NOT MEASUREMENT SENSITIVE

MIL-DTL-13924E
12 September 2018
SUPERSEDING
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18 March 1999

DETAIL SPECIFICATION

COATING, OXIDE, BLACK, FOR FERROUS METALS

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

1. SCOPE

1.1 Scope. This specification covers black oxide coatings applied to ferrous metals (carbon, low alloy, and corrosion resistant steels, and wrought iron). Black oxide coatings, with or without a supplementary preservative treatment (see 3.11), may be used where a black surface is required. Only very limited corrosion protection, under mildly corrosive conditions, is obtained as a result of black oxide coating (see 6.1). Black coatings are included in this specification with limitations as noted in 1.2. Unless contraindicated by contract, chrome-free finishes should be used in place of finishes containing hexavalent chromium, provided all performance criteria herein has been met and documented as OQE.

Comments, suggestions, or questions on this document should be addressed to: Director, U.S. Army Research Laboratory, Weapons and Materials Research Directorate, Specifications & Standards Office, Attn: RDRL-WMM-C, Brian Placzankis, Aberdeen Proving Ground, MD 21005-5069. 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 FSC MFFP

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1.2 Classification. Black oxide coatings covered by this specification should be of the following classes as specified (see 6.2 and 6.3.2).

Class 1	Alkaline oxidizing process (for wrought iron, cast and malleable irons, and plain carbon or low alloy steels).
Class 2	Alkaline chromate oxidizing process (for use on certain corrosion resistant steel alloys which are tempered at less than 900°F (482°C)).
Class 3	Fused salt oxidizing process (for corrosion resistant steel alloys which are tempered at 900°F (482°C) or higher).
Class 4	Alkaline oxidizing process (for other corrosion resistant steel alloys).

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

2.2 Government documents.

2.2.1 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 this document are those cited in the solicitation or contract.

DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-DTL-53072	-	Chemical Agent Resistant Coating (CARC) System Application Procedures and Quality Control Inspection

(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.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM B117	-	Operating Salt Spray (Fog) Apparatus
ASTM F519	-	Mechanical Hydrogen Embrittlement Testing of Plating Processes and Aircraft Maintenance Chemicals
ASTM D1735	-	Standard Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus
ASTM F22	-	Standard Test Method for Hydrophobic Surface Films by the Water-Break Test

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

SAE INTERNATIONAL

SAE AMS2485	-	Coating, Black Oxide
SAE AMS2759/9	-	Hydrogen Embrittlement Relief (Baking) of Steel Parts

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

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. REQUIREMENTS

3.1 Materials. The materials for the blackening processes shall be selected by the contractor from manufacturers that have demonstrated through qualitative testing and proven performance in accordance with this specification. The selected materials shall result in black coatings meeting all the applicable requirements of this specification.

3.2 Preparation of basis metal. Prior to the application of the black coatings, the basis metal shall be thoroughly cleaned. The cleaned surfaces shall be free of rust, scale, grease, oil, paint, or other foreign matter, and shall pass the water break test described in ASTM F22. Supplier validated cleaning materials and methods shall be selected at the option of the contractor. The cleaning process shall be performed without measurable abrasion or erosion unless abrasive blasting or hand tool cleaning is permitted by drawing or contract and dimensional tolerances are maintained. Should the cleaning process or application of coating create a possibility of hydrogen embrittlement, the contractor shall take the appropriate steps for stress relief as indicated within this specification.

3.2.1 Stress relief. Unless otherwise specified for a particular end item specification or drawing, after forming and hardening, and prior to cleaning and coating, objectionable residual stress in ferrous alloy parts having a hardness greater than 39 HRC shall be relieved by suitable heat treatment. The temperature shall be such that maximum relief is given without hardness being reduced to less than the specified minimum. Stress relief is not necessary where it has been demonstrated through regularly applied qualitative testing for hydrogen embrittlement for a period not to exceed 90 days of production evidenced in that the cleaning and coating process has no harmful effect on the coated part or a test sample in accordance with ASTM A519 and recorded as OQE.

3.2.2 Coating as a final process. Unless otherwise specified, the black coatings shall be applied after all machining, forming, welding, cold straightening and heat treatment have been completed.

3.3 Application of black coatings. The coating shall conform to the class specified. The specified black coating shall be applied under controlled procedural conditions, especially with respect to time and temperature, in accordance with the coating manufacturer's recommendations. All equipment, together with solutions or baths, shall be properly maintained and kept free of dirt or detrimental contaminants. The selected process shall not reduce the hardness of the parts being processed or expose the parts to temperatures in the temper embrittlement range of the material, nor shall it cause embrittlement of the steel.

3.3.1 Surface attack. The process shall not result in any attack of the surface, either pitting or intergranular.

3.4 Alkaline oxidizing solutions (classes 1 and 2). Class 1 coatings shall be formed from a boiling chrome-free alkaline oxidizing solution. Class 2 coatings shall be oxidized using chromated oxidizing compounds.

3.4.1 Rinsing. All classes of black oxide coated pieces shall be rinsed in ambient water.

3.4.2 Sealing. Class 1 coatings shall be sealed using non-chromated sealers in accordance with the sealer manufacturer's recommendations.

3.4.3 Chromic acid dip for Class 2 and Class 3 coatings. After the cold water rinse (see 3.4.1) the pieces should be dipped for a minimum of 30 seconds in a 0.06 percent solution (8 oz. chromic acid per 100 gallons water) of chromic acid maintained at a temperature of l50 to l90°F (66 to 88°C) and a pH of 2 to 3. After the chromic acid dip, parts should be dried without further rinsing by using warm dry air or comparable process that prevents flash rusting or staining.

3.5 Fused salt oxidizing (class 3). The temperature of the molten oxidizing salt of class 3 shall not be higher than 900°F (482°C). After suitable immersion, the treated parts shall be

withdrawn, cooled from eight to ten minutes and rinsed in hot water, l90°F (88°C), followed by thorough cold water rinsing to effect complete removal of excess blackening solution. After rinsing, the parts should be dried by warm dry air and given a chromic acid dip as outlined in 3.4.3.

3.6 Alkaline oxidizing (class 4). The black coating of class 4 shall be processed in accordance with instructions furnished by the suppliers of the raw materials and the resulting coating shall conform to the applicable requirements of this specification.

3.7 Coverage and color. Class 1, 2, 3, and 4 coatings (see 1.2) shall cover the basis metal completely and shall pass the smut test. The color shall be a uniform black. A slight amount of smut, which is inherent in the process, shall not be cause for rejection. There shall be no indication of any reddish-brown or green smut when tested as in 4.4.1. "Spottiness" or streaking shall be classified as unsatisfactory and shall be grounds for reprocessing.

3.8 Oxalic acid spot test (class 1, 2, and 3). The black oxide coatings of classes 1, 2, and 3, prior to the application of a preservative, shall pass the oxalic acid spot test for a good quality coating IAW SAE AMS2485 and as described in 4.4.2.

3.9 Resistance to salt spray (fog) (class 4 AISI type 300 series corrosion resistant steel only). The black coating, of class 4 (300 series only), prior to the application of a preservative, shall show no signs of corrosion after 96 hours of exposure in the salt spray test (see 4.4.3).

3.10 Hydrogen embrittlement relief treatment. Steel parts that are surface or through hardened at 39 HRC and above shall be given a hydrogen embrittlement relief treatment after application of the oxide coating. Coated springs or other parts subject to flexure shall not be flexed prior to the embrittlement relief treatment. If an embrittlement relief treatment is required, it shall follow the final water rinse. The embrittlement relief treatment precedes the supplementary preservative treatment.

3.11 Supplementary preservative. Materials for supplementary preservative treatments and methods of application shall be in accordance with the applicable requirements of the end item specification, or as otherwise specified. Unless otherwise specified, the supplementary preservative treatment shall be applied to the clean and dry parts immediately after the final stage of processing or embrittlement relief treatment.

3.12 Workmanship. The surface of the coated part shall be uniform in appearance and free of visible coating defects, such as blisters, pits, roughness, nodules, burning, cracks, or uncoated areas, and other defects that will affect the function of the coating. The coated parts shall be clean and free of damage.

4. VERIFICATION

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements (examinations and tests) as specified herein and documented (recorded) as Objective Quality Evidence (OQE) defined as a statement of fact, either quantitative or qualitative, pertaining to the quality of the product or service based on observations, measurements, or tests which can be verified. (Evidence will be expressed in terms of specific quality requirements or characteristics described herein). Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform or review any of the inspections set forth in this specification where such oversite is deemed necessary to ensure supplies and services conform to prescribed requirements.

4.1.1 Classification. All the tests required for the testing are classified as Objective Quality Evidence, for which necessary sampling techniques and the methods of testing are specified in this section.

4.2 Sampling.

4.2.1 Lot. A lot shall consist of coated parts of the same class, same basis metal, and approximately the same size and shape and coated under similar conditions. A maximum of 8 hours continuous production shall constitute a lot.

4.2.2 Selection. The number of random samples selected for nondestructive tests (visual inspection, coverage, color, smut, and workmanship), and destructive tests (oxalic acid spot test, salt spray test, and embrittlement relief test) from each lot of coated parts shall be defined by the procuring activity (see 6.2).

4.3 Acceptance and rejection. The selected coated parts shall meet the requirements in this specification for the represented lot to be acceptable. Failure of any test samples to meet the requirements in this specification shall constitute rejection of the entire lot which they represent. Unless otherwise specified (see 6.2), the tests shall be conducted and the test results accepted prior to shipment of the lot of coated parts represented by the test samples.

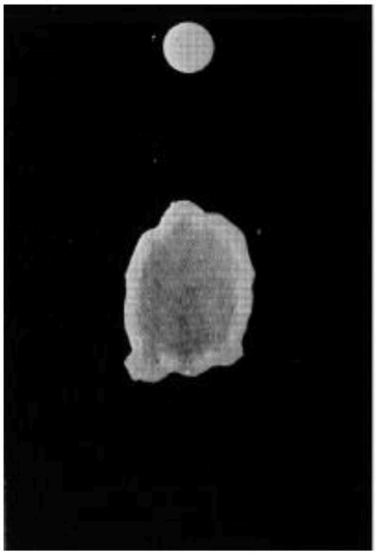
4.3.1 Surface attack. A sampling of the parts shall be examined for pitted surfaces or intergranular attack by viewing them at 10x.

4.4 Test procedures.

4.4.1 Smut test. The test shall be made prior to application of corrosion preventive compound or after degreasing to remove the preservative. Each black oxide coated piece shall be inspected visually under strong light to assure a satisfactory appearance. Each sample shall also be wiped with a clean white cloth for indications of smut (see 3.7). A

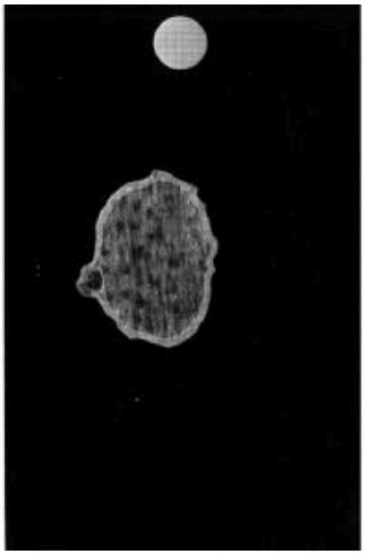
slight amount of smut which is inherent in the process is acceptable for all classes of coatings and shall not be cause for rejection.

4.4.2 Oxalic acid spot test (class 1, 2, and 3). The black oxide coated pieces of classes 1, 2, and 3 only, prior to the application of a preservative, shall be handled with clean cotton gloves or the equivalent. Each sample shall have deposited, on one flat spot of the black oxide coated surface, three drops (0.00676 oz. (0.2 ml)) of a five percent solution of oxalic acid. The reaction shall be observed after 30 seconds and up to eight minutes. After eight minutes the panel shall be rinsed with water and compared to figures 1, 2, and 3. A light gray center with a lighter border color (Figure 1) indicates a poor quality coating. A gray-black center with a light border (Figure 2) indicates a borderline quality coating. The coating shall conform to the good quality coating of Figure 3 to be acceptable. A black or dark brown center with a light border (Figure 3) indicates a good quality coating. A good quality coating may show a light border, indicating exposure of metal around the drops. Parts shall therefore be judged only on the color and exposure of the metal under the drops.



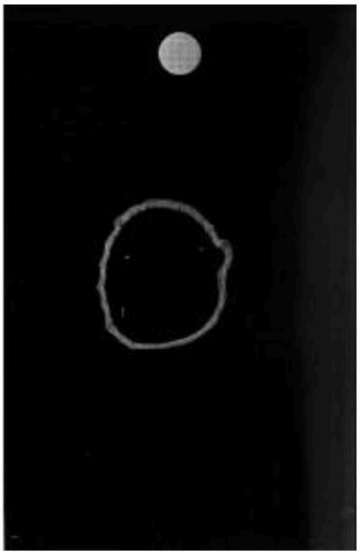
A poor quality coating.

Figure 1.



A borderline quality coating.

Figure 2.



A good quality coating.

Figure 3.

4.4.3 Resistance to salt spray (class 4). The coated pieces of class 4 (AISI type 300 series corrosion resistant steel) shall be subjected to a 5 percent salt spray (fog) test in accordance with ASTM B117. Exposure time for the black coatings, prior to the application of a preservative, or after vapor degreasing, shall comply with the requirements of 3.9.

4.4.4 Hydrogen embrittlement relief treatment. When specified (see 6.2), ferrous parts shall be baked in accordance with SAE AMS 2759/9, Table 1.

4.4.4.1 Hydrogen embrittlement relief test. Samples selected in accordance with 4.2.2 to determine the adequacy of the hydrogen embrittlement relief treatment, shall be tested in accordance with ASTM F519. When specified (see 6.2), the standard notched round bar specimen under load control described in Table I, which is a part of ASTM F519, shall be used. The lot shall be rejected if any coated specimen develops any crack or fails by fracture as a result of the test.

TABLE I. Materials and processing procedures.

Class	Applicability to ferrous metals	Process and possible chemicals	Approximate processing temperature	Approximate immersion time
1	Carbon steels, low alloy steels, wrought irons, cast and malleable irons	Alkaline oxidizing, NaOH, $NaNO_3$, H_2O	1 or 2 tanks boiling at 285 - 305°F (141 - 152°C)	5 to 60 min
2	Certain corrosion resistant steel alloys which are tempered at less than 482°C	Alkaline chromate, NaOH, $NaNO_3$, $Na_2Cr_2O_7$ 1/, H_2O	250°F ± 10°F (121°C ± 5°C)	30 to 45 min
3	For corrosion resistant steel alloys which are tempered at 900°F (482°C) or higher	Fused salt oxidizing, $Na_2Cr_2O_7$ 1/, and/or $K_2Cr_2O_7$ 1/	Molten bath 750 – 850°F (399 – 454°C)	30 min
4	For corrosion resistant steel alloys	Alkaline oxidizing, proprietary compounds plus H_2O	250 - 265°F (121 – 130°C)	15 to 30 min

1/ Chromium compounds.

5. PACKAGING

5.1 Packaging. Packaging requirements are not applicable to this specification.

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 black oxide coatings covered by this specification are military unique. Black oxide coatings are used on munition cases and gun barrels. These coatings are particularly suited for moving parts that cannot tolerate the dimensional buildup of a more corrosion resistant finish. They are not recommended on parts going into long term storage. Sometimes, long term storage is required and a protective preservative application is recommended or a desiccated package is utilized. The coatings present a pleasing black appearance frequently employed for decorative purposes or decrease in light reflection. A supplementary water displacing preservative coating such as MIL-PRF-16173, grade 3 or comparable material which will provide equal or superior corrosion protection may be specified.

CAUTION: High strength steel (39 HRC or greater hardness) may be subjected to "caustic embrittlement" that could lead to spontaneous cracking if under internal or applied stress during the blackening treatment.

6.2 Acquisition requirements. Acquisition documents should specify the following:

(a) Title, number, and date of this specification.

(b) Class of coating (see 1.2).

(c) Number of samples to be inspected from the lot of coated parts (see 4.2.2).

(d) Acceptance requirements prior to shipment of the coated parts, if different (see 4.3).

(e) Use the standard notched round bar specimen under load control described in annex A1, which is a part of ASTM F519 (see 4.4.4.1).

6.3 Miscellaneous notes.

6.3.1 Dimensional change. Black oxide coatings on iron and steel should produce no appreciable dimensional change of the treated piece. The dimensions shown on the drawings are, therefore, the dimensions after the application of the coatings.

6.3.2 Processing. Contractors may employ one of a number of trade name black finishing oxidizing materials or prepared chemical mixtures to apply the black oxide coating. The processing details should conform to MIL-HDBK-205, “Phosphatizing and Black Oxide Coating of Ferrous Metals”, or as recommended by the material supplier. Class 2 is used to process the 4XX series corrosion resisting steels, class 3 is used for 3XX and 4XX series, and class 4 is used to process those 300 series corrosion resisting steels which can meet the

special salt spray test criteria and for those 4XX series corrosion resisting steels which do not have any special salt spray test criteria. Table I provides an outline of the various processes.

6.3.3 Cast and malleable irons, and certain 400 series corrosion resistant steels. Cast and malleable irons and 400 series corrosion resistant steels of the martensitic type can also be effectively treated in Class 4 proprietary baths, but will not meet the salt spray requirement of austenitic 300 series corrosion resistant steels.

6.3.4 Class 4 coatings. Corrosion resisting steels of minimum composition l7Cr-7Ni can be effectively blackened by this process.

6.3.5 Rinsing. In order to obtain effective removal of blackening solution and ensure thorough rinsing, a combination of spray rinses with tank rinses or a properly operated double counter-flow rinse operation may be advantageous. Use of such a system may help reduce the amount of water required to obtain a desired rinsing criterion and facilitate meeting the EPA standard.

6.3.6 Ozone depleting chemicals. Classes I and II ozone depleting chemicals should be avoided when cleaning the basis metal.

Hazardous materials. For more information, please refer to the online list of toxic chemicals and hazardous substances maintained by the Environmental Protection Agency at https://www.epa.gov/epcra/consolidated-list-lists

These chemicals should be avoided whenever another finish can be substituted for the finish specified herein provided all performance criteria has been met and documented as OQE.

6.4 Subject term (key word) listing.

Non-hexavalent chrome dip
Chromic acid dip
Chromium compounds
Embrittlement relief
Oxalic acid spot test
Preservative
Salt spray
Smut test

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

CONCLUDING MATERIAL

Custodians:
Army – MR
Navy – OS
Air Force – 20

Review activities:
Army - MI, PT
Navy - AS, EC, SH
Air Force - 13, 19
DLA – DH

Preparing activity:
Army - MR
(Project MFFP-2018-007)

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