

METRIC

**MIL-STD-3021
w/CHANGE 2
4 March 2015
SUPERSEDING
MIL-STD-3021
w/CHANGE 1
13 July 2011**

DEPARTMENT OF DEFENSE MANUFACTURING PROCESS STANDARD

MATERIALS DEPOSITION, COLD SPRAY



AMSC N/A

AREA MFFP

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

FOREWORD

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

2. The purpose of this standard is to develop the manufacturing process controls for a cold spray operation utilizing a high-velocity jet of solid-phase particles. The jet temperatures are below the melting thresholds of many engineering materials. This allows the process to be used to apply deposits on a wide variety of substrates, such as, alloys, ceramics, and plastics, as well as to produce bulk materials and near-net shaped parts. The uniqueness of cold spray lies in its ability to produce a coating, provide dimensional restoration or even produce a near-net shaped part at temperatures well below the melting point of the powders being applied, thereby avoiding or minimizing many deleterious high temperature reactions which are characteristics of typical thermal spray processes. This ensures that the input powder materials do not experience any grain growth, oxidation or phase changes when deposited. It is this characteristic of cold spray that makes it attractive as a method for coatings or for dimensional restoration, and/or the production of bulk materials or near-net shaped, while retaining their own unique material properties.

3. This standard is required because the cold spray deposition process has significant differences from thermal spray coating technologies such as high velocity oxy-fuel (HVOF), detonation gun, plasma spray, flame spray, and arc spray. These process differences result in application criteria, process equipment, and operating parameters that are considerably different than previously documented processes. The objective is to enable the application of Cold Spray Deposition with the success of the aforementioned processes when properly applied.

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

MIL-STD-3021
w/Change 2

SUMMARY OF CHANGE NOTICE 2 MODIFICATIONS

The following modifications to MIL-STD-3021 Change Notice 1 have been made:

<u>Paragraph</u>	<u>Modification</u>		<u>Paragraph</u>	<u>Modification</u>		<u>Paragraph</u>	<u>Modification</u>
ii	Changed		4.2.1	Changed		4.3.2.3	Deleted
1.1	Changed		4.2.1.1	Changed		4.3.3	Deleted
1.2	Changed		4.2.1.2	Added		4.3.3.1	Deleted
1.2.1	Deleted		Table I	Changed		4.3.4	Deleted
1.3	Changed		4.2.2	Changed		4.3.5	Deleted
2.1	Changed		4.2.2.1	Changed		4.3.6	Deleted
2.2	Deleted / renumbered		4.2.2.1.1	Deleted		4.4	Deleted
2.2.1	Changed		4.2.2.2	Changed		4.4.1	Deleted
2.3	Deleted / renumbered		4.2.2.2.1	Added		4.3.1.1	Deleted
3.1	Changed		4.2.2.2.2	Added		5.1.1	Changed
3.1.1	Added		4.2.2.3	Added		5.1.3.1	Changed
Figure 1	Changed		Figure 3	Changed		5.1.3.2	Added
Figure 2	Changed		4.2.3	Changed		5.1.3.3	Changed
3.11	Added		4.2.3.1	Added		5.2.2	Changed
3.12	Added		4.2.5	Changed		5.2.3	Changed
3.13	Added		4.2.6	Added		5.2.4	Changed
3.14	Added		4.3	Changed		5.2.5	Added
3.15	Added		4.3.1	Changed		6.2	Changed
4.1	Changed		4.3.1.1	Added		6.3	Changed
4.1.1	Added		4.3.1.2	Added		6.3.1	Added
4.1.2	Added		4.3.2	Deleted		6.4	Changed
4.1.2.1	Added		4.3.2.1	Deleted		6.4.1	Deleted
4.1.2.1.1	Added		4.3.2.2	Deleted		6.5	Changed
4.1.2.2	Added		4.3.2.2.1	Deleted		6.6	Deleted
4.2	Changed		4.3.2.2.2	Deleted		APPENDIX A	Added

CONTENTS

<u>PARAGRAPH</u>	<u>PAGE</u>
<u>FOREWORD</u>	ii
1. <u>SCOPE</u>	
1.1 <u>Purpose</u>	1
1.2 <u>Process</u>	1
1.3 <u>Applications</u>	1
2. <u>APPLICABLE DOCUMENTS</u>	
2.1 <u>General</u>	2
2.2 <u>Non-Government publications</u>	2
2.3 <u>Order of precedence</u>	2
3. <u>DEFINITIONS</u>	
3.1 <u>Cold spray</u>	2
3.1.1 <u>Types</u>	3
3.2 <u>Nozzle</u>	3
3.3 <u>Powder lot</u>	3
3.4 <u>Simulated part</u>	4
3.5 <u>Substrate</u>	4
3.6 <u>Lot</u>	4
3.7 <u>Pass</u>	4
3.8 <u>Increment</u>	4
3.9 <u>Layer</u>	4
3.10 <u>Thermal spraying</u>	4
3.11 <u>Non structural applications</u>	4
3.12 <u>Structural applications</u>	4
3.13 <u>Cognizant Engineering Authority (CEA)</u>	4
3.14 <u>Acquisition Authority</u>	4
3.15 <u>Powder Specification</u>	4
4. <u>GENERAL REQUIREMENTS</u>	
4.1 <u>Process</u>	4
4.1.1 <u>Cold spray equipment</u>	5
4.1.2 <u>Material</u>	5
4.1.2.1 <u>Gas</u>	5
4.1.2.1.1 <u>Gas specifications</u>	5
4.1.2.2 <u>Coating powder</u>	5
4.2 <u>Required procedures and operations</u>	5
4.2.1 <u>Process control</u>	5
4.2.1.1 <u>Gauges, meters, and sensors</u>	6

CONTENTS

<u>PARAGRAPH</u>		<u>PAGE</u>
4.2.1.2	<u>Checks</u>	6
4.2.2	<u>Surface preparation</u>	6
4.2.2.1	<u>Cleaning</u>	6
4.2.2.2	<u>Handling and storage</u>	6
4.2.2.2.1	<u>Handling</u>	6
4.2.2.2.2	<u>Storage</u>	6
4.2.2.3	<u>Overspray protection</u>	8
4.2.3	<u>Abrasive blast</u>	8
4.2.3.1	<u>Blast contamination</u>	8
4.2.4	<u>Preheating</u>	8
4.2.5	<u>Cold spray deposition</u>	8
4.2.6	<u>Identification</u>	8
4.3	<u>Operator certification</u>	8
4.3.1	<u>Cold spray operator</u>	8
4.3.1.1	<u>Training</u>	9
4.3.1.2	<u>Continuance of certification</u>	9
5.	<u>DETAILED REQUIREMENTS</u>	
5.1	<u>General</u>	9
5.1.1	<u>Certification</u>	9
5.1.1.1	<u>Certification samples</u>	9
5.1.1.2	<u>Re-certification</u>	9
5.1.2	<u>Test specimens</u>	9
5.1.2.1	<u>Metallographic test specimens</u>	10
5.1.2.1.1	<u>Test specimen panel configuration</u>	10
5.1.2.2	<u>Bond strength test specimens</u>	10
5.1.2.2.1	<u>Size</u>	10
5.1.2.2.2	<u>Material</u>	10
5.1.2.2.3	<u>Deposit</u>	10
5.1.2.2.4	<u>Deposition thickness</u>	10
5.1.3	<u>Process approvals</u>	10
5.1.3.1	<u>Processing data</u>	10
5.1.3.2	<u>First article testing</u>	10
5.1.3.3	<u>Rework</u>	11
5.1.3.3.1	<u>Stripping</u>	11
5.1.3.4	<u>Metallographic test specimen</u>	11
5.1.4	<u>Process certification</u>	11
5.2	<u>Test methods</u>	11

CONTENTS

<u>PARAGRAPH</u>		<u>PAGE</u>
5.2.1	<u>Visual</u>	11
5.2.2	<u>Microscopic examination</u>	11
5.2.3	<u>Oxides and porosity</u>	12
5.2.4	<u>Bond strength test</u>	12
5.2.5	<u>Non-destructive testing</u>	12
6.	<u>NOTES</u>	
6.1	<u>Intended use</u>	13
6.2	<u>Acquisition requirements</u>	13
6.3	<u>Safety</u>	13
6.3.1	<u>Material safety data sheets (MSDS)</u>	14
6.4	<u>English units</u>	14
6.5	<u>Subject term (key word) listing</u>	14
<u>TABLE</u>		
I.	<u>Operating parameters</u>	6
<u>FIGURE</u>		
1.	<u>High pressure powder injection system</u>	3
2.	<u>Low pressure powder injection system</u>	3
3.	<u>Typical cold spray control sheet</u>	7
<u>APPENDIX</u>		
A.	<u>Guidance for Additional Qualification Requirements for Structural Applications</u>	15
CONCLUDING MATERIAL		17

1. SCOPE

1.1 Purpose. The procedures covered by this standard are intended to ensure that cold spray operations, either manual or automated, meet prescribed requirements. This standard ensures that cold spray operations are repeatable and consistent. However, additional qualification requirements may be required based on each specific application. Therefore, for structural applications and other applications that are considered critical - (safety critical or FOD critical), a qualification plan shall be provided by the Cognizant Engineering Authority (CEA) (see 3.13) and shall follow the recommendations specified in Appendix A and shall be specified in the contract or purchase order (see 6.2).

1.2 Process. Cold spray is a process whereby powder particles are utilized to form a coating, provide dimensional restoration and/or the production of bulk, or near net shaped, materials by means of supersonic impingement upon a substrate. The powders can be metal, ceramic, and polymeric or combinations thereof, typically range in particle size from 1.0 to 100 micrometers (μm) and are accelerated by injection into a high-velocity stream of gas. The powder particles remain solid throughout the deposition process, which is the primary difference between cold spray and traditional thermal spray processes. The high velocity gas stream is generated through the expansion of a pressurized, preheated, gas through a nozzle. The nozzle may be a contracting-expanding supersonic type or a contracting sonic type. The pressurized gas is expanded in order to achieve high velocity, with an accompanying decrease in pressure and temperature. The powder particles, initially carried by a separate gas stream, are injected into the nozzle either at the nozzle entrance (High Pressure Powder Injection) or at a lower pressure point downstream of the entrance (Low Pressure Powder Injection). The particles are then accelerated by the main nozzle gas flow and are impacted onto a substrate after exiting the nozzle. The solid particles that impact the substrate above a threshold (critical) velocity for the powder and substrate combination will deform and bond in a dense layer. As the process continues, particles continue to impact and form bonds with the previously deposited material resulting in a uniform deposition with very little porosity and high bond strength.

1.3 Applications. Applications for cold spray technology often occur in situations where conventional thermal metal spray technology cannot be successfully used and where cold spray will result in an improved deposit. These situations often occur when high temperatures cannot be tolerated by the substrate. Cold spray is often used for dimensional restoration and repair of mating surfaces, but is not limited to those applications. Good corrosion protection is achieved by dense cold sprayed deposits. Wear resistant, hard surfaces, such as MCrAlYs, can be deposited by cold spray when operated at its high-temperature end. The cold spray process has been used to produce dense, pure, thick and well bonded deposits of many metals and alloys, such as aluminum (Al), copper (Cu), nickel (Ni), tantalum (Ta), titanium (Ti), silver (Ag), and zinc (Zn), as well as stainless steel, nickel-base alloys (Inconels, Hastalloys), and bond coats, such as MCrAlYs. Cold spray can produce composites, such as metal-metal like copper-tungsten (Cu-W) or copper-chromium, metal-carbides like aluminum-silicon carbide (Al-SiC), and metal-oxides like aluminum-alumina. Polymeric materials have also been deposited by the cold spray process. Cold spray has been used to produce protective coatings and performance enhancing layers, ultra thick deposits for dimensional restoration, freeform and near net shape parts. Typical protective coatings produced by cold spray include MCrAlY coatings for high temperature protection and bond coats

MIL-STD-3021
w/Change 2

for thermal barriers, copper-chrome layers for oxidation protection, and corrosion resistant aluminum and zinc coatings for oil and auto industries and others.

2. APPLICABLE DOCUMENTS

2.1 General. 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 Non-Government publications. The following documents 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.

SAE INTERNATIONAL

SAE-AMS 2750 - Pyrometry

(Copies of this document are available from www.sae.org or SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001)

ASTM INTERNATIONAL

ASTM C633 - Standard Test Method for Adhesion or Cohesion
Strength of Thermal Spray Coatings.
ASTM E3 - Standard Guide for Preparation of Metallographic
Specimens.
ASTM E2109 - Standard Test Methods for Determining Area Percentage
Porosity in Thermal Sprayed Coatings.

(Copies of these documents are available from www.astm.org or ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.)

2.3 Order of precedence. 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. DEFINITIONS

3.1 Cold spray. Cold spray is a materials deposition process in which relatively small particles (ranging in size from approximately 1 to 100 micrometers (µm) in diameter) in the solid state are accelerated to high velocities (typically 300 to 1200 meters/second), and subsequently adhere to an appropriate substrate. Various terms—including “kinetic energy metallization,” “kinetic metallization,” “kinetic spraying,” “high-velocity powder deposition,” and “cold gas-dynamic spray method”—have been used to refer to this technique. In most instances, deformable

powder particles in a gas carrier are brought to high velocities through introduction into a nozzle, designed to accelerate the gas. The subsequent high-velocity impact of the particles onto the substrate disrupts the oxide films on the particle and substrate surfaces, pressing their atomic structures into intimate contact with one another under momentarily high interfacial pressures and temperatures.

3.1.1 Types. The two principal cold spray system configurations are depicted by Figures 1 and 2. The two configurations differ primarily in gas pressure, and powder injection location. Figure 1 shows a High Pressure Powder Injection System in which the main gas stream and the powder stream are both introduced into a mixing chamber, which can be separate from or part of, the inlet chamber of the nozzle. This configuration requires that the powder feeder be capable of high gas pressure. Figure 2 shows a Low Pressure Powder Injection System in which the powder stream is injected into the nozzle at a point where the gas has expanded to low pressure. Both systems can utilize compressed air, nitrogen and helium, but can utilize other inert gases as well.

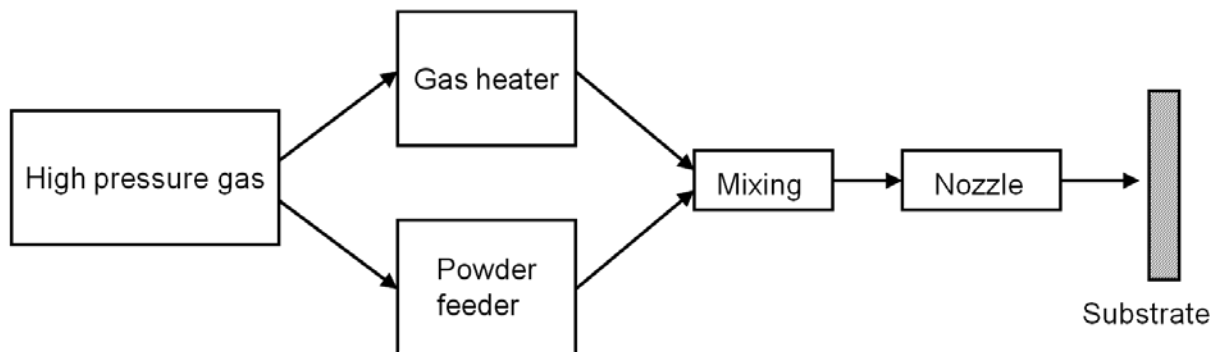


FIGURE 1. High Pressure Powder Injection System.

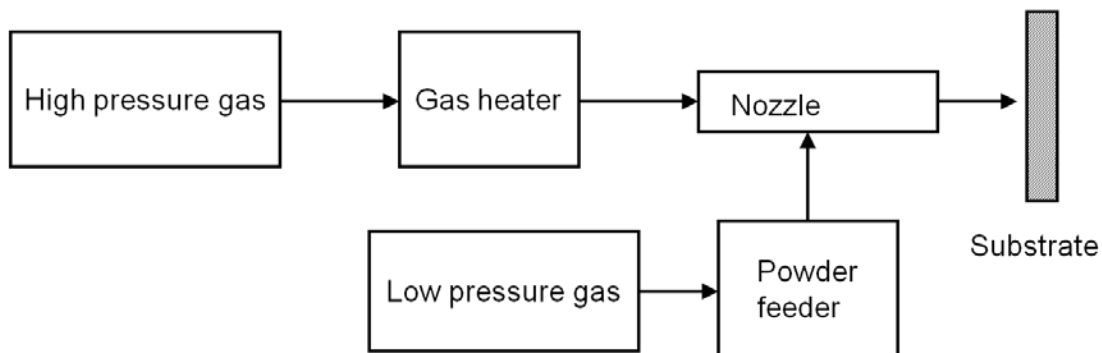


FIGURE 2. Low Pressure Powder Injection System.

3.2 Nozzle. A gas manifold designed to accelerate a gas to a velocity greater than Mach 1.

3.3 Powder lot. A powder lot is all the powder of a specified type manufactured at the same time by the same manufacturer.

3.4 Simulated part. A simulated part is a test piece or section with a similar configuration (surface condition, access, thickness, etc.) to the part it represents. The simulated part will be approved by the acquisition authority.

3.5 Substrate. The material, work piece or substance on which a coating is deposited.

3.6 Lot. A lot is all the parts of a similar configuration, coated sequentially on the same machine setup using the same batch of coating material and process parameters, within a shift or eight hours of operation, and presented for processor's inspection at one time. Any critical secondary processes, such as heat treatment, must be done at the same time for the components to be in the same lot.

3.7 Pass. A single traverse by the nozzle over the work piece.

3.8 Increment. The distance between adjacent passes (also called step size).

3.9 Layer. Multiple passes over the work piece that result in complete coverage. Successive layers result in increased deposit thickness.

3.10 Thermal spraying. A group of processes wherein metallic or nonmetallic materials are deposited in a molten or semi-molten condition to form a deposit. The feed material may initially be in the form of powder, ceramic rod, or wire.

3.11 Non structural applications. For applications where the cold spray deposit is not a significant load-bearing member and where failure or disbonds do not cause significant damage or reduction in service life. An example of this is a metallic coating.

3.12 Structural applications. For applications where the cold spray deposit is a significant load-bearing member or where failure or disbonds could cause significant damage or reduction in service life.

3.13 Cognizant Engineering Authority (CEA). The CEA is the member of the Procuring Activity (PA) that provides the technical input for the contract or purchase agreement.

3.14 Acquisition Authority. The Acquisition Authority is the activity that prepares the contract or purchase agreement and is normally considered to be the Procuring Activity (PA).

3.15 Powder Specification. A document that describes a specific powder (such as MIL-DTL-32495) and lists all the properties required to identify and distinguish it from other powders.

4. GENERAL REQUIREMENTS

4.1 Process. The process utilizes nitrogen, which has been compressed to propel metal powder onto the surface of a substrate. Alternatively, compressed air or helium may be used. The single layer deposition thickness produced by a moving nozzle can vary from 0.01 to 1.0 millimeter

(mm); depending on powder feed rate, nozzle traverse speed, and deposition efficiency. The cold spray nozzle is frequently handled by a robot. Multiple coating layers can result in deposits several centimeters thick. The width of a single pass can be between 2 and 12 mm, depending on nozzle design, and large surfaces can be coated through multiple, slightly overlapping, parallel passes.

4.1.1 Cold spray equipment. The process gas is introduced to a manifold system containing a gas heater and powder-metering device. The pressurized gas is heated to a preset temperature, often using a coil of an electrical resistance-heated tube. The gas is heated not to heat or soften spray particles, but instead to achieve higher flow velocities, which ultimately result in higher particle impact velocities. The high-pressure gas is introduced into the entrance of a nozzle (converging/diverging or converging only), where the gas accelerates to a high supersonic velocity as it expands in the nozzle. The gas cools as it expands in the spray nozzle, sometimes exiting the spray gun at below ambient temperature. The powder to be deposited is introduced by a separate gas stream either at the nozzle entrance or at a lower pressure location on the nozzle, where the powder mixes with the main gas stream and is accelerated by the gas stream.

4.1.2 Material.

4.1.2.1 Gas. The preferred gas is an inert gas or a mixture of inert gases. The concentration of oxygen, if any, should be kept to a minimum to avoid oxidation within the deposit.

4.1.2.1.1 Gas specifications. Gas specifications shall be acceptable to the cognizant engineering authority and shall be specified in the contract or purchase order (see (6.2)).

4.1.2.2 Feed stock powder. The powder shall be dry, free flowing, and thoroughly blended. Mixtures of powder stock with varying density and/or size particulates shall be kept from settling or stratifying in the feeder as long as that powder charge is utilized. The cold spray provider shall be cautioned to the importance of paying attention to the manufacturer's instructions pertaining to the storage and handling of finely divided metal powders. The powder specification shall be specified in the contract or purchase order (see (6.2)).

4.2 Required procedures and operations.

4.2.1 Process control. The cold spray process is optimized through the adjustment of critical process parameters. The critical parameters shall be tailored to the application and shall include, as a minimum, the part preparation, coating powder and coating data listed in Figure 3. Operational parameters are typically selected to achieve the most suitable deposit for the intended application at the lowest operational temperatures. The range of operation for the High Pressure Powder Injection System and the Low Pressure Powder Injection System is provided in Table I. The values in the table are representative of values currently in use but are not necessarily limiting values for the systems. A distinguishing feature of the cold spray process compared with conventional thermal spray processes is its ability to produce deposits with preheated gas temperatures below the melting temperature of the coating particle materials. Any change to a controlled parameter requires recertification, see 5.1.

TABLE I. Typical operating parameters.

	High Pressure Powder Injection System	Low Pressure Powder Injection System
Working gas	N ₂ , He, air	N ₂ , He, air
Gas pressure, MPa	0.5 – 7.0	0.5 – 3.5
Gas preheat, °C	20 - 1200	20 - 600
Gas flow rate, m ³ /hour	10 - 300	10 - 100
Maximum Gas Mach #	1 - 4	1 - 3
Powder flow rate, g/s	0.1 – 3.0	0.1 – 1.0
Particle size, μm	5 - 100	5 - 50

4.2.1.1 Gauges, meters, and sensors. Minimum accuracy on these instruments shall be defined in the contract or purchase order (see (6.2)).

4.2.1.2 Checks. Process control procedures shall be checked during cold spraying to assure that the specified operating parameters on the control sheet are maintained (see Figure 3 for minimum requirements). The nozzle shall be checked between runs for internal wear / deposition. The operator shall determine if the nozzle needs to be replaced and/or cleaned.

4.2.2 Surface preparation.

4.2.2.1 Cleaning. Surfaces to receive deposits shall be thoroughly cleaned to remove oil, grease, dirt, paint, oxides and other foreign material. Final cleaning shall take place no more than 4 hours prior to coating. Cleaning procedures shall not cause embrittlement, pitting, or any damage to the surfaces that are to be coated.

4.2.2.2 Handling and storage.

4.2.2.2.1 Handling. All surfaces requiring cold spraying shall be handled with clean lint-free gloves, tongs, or other means that will avoid surface contamination.

4.2.2.2.2 Storage. If a delay in spraying occurs beyond 4 hours but less than 20 hours (except for magnesium which shall be limited to 8 hours), special measures shall be employed to protect the surface to be coated from dust, dirt, moisture, and other contaminants such as flash rust or excessive oxidation that would reduce adhesion of the cold-sprayed deposit. Protection shall be in the form of covering or inserting the parts in clean plastic bags, storing parts in a moderate temperature oven maintained at approximately $95^{\circ} \pm 3^{\circ}$ C or in a vacuum chamber under low pressure. Should the delay in spraying, after proper surface preparation, exceed 20 hours the parts shall be reprocessed in accordance with 4.2.2.1.

MIL-STD-3021
w/Change 2

Vendor:		Sheet ____ of ____
Vendor Process #:		CEA:
Purchase Order Number:		
Part Number:		S/N:
Area to receive deposition:		
Cold Spray (CS) Manufacturer:		
CS System		Nozzle:
PART PREPARATION		
Base Material Type:		
Heat Treat Condition :		
Thickness:		
Surface Condition (clad, anodize, shot peened, etc.):		
Surface Roughness (if different than base):”		
Method of Cleaning:		
Masking Information:		
Grit Type and Size:		
Grit Blast Pressure (MPa) : <u>±</u> Suction: Pressure :		
COATING POWDER		
Powder material:	Powder size:	Material Lot # :
Supplier:	Powder material specification:	
COATING DATA		
Gas (1) Primary:		Temperature Deg. C <u> + </u>
Secondary Gas Type:		Temperature Deg. C <u> + </u>
Main Gas Pressure MPa <u> + </u>		Main Gas Flow (1) m ³ /hour. <u> + </u>
Feeder Gas Pressure MPa <u> + </u>		Feeder Gas Flow (2) m ³ /hour. <u> + </u>
Elapsed Time Between Surface Prep and Spraying:		
Powder Feed Rate (kg/hr):		
Nozzle to Work Distance:		
Deposition Angle:		
Notes:		
Traverse rate (mm/s): _____ Increment (mm): _____		Preheat Temp:
Deposition Thickness as Sprayed:		Method of Preheat:
Number of Passes Per Layer:		Number of Layers:
Hand-Held Gun: YES NO		Robotically Manipulated Gun: YES NO
Method of Cooling (if any):		
Notes:		
Certification #:		
Approval:		

FIGURE 3. Typical cold spray control sheet.

MIL-STD-3021
w/Change 2

4.2.2.3 Overspray protection. Areas adjacent to the area to be cold spray coated shall be suitably protected from overspray by masking or shielding.

4.2.3 Abrasive blast. Abrasive blasting used to provide an anchor tooth profile is commonly performed prior to applying thermal spray coatings but is not always recommended prior to cold spray. Certain materials require only oxide removal and others require virgin abrasive blast media. Care should be taken when performing abrasive blasting to avoid embedded grit in the substrate. If abrasive blast media is used by the manufacturer's process, it shall be specified in the contract or purchase order (see (6.2)).

4.2.3.1 Blast contamination. All blast media shall be free of contamination that will affect the base material, such as, utilizing a blasting cabinet that has been used for ferrous substrates in the past and is now being used for nonferrous substrates such as aluminum or magnesium.

4.2.4 Preheating. When required and as specified in the contract or purchase order (see 6.2), surfaces shall be preheated by a suitable and controllable source. The following shall apply:

- a. Preheating is performed to remove moisture and minimize the thermal shock effect encountered during deposition. Preheating of the substrate is also known to improve deposition efficiency and bond strength as well.
- b. Temperature of the part, during the preheating and coating application, shall be controlled to prevent discoloration, oxidation, distortion and other conditions detrimental to the coating or substrate.
- c. Temperature of the part after pre-heat and prior to spraying shall be measured using measurement sensors calibrated in accordance with SAE-AMS 2750.

CAUTION: Special care must be taken to avoid overheating alloys with low melting temperatures such as aluminum and magnesium or with heat treatment temperatures lower than gas temperatures.

4.2.5 Cold spray deposition. The cold spray material shall be deposited on the designated surfaces to a sufficient thickness to provide, after subsequent operations, a finished composition and thickness which will meet the engineering specifications.

4.2.6 Identification. Unless otherwise specified in the contract or purchase order (see 6.2), each coated part shall be identified by applying the symbol CS as a prefix to the serial number (e.g., CS S/N 1234) or as a prefix to the Federal manufacturer code on non-serialized parts. The prefix shall be applied in the same manner as specified on the engineering drawing for the part number unless otherwise specified in the contract or purchase order (see 6.2). In the event that available space precludes application of the symbol as a prefix, the symbol shall be applied as closely as possible to the serial number or Federal manufacturer code. In no case shall the symbol be applied as a suffix to the serial number or Federal manufactures code.

4.3 Operator qualification.

4.3.1 Cold spray operator. The cold spray operator shall demonstrate proficiency in the operation of the cold spray equipment and in the performance of other related items by successfully

preparing and coating a set of qualification test panels and specimens as described in 5.1.2 through 5.1.2.2.4. In the event an operator fails the test, the operator shall be permitted to submit three more samples of the qualification test panels or specimens for the specific failed test. If any of the second set fails to conform to the specified acceptance requirements, the operator shall be disqualified. If reexamination is still desired after failing to pass the second set the operator shall be required to submit a complete series of qualification test panels and specimens as specified in 5.1.2 through 5.1.2.2.4 after the operator receives the necessary training in the process prior to being retested. A cold spray operator of semiautomatic or automatic equipment shall meet all the requirements of a manual spray operator.

4.3.1.1 Training. For operators applying deposits for structural applications, formalized training shall be documented and retesting shall be required in the event of more than 3 months without use of the equipment or anytime quality standards are failed. Training programs shall be approved by the CEA.

4.3.1.2 Continuance of certification. The certification status of a cold spray operator may be maintained by a continuous record of satisfactory proficiency or by the annual successful preparation and coating of a set of test panels and specimens as described in 5.1.2. Re-certification of a cold spray operator shall be required when there is evidence of a lack of proficiency.

5. DETAILED REQUIREMENTS

5.1 General.

5.1.1 Certification. To ensure supplier capability to consistently deliver a satisfactory product, the supplier facilities, training and general procedures shall be certified by the CEA before parts for production are supplied unless otherwise specified in the contract or purchase order (see 6.2). The certification requirements shall be as required in 4.3 as a minimum. Additional requirements shall be provided by the CEA prior to process development and as specified in the contract or purchase order (see 6.2).

5.1.1.1 Certification samples. The supplier shall submit test samples as specified in 5.1.2, together with the sample parts required for examination by the acquisition authority. The supplier shall apply the cold spray to the base metal combination as specified by the acquisition authority. Sample parts submitted shall be prepared in accordance with 5.1.3.2.

5.1.1.2 Re-certification. In the event a supplier has not supplied cold sprayed parts or test specimens for over 1 year, the supplier shall be required to re-certify in a manner equivalent to the original certification unless otherwise specified in the contract or purchase order (see 6.2).

5.1.2 Test specimens. A minimum of three test specimen panel for metallographic microscopic examination and three test specimen panels for bond strength testing shall be cold sprayed. The microscopic examination specimens and bond strength specimens shall be fabricated from the same materials as the parts to receive the cold spray or as specified in the contract or purchase order (see 6.2).

MIL-STD-3021
w/Change 2

5.1.2.1 Metallographic test specimens. Test specimen panels to be used for microscopic examination shall be prepared as follows:

- a. Size: Approximately 40 x 40 x 10 millimeters.
- b. Material: Same as parts to receive the cold spray or as specified by the acquisition authority in the contract or purchase order (see 6.2).
- c. Deposition Material: Specified by acquisition authority in the contract or purchase order (see 6.2).
- d. Deposition thickness: 0.2 mm minimum unless otherwise specified in the contract or purchase order (see 6.2).

5.1.2.1.1 Test specimen panel configuration. Specimen configuration shall be specified in the contract or purchase order (see 6.2) and shall require the approval of the procuring activity.

5.1.2.2 Bond strength test specimens. The contract or purchase order (see 6.2) shall specify the pass/fail criteria for an acceptable bond strength for the specific application. The bond strength test specimen panels shall be prepared as follows:

5.1.2.2.1 Size. A minimum of three test specimen panels shall be prepared to the dimensions as specified in ASTM C633. Other specimen configurations may be used providing written permission is obtained from the acquisition authority, unless otherwise specified in the contract or purchase order (see 6.2).

5.1.2.2.2 Material. The substrate fixture shall be prepared using the alloy as selected in 5.1.2. The loading fixture can be any suitable material.

NOTE: The loading fixture shall be suitably identified (e.g., slotted along the outside diameter).

5.1.2.2.3 Deposit. The deposit (coating material) shall be as specified in the contract or purchase order (see 6.2).

5.1.2.2.4 Deposition thickness. The deposition thickness shall be at least 0.4 millimeters, unless otherwise specified in the contract or purchase order (see 6.2).

5.1.3 Process approvals.

5.1.3.1 Processing data. Operation sheets covering the process shall be established for each part number by the cold spray source. Figure 3 shows a typical operation sheet, the format is optional provided all pertinent information is provided. The engineering requirements as specified on the drawing shall prevail during any acceptance testing.

5.1.3.2 First article testing. The requirements for a first article test shall be processed in accordance with the procedure established in 5.1.3.1 and submitted to the acquisition authority for approval prior to production parts being cold sprayed. For structural applications a qualification plan shall be provided by the CEA and shall follow the recommendations specified in Appendix A and shall be specified in the contract or purchase order (see 6.2).

MIL-STD-3021
w/Change 2

5.1.3.3 Rework. Discrepant parts with damaged or imperfect deposits shall be reworked by a qualified source approved by the acquisition authority. The number of reworks will also be an acquisition authority decision based on degree discrepancy of and method of repair and shall be specified in the contract or purchase order (see 6.2).

5.1.3.3.1 Stripping. Reworking to completely remove the defective coating is permissible by minimally aggressive mechanical methods. Non-mechanical (chemical) stripping shall be subject to approval by the acquisition authority.

5.1.3.4 Metallographic test specimen. The test specimen panel for metallographic microscopic examination shall be prepared in accordance with 5.1.2.1 and cold sprayed along with the first production lot of parts and each subsequent lot (see 3.3) and submitted to the acquisition authority with the parts. If specified in the contract or purchase order (see 6.2), the cold spray supplier may submit photomicrographs of the coating in lieu of the test samples.

5.1.4 Process certification. Unless otherwise specified in the contract or purchase order (see 6.2), the supplier shall furnish three copies of a certificate stating the following information:

- a. Purchase order number.
- b. Part number and revision letter.
- c. Serial number of parts coated (when applicable).
- d. Cold spray lot numbers.
- e. Quantity and serial numbers of rejected parts stripped and recoated.
- f. Test results per 5.2.1 through 5.2.4.
- g. Statement of conformance to this standards latest revision.

5.2 Test methods.

5.2.1 Visual. All parts and assemblies (before and after machining) and test specimen panels shall be visually inspected to verify that cold spray deposit is adherent to the substrate material and has a uniform continuous surface free from spalling, chipping, flaking, cracking, and other objectionable imperfections.

5.2.2 Microscopic examination. A microscopic examination in the as-polished condition (no etchant) of a specimen cut perpendicular to the deposit surface shall not reveal any cracks, aligned porosity, excessive or massive oxides or porosity when examined at magnifications of 40x, 100x, and 200x in accordance with the procedures outlined in ASTM E3 and ASTM E2109. The first examination of the deposit cross-section will be performed at 40x to maximize the field of view and present a gross overview of the deposit. This will aid in defining the fields of view for the higher magnification inspection. The next examination is done at 100x and then 200x. It is also important to label each micrograph file with a marker such as a 50um bar in the lower right corner to provide a scale to the examiner. Etching is typically performed on a deposit to reveal grain size, grain boundaries and sub-structures. In accordance with ASTM E407 and the results desired, the cold spray deposit material shall determine the etchant number. The etchant composition and procedure shall be determined from the corresponding etchant number.

MIL-STD-3021
w/Change 2

5.2.3 Oxides and porosity. The CEA shall determine what the acceptable levels are for oxide and porosity and shall be specified in the contract or purchase order (see 6.2). Typically, excessive porosity is defined as greater than 5 percent at magnification up to 200x. Typically, excessive or massive oxides are defined as greater than 3 percent for deposits where oxygen is not an integral part of the material.

5.2.4 Bond strength test. Test specimen panels to be used for bond strength testing (see 5.1.2.2) shall be bonded (glued) to an uncoated specimen to form a single test specimen and the test specimen shall be tensile strength tested to verify conformance to the applicable coating material requirements as per ASTM C633. The CEA shall determine the level of frequency required for bond strength testing (i.e. every work shift) and shall be specified in the contract or purchase order (see 6.2). If not specified, the bond strength test shall be performed every shift. The test procedure is as follows:

- a. Remove masking materials.
- b. Verify that bonding end of the uncoated section of the test specimen has been cleaned, dried, and abrasive-blasted.
- c. Apply a thermosetting epoxy adhesive to the bonding ends of the coated and the uncoated sections of the test specimen.
- d. Accurately align and join the ends of the two specimens.

NOTE: A fixture may be used to facilitate bonding of the test specimen.

- e. Cure the adhesive in a hot air circulating oven at the temperature recommended by the manufacturer.
- f. After bonding, dress the edge of the coating flush with the outside diameter of the test specimen.

NOTE: An abrasive disc or wheel may be used for this purpose but care shall be taken that the disc or wheel moves parallel to the centerline of the test specimen. Care shall be taken during dressing to ensure the temperature does not increase to affect the bond adhesion.

- g. Test the bond specimen with a standard laboratory tensile tester equipped with universal joint grips for each end of the joined specimens. Use a fixture that assures uni-axial loading of the specimen. Set the no-load cross-head speed at approximately 1 millimeter per minute.

5.2.5 Non-destructive testing. The CEA shall require nondestructive testing to detect delamination or other inherent discontinuities of concern and it shall be specified in the contract or purchase order (see 6.2).

6. NOTES

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

MIL-STD-3021
w/Change 2

6.1 Intended use. This military standard is intended to ensure cold spray coating operations on parts for military components meet prescribed requirements.

6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number, and date of the standard.
- b. Specify a qualification plan for structural & critical applications (see 1.1 and 5.1.3.2).
- c. Powder and gas specification (see 4.1.2.1.1 and 4.1.2.2).
- d. If a minimum accuracy is required on the instruments (see 4.2.1.1).
- e. If required, specify the abrasive media for cleaning by abrasive blasting (see 4.2.3).
- f. If the surfaces are to be pre-heated (see 4.2.4).
- g. If the symbol to be applied is different (see 4.2.6).
- h. How the prefix is to be applied for identification (see 4.2.6).
- i. If the supplier's facility and general procedures needs to be certified before parts are supplied or waived (see 5.1.1).
- j. Additional requirements for specific applications (see 5.1.1).
- k. The manner in which the supplier needs to re-certify (see 5.1.1.2).
- l. If material of the test specimens is different from the material of the parts that are to be cold sprayed (see 5.1.2 and 5.1.2.1).
- m. If the coating thicknesses on the metallographic test specimens are different (see 5.1.2.1).
- n. Specify test specimen panel configuration (see 5.1.2.1.1).
- o. Specify the bond strength accept/reject criteria (see 5.1.2.2).
- p. If other configurations cannot be specified (see 5.1.2.2.1).
- q. Specify coating material (see 5.1.2.2.3).
- r. Specify coating thickness or range if different (see 5.1.2.2.4).
- s. Specify the number of reworks, if required (see 5.1.3.3).
- t. If photomicrographs can be submitted in lieu of the test samples (see 5.1.3.4).
- u. If the supplier can state different information than that listed (see 5.1.4).
- v. Specify acceptable levels are for oxide and porosity (see 5.2.3).
- w. Specify the level of frequency required for bond strength testing (see 5.2.4).
- x. Specify if nondestructive testing to detect delamination or other inherent discontinuities are required (see 5.2.5).

6.3 Safety. Use of cold spray equipment involves exposure to certain safety hazards, such as, pressurized gases and metal powders. Guidelines for the handling of these substances is found in OSHA Safety and Health Standards (29 CFR 1910), General Industry.

6.3.1 Material safety data sheets (MSDS). The contracting activity should be provided a material safety data sheet for each part at the time of contract award. The MSDS should be provided in accordance with OSHA section 1910.1200, 29 CFR Chapter XVII and found as part of FED-STD-313. OSHA section 1910.1200 requires reporting threshold criteria for known or suspected human carcinogens on MSDS 0.1 percent or greater, and 1 percent or greater for other health hazards. The MSDS should be included with each unit of issue of material covered by the specification, when specified. Contracting officers will identify those activities requiring copies of

MIL-STD-3021
w/Change 2

completed material safety data sheets prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

6.4 English units. When English divisions are required, units for meter, kilogram, meter per second, and mega Pascal may be converted to the English equivalent by multiplying them by the following conversion factors:

Metric SI unit	Multiply by	Equals	English
meter (m)	39.37	=	inch
meter (m)	3.28	=	foot
kilogram (kg)	2.205	=	pound
meter per second (m/s)	3.2808	=	feet/sec
mega Pascal (MPa)	145.038	=	pounds/sq. inch

6.5 Subject term (key word) listing.

Adhesion
Coating
Cohesion
Cold gas dynamic spray
High velocity powder
Impaction process
Kinetic energy metallization
Kinetic metallization
Kinetic spraying
Particles
Powder
Propelling gas
Thermal spray

6.6 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

MIL-STD-3021
w/Change 2
APPENDIX A

GUIDEANCE FOR ADDITIONAL QUALIFICATION REQUIREMENTS FOR STRUCTURAL APPLICATIONS

A.1 SCOPE

A.1.1 Scope: This appendix provides additional requirements for safety critical, FOD critical or structural applications (see 5.1.3.2). The requirements in this appendix should be tailored by the CEA to the specific application. This Appendix is a mandatory part of the standard. The information contained herein is intended for guidance only.

A.2 PROCEDURE

A.2.1 Understand Design Intent. The first step in identifying additional qualification requirements is to identify the critical design requirements of the component. These may include corrosion resistance, fatigue resistance or crack growth behavior to name a few.

A.2.2 Ensure Process Controls are Sufficient for Application. For safety critical items, it is typical that the entire process envelope be characterized. For example, metallographic test specimens for structural applications should be evaluated in the as-deposited thickness per the engineering drawing, half of that thickness and twice the thickness. This is in addition to the 0.2 mm minimum in 5.1.2.1. It is unlikely that 5% porosity would be sufficient for structural applications, so this acceptance criterion also needs specific review. For all structural applications each component must be inspected using surface and volumetric inspection in accordance with AWS D17.1. The acceptance criteria will be based on part criticality as defined by AWS D17.1.

A.2.3 Fixing Critical Process Parameters. The CEA must work with the cold spray provider to deliver application-unique requirements. The cold spray provider should then develop a cold spray deposition process that meets these requirements. Once the process has been developed, all critical parameters (see section 4 and 5) must remain fixed. If any parameter is changed the process must be re-certified.

A.2.4. Application-Specific Process Certification. Once the process has been fixed, the process must be certified using test data and pre-established acceptance criteria. An example test matrix for structural applications is shown in Table A.1. Table A.1 is an example only and is not a requirement. The CEA has the responsibility of establishing acceptance criteria for a specific application. Additional tests may be required or in some instances fewer tests may be required.

MIL-STD-3021
w/Change 2
APPENDIX A

Table A.1

EXAMPLE CERTIFICATION TEST MATRIX FOR STRUCTURAL APPLICATIONS

Test	Number of specimens ^{1/}	Acceptance Criteria
Metallographic examination (see 5.1.2.1)	20	MIL-STD-3021, Paragraph 5.1.2.1 Acceptance criteria defined by AWS D17.1 based on component criticality.
Bond strength	20 ^{2/}	10,000 psi minimum
Tensile testing (all deposit) ASTM E8/8M	10	Average/std div/high low tensile strength/yield strength (TS/YS) and elongation (EL) will be reported.
Impact test ASTM D2794	20 ^{2/}	No delamination or cracking based on visual observation.
Fatigue stress life (S-N) ASTM E466 (using open-hole test specimen geometry)	20	Standard S-N plot where: Specimen failure = loss of deposit structural integrity due to cracking. Statistical fatigue analysis of each condition shall be performed.
Fatigue stress life (S-N) ASTM E466 (if capability exists at testing facility)	25	Standard S-N plot of samples with deposit showing where deposit failure acoustic emission (AE) indications were found. 5 Specimens at 5 different stress levels
Fatigue stress life (S-N) ASTM E466 of deposit material only	25	Standard S-N plot using round or flat fatigue sample with geometry known to produce consistent results
Adhesion test (Bend around mandrel) ASTM B571	10	No delamination
Corrosion testing ASTM B117	10	No corrosion debit compared to baseline material system
Bearing ASTM E238	10 ^{2/}	Bearing strengths reported to CEA
^{1/} At least three lots of powder shall be used for these tests. ^{2/} Service temperature range (80 to 250 °F), if not specified.		

CONCLUDING MATERIAL

Custodian:

Army - MR
Navy - AS
Air Force - 20

Preparing activity:
Army - MR

Project MFFP-2014-005

Review activities:

Army – AV
Navy – EC, MC, SH
Air Force – 11, 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 <https://assist.dla.mil/>.