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Standard Specification for Metal Injection Molded Unalloyed Titanium Components for Surgical Implant Applications[1]

This standard is issued under the fixed designation F2989; 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 chemical, mechanical, and metallurgical requirements for three grades of metal injection molded (MIM) unalloyed titanium components in two types to be used in the manufacture of surgical implants.

1.2 The Type 1 MIM components covered by this specification may have been densified beyond their as-sintered density by post-sinter processing.

1.3 Values in either inch-pound or SI are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore each system shall be used independent of the other. Combining values from the two systems may result in non-conformance with the specification.

1.4 *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]

B243 Terminology of Powder Metallurgy

B311 Test Method for Density of Powder Metallurgy (PM) Materials Containing Less Than Two Percent Porosity

B923 Test Method for Metal Powder Skeletal Density by Helium or Nitrogen Pycnometry

E3 Guide for Preparation of Metallographic Specimens

E8/E8M Test Methods for Tension Testing of Metallic Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E165 Practice for Liquid Penetrant Examination for General Industry

E407 Practice for Microetching Metals and Alloys

E539 Test Method for Analysis of Titanium Alloys by X-Ray Fluorescence Spectrometry

E1409 Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique

E1447 Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by Inert Gas Fusion Thermal Conductivity/Infrared Detection Method

E1941 Test Method for Determination of Carbon in Refractory and Reactive Metals and Their Alloys by Combustion Analysis

E2371 Test Method for Analysis of Titanium and Titanium Alloys by Atomic Emission Plasma Spectrometry (Withdrawn 2013)[3]

E2626 Guide for Spectrometric Analysis of Reactive and Refractory Metals

F67 Specification for Unalloyed Titanium, for Surgical Implant Applications (UNS R50250, UNS R50400, UNS R50550, UNS R50700)

F601 Practice for Fluorescent Penetrant Inspection of Metallic Surgical Implants

F629 Practice for Radiography of Cast Metallic Surgical Implants

SI 10 American National Standard for Use of the International System of Units (SI): The Modern Metric System

2.2 *ISO Standards:*[4]

ISO 5832-3 Implants for Surgery—Metallic Materials—Part 3: Wrought Titanium 6-Aluminum 4-Vanadium Alloy

ISO 6892 Metallic Materials—Tensile Testing at Ambient Temperature

ISO 9001 Quality Management Systems—Requirements

2.3 *Aerospace Material Specifications:*[5]

AMS 2249 Chemical Check Analysis Limits, Titanium and Titanium Alloys

[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 F04.12 on Metallurgical 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.

[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 SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://aerospace.sae.org.

2.4 *MPIF Standards:*[6]
Standard 10 Determination of the Tensile Properties of Powder Metallurgy Materials
Standard 42 Determination of Density of Compacted or Sintered Powder Metallurgy Product
Standard 50 Preparing and Evaluating Metal Injection Molded Sintered/Heat Treated Tension Specimens
Standard 63 Density Determinations of MIM Components (Gas Pycnometry)
Standard 64 Terms Used in Metal Injection Molding

3. Terminology

3.1 Definitions of powder metallurgy and MIM terms can be found in Terminology B243 and MPIF Standard 64. Additional descriptive information is available in the Related Material Section of Vol. 02.05 of the *Annual Book of ASTM Standards*.

3.2 The materials produced by means of the metal injection molding process are designated by the prefix, "MIM", followed by the appropriate designation for the alloy grade. The MIM designates that it was made by metal injection molding.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *absolute density, n*—the value of density used to characterize a powder material with a particular chemical composition as if it were a fully dense material, completely free of porosity.

3.3.1.1 *Discussion*—For the purposes of this specification, the skeletal density (also referred to as pycnometer density) measured on the raw material powders using the pycnometry method of Test Method B923 shall be used to represent the absolute density of the particular chemical composition.

3.3.2 *debinding, v*—a step between molding and sintering where the majority of the binder used in molding is extracted by heat, solvent, a catalyst, or other techniques.

3.3.3 *feedstock, n—in metal injection molding*, a moldable mixture of metal powder and binder.

3.3.4 *feedstock batch, n*—a specified quantity of feedstock made up of the same lot of metallic powders and the same lot of binder materials mixed under the same conditions at essentially the same time.

3.3.5 *lot, n*—a specified quantity of components made up of the same batch of feedstock, debound, sintered, and post-processed under the same conditions at essentially the same time.

3.3.6 *metal injection molded component, n*—product fabricated by a metal injection molding process consisting of mixing metal powders with binders to make a feedstock, introducing this feedstock into a mold by injection or other means, debinding to remove the binders, and sintering.

3.3.7 *near net component, n*—a component that meets dimensional tolerance as built with little post processing.

3.3.8 *net component, n*—a component that meets dimensional tolerance as built with no post processing.

3.3.9 *pre-alloyed powder, n*—powder composed of two or more elements that are alloyed in the powder manufacturing process in which the particles are of the same nominal composition throughout.

3.3.10 *relative density, n*—the density ratio, often expressed as a percentage, of the density of a porous material to the absolute density of the same material, completely free of porosity.

3.3.11 *sintering, v*—the metallurgical bonding of particles in a MIM component resulting from a thermal treatment at a temperature below the melting point of the main constituent.

3.3.12 *Type 1, n*—a MIM component that may have been desified beyond its as-sintered density by post-sinter processing.

3.3.13 *Type 2, n*—a MIM component that shows the as-sintered density and was not densified after sintering.

4. Ordering Information

4.1 Include with inquiries and orders for material under this specification the following information:
4.1.1 Quantity,
4.1.2 ASTM specification and date of issue,
4.1.3 Grade (MIM 1, MIM 2 or MIM 3),
4.1.4 Type (1 or 2),
4.1.5 Units to be certified—SI or Inch-Pounds,
4.1.6 Component configuration (engineering drawing or 3D solid model, or both) and dimensional requirements,
4.1.7 Condition (5.2),
4.1.8 Mechanical properties (if applicable),
4.1.9 Finish (5.2),
4.1.10 Special tests (9, 10 and 11), if any, and
4.1.11 Other requirements.

5. Materials and Manufacture

5.1 Components conforming to this specification shall be produced by the metal injection molding process using unalloyed metal powders with major elemental composition meeting the chemical requirements of Table 1.

5.2 Post-sintering operations may be employed to achieve the desired density, shape, size, surface finish, or other component properties. The post-sintering operations shall be agreed upon between the supplier and purchaser.

5.3 The condition and finish of the components shall be agreed upon between the supplier and purchaser.

TABLE 1 Chemical Composition

	Composition for both Type 1 and Type 2 Composition, % (mass/mass)		
Element	Grade MIM 1	Grade MIM 2	Grade MIM 3
Nitrogen, max	0.03	0.03	0.05
Carbon, max	0.08	0.08	0.08
Hydrogen, max	0.015	0.015	0.015
Iron, max	0.20	0.30	0.30
Oxygen, max	0.18	0.25	0.30
Titanium	Balance	Balance	Balance

[6] Available from Metal Powder Industries Federation (MPIF), 105 College Rd. East, Princeton, NJ 08540, http://www.mpif.org.

6. Chemical Requirements

6.1 The components supplied under this specification shall conform to the chemical requirements in Table 1. The supplier shall not ship components with chemistry outside the requirements specified in Table 1.

6.2 Chemical analysis of the finished component or a representative sample shall be used for reporting all chemical requirements. Any representative sample shall be produced from the same feedstock batch, debound, sintered, and post processed concurrently with the finished components that it represents.

6.2.1 Requirements for the major and minor elemental constituents are listed in Table 1. Also listed are important residual elements. The percentage of titanium is determined by difference and need not be determined or certified.

6.2.2 Intentional elemental additions other than those specified in Table 1 are not permitted.

6.2.3 Analysis for elements not listed in Table 1 is not required to verify compliance with this specification.

6.3 *Product Analysis:*

6.3.1 Product analysis tolerances do not broaden the specified heat analysis requirements but cover variations in the measurement of chemical content between laboratories. The product analysis tolerances shall conform to the product tolerances in Table 2.

6.3.2 The product analysis is either for the purpose of verifying the composition of the manufacturing lot or to determine variations in the composition within the lot. Acceptance or rejection of the manufacturing lot of components may be made by the purchaser on the basis of this product analyses.

6.3.3 Samples for chemical analysis shall be representative of the component being tested. The utmost care shall be used in sampling titanium for chemical analysis because of its affinity for elements such as oxygen, nitrogen, and hydrogen. In cutting samples for analysis, therefore, the operation should be carried out insofar as possible in a dust-free atmosphere. Cutting tools should be clean and sharp. Samples for analysis should be stored in suitable containers.

6.3.4 Product analysis outside the tolerance limits allowed in Table 2 is cause for rejection of the product. A referee analysis may be used if agreed upon by the supplier and purchaser.

TABLE 2 Product Analysis Tolerance[A]

Element	Limit or Maximum of Specified Range %, (mass/mass)	Tolerance Under the Minimum or Over the Maximum Limit[B]
Nitrogen	up to 0.05	0.02
Carbon	0.10	0.02
Hydrogen	up to 0.015	0.002
Iron	up to 0.25	0.10
Iron	over 0.25	0.15
Oxygen	up to 0.20	0.02
Oxygen	over 0.20	0.03

[A] See AMS 2249.
[B] Under the minimum limit is not applicable for elements where only a maximum percentage is indicated.

6.3.5 For referee purposes, use Test Methods E539, E1409, E1447, E1941, and E2371 and Guide E2626 or other analytical methods agreed upon between the purchaser and the supplier.

7. Mechanical Requirements

7.1 *Tensile Properties:*

7.1.1 The components supplied under this specification shall conform to the mechanical property requirements in Table 3.

7.1.2 Test specimens shall be taken from a MIM component if possible, or from a representative sample or molded tensile specimen. A representative sample or molded tensile specimen may only be used only if the component configuration is such that a tensile specimen cannot be obtained from the component.

7.1.3 The number of tensile tests should be agreed upon between the supplier and the purchaser.

7.2 Representative samples or molded tensile specimens shall be produced from the same feedstock batch, debound, sintered and post processed concurrently with the finished components that they represent.

7.2.1 Specimens machined from components or representative samples shall be ground, or machined to final dimensions in accordance Test Methods E8/E8M.

7.2.2 Alternate tensile specimen geometries may be agreed upon between the purchaser and supplier. Some examples of the configurations for molded tensile specimens are described in MPIF Standards 10 and 50.

7.3 Specimens for tensile tests shall be tested in accordance with Test Methods E8/E8M. Tensile properties shall be determined using a strain rate of 0.076 to 0.178 mm/mm/min [0.003 to 0.007 in./in./min] through yield and then the crosshead speed may be increased so as to produce fracture in approximately one additional minute.

7.4 Should any test piece not meet the specified requirements, test two additional representative test pieces, in the same manner, for each failed test piece. The lot shall be considered in compliance only if all additional test pieces meet the specified requirements.

7.5 Tensile test results for which any specimen fractures outside the gauge length shall be considered valid if both the elongation and reduction of area meet the minimum requirements specified. If either the elongation or reduction of area is less than the minimum requirement, invalidate the specimen and retest. Retest one specimen for each invalidated specimen.

8. Dimensions and Permissible Variation

8.1 *Units of Measure:*

8.1.1 *Selection*—This specification requires that the purchaser selects the units (SI or inch-pound) to be used for product certification. In the absence of a stated selection of units on the purchase order, this selection may be expressed by the purchaser in several alternate forms listed in order of precedence.

8.1.2 If the purchaser and supplier have a history of using specific units, these units shall continue to be certified until expressly changed by the purchaser.

TABLE 3 Mechanical Requirements

	Grade MIM 1		Grade MIM 2		Grade MIM 3	
	Type 1 Densified	Type 2 Sintered	Type 1 Densified	Type 2 Sintered	Type 1 Densified	Type 2 Sintered
Ultimate Tensile Strength	405 MPa min [58 750 psi]	370 MPa min [53 650 psi]	460 MPa min [66 500 psi]	420 MPa min [61 000 psi]	545 MPa min [79 000 psi]	495 MPa min [71 800 psi]
Yield Strength (0.2 % offset)	350 MPa min [50 750 psi]	315 MPa min [45 700 psi]	380 MPa min [55 100 psi]	360 MPa min [52 200 psi]	430 MPa min [62 350 psi]	390 MPa min [56 500 psi]
Elongation[A]	24 % min	23 % min	18 % min	17 % min	12 % min	10 % min
Reduction of Area	25 % min	25 % min	20 % min	20 % min	15 % min	15 % min

[A] Elongation of material 1.575 mm [0.062 in.] or greater in diameter (D) or width (W) shall be measured using a gauge length of 2 in. or 4D or 4W. The gauge length shall be reported with the test results. The method for determining elongation of material under 1.575 mm [0.062 in.] in diameter or thickness may be negotiated. Alternately, a gauge length corresponding to ISO 6892 (5.65 times the square root of S_o, where S_o is the original cross-sectional area.) may be used when agreed upon between the supplier and purchaser.

8.1.3 In the absence of historic precedence, if the units used to define the product on the purchaser's purchase order, specification, and engineering drawing are consistent, these units shall be used by the supplier for product certification.

8.1.4 If the purchaser's selection of units is unclear, the units of measure shall be agreed upon between the purchaser and supplier.

8.1.5 *Conversion of Units*—If the supplier's test equipment does not report in the selected units, the test equipment units may be converted to the selected units for certification purposes. Accurate arithmetic conversion and proper use of significant digits should be observed when performing this conversion. ASTM SI 10 provides guidelines for the use of SI units. Annex A provides conversion tables and Annex B provides rules for conversion and significant digits.

9. Microstructure

9.1 Alpha case is not permitted on net components when examined on a metallurgical cross section at 100× magnification.

9.2 The alpha case requirement on near net components shall be agreed upon between supplier and purchaser.

9.3 The microstructural requirements and frequency of examinations shall be mutually agreed upon by the supplier and purchaser. Specimen preparation shall be in accordance with Guide E3 and Practice E407.

10. Density

10.1 The relative density of the finished component shall be a minimum of:

10.1.1 *Type 1*—98 % of the absolute density of the prealloyed metal powder lot used to make the component.

10.1.2 *Type 2*—96 % of the absolute density of the prealloyed metal powder lot used to make the component.

10.2 The density of the finished component shall be measured per Test Method B311, MPIF Standard 42, or MPIF Standard 63.

10.3 The absolute density of the prealloyed metal powder shall be measured in accordance with Test Method B923.

10.4 The component measured density shall be reported on the test report in units of g/cm^3. The component relative density shall be reported as a percent of the absolute density of the prealloyed metal powder lot used to make the component.

11. Nondestructive Examination

11.1 *Fluorescent Penetrant Examination*—When required by the purchaser, each individual component shall be subject to fluorescent penetrant examination in accordance with Practice E165 or F601, as appropriate for the surface condition of the component being tested. Acceptance criteria and a sampling plan other than 100 % inspection shall be agreed upon between the supplier and purchaser.

11.2 *Radiographic Examination*—When required by the purchaser, each individual component shall be subject to radiographic examination in accordance with Practice F629. Acceptance criteria and a sampling plan other than 100% inspection shall be agreed upon between the supplier and purchaser.

11.3 Other methods of nondestructive inspection may be used as agreed upon by the supplier and purchaser.

12. Significance of Numerical Limits

12.1 The following applies to all specified numerical limits in this specification. To determine conformance to these limits, an observed or calculated value shall be rounded to the nearest unit in the last right hand digit used in expressing the specification limit, in accordance with the Rounding Method of Practice E29.

13. Certification

13.1 The supplier shall provide a certification that the components were tested in accordance with this specification and met all requirements. A report of the test results shall be furnished to the purchaser at the time of shipment.

14. Quality Program Requirements

14.1 The supplier shall maintain a quality program as defined in ISO 9001, or similar quality program.

15. Keywords

15.1 metal injection molded components; metals (for surgical implants); orthopedic medical devices; titanium alloys; titanium components (for surgical implants)

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 Purpose

X1.1.1 The purpose of this specification is to characterize the chemical, physical and mechanical properties of metal injection molded, unalloyed titanium components to be used in the manufacture of surgical implants.

X1.2 Chemistry, Process History and Mechanical Properties

X1.2.1 The chemical composition requirements in this specification for MIM unalloyed titanium components is the same as Specification F67 for wrought titanium, except the maximum oxygen level of Grade MIM 3.

X1.2.2 The choice of composition and mechanical properties is dependent upon the design and application of the medical component.

X1.3 Fatigue

X1.3.1 It is recommended that users evaluate fatigue properties for MIM components that experience dynamic loads in service.

X1.4 Binders

X1.4.1 The binders mixed with the metal powders to make the MIM feedstock are almost completely removed from the molded component during the debinding step(s) that occur prior to sintering. Any residual binder materials are decomposed to their elemental constituents during the sintering cycle. The effect of the binders on the chemical composition of the MIM components is controlled through the chemical requirements in Table 1.

X1.5 Units of Measure

X1.5.1 *ASTM Policy*—ASTM is promoting the use of rationalized SI (metric) units in their standards. The F04.12 Committee has written this specification to facilitate the transition by the medical materials industry to SI units of measure between now and 2018. In the first phase of this transition, running to 2013, the specifications will be structured to allow the use of either SI or inch-pound units. The choice of primary units in each specification will be determined by the industry using the specification. The change to SI units during this period may be initiated by the purchaser through his purchase documentation. In the second phase of this transition the specifications will be written with SI as the primary units. Harmonization with corresponding ISO documents will be considered when assigning the SI values.

X2. BIOCOMPATIBILITY

X2.1 The alloy composition covered by this specification has a long history of successful clinical application in soft tissue and bone implants in humans, with a well-characterized level of biological response.

X2.2 No known surgical implant material has ever been shown to be completely free from adverse reactions in the human body. Long-term clinical experience of the use of the material referred to in this specification, however, has shown that an acceptable level of biological response can be expected, if the material is used in appropriate applications.