

# **M5 Alloy Specification for TREAT Conceptual Fuel Cladding Selection**

Clemente J. Parga

March 2018



The INL is a U.S. Department of Energy National Laboratory  
operated by Battelle Energy Alliance

# **M5 Alloy Specification for TREAT Conceptual Fuel Cladding Selection**

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**March 2018**

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**Prepared for the  
U.S. Department of Energy  
National Nuclear Security Administration  
Under DOE Idaho Operations Office  
Contract DE-AC07-05ID14517**

## **Specification**

Project No. 31772

# **M5 Alloy Specification for TREAT Conceptual LEU Fuel Cladding Selection**



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## REVISION LOG

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	Revision:	0
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**1. SCOPE****1.1 Introduction**

The Idaho National Laboratory (INL) Transient Reactor Test Facility (TREAT) is a nuclear test facility designed to provide safety data for fuel designs by simulating conditions during various types of nuclear excursions. The M5 alloy cladding material for the conceptual TREAT Low Enriched Uranium fuel core shall be supplied according to this specification.

**1.2 Definitions**

For the purpose of this specification, the following terms are identified:

- 1.2.1 *Purchaser.* Organization requesting/contracting for the material
- 1.2.2 *Fabricator.* Organization responsible for the material fabrication
- 1.2.3 *Specification.* All parts and attachments of this document, its references, drawings, and standards, as may be modified by contractual document

**2. APPLICABLE DOCUMENTS****2.1 Applicable Standards**

The applicable portions of the following documents, as defined herein, form a part of this specification. Where there is a conflict between the documents cited and the latest revision, thereof, the Fabricator shall notify the Purchaser of the conflict and use the latest revision, unless otherwise directed by the Purchaser.

**2.1.1 ANSI/ASME Standards**

NQA-1-2008/2009 addenda	Quality Assurance Program Requirements for Nuclear Facilities
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**2.1.2 ASTM Standards**

ASTM B352-11	Standard Specification for Zirconium and Zirconium Alloy Sheet, Strip, and Plate for Nuclear Application
ASTM B350	Standard Specification for Zirconium and Zirconium Alloy Ingots for Nuclear Applications
ASTM E8	Standard Test Methods for Tension Testing of Metallic Materials
ASTM G54	Standard Practice for Simple Static Oxidation testing
ASTM B614	Standard Practice for Descaling and Cleaning Zirconium and Zirconium Alloy Surfaces
ASTM E112	Standard Test Methods for Determining Average Grain Size

**3. MATERIAL SPECIFICATION**

The sheet material shall be fabricated from reactor grade zirconium alloy, cold rolled, and vacuum annealed to full recrystallization in conformance to this specification and supporting documents.

**3.1 Ordering Information**

- A. Material: M5® alloy AREVA Inc.
- B. Condition: Cold-Rolled and High Vacuum Annealed to Full Recrystallization. Cold rolling will be performed with an adequate lubrication to produce a smooth surface finish and to avoid the pickup of foreign metals on the mill rolls. Depending on the amount of cold-work percent, the process annealing shall be conducted at a temperature between 510 to 790°C and the material held at temperature for a minimum of one hour, or as needed to ensure full recrystallization. Any significant grain growth after full recrystallization is not acceptable (e.g., avoid an increase in ASTM grain size value, G, caused by grain growth ( $\Delta G < 1$ ) after recrystallization). The ASTM grain size number shall be measured and reported according to the ASTM E112 standard using the planimetric procedure. The annealing shall be performed under a high vacuum

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atmosphere of  $< 1 \times 10^{-4}$  Torr or better. The vacuum furnace must be purged for at least three cycles before starting the thermal processing. The purging cycle will consist of flowing high-purity argon (99.996% Ar  $-65^{\circ}\text{C}$ , or  $-85^{\circ}\text{F}$ , dew point) until reaching a slight over-pressure, then evacuating the furnace to high vacuum ( $< 1 \times 10^{-4}$  Torr), and repeating this process for at least three times. If required for dimensional conformance, a final light cold-rolled pass with polished rollers can be performed avoiding the introduction of significant cold-work ( $< 5\%$  reduction in thickness). No reannealing is permitted after final light cold-rolled pass.

- C. Finish: Mechanically descaled and conditioned or ground to produce a surface roughness with the following parameters:  $R_{\text{RMS}} < 0.8 \mu\text{m}$ ;  $R_a < 0.8 \mu\text{m}$ ;  $R_{\text{max}} < 3.2 \mu\text{m}$   $R_t < 5 \mu\text{m}$ .
- D. Form: Sheet.
- E. Edge: edges shall be square, no more than 0.0025 inch out of perpendicular with the top/bottom face.
- F. Dimensions: thickness =  $0.026 \pm 0.002$  inch; width =  $11 \pm 0.005$  inch; flatness =  $\pm 0.005$  inch.
- G. Grade designation: Generic alloy composition is 99 weight percent Zirconium, plus 1 weight percent Niobium. Precise chemical composition and thermo-mechanical processing are proprietary information of AREVA Inc.

### 3.2 Alloying Elements and Impurity Level Requirements and Analyses

The material's constituent concentrations shall be in conformance with the alloying elements and impurity levels delimited in Table 1 and 2, respectively.

The chemical analysis of the material produced to this specification shall be made by the manufacturer on the ingot in accordance with ASTM B350. This analysis can be performed by the manufacturer on the ingot itself, or on intermediate or final products with the same frequency and in the same positions relative to the ingot as required in ASTM B350. The chemical analysis of hydrogen, oxygen, and nitrogen shall be determined on the finished product.



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Table 1. Alloying elements chemical composition range for M5 (Zr-1Nb alloy).

Element	Composition Range, Weight %	
Zirconium, Zr	Balance Weight % Zr	
Niobium, Nb	$\geq 0.8$	$\leq 1.2$
Oxygen, O	$\geq 0.12$	$\leq 0.14$
Sulfur, S	$\geq 0.0010$	$\leq 0.0035$

Table 2. Maximum impurity concentration in M5 (Zr-1Nb alloy).

Impurity Element	Maximum Allowable Content, ppm	Impurity Element	Maximum Allowable Content, ppm	Impurity Element	Maximum Allowable Content, ppm
Aluminum, Al	$\leq 75$	Copper, Cu	$\leq 50$	Nickel, Ni	$\leq 70$
Nitrogen, N	$\leq 80$	Tin, Sn	$\leq 100$	Lead, Pb	$\leq 30$
Boron, B	$\leq 0.5$	Iron, Fe	$\leq 500$	Silicon, Si	$\leq 120$
Cadmium, Cd	$\leq 0.5$	Hafnium, Hf	$\leq 100$	Tantalum, Ta	$\leq 100$
Calcium, Ca	$\leq 30$	Hydrogen, H	$\leq 25$	Titanium, Ti	$\leq 50$
Carbon, C	$\leq 100$	Magnesium, Mg	$\leq 20$	Tungsten, W	$\leq 100$
Chromium, Cr	$\leq 150$	Manganese, Mn	$\leq 50$	Uranium, U	$\leq 3.5$
Cobalt, Co	$\leq 10$	Molybdenum, Mo	$\leq 50$	Vanadium, V	$\leq 50$

### 3.3 Mechanical Properties Requirements and Testing

The mechanical properties of the material shall conform to the requirements prescribed in Table 3.

The tensile testing of sheet material will be in the longitudinal and transverse direction. The testing will be performed in accordance to ASTM E8. For each lot of 4000 lb [1800 kg] or fraction of material, two random samples shall be tested for mechanical properties.

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Table 3. Mechanical properties requirements.

Condition	Specimen Orientation	Test Temp. (°C)	0.2% Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Uniform Elongation (%)	Total Elongation (%)
Cold-Worked and Fully Recrystallized	Longitudinal (Rolling Direction)	15 - 25	≥ 360	≥ 470	≥ 10	≥ 25%
	Transverse	15 - 25	≥ 395	≥ 415	≥ 9	≥ 25%

**3.4 Oxidation Properties Testing and Requirements**

For oxidation testing, two sets of samples (three samples per set) with at least 400 mm<sup>2</sup> total surface area chosen at random from each lot of 4000 lb [1800 kg], or fraction of material, shall be oxidation tested in still air at 1080°F [600°C] and 1260°F [700°C] for 5, 10 and 20 hours (one sample per temperature-time combination) in accordance with ASTM G54.

The tested coupons will not be descaled or stripped of their oxide layer after testing. The samples' oxidation will be evaluated by 1) recording macrographs at a suitable magnification to appreciate the pre and posttest appearance of each sample, 2) mass change per unit area measurements and 3) total depth of attack measurements will be reported.

**3.5 Summary of Testing and Analyses Requirements**

Documentation on the testing and analyses required by this specification will be recorded and supplied to the Purchaser by the Fabricator for compliance of material. The following documents are required:

- Gain size determination in accordance to ASTM E112.
- Tensile testing in accordance to ASTM E8.
- Chemical composition analyses as specified in ASTM B350.
- Oxidation testing in accordance to ASTM G54.

**4. FABRICATION**

All components to be used shall be tagged with a status indicator demonstrating compliance with this specification. This tag demonstrates receipt inspection has been completed and the material verified in compliance.

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**4.1 Cleanliness**

Components shall be cleaned, handled and stored in a manner that keeps them clean, dry and protected from damage. Prior to heat treatment, clean sheet material in an ultrasonic bath using deionized water for 20 min, rinsed with alcohol and oven dried at 130°C for 2 hours. Rinsing shall be done with 100% not reconstituted purity (200 proof) ethyl alcohol. Other procedures provided in ASTM B614 may be used as a substitute for these cleaning requirements.

**4.2 Appearance**

Sheet shall be visually free from cracks, seams, slivers, blisters and other imperfections.

**4.3 Inspection**

Each sheet shall be dimensionally inspected prior to shipment to confirm compliance with specified dimensions and tolerances. A report documenting this inspection shall be provided to the Purchaser.

**4.4 Cleaning, Handling and Shipping Requirements**

Sheet material shall be cleaned as specified in Section 4.1. Similarly, the material shall be packaged with the aim of keeping it clean, dry and protected from damage during shipping.

**5. QUALITY ASSURANCE**

The Fabricator shall document, implement, and maintain a quality program in compliance with NQA-1-2008/2009 addenda and any additional quality assurance requirements of this specification.

The Purchaser may conduct onsite reviews, at the Purchaser's discretion, of the Fabricator's manufacturing, inspection and test operations. The Fabricator shall make available procedures, manufacturing, and inspection records relevant to fabrication.

Unless otherwise specified, the Fabricator shall be responsible for the performance of all tests and inspections required prior to submission of records to the Purchaser for acceptance. The performance of these tests and inspections does not limit the right of the Purchaser to conduct additional tests and inspections deemed necessary to assure conformance with the requirements of this specification.

**5.1 Certification**

A certification package shall be provided for each component. The certification package shall include the following:

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- Copies of the Certified Material Test Reports, (CMTR) for the fabricated items.
- Certificate of Conformance (CofC) Statement that the material has been processed per the requirements contained in this specification and meets the requirements.
- Inspections reports demonstrating compliance with manufacturing, inspection, and test operations requirements.

**6. OTHER REFERENCES**

1. AREVA, M5 Sheet (0.026" thick) Material Inspection Certificate, Manufacture Order 64370-10, INL PO148311, 05-26-2015, p. 1-21.
2. W. R. Lloyd, J. A. Simpson, B. D. Coryell, "Mechanical Properties Evaluation of Zirconium Alloys (22°C – 820°C)," INL/LTD-16-38460, April 2016, p. 1-91.
3. R.T. Webster, Zirconium and Hafnium, "Properties and Selection: Nonferrous Alloys and Special-Purpose Materials," Vol.2, ASM Handbook, ASM International, 1990, p.661-669.