

METRIC

MIL-STD-3021

4 August 2008

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.

MIL-STD-3021

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. Moreover, the deleterious effects of deposit oxidation, evaporation, and residual stresses are avoided.

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, Weapons and Materials Research Directorate, Materials Applications Branch, Specifications and Standards Office, Attn: AMSRD-ARL-WM-MC, Aberdeen Proving Ground, MD 21005-5069 or emailed to rsquilla@arl.army.mil. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <http://assist.daps.dla.mil/>.

MIL-STD-3021

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
1.2.1 <u>Types</u>	1
1.3 <u>Applications</u>	1
2. <u>APPLICABLE DOCUMENTS</u>	
2.1 <u>General</u>	2
2.2 <u>Government documents</u>	2
2.2.1 <u>Specifications, standards, and handbooks</u>	2
2.3 <u>Non-Government publications</u>	3
2.4 <u>Order of precedence</u>	3
3. <u>DEFINITIONS</u>	
3.1 <u>Cold spray</u>	3
3.2 <u>Nozzle</u>	4
3.3 <u>Powder lot</u>	4
3.4 <u>Powder meter wheel</u>	4
3.5 <u>Simulated part</u>	4
3.6 <u>Substrate</u>	4
3.7 <u>Lot</u>	4
3.8 <u>Pass</u>	4
3.9 <u>Increment</u>	4
3.10 <u>Layer</u>	4
3.11 <u>Thermal spraying</u>	4
4. <u>GENERAL REQUIREMENTS</u>	
4.1 <u>Application</u>	4
4.2 <u>Process</u>	5
4.2.1 <u>Cold spray equipment</u>	5
4.2.2 <u>Material</u>	5
4.2.2.1 <u>Gas</u>	5
4.2.2.1.1 <u>Gas specifications</u>	5
4.2.2.2 <u>Coating powder</u>	5
4.3 <u>Required procedures and operations</u>	6
4.3.1 <u>Process control</u>	6
4.3.1.1 <u>Gauges, meters, and sensors</u>	6
4.3.1.2 <u>Checks</u>	6
4.3.2 <u>Surface preparation</u>	6
4.3.2.1 <u>Cleaning</u>	6

MIL-STD-3021

CONTENTS

<u>PARAGRAPH</u>		<u>PAGE</u>
4.3.2.2	<u>Handling and storage</u>	6
4.3.2.2.1	<u>Handling</u>	7
4.3.2.2.2	<u>Storage</u>	7
4.3.2.3	<u>Overspray protection</u>	7
4.3.3	<u>Abrasive blast</u>	7
4.3.3.1	<u>Blast contamination</u>	7
4.3.4	<u>Preheating</u>	7
4.3.5	<u>Coating deposition</u>	7
4.3.6	<u>Identification</u>	9
4.4	<u>Operator certification</u>	9
4.4.1	<u>Cold spray operator</u>	9
4.4.1.1	<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>	10
5.1.2	<u>Test samples</u>	10
5.1.2.1	<u>Metallographic test specimens</u>	10
5.1.2.1.1	<u>Specimen configuration</u>	10
5.1.2.2	<u>Bond strength 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>Sample part</u>	11
5.1.3.3	<u>Rework</u>	11
5.1.3.3.1	<u>Stripping</u>	11
5.1.3.4	<u>Metallographic test sample</u>	11
5.1.4	<u>Process certification</u>	11
5.2	<u>Test methods</u>	11
5.2.1	<u>Visual</u>	11
5.2.2	<u>Micro examination</u>	11
5.2.3	<u>Oxides and porosity</u>	12
5.2.4	<u>Bond strength test</u>	12
6.	<u>NOTES</u>	
6.1	<u>Intended use</u>	12
6.2	<u>Acquisition requirements</u>	12

MIL-STD-3021

CONTENTS

<u>PARAGRAPH</u>		<u>PAGE</u>
6.3	<u>Issue of DoDISS</u>	13
6.4	<u>Safety</u>	13
6.4.1	<u>Material safety data sheets (MSDS)</u>	13
6.5	<u>English units</u>	13
6.6	<u>Subject term (key word) listing</u>	14

TABLE

I.	<u>Operating parameters</u>	6
----	-----------------------------------	---

FIGURE

1.	<u>High-pressure system</u>	2
2.	<u>Low-pressure system</u>	2
3.	<u>Typical cold spray control sheet</u>	8

CONCLUDING MATERIAL.....	14
--------------------------	----

MIL-STD-3021

1. SCOPE

1.1 Purpose. The procedures covered by this standard are intended to ensure that cold spray coating operations, either manual or automated, meet prescribed requirements. This process can be used to restore dimensionally discrepant parts, or parts requiring protection from corrosion and wear (e.g. abrasion, cavitations, and erosion) but not limited to these applications.

1.2 Process. This standard describes the process requirements for surfacing by means of cold spray deposition. The term “cold spray” has been used to describe this process because both the temperature of the powder-laden gas jet and the temperature of the powder material are low enough to prevent a phase change or stress in the deposit or substrate. Cold spray is a process whereby metal powder particles are utilized to form a deposit by means of ballistic impingement upon a substrate. The metal powders range in particle size from 5 to 100 micrometers (μm) and are accelerated by injection into a high-velocity stream of gas. The high velocity gas stream is generated through the expansion of a pressurized, preheated, gas through a nozzle. 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 or at a lower pressure point downstream of the entrance. 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 the substrate and form bonds with the previously deposited material resulting in a uniform deposition with very little porosity and high bond strength.

1.2.1 Types. The two principal cold spray system configurations are depicted by Figures 1 and 2. The two configurations differ in the carrier gas, gas pressure, and powder injection location. Figure 1 shows a high-pressure system in which the main gas stream and the powder stream are both introduced into the inlet chamber of the nozzle. This configuration requires that the powder feeder be capable of high gas pressure and is most often used in stationary cold spray systems. High-pressure systems utilize higher pressure gases and often have a dedicated gas compressor. A low molecular weight gas, such as helium, is sometimes used as the accelerating gas when particles must be brought to very high velocity. Figure 2 shows a system in which the powder stream is injected into the nozzle at a point where the gas has expanded to low pressure. Atmospheric pressure air, drawn by the lower pressure nozzle injection point, is used for powder transport from the feeder. Since this system does not require a pressurized feeder, it is often used in portable cold spray systems. The low-pressure system generally utilizes readily available compressed air, but can utilize nitrogen as well.

1.3 Applications. The superior qualities of cold sprayed deposits are often required by the application. For example, the high heat transfer coefficient and electrical conductivity of cold sprayed deposits favor its use in electronic 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. Good corrosion protection is

MIL-STD-3021

achieved by dense, impermeable cold sprayed deposits. Wear resistant, hard surfaces, such as MCrAlYs, can be deposited by cold spray when operated at its high-temperature end.

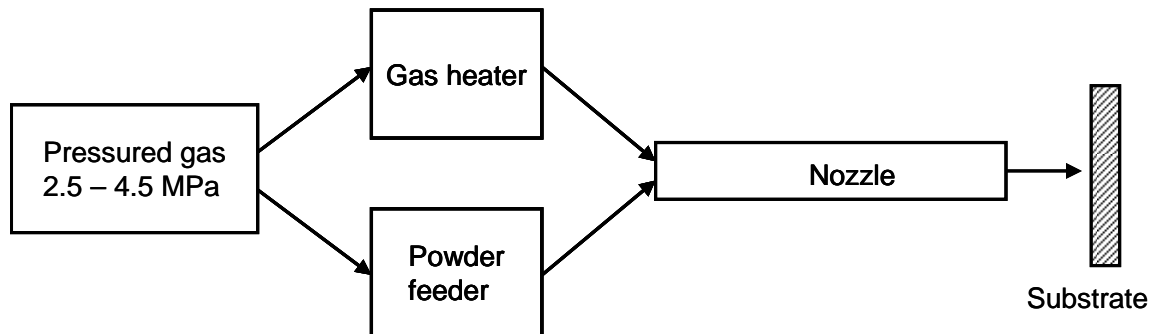


FIGURE 1. High-pressure system.

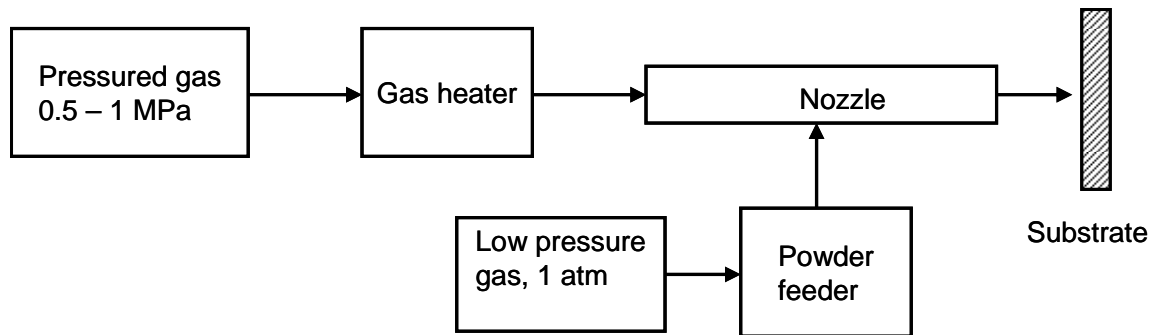


FIGURE 2. Low-pressure system.

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3, 4, or 5 of this standard. This section does not include documents cited in other sections of this standard 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, 4, or 5 of this standard, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks 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.

MIL-STD-3021

DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-S-5002 - Surface Treatments and Inorganic Coatings for Metal Surfaces of Weapons Systems

(Copies of these documents are available online at <http://assist.daps.dla.mil/quicksearch/> or <http://assist.daps.dla.mil/> or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

2.3 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 (DoD Adopted).

(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 (DoD Adopted)

ASTM E3 - Standard Guide for Preparation of Metallographic Specimens (DoD Adopted).

ASTM E407 - Standard Practice for Micro-Etching Metals and Alloys (DoD Adopted).

(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.)

2.4 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 5 to 100 micrometers (μm) in diameter) in the solid state are accelerated to high velocities (typically 300 to 1200 meters/second), and subsequently develop a coating or deposit by impacting 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

MIL-STD-3021

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.

3.2 Nozzle. A gas manifold designed to accelerate a gas to high velocity.

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

3.4 Powder meter wheel. A perforated wheel which meters powder feed rate through rotation speed.

3.5 Simulated part. A simulated part is a test piece or section with a similar surface configuration to the part it represents. The simulated part will be approved by the acquisition authority.

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

3.7 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.

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

3.9 Increment. The distance between adjacent passes.

3.10 Layer. Multiple passes over the work piece that result in complete coverage.

3.11 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.

4. GENERAL REQUIREMENTS

4.1 Application. 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), commercially pure titanium (Ti), silver (Ag), and zinc (Zn), as well as stainless steel, nickel-base alloys (Inconels, Hastalloys), and bondcoats, 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. Cold spray has been used to produce protective coatings and performance enhancing layers, ultra thick coatings, freeform and near net shape substrates. Typical protective coatings produced by cold spray include MCrAlY coatings for high temperature protection and bond coats for thermal barriers, copper-chrome layers for oxidation protection, and corrosion resistant aluminum and zinc coatings for oil and auto industries and others.

MIL-STD-3021

4.2 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 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. Large sizes and shapes can be spray fabricated and geometrical features can be easily incorporated during spray preparation and subsequent machine finishing. Moreover, by controlling the feedstock composition, one could vary the deposit microstructure and composition to produce functionally graded materials and other special structures.

4.2.1 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 high velocity (Mach numbers ranging from 1 to 4) 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.

4.2.2 Material.

4.2.2.1 Gas. The gas may be (but not restricted by) any of the following:

- a. Nitrogen (N₂).
- b. Helium (He).
- c. Mixture of nitrogen and helium (N₂ + He).
- d. Air.

4.2.2.1.1 Gas specifications. Gas specifications, if used by the processor for procurement, shall be acceptable to the cognizant engineering organization.

4.2.2.2 Coating powder. The powder for coating 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 mass median particle size shall be between 5 and 100 micrometers in diameter. The user should be cautioned to the importance of paying attention to the manufacturer's instructions pertaining to the storage and handling of finely divided metal powders. If the powder to be used by the manufacturer requires that it is controlled by a specification, it shall be specified in the contract or purchase order (see (6.2)).

4.3 Required procedures and operations.

MIL-STD-3021

4.3.1 Process control. The cold spray process is optimized through the adjustment of control parameters. These parameters include the gas preheat temperature, gas pressure, nozzle geometry, throat size, powder feed rate, and spray distance. A critical process parameter is the feedstock powder material itself—primarily particle size distribution and particle attributes such as oxide layer and mechanical properties, which influence the ability to form a compacted deposit. Operational parameters are typically selected to achieve the most suitable deposit for its intended application at the lowest operational temperatures. The distinguishing feature of the cold spray process compared with conventional thermal spray processes is its ability to produce deposits with preheated gas temperatures in the range of 0 to 800° C, a range that is generally lower than the melting temperature of the coating particle materials. The nozzle exit temperature is substantially lower than the gas pre-heat temperature, further lowering the temperature excursions experienced by the feedstock particles and substrate materials. The range of operation for the stationary and portable cold spray systems 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.

4.3.1.1 Gauges, meters, and sensors. If a minimum accuracy is required on these instruments, it shall be defined in the contract or purchase order (see (6.2).

4.3.1.2 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). 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.

TABLE I. Operating parameters.

	High-Pressure System	Low-Pressure System
Working gas	N ₂ , He, air	N ₂ , air
Gas pressure, MPa	2.5 – 4.5	0.5 – 1.0
Gas preheat, °C	20 - 800	20 - 550
Gas flow rate, m ³ /hour	50 - 150	15 - 30
Maximum Gas Mach #	1 - 3	1 - 3
Powder flow rate, g/s	0.1 – 1.0	0.1 – 1.0
Particle size, µm	5 - 100	10 - 80

4.3.2 Surface preparation.

4.3.2.1 Cleaning. Surfaces to receive deposits shall be thoroughly cleaned to remove oil, grease, dirt, paint and other foreign material. Final cleaning shall take place no more than four hours prior to coating. Cleaning procedures shall not embrittle, pit, or damage surfaces to be coated.

4.3.2.2 Handling and storage.

MIL-STD-3021

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

4.3.2.2.2 Storage. If a delay in spraying occurs beyond 2 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. Alternative methods may be to store parts overnight in a moderate temperature oven maintained at approximately $95^{\circ} \pm 3^{\circ} \text{C}$ 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 4.3.2.1.

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

4.3.3 Abrasive blast. When required, all surfaces designated for cold spray coating shall be cleaned by abrasive blasting with the abrasive media specified in the qualified procedure.

4.3.3.1 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.

4.3.4 Preheating. When required 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 preheat and prior to spraying shall be measured using the appropriate pyrometric devices in accordance with AMS 2750.

CAUTION: Special care must be taken to avoid overheating nonferrous alloys with low melting temperatures such as aluminum and magnesium.

4.3.5 Coating deposition. The coating 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.

MIL-STD-3021

VENDOR:		Sheet ____ of ____	
VENDOR PROCESS #:			
PURCHASE ORDER NUMBER:			
PART NUMBER:		S/N:	
AREA TO RECEIVE DEPOSITION:			
COLD SPRAY (CS) MANUFACTURER:			
PART MATERIAL:			
CS TYPE:		NOZZLE:	
<u>PART PREPARATION</u>			
METHOD OF CLEANING:			
MASKING INFORMATION:			
GRIT TYPE AND SIZE:			
GRIT BLAST PRESSURE (MPa) : _____ \pm Suction : _____ Pressure : _____			
GAS (1) PRIMARY:		Temperature Deg. C _____ \pm	
Main Gas Pressure MPa _____ \pm		Main Gas Flow (1) m ³ /hour. _____ \pm	
Feeder Gas Pressure MPa _____ \pm		Feeder Gas Flow (2) m ³ /hour. _____ \pm	
<u>COATING POWDER</u>			
Powder material: _____		Powder size: _____	
Supplier: _____		Material Lot # : _____	
<u>COATING DATA</u>			
Elapsed Time Between Surface Prep and Spraying: _____			
Powder Feed Rate (kg/hr): _____			
Powder Meter Wheel: _____			
Nozzle to Work Distance: _____			
Traverse rate (mm/s): _____		Preheat Temp: _____	
Increment (mm): _____			
Deposition Thickness as Sprayed: _____		Method of Preheat: _____	
Number of Passes Per Layer: _____		Number of Layers: _____	
Method of Cooling (if any): _____			
NOTES:			
CERTIFICATION #: _____			
APPROVAL: _____			

FIGURE 3. Typical cold spray control sheet.

MIL-STD-3021

4.3.6 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 manufacturer code.

4.4 Operator certification

4.4.1 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 two more samples of the qualification test panels or specimens which failed to pass the specified test. If any of the second set fail 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. In addition, the operator shall be required to submit one additional set of the qualification test panels or specimens which resulted in the initial disqualification. A cold spray operator of semiautomatic or automatic equipment shall meet all the requirements of a manual spray operator.

4.4.1.1 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 and general procedures shall be certified before parts for production are supplied unless otherwise specified in the contract or purchase order (see 6.2). If such certification is to be waived by the acquisition authority it must be specified in the contract or purchase order.

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.

MIL-STD-3021

5.1.1.2 Re-certification. In the event a supplier has not supplied cold sprayed parts or test specimens for over one 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 samples. A minimum of one panel for micro-examination and three bond strength specimens shall be cold sprayed. The microscopic examination panels and bond specimens shall be fabricated from the same materials as the parts to received the cold spray or as specified by the acquisition authority.

5.1.2.1 Metallographic test specimens. Test panels 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.
- c. Deposition Material: Specified by acquisition authority.
- d. Deposition thickness: 0.2 to 0.3 millimeters unless otherwise specified in the contract or purchase order (see 6.2).

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

5.1.2.2.1 Size. Three specimens 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. One specimen of the selected alloy as specified in 5.1.2 and one specimen of any suitable material for the uncoated end of the test specimen.

5.1.2.2.3 Deposit. The deposit shall be as specified by the acquisition authority (see 6.2).

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

5.1.2.2.4 Deposition thickness. The deposition thickness shall be at least 0.4 millimeters, unless other 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

MIL-STD-3021

is optional provided all pertinent information is provided. Processing procedures shall not be dependent upon part function or the critical nature when in service. The engineering requirements as specified on the drawing shall prevail during any acceptance testing.

5.1.3.2 Sample part. A sample part, or a simulated part 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.

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 and method of repair.

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 sample. Metallographic test samples 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), test panels, and test specimens, 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 Micro examination. A microscopic examination of a specimen cut normal to the coating surface shall not reveal any cracks, excessive or massive oxides or porosity when examined at a magnification of 100x in accordance with the procedures outlined in ASTM E3 and ASTM E407. In accordance with ASTM E407 and the results desired, the cold spray deposit

MIL-STD-3021

material shall determine the etchant number. The etchant composition and procedure shall be determined from the corresponding etchant number.

5.2.3 Oxides and porosity. Excessive porosity shall be defined as not greater than 5 percent when viewed at 200x after etching. Excessive or massive oxides shall be defined as not greater than 3 percent for coatings where oxygen is not an integral part of the material. Examination of the part for oxides shall be at 100x after etching.

5.2.4 Bond strength test. A bond strength test specimen (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:

- 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 uniaxial loading of the specimen. Set the no-load cross-head speed at approximately 1 millimeter per minute.

6. NOTES

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

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.

MIL-STD-3021

- b. If the powder is to be controlled by a specification (see 4.2.2.2).
- c. If a minimum accuracy is required on the instruments (see 4.3.1.1).
- d. If the surfaces are to be pre-heated (see 4.3.4).
- e. If the symbol to be applied is different (see 4.3.6).
- f. How the prefix is to be applied for identification (see 4.3.6).
- g. If the supplier's facility and general procedures needs to be certified before parts are supplied or waived (see 5.1.1).
- h. The manner in which the supplier needs to re-certify (see 5.1.1.2).
- i. If the coating thicknesses on the metallographic test specimens are different (see 5.1.2.1).
- j. Specify specimen configuration (see 5.1.2.1.1).
- k. Specify the bond strength accept/reject criteria (see 5.1.2.2).
- l. If other configurations can not be specified (see 5.1.2.2.1).
- m. Specify coating material (see 5.1.2.2.3).
- n. Specify coating thickness or range if different (see 5.1.2.2.4).
- o. If photomicrographs can be submitted in lieu of the test samples (see 5.1.3.4).
- p. If the supplier can state different information than that listed (see 5.1.4).

6.3 Issue of DoDISS. When this standard is used in acquisition, the applicable issue of the DoDISS must be cited in the solicitation (see 2.2.1 and 2.2.2).

6.4 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.

6.4.1 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 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.

6.5 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

MIL-STD-3021

6.6 Subject term (key word) listing.

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

CONCLUDING MATERIAL

Custodian:

Army - MR
Navy - AS
Air Force - 11

Preparing activity:

Army - MR

Project MFFP-2007-006

Review activities:

Army – AV, PT
Navy – EC, MC, NP, 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 <http://assist.daps.dla.mil/>.