The magnetic permeability is a function of the final implant's size, dimensions, and placement within the intended application. The possibility of selective dealloying may occur during degradation and periodic magnetic permeability measurements during *in-vitro* absorption testing may be used to determine nonmagnetic retention properties. The magnetic response of low permeability materials may be measured in accordance with **A342/A342M**.

6.4.2 The minerals and any ensuing extraction and refining process should be assessed for the potential presence of radioactivity in the absorbable metallic device. If the assessment determines that a risk of potentially significant radioactive carryover is present, the radioactivity, defined as the sum of the mass activity of U238, Ra226, and Th232, as determined by γ -spectroscopy on the material, should meet the suggested limits of **ASTM F2393**, unless justified otherwise. Radioactivity measurements may be conducted according to **D3648**.

6.5 Microstructural:

6.5.1 Microstructural features will primarily depend on the chemical composition and the level of processing (e.g. whether the material is wrought or cast).

6.5.2 Wrought materials may exhibit a refined microstructure and the grain size should be evaluated and reported according to **E112** as equiaxed, uniform, mixed, or duplex. If analytically beneficial, a more precise grain boundary determination should be considered through utilization of one of the electron backscatter diffraction (EBSD) methods that are described in **ASTM E1382**, **E2627**, and **ISO 13067**, all of which are suited for both cast and wrought alloys.

6.5.3 Casting alloy is usually in the form of shot, bar or ingots and may exhibit lamellar, dendritic, and/or equiaxed microstructural characteristics after melting into castings followed by heat treating.

6.5.4 Metastable martensitic phase transformations such as ($\gamma\text{Fe}, \gamma\text{Mn} \rightarrow \alpha'$) may occur in specific Fe-Mn compositions during cold working and magnetic permeability measurements may be used to verify nonmagnetic stability.

6.5.5 A magnetic surface layer may be formed on high manganese (Mn) containing alloys as a result of thermal oxidation of Mn during hot working or annealing operations and may increase the magnetic permeability. The magnetic surface layer shall be removed from the finished product prior to its use as a medical device (see material standards **F2229** Appendix X1.7 and **F2581** Appendix X1.6).

6.5.6 The presence of nonmetallic inclusion types such as sulfide, alumina, silicate, and globular oxides in the microstructure of Fe-base materials may affect localized corrosion resistance and absorption rate. Such inclusions may be classified according to **E45** and/or **E1245** in order to allow assessment of their potential effect on localized corrosion resistance and absorption rate.

6.5.7 The presence of a cold worked, annealed, or thermally aged metallurgical condition shall be documented, since these factors influence the initial mechanical, physicochemical, and degradation properties.

6.5.8 Metallographic etching methods, such as **E407** or **E340**, may be used to detect decarburization (superficial carbon oxidation) due to high-temperature thermal processing in order to evaluate compositional uniformity and the reduction in initial surface hardness.

6.5.9 The presence of a carbide network in the microstructure may affect the localized absorption rate. The carbide network can be evaluated according to **A262**.

6.5.10 Other unique microstructures obtained by nanoprocessing, metal injection molding, powder consolidation, hot isostatic pressing, or additive manufacturing methods, may influence the overall absorption rate and should be specified accordingly.

6.6 Mechanical:

6.6.1 Tensile properties of bar, wire, sheet, strip, plate, tubing or forged wrought material and castings produced from casting alloy may be determined according to **ASTM E8/E8M**, **ASTM B557M**, or **ISO 6892-1** in order to determine initial mechanical properties.

6.6.2 Hardness testing according to **E18** or **E384** may be used to characterize initial wear resistance or abrasion properties.

6.6.3 Bend mandrel diameter testing of sheet, strip, and tubing materials according to **E290** may be useful to verify that materials intended for medical device applications have satisfactory formability for fabrication into surgical implants.

6.7 Inspection:

6.7.1 Visual inspection of wrought materials may be used to characterize surface discontinuities and surface finish.

6.7.2 Visual inspection of castings may be used to characterize the presence of surface discontinuities, scale, casting shell, and weld repair.

6.7.3 Fluorescent penetrant inspection (FPI) of castings according to **F601** may be used to evaluate surface and sub-surface discontinuities.

6.7.4 Radiographic evaluation of castings according to **F629** may be used to evaluate internal porosity and differential density indications. Computed Tomography (CT) imaging in accordance with the methodology described in **E1441** may also be considered as an alternative means to evaluate internal porosity.

6.7.5 Contact and immersion ultrasonic examination of wrought materials according to **AMS 2630** and **AMS 2632** may be used to characterize internal porosity and sub-surface discontinuities.

6.7.6 It is recognized that absorbable metallic materials may be intentionally designed to provide a level of surface or

⁶ Rare Earth Technology Alliance & American Chemical Council, *The Economic Benefits of the North American Rare Earth Industry*, Report, April 2, 2014.