INCH-POUND

MIL-DTL-32502
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DETAIL SPECIFICATION

COATING, COBALT-PHOSPHORUS ALLOY, NANOCRYSTALLINE (ELECTRODEPOSITED)

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

1. SCOPE

1.1 Scope. This specification covers an electrodeposited nanocrystalline cobalt-phosphorus alloy on metal surfaces. It is generally applied as a protective coating to steel alloys but may be applied to other metals such as, but not limited to, aluminum, copper, stainless steel, nickel, cobalt and their alloys.

1.2 Restriction.

1.2.1 Application of nanocrystalline cobalt-phosphorous alloy plating to steel parts having a hardness of 48 HRC (ultimate tensile strength of 238 ksi [1641 MPa]) or higher should not be performed unless authorized by the design documentation of the cognizant engineering organization. Application of nanocrystalline cobalt-phosphorous alloy plating for repair of steel parts having a hardness of 48 HRC (ultimate tensile strength of 238 ksi [1641 MPa]) or higher should not be performed unless specific approval has been received from the cognizant engineering organization.

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.

Comments, suggestions, or questions on this document should be addressed to: Commander, Naval Air Warfare Center, Aircraft Division, Code 4L8000B120-3, Highway 547, Lakehurst, NJ 08733-5100 or emailed to michael.sikora@navy.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.

AMSC N/A AREA MFFP

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.

DEPARTMENT OF DEFENSE SPECIFICATION

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

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

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.

ASTM INTERNATIONAL

ASTM B487	-	Standard Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
ASTM B571	-	Standard Practice for Qualitative Adhesion Testing of Metallic Coatings
ASTM B117	-	Standard Practice for Operating Salt Spray (Fog) Apparatus
ASTM E384	-	Standard Test Method for Knoop and Vickers Hardness of Materials
ASTM F519	-	Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments

(Copies of these documents are available online at http://www.astm.org/.)

SAE INTERNATIONAL

SAE AMS 2430	-	Shot Peening, Automatic
SAE AMS 2759/11	-	Stress Relief of Steel Parts

(Copies of these documents are available online at http://www.sae.org/.)

2.4 Order of precedence. 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 Materials. The materials used shall produce deposits which meet the requirements of this specification.

3.2 General requirements.

3.2.1 Basis metal. The basis metal (parts) submitted for the plating process shall be free from visible defects such as blemishes, prior pitting from corrosion, nicks, scratches, burrs or other geometrical or base metal defects that could be detrimental to the appearance or performance of the plating. As part of the plating process, the basis metal (parts) shall receive the necessary cleaning and surface activation to yield a deposit that is in accordance with the requirements of this specification.

3.2.2 Preplating operations. Prior to plating, items to be plated shall have undergone all required heat treatments and mechanical operations (machining, welding, brazing, forming, punching, grinding and similar operations) (see 6.2).

3.2.3 Stress relief treatment. All steel parts having an ultimate tensile strength of 150,000 psi or greater, which are machined, ground, cold formed or cold straightened, shall be stress relieved (see 6.2). Stress relief shall conform to requirements in SAE AMS 2759/11. When peening is required, thermal stress relief shall be performed prior to shot or rotary flap peening (see 6.2).

3.2.4 Peening. A reduction in the fatigue life of plated parts may be expected. Parts designed for unlimited fatigue life under dynamic loads should be peened prior to plating. When specified in the contract or order, parts shall be peened prior to plating in accordance with SAE AMS 2430 (see 6.2). Unless otherwise specified, such peening shall be accomplished on all surfaces for which the plating is required and on all immediate adjacent surfaces that contain notches, fillets or other abrupt changes of section size.

3.2.5 Cleaning. The plating shall be applied over a surface free from water breaks. The cleaning procedure shall not produce pitting, intergranular attack, or hydrogen embrittlement of the basis metal and shall preserve dimensional requirements. Metal parts shall be cleaned in accordance with MIL-DTL-5002 or cleaned by methods that shall neither damage the substrate nor interfere with the adhesion of the deposit.

3.3 Processing requirements.

3.3.1 Plating application. Unless otherwise specified in the contract or order, parts shall be plated with nanocrystalline cobalt-phosphorus alloy onto a properly prepared surface directly on the basis metal without a coating of another metal underneath, except that of a preliminary plating of nickel or other suitable metals as applicable to ensure adhesion of the basis metal being plated. In no case shall any underplate be substituted for any part of the specified nanocrystalline cobalt-phosphorous alloy plating thickness.

3.3.2 Coverage. All surfaces of the part, except those which cannot be touched by a sphere 0.75 inch in diameter, shall be plated to the specified thickness. Unless otherwise specified in the contract or order, surfaces such as holes, recesses, threads and other areas where a controlled deposit cannot be obtained under normal plating conditions, may be under the specified thickness limit provided visual plating coverage is shown. The plate shall be uniform in thickness on significant surfaces with the exception of build-up on exterior corners or edges which shall be permitted provided finished drawing dimensions are met.

3.3.3 Contact marks. The size and number of contact marks shall be at a minimum or consistent with good practice. The location of contact marks shall be in areas of minimum exposure to service environmental conditions important to the function of the part.

3.3.4 Hydrogen embrittlement relief. The electrodeposition process and cleaning method shall not cause hydrogen embrittlement in the metal substrate. Unless otherwise specified in the contract or order (see 6.2), steel parts that are surface or through hardened to 180,000 psi (40 HRC) and higher and steel fasteners with a tensile strength 150,000 psi (34 HRC) and higher, shall be given a hydrogen embrittlement relief baking at 375 °F ±25 °F (191°C ± 14 °C) for a minimum of 23 hours within 4 hours after completion of plating. Plated springs and other parts subject to flexure shall not be flexed prior to the embrittlement relief treatment.

3.4 Electrodeposited coating requirements.

3.4.1 Appearance. The deposits shall be smooth, adherent, and free from visible blisters, pits, porosity, cracks and other defects. The plating shall show no indication of contamination or improper operation of equipment used to produce the deposit, such as excessively powdered or darkened plating, build-up and other defects. Superficial staining which has been demonstrated to result from rinsing or slight discoloration resulting from baking operations to relieve embrittlement, shall not be cause for rejection. All details of workmanship shall conform to the best practice for high quality plating.

3.4.2 Thickness. The as-plate thickness shall be as specified on the part drawing. Unless otherwise specified in the contract or order (see 6.2), the minimum coating thickness shall be 0.002 inch on all visible surfaces. There shall be no bare areas. The plating shall be uniform in thickness on significant surfaces with the exception of build-up on exterior corners or edges which shall be permitted provided drawing dimensions are met. Deposit thickness shall be in accordance with ASTM B487 or another method approved by the procuring activity (see 4.5.1).

3.4.3 Adhesion. The plating and any underplate shall be tightly adherent to the substrate in accordance with ASTM B571. When examined at a magnification of 4X or greater, neither the nanocrystalline cobalt-phosphorus alloy nor any electrodeposited underplate(s) shall show separation from the basis metal or from each other. The formation of cracks in the plating or the basis metal which do not result in flaking, peeling, or blistering of the plating shall not be cause for rejection (see 4.5.2).

3.4.4 Corrosion resistance. After exposure to the salt spray fog for 200 hours in accordance with ASTM B117 as specified in 4.5.3, the specimens shall show no basis metal corrosion products when examined at normal reading distance.

3.4.5 Hardness. Hardness shall be a minimum of 510 HV_{100} (Vickers Hardness at 100 grams force), determined on a metallographic cross section in accordance with ASTM E384 as specified in 4.5.4. Plating thickness for determination of microhardness shall be 0.002 inch minimum.

4. VERIFICATION

4.1 Classification of inspection. The inspection requirements specified herein are classified as follows:

a. First article inspection (see 4.2).
b. Conformance inspection (see 4.3).

4.2 First article inspection.

4.2.1 Control records. When specified in the contract or order (see 6.2), the supplier shall maintain a record of each processing bath, showing all additional chemicals or treatment solutions to the unit, the results of all chemical analyses performed, and the quantity of parts plated during operation (see 6.2 and 6.4).

4.2.2 Production control. The equipment, procedures and operations employed shall be capable of producing electrodeposited platings as specified in this document and Table I.

4.2.3 Frequency of tests. Specimens prepared and tested in accordance with Table I shall be made once each month or as specified by the procuring activity.

TABLE I. First article tests and specimens.

Test	Requirement	Test method paragraph
Appearance	3.4.1	Visual inspection
Thickness	3.4.2	4.5.1
Adhesion	3.4.3	4.5.2
Corrosion resistance	3.4.4	4.5.3
Hardness	3.4.5	4.5.4
Hydrogen embrittlement relief	3.3.4	4.5.5

Note: Unless otherwise specified, standard alloy sheets shall be used for first article test specimens. The selection shall be at the option of the supplier and representative of articles plated.

4.3 Conformance inspection.

4.3.1 Lot. A lot shall consist of plated articles of the same material and condition, and of approximately the same size and shape, plated under similar conditions and submitted for inspection at the same time. Unless otherwise specified by the procuring activity, conformance testing of each inspection lot shall be performed in accordance with Table II.

TABLE II. Conformance tests and specimens.

Test	Requirement	Test method paragraph
Appearance	3.4.1	Visual inspection
Thickness	3.4.2	4.5.1
Adhesion	3.4.3	4.5.2
Hardness	3.4.5	4.5.4

Note: Unless otherwise specified, standard alloy sheets shall be used for production control specimens. The selection shall be at the option of the supplier and representative of articles plated.

4.4 Sampling.

4.4.1 Sampling for inspection. The number of random samples selected for nondestructive tests from each inspection lot shall be determined by the procuring activity (see 6.2). For destructive tests, a random sample of four items shall be selected from each inspection lot, or four separate specimens shall be prepared in accordance with 4.4.2 to represent each inspection lot. If the number of items in an inspection lot is four or less, the number of items in the sample shall be determined by the procuring activity (see 6.2).

4.4.2 Separate specimens. When the plated articles are not readily adaptable to the tests specified in 4.5, the test may be made by the use of separate specimens plated concurrently with the articles represented. The separate specimens shall be of a basis metal equivalent to that of the articles represented, and shall be a minimum of 3 x 6 x 0.040 inches for corrosion testing and 1 x 4 x 0.040 inches for adhesion, thickness and hardness testing. Hydrogen embrittlement testing shall utilize ASTM F519, Type 1a.1 specimens. The specimens shall be introduced into a lot at regular intervals prior to the cleaning operations preliminary to plating and shall not be separated until after completion of the processing.

4.5 Test methods.

4.5.1 Thickness. The testing shall be performed in accordance with ASTM B487 or by another method approved by the procuring activity. Failure to meet requirements of 3.4.2 shall be cause for rejection.

4.5.2 Adhesion. The testing shall be performed in accordance with ASTM B571 or by another method approved by the procuring activity. Platings that demonstrate the capability to withstand final grinding operations shall be considered satisfactory.

4.5.3 Corrosion resistance. The test for corrosion resistance shall be performed on the specimens with 0.002 inch minimum thickness selected in accordance with 4.4.1. The specimens shall be subjected to a continuous test of 200 hours in 5 percent salt spray fog in accordance with ASTM B117. Appearance of cobalt-phosphorus oxidation products may be misinterpreted as red rust. Where identification of red rust is difficult, ferroxyl test shall be used (see 6.4). Visual evidence of corrosion of the basis metal, in accordance with 3.4.4, shall be cause for rejection of the entire lot.

4.5.4 Hardness. Hardness testing shall be conducted on samples selected as specified in 3.4.5. The testing shall be performed in accordance with ASTM E384 or by another method approved by the procuring activity.

4.5.5 Hydrogen embrittlement relief test. Four (4) each type 1a.1 test specimens in accordance with annex A1 of ASTM F519 (4340 steel at HR_c 51-54) shall be plated and hydrogen embrittlement relief treated similar to the parts, including preplating operations. For test purposes, the plating thickness shall be 0.002 inch minimum thickness measured on the smooth section of the specimen, but with visual plating at the root of the notch. The specimens shall be loaded in accordance with ASTM F519 to 75 percent Notch Fracture Strength (NFS) for 200 hours. Test failure shall be cause for rejection of the entire lot.

4.6 Resampling and retesting. If any test fails to meet specified requirements, the parts in that lot may be stripped, pretreated, plated and post treated as specified herein and retested. Alternatively, all parts in the lot may be inspected for the non-conforming attribute, and the non-conforming parts may be stripped, pretreated, plated, and post treated as specified herein and then retested. When specified in the contract or order, for testing, if hydrogen embrittlement fails to meet test requirements, retesting in accordance with the procedures of ASTM F519 is permitted.

4.7 Stripping and re-plating. When stripping is performed, the method shall be approved by the procuring activity and shall not roughen, pit, or embrittle the basis metal or adversely affect part dimensions. When parts have been stripped and re-plated, the reprocessing shall be documented and the procuring activity shall be informed.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's 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 Intended use. The electrodeposited nanocrystalline cobalt-phosphorus alloy is designed as a hexavalent chromium free plating process to provide corrosion resistance, wear resistance or to perform dimensional restoration of damaged substrates. Wear resistance may differ from that of engineering hard chromium.

6.2 Acquisition requirements. Acquisition documents should specify the following:

(a) Title, number and date of this specification.
(b) Operations to be performed on the items after plating or further processed, if different than specified (see 3.2.2).
(c) Certification of stress relief, if required (see 3.2.3).
(d) If peening is required (see 3.2.4)
(e) If hydrogen embrittlement relief treatment is not required (see 3.3.4).
(f) Thickness of plating, as specified (see 3.4.2).
(g) Control record requirement (see 4.2.1 and 6.4)
(h) Lot size, if different (see 4.4.1).
(i) Number of samples to be inspected from the lot for nondestructive tests, and for destructive tests if the number of items in a lot is four or less (see 4.6).
(j) Packaging requirements (see 5.1).

6.3 Part fabricator peening. Peening must be performed by or on behalf of the part fabricator, unless specifically delegated to the plating processor.

6.4 Control records availability. Upon request of the procuring activity, such records as well as reports of the test results must be made available. These records must be maintained for not less than one year after completion of the contract or purchase order.

6.5 Precipitation hardening. Post plating precipitation hardening heat treatment is not controlled by this specification. If performed, final heat treatment operations should be approved by the cognizant engineering organization to not adversely affect condition of the basis metal.

6.6 Properties. The electrodeposition process produces coatings with an average grain size between 5-15 nm and a composition between 1.0 - 2.0 weight percent phosphorus and balance cobalt. Grain size estimation by XRD (X-Ray Diffraction) testing may obtained by applying Scherrer's line broadening analysis. The composition of the coating may be obtained by SEM-EDX (Scanning Electron Microscope, Energy Dispersive X-ray spectroscopy).

6.7 Ferroxyl test. The ferroxyl test may be used to identify corrosion products on plated samples following exposure to the salt spray fog. A sheet of filter paper or other suitable adsorbent paper, saturated in the ferroxyl solution must be applied for 10 minutes to the flat surface of the specimen or the article. Complete contact of the filter paper with the plated test specimen must be ensured using strokes with a soft bristle brush. Filter paper must be kept saturated during the duration of the 10 minute test. Dark blue spots are in indication of iron corrosion products, while red spots are in indication of cobalt corrosion products. The approximate solution composition must be as follows:

Potassium ferricyanide ($K_3Fe(CN)_6$) 1 gm
Sodium Chloride (NaCl) 10 gm
Water (distilled or deionized) to make 1 liter

6.8 Equipment and processing. Plating tanks made of polyvinyl chloride have been found to be satisfactory. Tank liners may be employed for compatibility with hard chrome plating infrastructure. An exhaust system is recommended to remove steam. Anodes of high purity cobalt in titanium baskets are preferred. Parts with deeply recessed areas may require auxiliary anodes, cobalt wire or cobalt plated materials. Plating can be accomplished at temperature of 185 °F (85 °C). The use of solution level controller technology is recommended to minimize volume changes associated with evaporative losses during electrodeposition. A pulse power supply (low frequency) is required to obtain a square pulse waveform with a frequency of 25Hz and a duty cycle of 50 percent with a maximum voltage of 20V at a current density of $125mA/cm^2$ (116 ASF).

6.9 Ozone depleting chemicals. Classes I and II ozone depleting chemicals should be avoided when cleaning the basis metal. A list of ozone depleting chemicals can be found at http://www.epa.gov/spdpublc/science/ods/index.html.

6.10 Material Safety Data Sheet (MSDS). 29 CFR 1910.1200 requires that the MSDS for each hazardous chemical used in an operation must be readily available to personnel using the material. Contracting officers should identify the activities requiring copies of the MSDS.

6.11 Subject term (key word) listing:

Electroplating

CONCLUDING MATERIAL

Custodians:
Army - MR
Navy - AS
Air Force - 20

Preparing activity:
Navy - AS

(Project MFFP-2014-003)

Review activities:
Army - AR, AT, MI, AV
Navy – SH, OS
Air Force –99

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.