INCH-POUND

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DETAIL SPECIFICATION

ALUMINUM-BASED POWDERS FOR COLD SPRAY DEPOSITION

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 <u>Scope.</u> This specification covers requirements intended for use in the procurement of aluminum and aluminum-based alloy powders that will be used to produce deposits utilizing a cold spray materials deposition process for the purpose of parts repair, coatings, or fabrication of freestanding structures. Cold spray is a process whereby metal powder particles are utilized to form a deposit by means of ballistic impingement upon a substrate in order to produce coatings or free-standing structures. This cold spray process is explained in the manufacturing process standard MIL-STD-3021, "Materials Deposition, Cold Spray".

Comments, suggestions, or questions on this document should be addressed to: Director, U.S. Army Research Laboratory, Weapons and Materials Research Directorate, Specifications & Standards Office, Attn: RDRL-WMM-D, Aberdeen Proving Ground, MD 21005-5069 or emailed to <u>richard.j.squillacioti.civ@mail.mil</u>. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <u>https://assist.dla.mil/</u>.

AMSC N/A

AREA MFFP

DISTRIBUTION STATEMENT A: Approved for public release; distribution is unlimited.

1.2 <u>Classification</u>. The aluminum and aluminum-based alloy powders are of the following classes, as specified (see 6.2).

Class A - Commercially Pure (CP) aluminum powder.

Class B - High Purity (HP) aluminum powder.

Class C - Aluminum alloy 6061 powder.

Class D - Aluminum alloy 5056 powder

1.3 <u>Part or Identifying Number (PIN).</u> PINs to be used for powders acquired by this specification are created as follows:



2. APPLICABLE DOCUMENTS

2.1 <u>General.</u> The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3 and 4 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 <u>Specifications, standards, and handbooks</u>. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of this document are those cited in the solicitation or contract.

DEPARTMENT OF DEFENSE STANDARDS

MIL-STD-3021 - Materials Deposition, Cold Spray

(Copies of this document are available online at http://quicksearch.dla.mil/.)

2.2.2 <u>Other Government documents, drawings, and publications.</u> The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

THE NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY (NIST)

NIST Special Publication 960-1	-	Particle Size Characterization NIS	
		Recommended Practice Guide	

(Copies of this document are available online at <u>http://www.nist.gov/publication-portal.cfm.</u>)

2.3 <u>Non-Government publications</u>. The following documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

Standard Test Method for Sieve Analysis of Metal Powders
Standard Practices for Sampling Metal Powders
Standard Test Method for Particle Size Distribution of
Metal Powders and Related Compounds by Light
Scattering
Standard Test Methods for Flow Rate of Metal
Powders Using the Carney Funnel
Standard Test Methods for Chemical Analysis of
Aluminum and Aluminum-Base Alloys
Standard Test Methods for Determination of Carbon,
Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel,
and Cobalt Alloys by Various Combustion and Fusion
Techniques
Standard Test Method for Compositional Analysis by
Thermogravimetry

(Copies of these documents are available from <u>www.astm.org</u>)

NATIONAL FIRE PROTECTION ASSOCIATION (NFPA)

NFPA 484 - Standard for Combustible Metals

(Copies of these documents are available from http://www.nfpa.org/)

2.4 <u>Order of precedence</u>. Unless otherwise noted herein or in the contract, in the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 <u>First article</u>. When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.4.

3.2 <u>Material.</u> The aluminum and aluminum-alloy powders shall meet the requirements cited in 3.2.1 thru 3.5 of this specification.

3.2.1 <u>Chemical composition</u>. The chemical composition of the powders, excluding oxygen, shall be of the following percentages by weight (wt.) from Table I when determined in accordance with 4.7.1.

EI EMENT	WT. %	WT. %	WT. %	WT. %
	Class A	Class B	Class C	Class D
Chromium (Cr)			0.04 - 0.35	0.05 - 0.20
Copper (Cu)	0.05 max	0.01 max	0.15 - 0.40	0.10 max
Iron (Fe)	0.2 max	0.01 max	0.70 max.	0.40 max
Magnesium (Mg)			0.8 - 1.2	4.5 -5.6
Manganese (Mn)	0.05 max	0.005 max	0.15 max.	0.05 - 0.20
Zinc (Zn)			0.25 max.	0.10 max
Silicon (Si)	0.1 max		0.40 - 0.80	0.30 max
Titanium (Ti)	0.05 max		0.15 max.	
Other, max. Each	0.05 max	0.005 max	0.05 max.	0.05 max.
Other, max. Total	0.15 max	0.015 max	0.15 max.	0.15 max.
Aluminum (Al)	99.4 min	99.95 min	Balance	Balance

 TABLE I.
 Chemical composition of powders (wt. %).

3.2.2 <u>Non-metallic impurities</u>. For all classes the total moisture and volatiles, loss on ignition shall not exceed 0.1 wt. % in accordance with 4.7.2.1. Oxygen, present as Al_2O_3 , shall not exceed 0.8 wt. % as measured in accordance with 4.7.2.2.

3.3 <u>Particle size distribution</u>. The size distribution for all powders shall be as specified in Table II as measured in accordance with 4.7.3 or as specified in the contract or purchase order (see 6.2).

3.4 <u>Quality</u>. All powders, as received by the purchaser, shall be thoroughly blended, dry, free flowing and free from foreign materials, clumps and individual particles as determined in accordance with 4.7.4.

3.4.1 <u>Agglomerates</u>. All powders, as received by the purchaser, shall be free of agglomerates exceeding 120µm in size as measured in accordance with 4.7.4.1.

3.4.2 <u>Flowability</u>. The flowability of all the powders through a Carney Funnel shall be greater than 1.0 g/s as measured in accordance with 4.7.5.

TABLE II. Particle size distribution (wt. %) by sieve or light scattering analysis.

Particle Size	% by mass	
(µm)	Min	Max
106	100	
90	99	
53	75	90
38	45	65
20	2	15
10		1

3.5 <u>Manufacturing Process</u>. The manufacturing processes for the production of the aluminum powders shall be as specified in the contract or purchase order (see 6.2) and shall produce a powder that meets or exceeds the requirements of 3.2, 3.3, and 3.4.

4. VERIFICATION

4.1 <u>Classification of inspections</u>. The inspection requirements specified herein are classified as follows:

- a. First article inspection (see 4.4).
- b. Conformance inspection (see 4.5).

4.2 <u>Testing responsibility and facilities.</u> Unless otherwise specified in the contract or purchase order (see 6.2), the contractor is responsible for the performance of all the requirement tests as specified herein. Unless otherwise specified in the contract or purchase order (see 6.2), the contractor may use his own or any other facilities suitable for the performance of the requirement tests specified herein, unless disapproved by the Government. The Government reserves the right to perform or check any of the inspections set forth in this specification where such inspections are

deemed necessary to assure supplies and services conform to prescribed requirements and to determine the validity of the certifications.

4.3 <u>Inspection lot</u>. A lot of material shall be defined as the amount produced of the same composition or composition blend, the same particle size, or as specified in Table II, and the same condition and manufactured at the same time from the same batches of basic materials by the same manufacturing process, and submitted for vendor's inspection at the same time.

4.4 <u>First article inspection</u>. When required (see 6.2), the first article samples submitted in accordance with 3.1 shall be examined for all the provisions of this specification applicable to end item examination and shall utilize the same requirements and test methods as the production acceptance inspection shown in 4.5.

4.4.1 First article tests. First article tests shall consist of all the tests specified in 4.7.

4.5 <u>Conformance inspection</u>. The tests under 4.7 shall serve as a basis for the acceptance of individual production lots.

4.6 <u>Sampling</u>. Test samples shall be taken from the master lot in accordance with ASTM B215, Practice B. Sufficient powder shall be taken from each inspection unit to perform at least three (3) evaluations for each test.

4.7 Test methods.

4.7.1 <u>Chemical composition.</u> Samples for chemical analysis (see 3.2.1) shall be prepared and tested in accordance with ASTM E34, or by one of the following methods; Inductively Coupled Plasma - Optical Emission Spectrometry (ICP-OES) or Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) or as specified in the contract or purchase order (see 6.2).

4.7.2 Non-metallic impurities.

4.7.2.1 <u>Moisture and volatiles</u>. The wt. % of the non-metallic impurities moisture and volatiles (see 3.2.2) shall be measured by the thermogravimetric method in accordance with ASTM E1131. For this test only inert gas purge shall be used, and the maximum temperature shall be 500° C.

4.7.2.2 <u>Oxygen content</u>. Oxygen content (see 3.2.2) shall be determined by inert gas fusion, as described in ASTM E1019 or as specified in the contract or purchase order (see 6.2).

4.7.3 <u>Particle size distribution</u>. Powder (see 3.3) shall be tested in accordance with ASTM B214 (sieve analysis) or ASTM B822 (light scattering) or as specified in the contract or purchase order (see 6.2).

4.7.4 <u>Quality</u>. The requirements specified in 3.4 shall be determined by visual inspection or as specified in the contract or purchase order (see 6.2).

4.7.4.1 <u>Agglomerates</u>. The size of the agglomerates (see 3.4.1) shall be measured in accordance with guidelines found in National Institute of Standards and Technology Special Publication 960-1, Section 5, Size Characterization by Microscopy-Based Techniques, unless otherwise specified in the contract or purchase order (see 6.2). Optical or electronic microscopes may be used. The dry powder sample preparation technique shall be used. The sizes of all particles and agglomerates within a square perimeter shall be characterized, where the perimeter contains at least 300 particles. The longest dimension of each individual particle and agglomerate of particles shall be less than 120 microns. As an alternative to the microscopy-based technique, a sieve analysis can be used. If a sieve analysis is used then a 99.5% of a 100 gram sample of the powder shall pass through a 140 mesh U.S. standard sieve.

4.7.5 <u>Flowability</u>. The flow of particles (see 3.4.2) through a feeder and spray gun shall be determined by means of a Carney Funnel, in accordance with ASTM B964, either Method 1 or 2 or as specified in the contract or purchase order (see 6.2). If necessary, the powder may be dried prior to performing the flow test. Drying can be accomplished by heating to greater than 200°C in an inert atmosphere for one hour and subsequently cooling to room temperature within the atmosphere.

5. PACKAGING

5.1 <u>Packaging.</u> For acquisition purposes, the packaging and packing requirements shall be as specified in the contract or order (see 6.2). However, if no requirements are specified in the contract or purchase order the packing of the powder shall be sealed within an inert atmosphere in electrically conductive plastic pouches in accordance with NFPA 484 (Sec. 12.4.1.4 - 12.4.1.6). Individual filled pouches shall not weigh more than 20 pounds. The pouches shall in turn be packaged within containers of sufficient quality to preserve the integrity of the contents against the normal and expected hazards of transportation. These filled containers shall not weigh more than 100 pounds. When packaging of materiel components are to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging activities within the Military Service or Defense Agency, or within the military service's system commands. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

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

6.1 <u>Intended use</u>. The powders specified herein are intended to be used with the Cold Spray process as specified in MIL-STD-3021, entitled: "Materials Deposition, Cold Spray". This standard (MIL-STD-3021) establishes the manufacturing controls for aluminum-based powders that are utilized for the 'Cold Spray' Process for repairing, coating, and the fabrication of freestanding structures, such as, Additive Manufacturing purposes. Cold Spray is a low-temperature, solid state consolidation process, whereby metal or combinations of metallic and

non-metallic particles are injected into a high-velocity gas stream and are directed upon a suitable substrate where they impact and consolidate to form a coating or freestanding structure, without melting. This specification (MIL-DTL-32495) will outline the requirements for chemical composition, particle size and distribution for aluminum-based powders that are utilized by the Cold Spray Process to; (1) produce aluminum and/or aluminum alloy coatings on metallic and/or nonmetallic substrates including but not limited to steel, aluminum, ceramics and polymers, (2) produce deposits for dimensional restoration on metallic and/or nonmetallic substrates, or (3) produce near net shaped parts. The intended use for each class is as follows:

Class A: Commercially Pure (CP) Aluminum powder is intended to be used to produce sacrificial coatings with 0.5 WT. % trace elements and for applications where strength is not a critical requirement.

Class B: High Purity (HP) Aluminum powder is intended to be used to produce sacrificial coatings with 0.05 WT. % trace elements and for applications where strength is not a critical requirement.

Class C: Aluminum Alloy 6061 powder is intended to be used to produce coatings that have increased strength and hardness and to provide a material that is compatible with 6061 aluminum alloy substrates and for applications where increased strength and hardness are required. Cold spray 6061 aluminum materials have the capacity of achieving an ultimate tensile strength and hardness greater than or equal to 6061 wrought aluminum heat treated to the T-6 condition.

Class D: Aluminum alloy 5056 powder is intended to be used to produce coatings that have increased strength and hardness and to provide a material that is compatible with 5056 aluminum alloy substrates and for applications where increased strength and hardness are required. Cold spray 5056 aluminum materials have the capacity of achieving an ultimate tensile strength and hardness greater than or equal to 5056 wrought aluminum in the fully-worked condition.

- 6.2 <u>Acquisition requirements</u>. Acquisition documents should specify the following:
 - (a) Title, number and date of this specification.
 - (b) Specify class of powders (see 1.2)
 - (c) When first article is required (see 3.1 and 4.4).
 - (d) Acceptable particle size distribution and method to use in determining particle size distribution (see 3.3).
 - (e) Process to use for the manufacturing of the powders (see 3.5).
 - (f) If other than the contractor is responsible for the performance of all the required tests (see 4.2).
 - (g) If the contractor can't use his own facility or any other facility for testing (see 4.2).
 - (h) If the testing and preparation for chemical analysis is different (see 4.7.1).
 - (i) If the testing and preparation for non-metallic impurities is different (see 4.7.2).
 - (j) If particle size test technique is different (see 4.7.3).
 - (k) If the method for determining quality is different (see 4.7.4).
 - (l) If the method for determining size of the agglomerates is different (see 4.7.4.1).

- (m) If the method for determining the flow of particles is different (see 4.7.5).
- (n) Packaging and packing requirements if different (see 5.1).
- (o) Specify shelf life requirements (see 6.3).

6.3 <u>Shelf-life</u>. This specification covers items where the assignment of a Federal shelf-life code is a consideration. Specific shelf-life requirements should be specified in the contract or purchase order, and should include, as a minimum, shelf-life code, shelf-life package markings in accordance with MIL-STD-129 or FED-STD-123, preparation of a materiel quality storage standard for type II (extendible) shelf-life items, and a minimum of 85 percent shelf-life remaining at time of receipt by the Government. These and other requirements, if necessary, are in DoD 4140.27-M, Shelf-life Management Manual. The shelf-life codes are in the Federal Logistics Information System Total Item Record. Additive information for shelf-life management may be obtained from DoD 4140.27-M, or the designated shelf-life Points of Contact (POC). The POC should be contacted in the following order:

- (1) The Inventory Control Points that manage the item and
- (2) The DoD Service and Agency administrators for the DoD Shelf-Life Program.

Appropriate POCs for the DoD Shelf-Life Program can be contacted through the DoD Shelf-Life Management website: <u>https://www.shelflife.hq.dla.mil/</u>

6.4 Subject term (key word) listing.

Adhesion Cohesion Cold gas dynamic spray High velocity powder deposition Impaction process Kinetic energy Kinetic metallization Kinetic spraying Laser diffraction Particles Propelling gas Thermal spray

6.5 <u>Changes from previous issue</u>. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

CONCLUDING MATERIAL

Custodians: Army – MR Navy - AS Air Force - 20 Preparing activity: ARMY – MR (Project MFFP-2014-010)

Review activities: Army – AV, PT Navy - EC, MC, SH Air Force - 19, 84, 99 DLA – DH, GS4

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at <u>https://assist.dla.mil/</u>.