INCH POUND

MSFC-SPEC-3706
REVISION A
EFFECTIVE DATE: December 10, 2020

National Aeronautics and
Space Administration

George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

EM30

MSFC TECHNICAL STANDARD

SPECIFICATION FOR 60Ni-40Ti BILLETS

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DOCUMENT HISTORY LOG

Status (Baseline/ Revision/ Canceled)	Document Revision	Effective Date	Description
Baseline	-	04/22/2016	Initial Release.
Revision	A	12/10/2020	Updated sections 6.4 Corrosion Corrosion characterization in various environments is described in NASA/CR-2016-218220, prepared for Marshall Space Flight Center under Contract NNM12AA41C, “Electrochemical, Polarization, Crevice Corrosion Testing of Nitinol 60, A Supplement to the ECLSS Sustaining Materials Compatibility Study”. R.E. Lee, Jacobs ESSSA Group, Huntsville, Alabama. This report is located on NASA Technical Reports Server (NTRS), which contains Scientific and Technical Information (STI). 6.5 Stress Corrosion Cracking (SCC) Resistance SCC resistance characterization is described in NASA/TM-2016-218230, “Stress Corrosion Evaluation of Nitinol 60 for the International Space Station Water Recycling System”. P.D. Torres, Marshall Space Flight Center, Huntsville, Alabama. This Technical Memorandum (TM) is located on NASA Technical Reports Server (NTRS), which contains Scientific and Technical Information (STI).

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1. SCOPE

This specification establishes the chemical composition, heat treatment, hardness, and tensile requirements and their verification of consolidated Nickel (Ni) Titanium (Ti) powdered metal billets made by the Hot Isostatic Pressing (HIP) method.

This standard applies the following: All mandatory actions (i.e., requirements) are denoted by statements containing the term, “shall.” The terms: “may” or “can” denote discretionary privilege or permission; “should” denotes a good practice and is recommended, but not required; “will” denotes an expected outcome; and “are/is” denotes descriptive material.

2. APPLICABLE DOCUMENTS

Unless otherwise noted the latest revision of the following documents are applicable to the extent specified herein. In the event of a conflict between the documents referenced herein and the contents of this specification, the contents of this specification shall take precedence. The contractor may pursue substituting equivalent specifications and documents to the ones identified herein as long as the substitution does not compromise the intent of the specifications and documents identified herein and is approved by NASA/MSFC before implementation.

2.1 Non-Government Publications

ASTM E3, Standard Guide for Preparation of Metallographic Specimens

ASTM E8, Standard Test Methods for Tension Testing of Metallic Materials

ASTM E18, Standard Test Methods for Rockwell Hardness of Metallic Materials

ASTM E1097, Standard Guide for Determination of Various Elements by Direct Current Plasma Emission Spectrometry

ASTM E1409, Standard Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by Inert Gas Fusion

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

ASTM E2465, Standard Test Methods for Analysis of Ni-Base Alloys by Wavelength Dispersive X-Ray Fluorescence Spectrometry

ASTM E2594, Standard Test Method for Analysis of Nickel Alloys by Inductively Coupled Plasma Atomic Emission Spectrometry

CGA G-11.1, Compressed Gas Association Commodity Specification for Argon

3. REQUIREMENTS

This specification is applicable for powdered metal billets consolidated using the preferred method of HIP. The billets are expected to possess the following composition, characteristics, and properties.

3.1 Chemical Composition

Chemical composition shall conform to the percentages by weight shown in Table I and be homogeneous throughout the billet.

TABLE I. Chemical Composition

Element	Min	Max
Titanium	39.0	41.0
Carbon	0	0.05
Oxygen	0	0.08
Nickel	balance	balance

No more than 0.2 weight per cent total of tramp element contamination shall be in the composition. Tramp elements are any other elements other than nickel and titanium. Some, but not all, examples of tramp elements include oxygen, carbon, iron, cobalt, chromium, aluminum, and nitrogen.

3.2 Heat Treatment

Classification Types (see 6.3)

Type I – Fully hardened condition (see 3.2.4)

Type II – Annealed condition (see 3.2.5)

3.2.1 Billet Sectioning and Section Identification

Each billet shall be sectioned prior to heat treatment. Billet sections shall be identified with a unique identifier relating back to the billet. Section thickness shall be one inch (± 0.1) or less to obtain complete through hardness. Sections with thickness greater than one inch (± 0.1) may experience incomplete through hardness.

3.2.2 Argon Gas Purge

The heat treat furnace shall be purged with Argon after loading material and prior to heat treatment to remove moisture and the ambient atmosphere.

3.2.3 Argon Gas Moisture Content

Argon gas shall be dry, as per CGA G-11.1, quality verification level (QVL) C or QVL D.

3.2.4 Fully Hardened Heat Treatment

Billets and/or sections shall be heated to 1,832˚F (± 25˚) in Argon for a minimum of two hours (±15 minutes), and then immediately quenched in room temperature water. Tap water is an acceptable quench medium. Billet or billet section orientation is not important during quench insertion.

3.2.5 Anneal Heat Treatment

Billets and/or billet sections shall be annealed at 1,922 ˚F (± 25˚) in Argon for a minimum of two hours (±15 minutes) and furnace cooled to ambient temperature. The Argon atmosphere shall be maintained during furnace cool down.

3.2.6 Post Fully Hardened and Annealed Heat Treatment Microstructures

When fully hardened, one microstructure from each billet and/or billet section shall be recorded. When annealed, one microstructure from each billet and/or billet section shall be recorded. The location of the microstructure relative to the billet and/or billet section shall be recorded. All microstructure metallographic specimens shall be prepared per ASTM E3 and etched with an aqueous solution of 1 volume % concentrated HF + 10 volume % concentrated HNO_3 in distilled water.

3.2.7 Hardness

Billet and/or billet section hardness readings shall be taken at three locations, three readings per location shall be taken from perimeter to the center. Hardness shall be within (± 1 HRC) across the cross section. The location of the section relative to the billet shall be recorded. For the fully hardened condition, readings shall be a minimum of HRC 58. For the annealed condition, readings shall be in the range of 32 to 36 HRC.

3.2.8 Heat Treatment Documentation

All heat treatment process details shall be documented and furnished to the purchaser upon request.

3.3 Mechanical Testing

3.3.1 Fully Hardened Tensile Strength

Ultimate tensile strength in any direction shall be a minimum of 146 ksi tested per ASTM E8 after fully hardened heat treatment per 3.2.4.

3.3.2 Mechanical Test Specimen Labeling and Preservation

Each test specimen shall be provided an identifying heat treatment condition label and will also correspond to each of the test results, and packaged to protect and prevent damage to the fracture surfaces for future failure analysis. Mechanical testing results of 3.3.1 and the test specimens corresponding to the stress/strain curves shall be provided to the purchaser.

3.4 Non-destructive Evaluation (NDE)

NDE shall be as specified on the purchase order.

4. VERIFICATION

4.1 Chemical Composition Testing

a. The billet alloying element(s) shall be determined either by Direct Current Plasma (DCP) Emission Spectrometry Analysis according to ASTM E1097, and/or Inductively-Coupled Plasma (ICP) Optical Emission Spectrometers according to ASTM E2594, and/or X-Ray Fluorescence Spectrometry according to ASTM E2465, and/or equivalent best method(s).
b. Carbon shall be measured by combustion per ASTM E1941, or by equivalent best method.
c. Oxygen and nitrogen shall be measured by inert gas fusion per ASTM E1409 or equivalent best method.
d. Certification documents shall be provided by the vendor that verifies the chemical composition and how it was derived.

4.2 Heat Treatment Verification

4.2.1 Billet Section Thickness and Identification

a. Each billet section thickness and its location relative to the billet shall be recorded in the heat treatment process control sheets.
b. Billet section thickness greater than one inch (±0.1) shall be cause for rejection, unless the purchase order requires thicker sections and specifies that the complete through hardness is not required.
c. Heat treatment process control sheets shall record when complete through hardness is not required as per the purchase order.

4.2.2 Argon Gas Purge Control

The Argon gas purge of the furnace shall be recorded in the heat treatment process control sheet.

4.2.3 Argon Gas Moisture Content Control

The Argon gas moisture content from the purchase invoice certificate of analysis (CoA) shall be recorded in the heat treatment process control sheet.

4.2.4 Fully Hardened Heat Treatment Process Control

The temperature, time, Argon gas atmosphere, and water quench shall be recorded in the heat treatment process control sheets.

4.2.5 Anneal Heat Treatment Process Control

The temperature, time, Argon gas atmosphere, and furnace cool time shall be recorded in the heat treatment process control sheets.

4.2.6 Post Fully Hardened and Annealed Microstructure Comparison

A metallographic specimen shall be prepared and etched per 3.2.6. Comparisons of Figure 1 as HIP furnace cooled to Figure 2 after fully hardened heat treatment clearly is an indicator whether the HIP billet and/or billet section has received full hardened heat treatment. A fully hardened condition photomicrograph and its location relative to the billet and/or section(s) shall be attached to the heat treatment process control sheet. When applicable, an annealed heat treatment condition photomicrograph and its relative location to the billet and/or section(s) shall be attached to the heat treatment process control sheet. The HIP furnace cooled microstructure shown in Figure 1 and the annealed heat treatment condition shown in Figure 3 result in microstructures that are essentially the same. Digital copies of the photomicrograph(s) with the corresponding specimens examined shall be provided to the purchaser upon request.

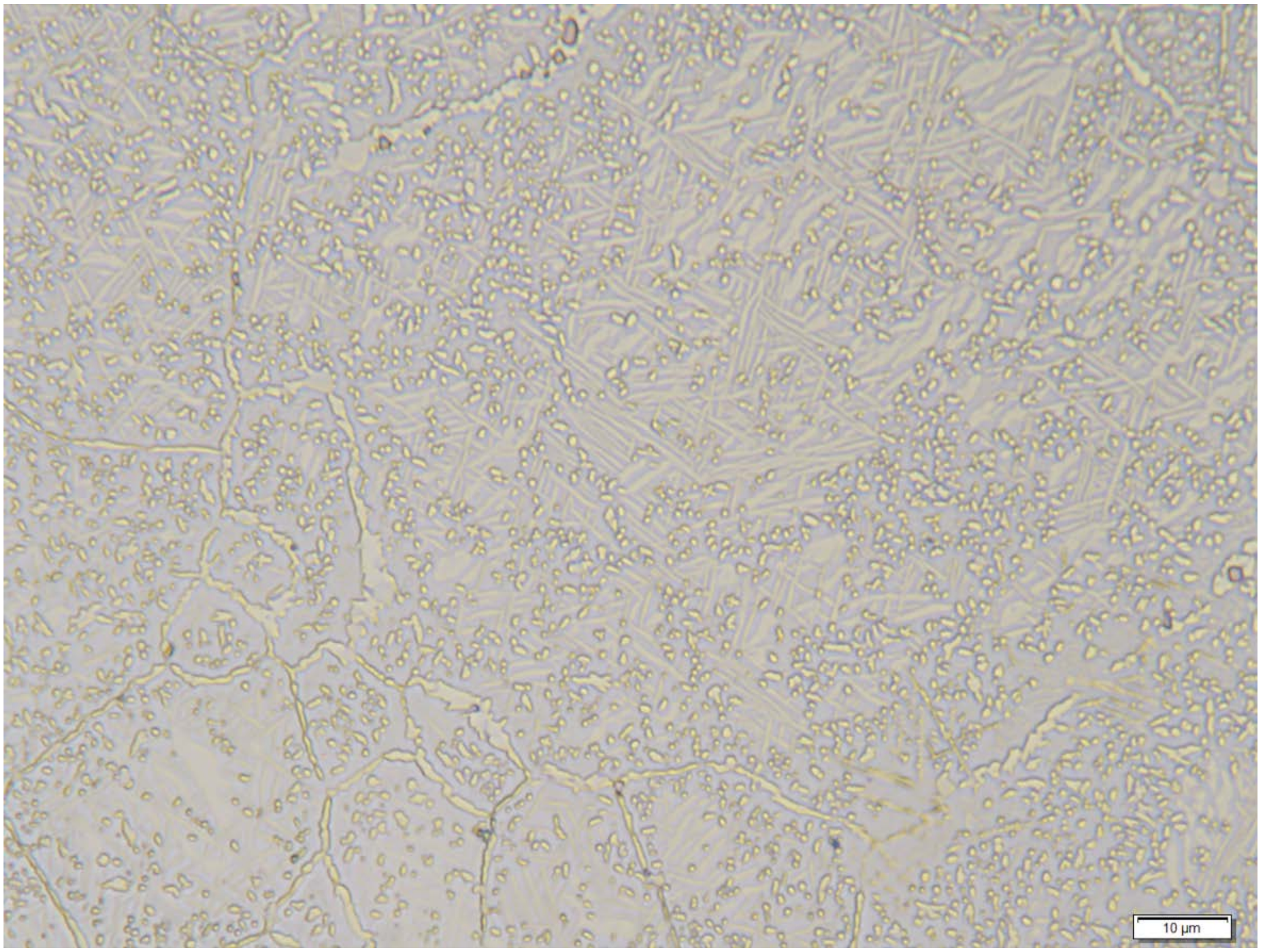

FIGURE 1. Typical microstructure by HIP with the HIP chamber furnace cooled

Typical microstructure by HIP with HIP chamber furnace-cooling. This microstructure and the annealed heat treatment condition are essentially the same. Note the fine secondary phases that are found at intergranular and intragranular locations. Within the grains, secondary phases form as nodules and fine platelets. Acicular phases can also be found within the grains and at grain boundaries. Etched with aqueous solution of 1 volume % concentrated HF + 10 volume % concentrated HNO_3 in distilled water.

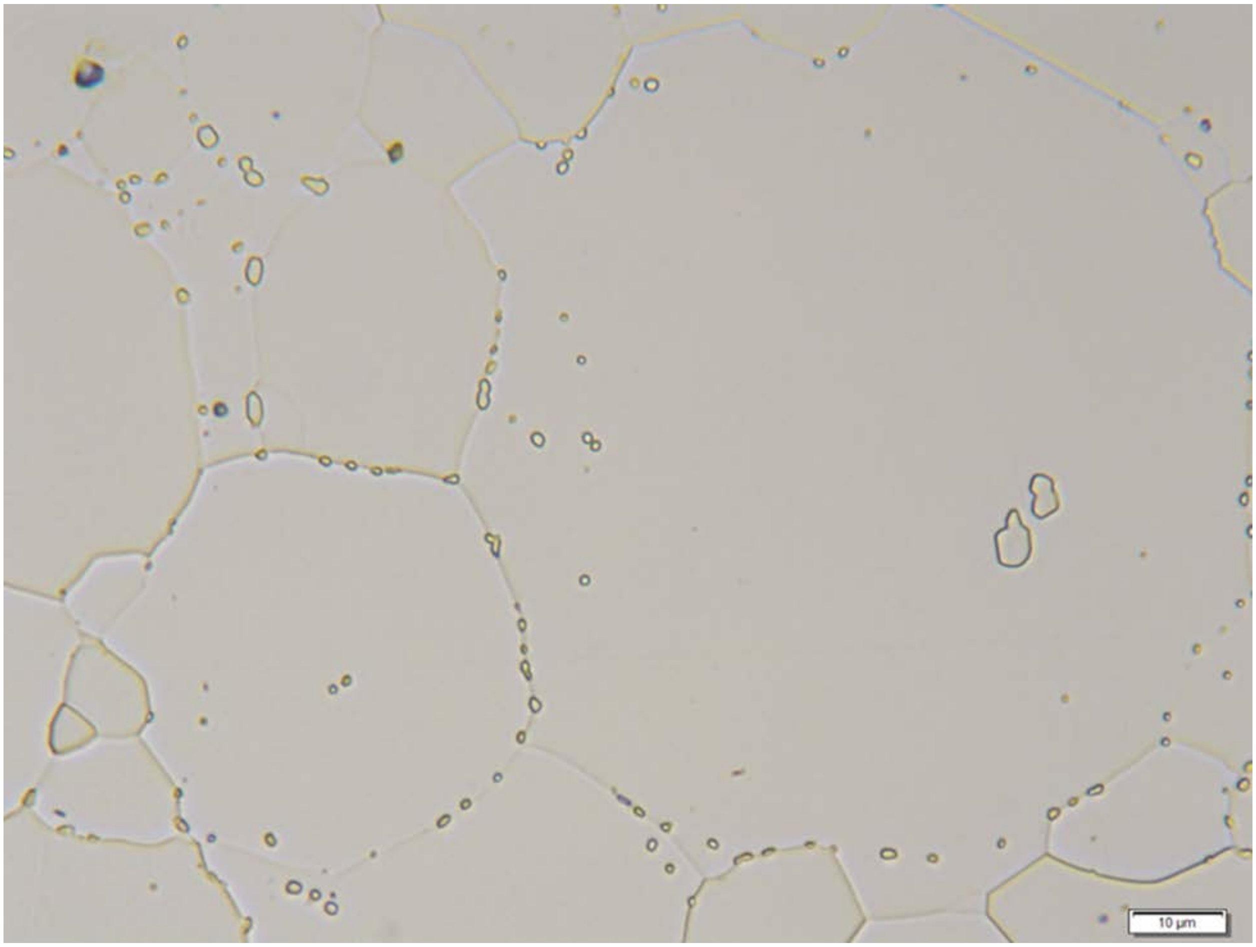

FIGURE 2. Typical microstructure after HIP with fully hardened heat treatment

Typical microstructure after HIP with fully hardened heat treatment which includes a water quench. Note, some retained secondary phases remain along grain boundaries and within the grains. Etched with aqueous solution of 1 volume % concentrated HF + 10 volume % concentrated HNO_3 in distilled water.

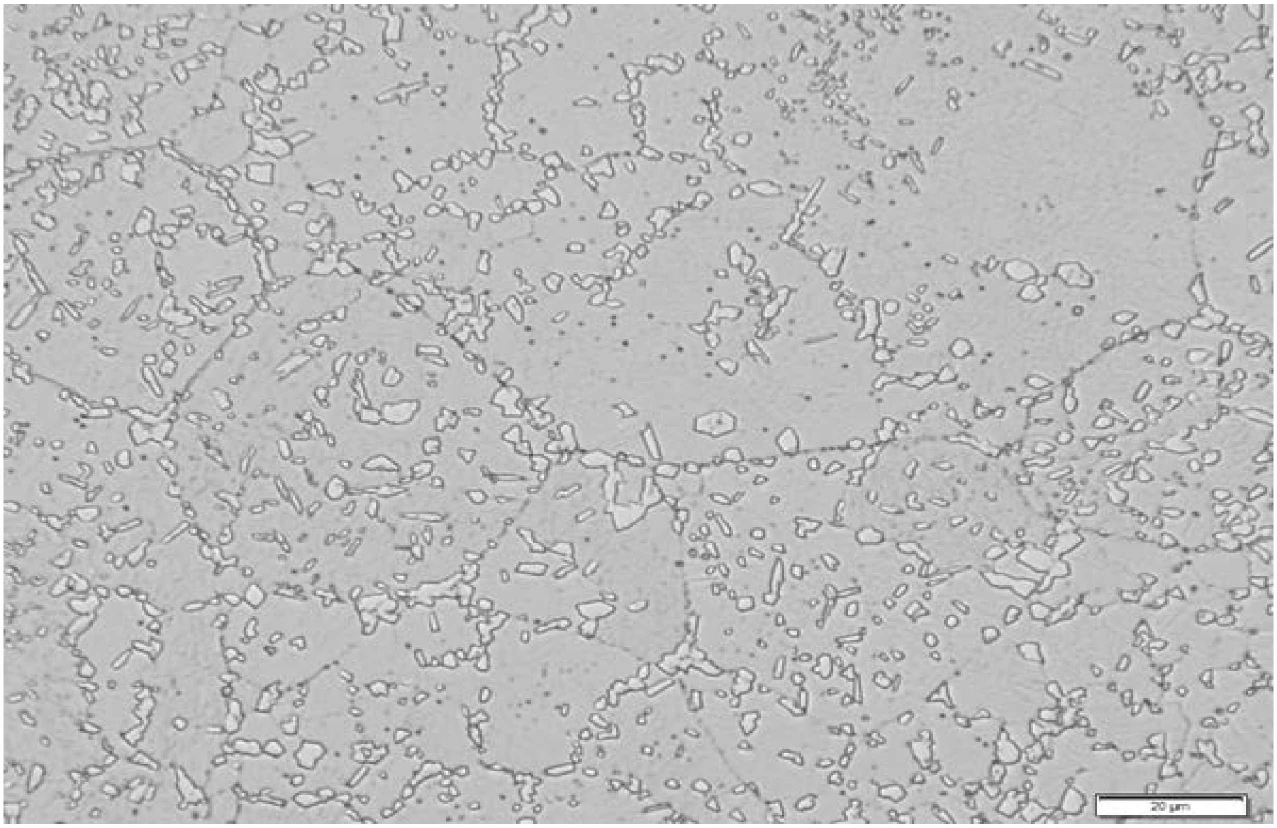

FIGURE 3. Typical microstructure with annealed heat treatment

Typical microstructure by HIP and annealed. Etched with 1 volume % concentrated HF + 10 volume % concentrated HNO_3 solution in distilled water.

4.2.7 Post Fully Hardened and Post Anneal Hardness Test

Hardness readings per ASTM E18 shall be recorded in the Heat Treatment Process Control Sheet for each applicable heat treat condition. A minimum of three readings shall be taken across the cross section, recorded, and provided to the purchaser. Per 3.2.7, for the fully hardened condition, an average shall be used to meet hardness requirements providing no individual hardness reading falls below the HRC 58 minimum. For the annealed condition, an average shall be used to meet the hardness requirement providing all hardness readings are in the 32 to 36 HRC range.

4.2.8 Heat Treatment Records

Upon the purchaser's request, documentation shall be provided with each billet and or billet sections as follows:

a. Furnace thermocouple set point temperature(s) and time duration.
b. Billet and or billet section(s) close proximity (load) thermocouple(s) temperature readings with corresponding time duration throughout the heat treatment process. Provide an explanation if a proximity (load) thermocouple is not placed in close proximity to the billet and/or billet sections.
c. An explanation of labeling and/or a legend distinguishing the type of heat treatment (anneal, fully hardened, quench, cool).

4.3 Mechanical Test Verification

4.3.1 Fully Hardened Tensile Testing

A minimum of five tensile test specimens shall be cut from the billet and/or billet section according to a cut plan approved by the purchaser, and identified as in the fully hardened condition. Tensile testing shall be conducted per ASTM E8. A minimum of 16 micro-inch arithmetic average roughness shall be maintained in the gage section. Surface grinding may be required to achieve the required surface finish. Ultimate strengths shall be determined according to ASTM E8.

4.3.2 Mechanical Test Specimens, Reports, and Records

All mechanical test specimens shall be returned to the purchaser from the testing facility/vendor along with each test result(s) and report(s) which identifies the heat treatment condition. This includes all testing parameters and testing results. Stress-strain curves shall be provided for each tensile test. Each test specimen shall be identified by the same labeling as in the test report.

4.4 Non-destructive Evaluation (NDE) Results

If required on the purchase order, NDE results shall be provided to the purchaser.

5. PACKAGING

Not applicable.

6. NOTES

This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.

6.1 Typical Physical Properties

Typical physical properties of 60Ni-40Ti material measured after Hot Isostatic Pressing (HIP) and heat treatment process are as follows: (Source NASA TM 2012-216056)

Thermal conductivity ~ 9 W/m ˚K
Thermal expansion ˚C ~11.2 × 10^{-6} /˚C
Magnetic None
Electrical resistivity ~1.04 × 10^{-6} / Ω·m

6.2 Part Distortion

To reduce distortion during quenching, machine the billet into smaller sections. Electrical-discharge machining (EDM) is a preferred method because of reduced residual stresses, which reduces 60Ni-40Ti part distortion.

6.3 Heat Treatment Types

The heat treatment is fully reversible. Billet sections can be supplied in the annealed condition and heat treated to the fully hardened condition. Similarly fully hardened material can be heat treated to the annealed condition.

6.3.1 Type I

Type I is the fully hardened condition, which may experience part distortion for complex geometries. To minimize distortion, fixtures may be required during heat treatment and subsequent machining. Additionally, parts may be machined to near net shape, fully hardened, and then final machined.

6.3.2 Type II

Type II is the annealed condition. Annealing results in reduced strength and hardness, which facilitates machining billet sections. Annealing is not essential to obtain the fully hardened condition and is applicable when reduced strength and reduced hardness are desired.

6.4 Corrosion

Corrosion characterization in various environments is described in NASA/CR-2016-218220, prepared for Marshall Space Flight Center under Contract NNM12AA41C, “Electrochemical, Polarization, Crevice Corrosion Testing of Nitinol 60, A Supplement to the ECLSS Sustaining Materials Compatibility Study”. R.E. Lee, Jacobs ESSSA Group, Huntsville, Alabama. This report is located on NASA Technical Reports Server (NTRS), which contains Scientific and Technical Information (STI).

6.5 Stress Corrosion Cracking (SCC) Resistance

SCC resistance characterization is described in NASA/TM-2016-218230, “Stress Corrosion Evaluation of Nitinol 60 for the International Space Station Water Recycling System”. P.D. Torres, Marshall Space Flight Center, Huntsville, Alabama. This Technical Memorandum (TM) is located on NASA Technical Reports Server (NTRS), which contains Scientific and Technical Information (STI).

6.6 Rolling Contact Fatigue

Rolling contact fatigue has been characterized by the joint efforts of NASA Glenn Research Center and NASA Marshall Space Flight Center Materials and Processes Laboratory. The testing standard used for characterization is based on ASTM Special Technical Publication (STP) 771 except that the steel balls were not intentionally roughened. Research testing results can be found in “Rolling Contact Fatigue of Superelastic Intermetallic Materials (SIM) for Use as Resilient Corrosion Resistant Bearings,” Tribology Letters, Vol. 57, No. 3, 2015.

APPENDIX A. Nickel-Titanium (NiTi) Powder Example

A.1 Scope

Use of NiTi powder conforming to Appendix A has produced billets meeting the requirements of this specification. Appendix A is not mandatory and vendors skilled in powder-metal processing may use their own powder requirements. Appendix A is for information only.

A.2 Applicable Documents

Unless otherwise noted the latest revision of the following documents are applicable to the extent specified herein. In the event of a conflict between the documents referenced herein and the contents of this Appendix, the contents of this Appendix shall take precedence. The contractor may pursue substituting equivalent specifications and documents to the ones identified herein as long as the substitution does not compromise the intent of the specifications and documents identified herein and is approved by NASA/MSFC before implementation.

ASTM B213, Standard Test Methods for Flow Rate of Metal Powders Using the Hall Flowmeter Funnel

ASTM B214, Standard Test Method for Sieve Analysis of Metal Powders

ASTM B527, Standard Test Method for Determination of Tap Density of Metal Powders and Compounds

ASTM E1097, Standard Guide for Determination of Various Elements by Direct Current Plasma Atomic Emission Spectrometry

ASTM E1172, Standard Practice for Describing and Specifying a Wavelength-Dispersive X-Ray Spectrometer

ASTM E1409, Standard Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by Inert Gas Fusion

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

ASTM E1479, Standard Practice for Describing and Specifying Inductively-Coupled Plasma Atomic Emission Spectrometers

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

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A.3 Ni-Ti Powder Metal

The nickel (Ni) and titanium (Ti) shall be pre-alloyed, gas atomized nickel titanium (Ni-Ti) powder.

A.3.1 Powder Chemical Composition

Chemical composition shall conform to the percentages by weight shown in Table A-I.

TABLE A-I. Powder Metal Chemical Composition

Element	Min	Max
Titanium	39.0	41.0
Carbon	0	0.05
Oxygen	0	0.08
Hydrogen	0	0.005
Nickel	balance	balance

No more than 0.2 weight per cent total of tramp element contamination shall be in the composition. Tramp elements are any other elements other than nickel and titanium. Some, but not all, examples of tramp elements include oxygen, carbon, iron, cobalt, chromium, aluminum, and nitrogen.

A.3.2 Powder Lots

Powder shall be produced in heats/lots.

A.3.3 Powder Processing Atmosphere

Each powder heat/lot shall be produced in an appropriate non-contaminating atmosphere.

A.3.4 Powder Blending

The powder from all production runs shall be thoroughly blended.

A.3.5 Powder Physical Requirements

A.3.5.1 Powder Size/Oversize Remnant Particles

Powder size when sieved through a standard 35 mesh sieve shall have no more than 3 per cent oversized remnant particles, by weight. Powders when sieved through a 60 or higher number standard mesh sieve shall have no more than 5 percent oversized remnant particles, by weight. The preferred powder size is 60 mesh. The following table A-II is an example of the oversized powder limits relative to mesh sieve size (thus particle size).

TABLE A-II. Mesh Sieve and Oversized Powder Limits

Mesh Size	Oversize Powder Limits
-35 mesh (-500 micron)	3%
-60 mesh (-250 micron)	5%
-270 mesh (-53 micron)	5%

A.3.5.2 Particle Dryness

Powder particles shall be dry per A.4.5.2.

A.3.5.3 Particle Tap Density

The powder particle tap density shall be determined and supplied to the purchaser upon request.

A.3.5.4 Particle Flow Rate

The powder particle flow rate shall be determined and supplied to the purchaser upon request.

A.4 Powder Verification

Use of unalloyed powder consisting of individual Ni and Ti constituents shall be cause for rejection.

A.4.1 Powder Chemical Composition Testing

The powder alloying element(s) shall be determined by Guide for Direct Current Plasma (DCP) according to ASTM E1097, Inductively-Coupled Plasma (ICP) Optical Emission Spectrometers according to practice ASTM E1479, and X-ray spectrometry according to ASTM E1172 or equivalent method(s). Carbon shall be measured by combustion per ASTM E1941 or best equivalent method. Hydrogen shall be measured by inert gas fusion per ASTM E1447, or best method and/or vacuum hot extraction by best method. Oxygen and nitrogen shall be measured by inert gas fusion per ASTM E1409, or by best method. Certification documents shall be provided by the vendor that verifies the chemical composition of the powder and how it was derived.

A.4.2 Powder Heat/Lot Characteristics

A heat/lot shall be powder produced from one production run. When approved by the purchaser, a heat/lot shall be powder produced in a series of consecutive runs in the same equipment under the same fixed parameters. A specimen shall be taken from each heat/lot of powder for chemical testing and certification.

A.4.3 Non-contaminating Atmosphere

A non-contaminating atmosphere shall consist of a non-reacting inert gas. Acceptable non-reacting inert gasses shall be Argon or Helium. To prohibit nitride growth, use of Nitrogen as an inert gas shall be prohibited.

A.4.4 Powder Blending Verification

Blends shall identify each heat/lot of which it is comprised. Each heat/lot shall be recorded by the vendor with each heat/lot and each blend identified by its own unique name/number/alphabetic characters. Certification shall identify each heat/lot and blend and their individual chemical composition.

A.4.5 Powder Particle Verification

A.4.5.1 Powder Size/Oversize Remnant Particles Verification

Powder size shall be determined by ASTM B214. Oversized remnant particles in excess of three per cent, by weight, when sieved through a standard 35 mesh sieve, shall be cause for rejection. Oversized remnant particles in excess of 5 percent by weight, when sieved through a 60 or higher number standard mesh sieve, shall be cause for rejection.

A.4.5.2 Particle Dryness Test

To determine if the powder is dry, a specimen shall be weighed, then heated to 212 ˚F in a non-contaminating atmosphere and re-weighed to determine if there is any weight loss. Weight loss shall not be greater than one-percent.

A.4.5.3 Particle Tap Density Test

The powder particle tap density shall be determined per ASTM B527.

A.4.5.4 Particle Flow Rate

The powder particle flow rate shall be determined per ASTM B213.

A.5 Powder Packaging

If powder is to be shipped to a separate processing facility for hot isostatic pressing, then the powder shall be packaged in sealed metal or plastic containers suitable for transportation, storage, contamination prevention, and moisture intrusion prevention. Relative humidity within the containers shall be maintained below 40 percent and shall be measured by mechanical gage, electromechanical gage, or by a desiccant that changes color. Labels on each container shall contain minimum information as follows:

a. Vendor ID
b. Alloy type
c. Powder heat/lot number
d. Powder size including each mesh fraction
e. Net powder weight
f. Purchase order number

A.6 Notes

A6.1 Contamination Prevention

Cleanliness prevents contamination and is important to achieving good material properties.

A.6.2 Contamination Sources

Contamination typically is due to tramp elements consisting of oxygen, carbon, iron, cobalt, chromium, aluminum, nitrogen.

A.6.3 Data

Upon the purchaser’s request, data to be provided includes the following:

a. Powder blend identified by its own unique name and/or number and/or alphabetic characters.
b. Blends shall identify each heat/lot of which it is comprised.
c. Weight of powder
d. Purchase Order number
e. Chemistry certification (CoA) for each heat/lot and blend and how each element was derived
f. Particle size(s) certification by weight percent for each size
g. Tap density
h. Flow rate

APPENDIX B. Hot Isostatic Pressing (HIP) Example

B.1 Scope

Use of the HIP conforming to Appendix B has produced billets meeting Specification requirements. Appendix B is not mandatory and vendors skilled in the HIP process may use their own HIP requirements. Appendix B is for information only.

B.2 Applicable Documents

Unless otherwise noted the latest revision of the following documents are applicable to the extent specified herein. In the event of a conflict between the documents referenced herein and the contents of this Appendix, the contents of this Appendix shall take precedence.

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

ASTM E3, Standard Guide for Preparation of Metallographic Specimens

ASTM E18, Standard Test Methods for Rockwell Hardness of Metallic Materials

ASTM E1097, Standard Guide for Determination of Various Elements by Direct Current Plasma Atomic Emission Spectrometry

ASTM E1172, Standard Practice for Describing and Specifying a Wavelength-Dispersive X-Ray Spectrometer

ASTM E1409, Standard Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by Inert Gas Fusion

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

ASTM E1479, Standard Practice for Describing and Specifying Inductively-Coupled Plasma Atomic Emission Spectrometers

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

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B.3 Hot Isostatic Pressing (HIP)

B.3.1 Fill Process

A suitable HIP container, such as that described in Appendix C, shall be filled with Ni-Ti powder through the fill tube in a glove box. The powder filled container shall be attached to a vacuum pump and evacuated. To remove moisture, at 0.05 Torr begin heating to 175 ˚F and hold until 0.005 Torr with a leak rate of less than 0.01 Torr per minute is achieved. The container shall then be sealed by tungsten inert gas (TIG) welding the fill tube. Use of any other procedures shall be fully disclosed by the vendor to the purchaser.

B.3.2 HIP Process

The sealed container shall undergo HIP. At a minimum the HIP shall include heating to 1,800˚F (± 25˚) at 15,000 psi (± 500 psi) for four hours (+15/-0 minutes) for consolidation. Turn off heat and release pressure. The sealed HIP container shall be left in the HIP chamber after heat and pressure have been turned off to furnace cool (furnace anneal), or shall be in accordance to the purchase order instructions. HIP parameters shall be varied to accommodate larger or smaller sized containers. These parameters shall be in accordance with the purchase order instructions.

B.3.2.1 End Piece Hardness Readings

The end pieces of the HIP container shall be cut off and three hardness readings shall be taken on the exposed NiTi material within the end piece from each perimeter to the center. Hardness shall be uniform across the cross section and readings shall be in the range of Hardness Rockwell C (HRC) 32-36. The end piece may be ground flat for hardness testing consistency.

B.3.2.2 Billet Appearance

The billet's shape shall be uniform and symmetric. The design and manufacture of the HIP container must be appropriate to achieve a uniform and symmetric billet.

B.3.2.3 Microstructure

The microstructure after HIP shall be recorded. The location relative to the billet in which the microstructure was examined then recorded will also be recorded and included in results.

B.3.2.4 Billet Density

The billet density shall in the range of 0.240 to 0.246 pounds per cubic inch.

B.3.2.5 Billet Chemical Composition

Chemical composition shall conform to the percentages by weight shown in Table B-I.

TABLE B-I. Billet Chemical Composition

Element	Min	Max
Titanium	39.0	41.0
Carbon	0	0.05
Oxygen	0	0.08
Nickel	balance	balance

No more than 0.2 weight per cent total of tramp element contamination shall be in the composition. Tramp elements are any other elements other than nickel and titanium. Some, but not all, examples of tramp elements include oxygen, carbon, iron, cobalt, chromium, aluminum, and nitrogen.

B.4 HIP Process Verification

B.4.1 Fill Process Control Sheet

Each step identified in paragraph B.3.1 shall be documented in a process control sheet with appropriate signatures. Use of inspector stamps and initials are acceptable.

B.4.2 HIP Process Control Sheet

Each step identified in paragraph B.3.2 shall be documented in a process control sheet with appropriate signatures. Use of inspector stamps and initials are acceptable.

B.4.2.1 End Piece Hardness Test

Hardness readings per ASTM E18 shall be recorded in the HIP Process Control Sheet. Three readings shall be taken across the cross section as per B.3.2.1, from each perimeter to the center. Any deviation from the HRC 32-36 range shall be cause for rejection.

B.4.2.2 Billet Shape Verification

The billet shape shall be recorded in the HIP Process Control Sheet. Any deviation from a uniform and symmetric appearance shall be cause for rejection.

B.4.2.3 Microstructure

A metallographic specimen shall be prepared per ASTM E3 and etched with aqueous solution of 1 volume % concentrated HF + 10 volume % concentrated HNO_3 in distilled water**.**

A photomicrograph shall be attached to the HIP process control sheet. Digital copies of the photomicrograph shall be provided to the purchaser upon request.

B.4.2.4 Billet Density Determination

Billet density shall be determined per ASTM B923. Failure to be in the range of 0.240 to 0.246 pounds per cubic inch shall be cause for rejection.

B.4.2.5 Billet Chemical Composition Testing

The billet alloying element(s) shall be determined by Guide for Direct Current Plasma (DCP) Emission Spectrometry Analysis according to ASTM E1097, Inductively-Coupled Plasma (ICP) Optical Emission Spectrometers according to practice ASTM E1479, and X-ray spectrometry according to ASTM E1172 or an equivalent method. Carbon shall be measured by combustion per ASTM E1941 or best method. Hydrogen shall be measured by inert gas fusion per ASTM E1479 or best method and/or vacuum hot extraction by best method. Oxygen and nitrogen shall be measured by inert gas fusion per ASTM E1409 or by best method. Certification documents shall be provided by the vendor that verifies the chemical composition of the billet and how it was derived.

B.5 Packaging

Not applicable.

B.6 HIP Process Records

Upon the purchaser’s request, documentation shall be provided with each billet as follows:

a. Digital photos and dimensions of the billet while still in the HIP container.
b. HIP container chamber set point temperatures and time which includes each zone and relative location to the HIP container/billet.
c. Any proximity (load) thermocouple readings including temperature and time.
d. Set point pressure(s) and actual pressure measured and recorded relative to time and temperature.
e. The inert gas that provided the isostatic pressure.

APPENDIX C. Hot Isostatic Pressing (HIP) Container Example

C.1 Scope

The HIP process conforming to Appendix C has produced billets meeting Specification requirements. Appendix C is not mandatory and vendors skilled in the HIP process may use their own HIP container design and HIP process. Dimensions and material of this HIP container may be varied to accommodate the billet size and keeping within HIP equipment capacity. The method of construction and materials can also be varied to meet project requirements. Appendix C is for information only.

C.2 Applicable Documents

Unless otherwise noted the latest revision of the following documents are applicable to the extent specified herein. In the event of a conflict between the documents referenced herein and the contents of this Appendix, the contents of this Appendix shall take precedence.

> ASTM A1011, Standard Specification for Steel, Sheet and Strip, Hot-Rolled, Carbon, Structural, High- Strength Low-Alloy, High-Strength Low-Alloy with Improved Formability, and Ultra-High Strength
>
> ASTM Headquarters – 100 Barr Harbor Drive PO Box C700 West Conshohocken, PA 19428-2959

C.3 Container Material

Container material may be steel conforming to ASTM A1011, Grade 36, Type 2, or any steel with sufficient ductility to enable the HIP process.

C.4 Container Parts

C.4.1 Tube Dimensions

The tube for the HIP container body may be a standard tube but must possess enough wall thickness for welding and to prevent deformation during the HIP. As an example, the tube may have an outer diameter (OD) of 5 inches and an inner diameter (ID) of 4.75 inches, and length of 13.75 inches.

C.4.2 End Caps

The end caps for the container may be made from the same material as the tube. For an example of dimensions, a diameter of 4.875 inches and a thickness of 0.25 inches.

C.4.3 Fill Tube

The fill tube may have an OD of 0.5 inches and an ID 0.34 inches, and length of 10 to 20 inches.

C.5 Fabrication Steps

C.5.1 Clean Parts

All parts may be cleaned using best shop practices. A cleaning method found to work is to use a dilute phosphoric acid, rinse with distilled water, and allow to dry. The cleaning process is very important for the container. Contamination (grease/oil) and oxides can change the chemistry of the billet.

C.5.2 End-Cap Hole

A 0.5 inch hole may be drilled in the middle of one end cap. The end-cap will be re-cleaned and allowed to dry.

C.5.3 Weld Fill Tube

One end of the fill tube may be welded to the end-cap with hole in the middle. The fill tube may be aligned with the end-cap hole. Tungsten inert gas (TIG) may be used to weld the fill tube to the end-cap and the end-caps to the container body. Clean the assembly after welding as per C.5.1 and allow to dry.

C.5.4 Fill the Tube with Argon

The tube (container body) may be filled with Argon gas. The Argon gas will minimize oxide formation inside the tube when the end caps are welded. Oxides on the tube inner surface can cause billet contamination.

C.5.5 Weld End Caps

End caps may be welded individually using TIG.

C.6 Notes

C.6.1 Fabrication Procedure Source

Much of the HIP container fabrication procedure was provided by Puris, LLC. Contact information is as follows: Puris LLC, 78 Northpointe, Drive, Bruceton, Mills, WV 26525, 304-777-4270, http://www.purisllc.com. Primary Point of Contact (POC): Eric Bono, VP Engineering Solutions 412-260-8048, ebono@purisllc.com.

C.6.2 Puris, LLC is not a Sole Source

This vendor is not designated as a sole source.

APPENDIX D. Acronyms

ASTM	ASTM International (formerly American Society for Testing and Materials)
CGA	Compressed Gas Association
CoA	Certificate of Analysis
DCP	Direct Current Plasma Emission Spectrometry Analysis
˚F	degrees Fahrenheit
˚C	degrees Centigrade
EDM	Electrical Discharge Machining
HF	Hydrofluoric Acid
HIP	Hot Isostatic Pressing
HNO_3	Nitric Acid
HRC	Hardness Rockwell C
ICP	Inductively-Coupled Plasma (Optical Emission Spectrometer)
ID	Inner Diameter
ksi	Kilo Pound Per Square Inch
MAPTIS	Material and Processes Technical Information System
MSFC	Marshall Space Flight Center
NASA	National Aeronautics and Space Administration
NDE	Non Destructive Evaluation
Ni	Nickel
Ni-Ti	Nickel Titanium
OD	Outer Diameter
psi	pounds per a square inch
QVL	Quality Verification Level
RCF	Rolling Contact Fatigue
SCC	Stress Corrosion Cracking
Ti	Titanium
TIG	Tungsten Inert Gas
TM	Technical Memorandum
TR	Technical Report
Wt%	Weight Per Cent

APPENDIX E: Verification Matrix

N/A - Not Applicable; A - Analysis; D - Demonstration; I - Inspection; T - Test

Requirement Paragraph #	Requirement Paragraph Title	Verification Method	Verification Paragraph #	Verification Paragraph Title
3.1	Chemical Composition	T	4.1	Chemical Composition Testing
3.2	Heat Treatment	N/A	4.2	Heat Treatment Verification
3.2.1	Billet Sectioning and Section Identification	I	4.2.1	Billet Section Thickness and Identification
3.2.2	Argon Gas Purge	I	4.2.2	Argon Gas Purge Control
3.2.3	Argon Gas Moisture Content	I	4.2.3	Argon Gas Moisture Content Control
3.2.4	Fully Hardened Heat Treatment	I	4.2.4	Fully Hardened Heat Treatment Process Control
3.2.5	Anneal Heat Treatment	I	4.2.5	Anneal Heat Treatment Process Control
3.2.6	Post Fully Hardened and Annealed Heat Treatment Microstructure	I	4.2.6	Post Fully Hardened and Annealed Microstructure Comparison
3.2.7	Hardness	T	4.2.7	Post Fully Hardened Post Anneal Hardness Test
3.2.8	Heat Treatment Documentation	I	4.2.8	Heat Treatment Records
3.3	Mechanical Testing	N/A	4.3	Mechanical Test Verification
3.3.1	Fully Hardened Tensile Strength	T	4.3.1	Fully Hardened Tensile Testing
3.3.2	Mechanical Test Specimen Labeling and Preservation	I	4.3.2	Mechanical Test Specimens, Reports, and Records
3.4	Non-destructive Evaluation (NDE)	T	4.4	NDE Results