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TECHNICAL REPORT

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CORROSION RESISTANCE OF BLACK OXIDE
COATINGS ON MILD AND CORROSION RESISTANT STEELS

By

Linden H. Wagner

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Engineering Support of Standardization Project X.O. 76502-9001

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Approved by:

G. O. Inman

G. O. INMAN
Acting Laboratory Director

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Rock Island Arsenal
Rock Island, Illinois

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ABSTRACT

In the revision of Military Specification MIL-C-13924A, dated 29 December 1956, the various processes were reviewed and evaluated for their effectiveness in blackening mild steel, 300 and 400 corrosion resistant steels. The corrosion resistance of these coatings were also investigated. A low temperature Class 4 proprietary alkaline-oxidizing process was proposed for blackening only the 300 series stainless steels in lieu of the fused salt oxidizing process which operates at 760°F.

The Class 2 alkaline-chromate process did not blacken the 300 series stainless steels. Class 3 and Class 4 processes did. The mild steel control panels, coated and uncoated, failed the salt spray test in one hour. The electrodeposited coating failed in three to four hours. Freshly abraded control stainless steel panels 302, 310 and 321 were without rust after 500 hours. Proprietary coatings A-1 and B applied to freshly abraded 302 and 310 series stainless steel panels were without rust after 500 hours. On the 321 series stainless steel panels, initial rust was noted on Coating A-1 after 165 hours and on Coating B after 141 hours.

The 300 series stainless steels specimens coated with the type 3 (molten dichromate process) failed in the salt spray test in one hour.

RECOMMENDATION

It is recommended that:

1. Proprietary compounds A-1 and B be used in the Class 4, low temperature alkaline-oxidizing process for blackening type 300 stainless steels only, and not be used on mild steel, or type 400 corrosion resistant steel.
2. The Class 4 process be used in place of the Class 3, fused salt oxidizing process, for blackening type 300 stainless steels, because of superior corrosion resistance and simplicity of application.

**CORROSION RESISTANCE OF BLACK OXIDE
COATINGS ON MILD AND CORROSION RESISTANT STEELS**

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CORROSION RESISTANCE OF BLACK OXIDE COATINGS ON MILD AND CORROSION RESISTANT STEELS

OBJECT

To determine the effectiveness of low temperature alkaline proprietary processes for blackening stainless steels.

To determine the corrosion resistance of the alkaline proprietary coatings applied to 300 series stainless steels and M1, gas cylinders.

To revise MIL-C-13924A according to the findings of this investigation.

INTRODUCTION

Military Specification, "Coating, Oxide, Black, for Ferrous Metals," MIL-C-13924A, dated 26 December 1956, covers black oxide coatings applied to ferrous metals (wrought iron, carbon, low alloy, and corrosion resistant steels). (1,2,3,4,5) Black sulfide coatings are not included in this specification.

The black oxide coating processes are classified as follows:

Class 1 - Alkaline oxidizing process (for wrought iron, plain carbon, and low alloy steels)

Class 2 - Alkaline-chromate oxidizing process (for corrosion resistant steels)

Class 3 - Fuzed salt oxidizing process (for all grades corrosion resistant alloy and carbon steels having a draw temperature above 900°F.)

This standardization project was concerned with the following problem: The Class 3 process for corrosion resistant steels utilizes molten dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7$) at 760°F. in the blackening of these steels. This is a hazardous operation, and it is difficult for the Department of the Army to locate contractors who are willing to use it. As a result, it was brought to the attention of the Department of the Army that a low temperature, commercially available blackening material had been used to produce satisfactory black coatings on corrosion resistant steels. Commercial samples of these materials were obtained; baths were prepared, and several types of steel specimens were processed. The coatings were comparatively evaluated for resistance to corrosion in the salt spray test. A revision

of the specification was made to include the Class 4 proprietary coatings in MIL-C-13924B, dated 10 July 1964. (FSC6850, Project No. 6850-0103). Appendix A, Rock Island Arsenal Report on "Alkali Black Finishes" describes the blackening process and properties of the coating. It is included in this report for informational use.

PROCEDURE AND RESULTS

The materials used in the tests, their treatment, and the results of the salt spray tests are listed below as follows:

Materials

Mild carbon steel panels, 2" X 3" X 1/16", SAE #1020. Austenitic stainless steel panels, 2" X 4" X 1/8", AISI #302; 2" X 3" X 1/16", #310, and #321 Martensitic stainless steel; M1, gas cylinders, AISI #410. The chemical composition of the steels is shown in Table I. (6)

These items were sandblasted to renew the surface, and cleaned in an alkaline derust MIL-C-14460 Type 2 solution with periodic reversal of current. After a hot and cold water rinse, they were blown dry with air.

Treatments

Process #1 - Controls. Representative items of the above were selected as controls.

Process #2 - (Passivation). The clean items were treated as outlined in Table V-A, par. 5.4.1(b) to par. 5.4.2(b) of Spec. MIL-STD-171A(ORD), dated 11 October 1960. The procedure is outlined as follows. Cleaning and passivating:

(a) Alkaline derust method was used instead of finish 4.3 (Spec. TT-C-490, Method II.)

(b) Immerse for 30 minutes in a hot (120° to 130°F.) aqueous solution containing 20 percent by volume of nitric acid (sp. gr., 1.42) and 2 percent by weight of sodium dichromate.

(c) Rinse in clean hot water.

(d) Immerse for one (1) hour in a hot (140° to 160°F.) aqueous solution containing 5 percent by weight of sodium dichromate.

TABLE I
CHEMICAL COMPOSITION OF COMMON AND CORROSION RESISTANT STEELS

Material		Element and Percentage							
AISI	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Chromium	Nickel	Titanium	
302	0.15	2.00	0.045	0.03	1.0	17-19	8-10	----	
310	0.25	2.00	0.045	0.03	1.5	24-26	19-22	----	
321	0.08	2.00	0.045	0.03	1.0	17-19	9-12	5 X Carbon, Min.	
410	0.15	1.00	0.040	0.03	1.0	11.5-13.5	----	----	
SAE 1020	0.18-0.23	0.30-0.60	----	----	----	----	----	----	

(e) Rinse in clean hot water.

(f) Rinse in hot (160° to 210°F.) aqueous solution maintained at pH 3 to pH 5 by the addition of flake chromic acid or proprietary mixture of chromic and phosphoric acids.

Process #3 - (Code A). The clean items were treated in accordance with "Instructions for the Black Passivation of Corrosion Resistant Steel," a Bureau of Naval Weapons publication, Code Ident. 10001, OD 15269, dated 15 February 1961. Proprietary compound A was then applied as outlined in the following processing procedure:

(a) Immerse the parts in a commercial type alkali cleaner in the recommended proportions and operating temperature for sufficient time to produce a chemically clean surface. (An alkaline derust method was used).

(b) Rinse in clean, running water.

(c) Immerse the parts in a 50% muriatic acid solution (50% by volume acid to 50% by volume water) for 5 minutes.

(d) Rinse in clean, running water.

(e) Immerse parts in 40% sulfuric acid solution (40% by volume sulfuric acid to 60% by volume water) for 1 to 1-1/2 minutes.

(f) Rinse parts in clean, running water.

(g) Immerse in black passivating bath operating at a boiling temperature of 255° - 260°F. (This is an alkali-nitrate salt mixture which is mixed with sufficient water to make a black passivating solution with a boiling point of 255° - 260°F.) Immersion time shall be of sufficient duration to produce a uniform black finish.

(h) Rinse thoroughly in clean, running water.

(i) Remove and blow-dry with air.

Process #4 - (Code B). Proprietary compound B was applied as outlined in the processing procedure of the supplier for blackening stainless steel, plain steel, nickel alloys, invars, monels, cast and malleable iron. The items were immersed for 1 to 1-1/2 minutes in an activator bath containing 3 lbs. of crystalline material per gallon maintained at 180°F. The items were removed, rinsed in water and placed in the blackening bath con-

taining 4.5 to 5.0 lbs. per gallon maintained at 250° to 255°F. for 30 minutes. They were removed, rinsed thoroughly in water, and dried with air.

Process #5 - (MIL-C-13924 Class 2). The clean items were blackened by the alkaline chromate process⁽⁷⁾ outlined in Spec. MIL-C-13924A, Class 2, operated at 290°F. for a period of 30 minutes. They were removed, rinsed thoroughly and dried with air.

Process #6 - (MIL-C-13924 Class 3). The clean items were blackened by the fused salt oxidizing process ($\text{Na}_2\text{Cr}_2\text{O}_7$) outlined in Spec. MIL-C-13924A, Class 3 at 700° to 760°F. for 30 minutes. The process is described in MIL-HDBK-205.⁽⁸⁾

Process #7 - (Black chromium electrodeposit). The clean items were coated for 30 minutes with electrodeposited black chromium as outlined in Spec. MIL-P-14538(ORD).^(9,10,11)

The items with the above finishes were subjected to the 5% salt spray test outlined in Fed. Test Method Std. No. 151a, Method 811.1, dated 6 May 1959. They were comparatively evaluated. The surface treatment of the items, the rust ratings, and the hours in test are shown in Table II.

Table II shows that the mild steel panels (1020) given treatments 1 through 6 failed after one (1) hour of salt spray exposure. Treatment 7, however, afforded from 3-4 hours protection. The condition of the panels after exposure is shown in Figure 1.

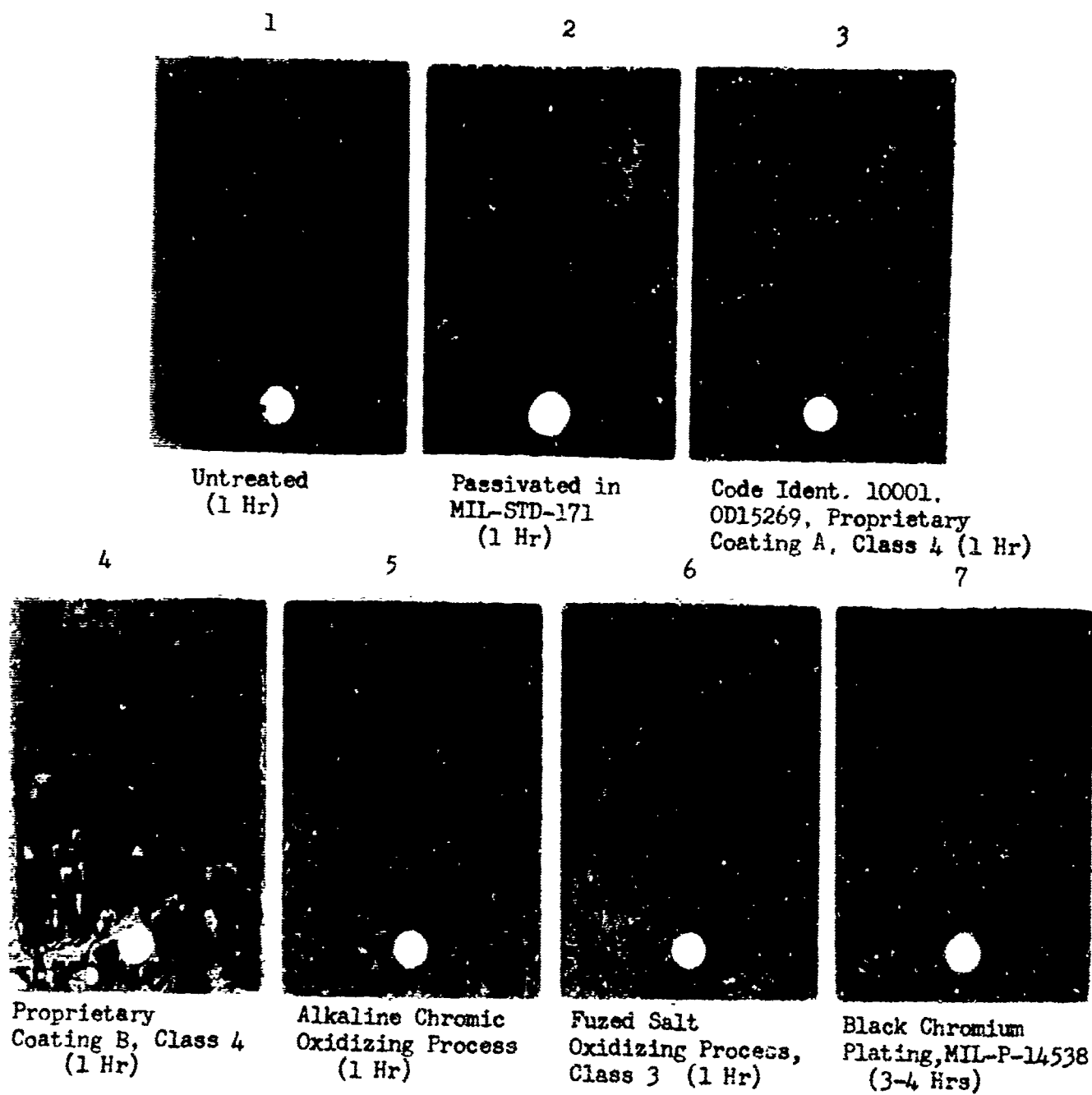
Table II shows that the treated series 300 alloy steels were of similar corrosion resistance within the group. The Class 2 process did not produce a coating. The condition of the panels after exposure is shown in Figures 2, 3 and 4, respectively.

The coatings on the M1, gas cylinders, #410 (1 through 6) failed to afford protection after one (1) hour exposure. Treatment 7 afforded from 6 to 10 hours of protection. The condition of the cylinders after exposure is shown in Figure 5.

Soon after the above tests were completed the supplier of Compound A, in view of the adverse findings, submitted sample A-1, a revised formulation. In order to evaluate this sample, the following procedure was established. Mild steel panels of #1020 and corrosion resistant panels of #302, #310, and #321 were resurfaced, first with #150 and finally with #240 grit aluminum oxide cloth. The panels were vapor degreased in trichloroethylene and placed in a desiccator. New pickle, activator, and blackening baths

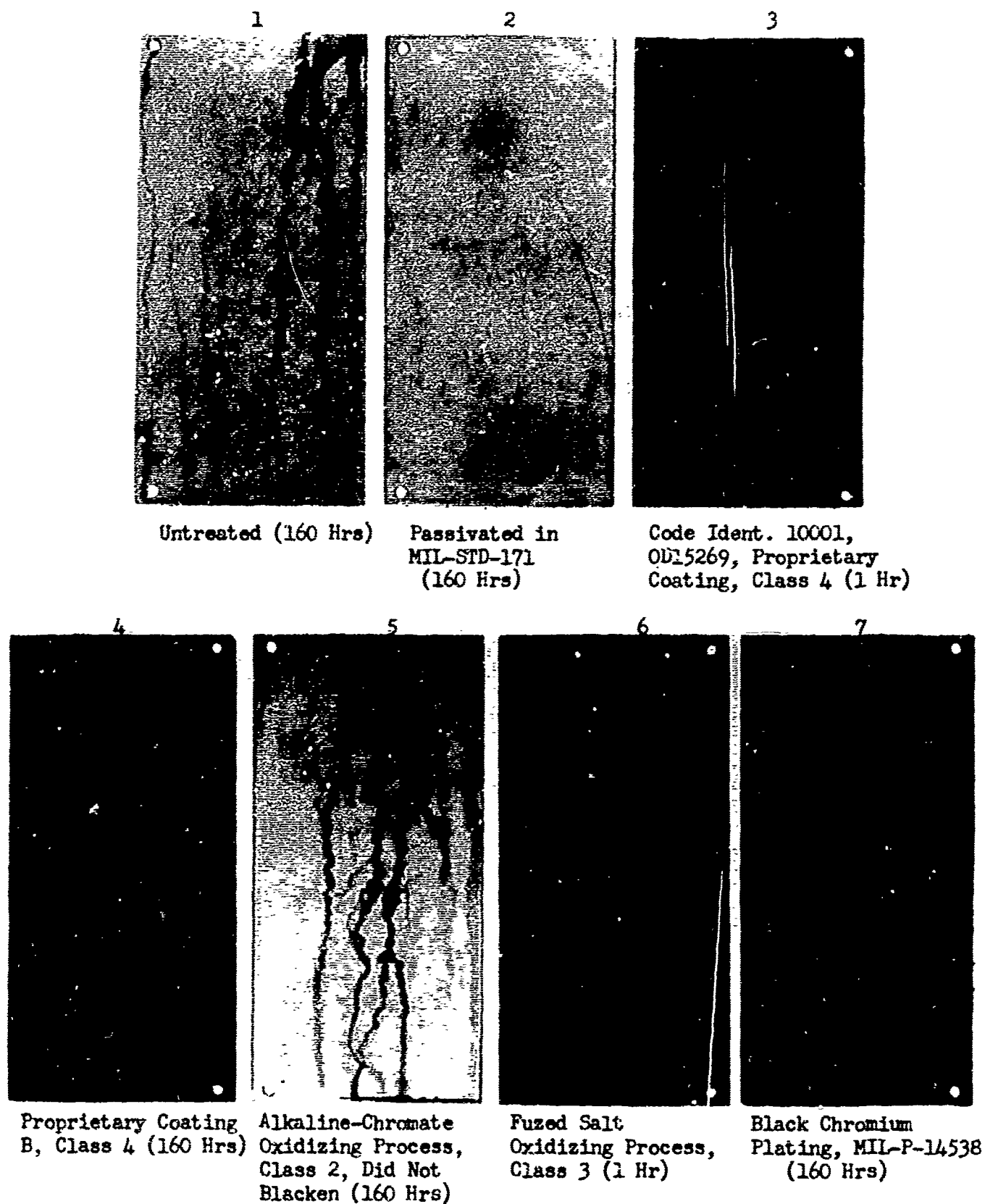
TABLE II
RESULT OF SALT SPRAY TEST ON BARE, PASSIVATED AND APPLIED FINISHES

Process Treatment	1		2		3		4		5		6		7	
	Bare Controls		MIL-STD-171A(ORD)		Code Ident.		Compound B Test		Alkaline Chromate Process		Molten Dichromate Process		Electrolytic Black Chromium	
	1	2	1	2	A	A-1	1	2						
Steel	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 3-4	Fail 3-4
#302	Pass 160	Pass 500	Pass 160	Pass 160	Fail 1	Pass 500	Pass 160	Pass 500	Did not blacken 160	Fail 1	Fail 1	Pass 160	Pass 160	Pass 160
#310	Pass 160	Pass 500	Pass 160	Pass 160	Fail 1	Pass 500	Pass 160	Pass 500	Did not blacken 160	Fail 1	Fail 1	Pass 160	Pass 160	Pass 160
#321	Pass 160	Pass 500	Pass 160	Pass 160	Fail 1	Pass 165	Pass 160	Pass 141	Did not blacken 160	Fail 1	Fail 1	Pass 160	Pass 160	Pass 160
#410 M1, Cyl.	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 1	Fail 6-10	Fail 6-10



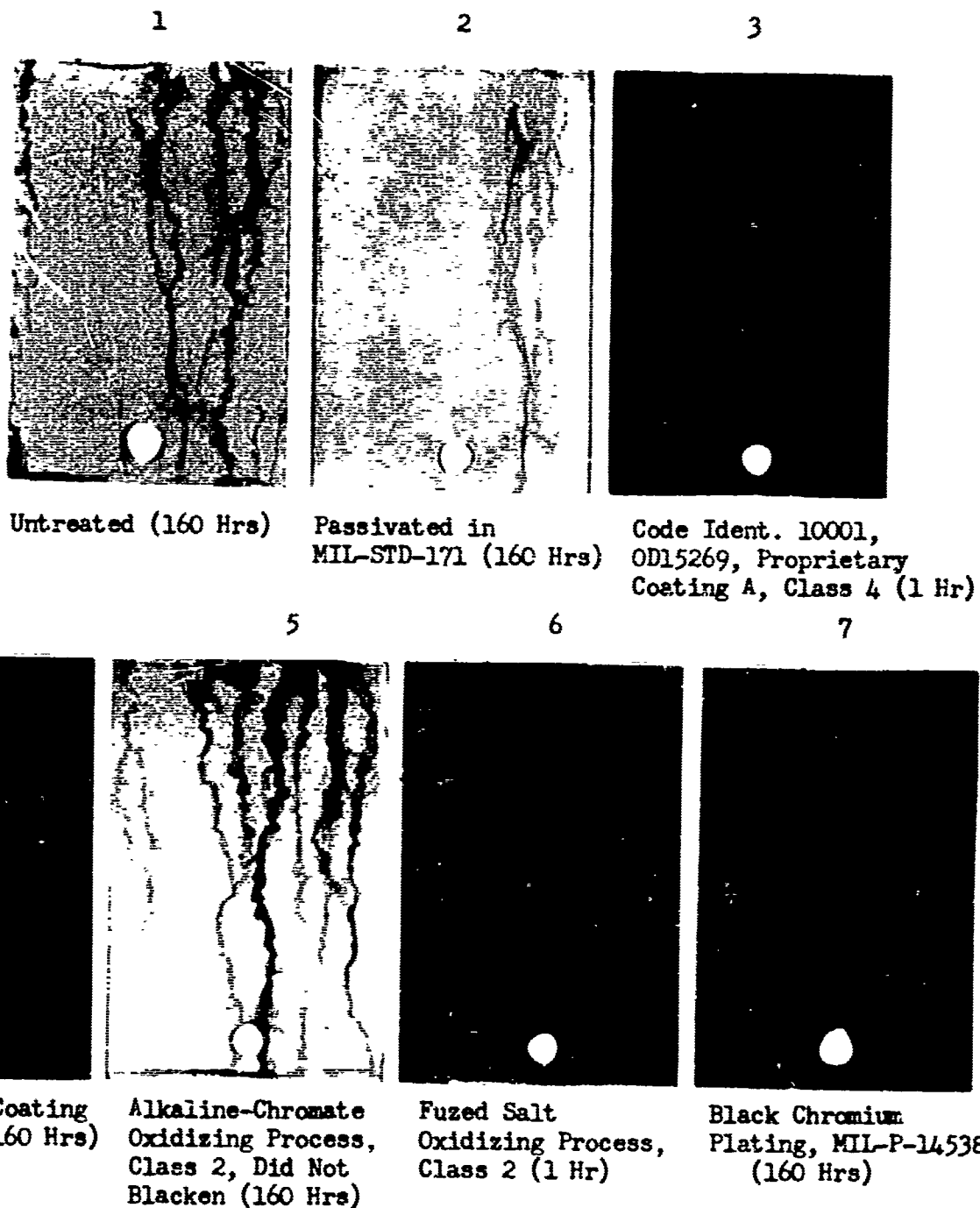
SALT SPRAY (FOG) EXPOSURE TEST ON
MILD STEEL SAE 1020 PANELS

Figure 1



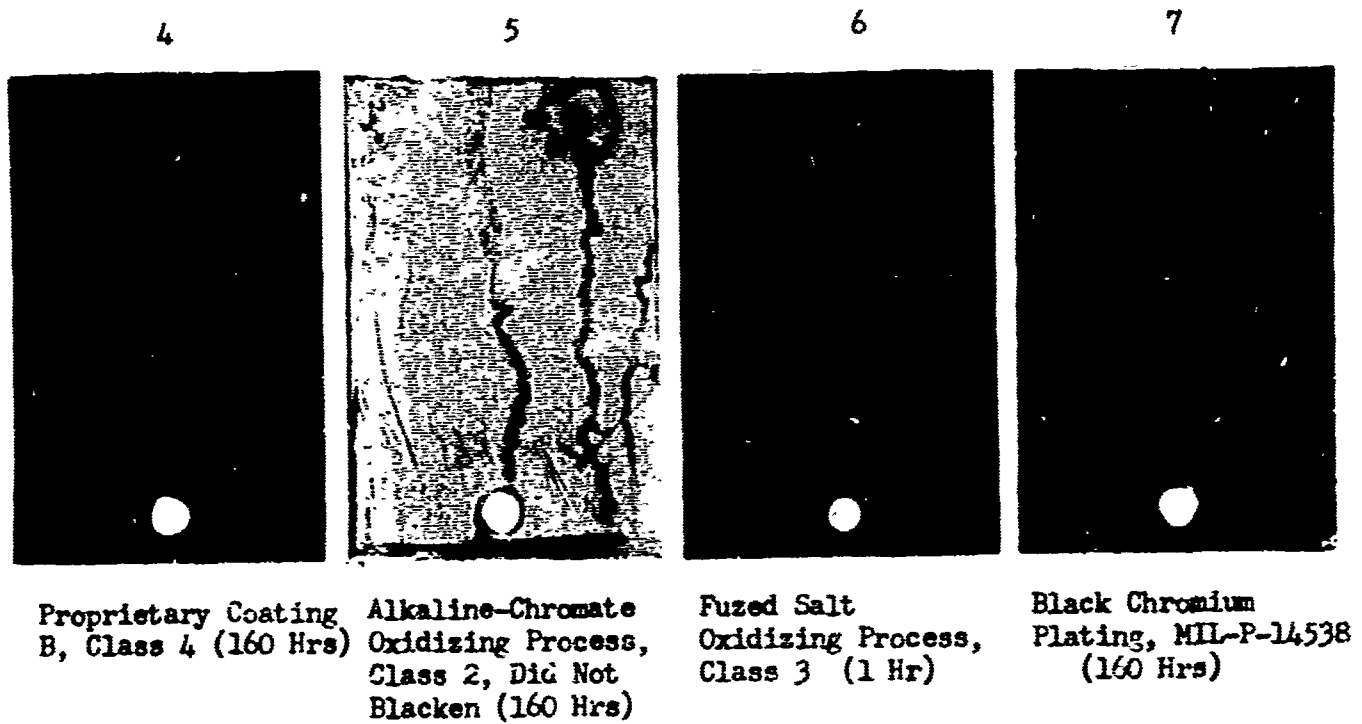
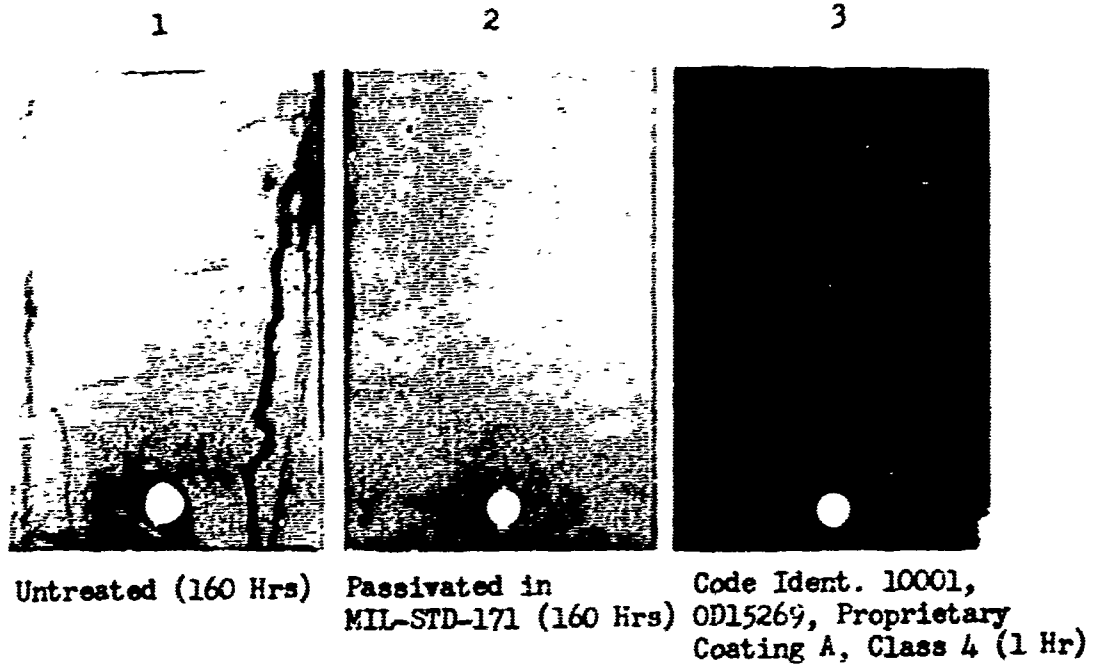
SALT SPRAY (FOG) EXPOSURE TEST ON
#302 STAINLESS STEEL PANELS

Figure 2



SALT SPRAY (FOG) EXPOSURE TEST #310 STAINLESS STEEL PANELS

Figure 3

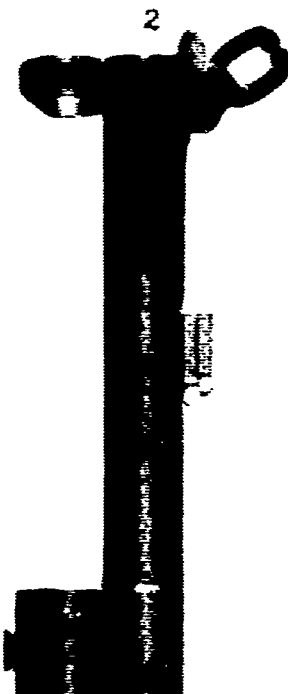


SALT SPRAY (FOG) EXPOSURE TEST #321 STAINLESS STEEL PANELS

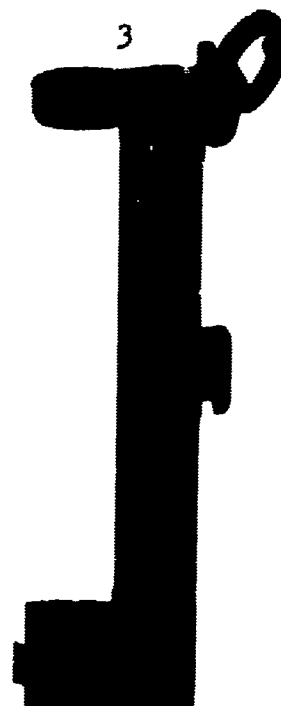
Figure 4



Untreated (1 Hr)



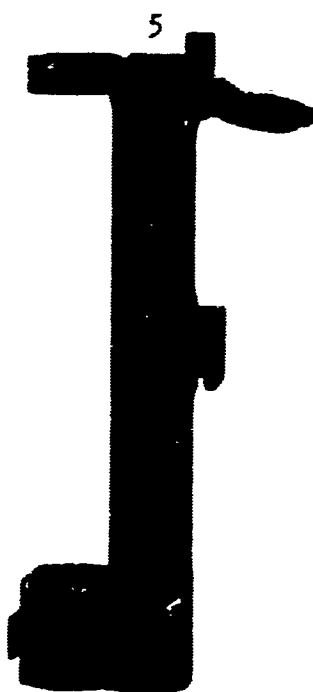
Passivated in
MIL-STD-171 (1 Hr)



Code Ident 10001,
OD15269, Proprietary
Coating A, Class 4 (1 Hr)



Proprietary Coating
B, Class 4 (1 Hr)



Alkaline-Chromate
Oxidizing Process,
Class 2 (1 Hr)



Fuzed Salt
Oxidizing Process,
Class 3 (1 Hr)



Black Chromium
Plating, MIL-P-14538
(6-10 Hrs)

SALT SPRAY (FOG) EXPOSURE-TEST ON #410 STAINLESS STEEL GAS CYLINDERS

Figure 5

were used prior to processing the freshly abraded panels. The blackening procedures for processing proprietary compounds A-1 and B were as follows:

<u>Compound A-1</u>	<u>Compound B</u>
<u>Acid Pickle</u>	<u>Activator Solution</u>
1 part (by vol.) water	350 - 360 gms/liter
2 parts (by vol.) hydro-chloric acid	
Room Temperature	Processing Temperature 180°F.
<u>Immersion Time (minutes)</u>	
2-5	1-1/2 - 2
<u>Water Rinses</u>	
1 warm	1 warm
1 cold	1 cold
<u>Blackening Bath</u>	
450 gms/liter	550 - 600 gms/liter
<u>Processing Temperature</u>	
250° - 260°F.	250° - 260°F.
<u>Immersion Time (minutes)</u>	
30	30
<u>Water Rinses</u>	
1 warm	1 warm
1 cold	1 cold
Dry with air	Dry with air
<u>Stop-Off Wax</u>	
to seal edge of panel	to seal edge of panel

In order to evaluate the corrosion resistance of the proprietary coatings the following comparative procedure was established. Three (3) sets of panels were prepared in duplicate: One (1) set of physically abraded panels were used as controls. The second (2) set was blackened with Compound A-1. The third (3) set was blackened with Compound B. The control and the two blackened sets were simultaneously placed in the salt spray (fog) cabinet.

The panels were inspected periodically and the following observations were noted. They are shown in Table II, as test 2.

Set 1, Controls. The mild steel panels, 1020, were about 90-95% rusted after one hour. The stainless steel panels #302, #310 and #321 were without rust after 500 hours.

Set 2, Blackened with Compound A-1. The mild steel panels, 1020, were about 5 to 15% rusted after one (1) hour. The stainless steel panels #302 and #310 were without rust after 500 hours. The #321 panels showed initial rust after 16⁵ hours. After 500 hours these latter panels were from 15 to 20% rusted.

Set 3, Blackened with Compound B. The mild steel panels, 1020, were from 10 to 15% rusted after one (1) hour. The stainless steel panels #302 and #310 were without rust after 500 hours. The #321 panels showed initial rust after 141 hours of exposure. After 237 hours exposure the panels were from 20 to 25% rusted.

In the previous test shown on Figures 2, 3, and 4, proprietary Compound B coated on series #302, #310 and #321, previously cleaned in an alkaline-derust solution, passed 160 hours in the salt spray test, whereas, Compound A failed in one (1) hour. The revised formulation, Compound A-1, was equally as affective as Compound B when tested as above on freshly abraded panels.

DISCUSSION

Sandblasted mild steel panels and M1 gas cylinders, subjected to the various blackening processes have been shown to exhibit poor corrosion resistance in the salt spray test. No alkaline-oxidizing process afforded more than one-half to one hour protection on the mild steel panels. The electrodeposited black chromium process afforded the best protection on the mild steel panels and the M1 gas cylinders.

It was shown that the proprietary blackening compounds A and B blackened the #302, #310 and the #321 stainless steel panels; whereas, the alkaline-chromate process of Class 2 did not. It did, however, blacken the M1 gas cylinders of corrosion resistant steel #410. In lieu of the Class 2 process, the fuzed salt oxidizing process is recommended to blacken the 300 and 400 series steels; however, because this process operates at 760°F., only items that can be heated to this temperature without damage may be blackened. Furthermore, the coating has poor resistance to

corrosion. Since this process is conducted at high temperature with inherent hazards, the Class 4 proprietary blackening process was proposed. This low temperature process is much more applicable to industrial applications.

The protection afforded by the Class 4 proprietary coatings on the 300 series stainless steels is superior in resistance to corrosion to the Class 3 molten dichromate process.

On the 302 and 310 series stainless steels the Class 4 coatings were superior in their resistance to corrosion in comparison with the controls.

On the 321 series stainless steels the uncoated panels were more resistant to corrosion than the coated panels. The resistance to corrosion was 500 hours against 165 hours. It has been shown that initial cleaning, activation, and passivation of the surfaces affects the protection that can be attained. The alkaline-derust solution with periodic reversal of the current removed the superficial rust and the more soluble material from the panels and the gas cylinders. Activation of the surface by acids, or acid salts, tends to etch the surface lightly, effects removal of the surface contaminants, and makes the surface more receptive to the alkaline-oxidizing process. Passivation of the surface in a nitric acid-sodium dichromate solution tends to remove the more corrodable elements and leave the less corrodable material. However, where inclusions of mild steel are heterogeneously distributed in the surface, pitting can be noted in the salt spray test.

The protection afforded by the Class 4 compounds is dependent on the cleaning and preparation of the basis metal prior to the application of the oxide coatings.

When corrosion products from a corroding area runs down over an uncorroded area, corrosion to that second area is accelerated.

In this work it was found that physical abrading of the surface of 300 series stainless steel panels with #150 and #240 grit aluminum oxide, and subsequently cleaned in trichloroethylene, provided a clean surface that was capable of withstanding over 500 hours of salt spray exposure. Proprietary coatings applied to this surface were found to provide protection over a similar period of time.

It is known that the primary purpose of the black oxide coating is to blacken steel for decorative purposes.

This work was performed with the thought in mind that it would be best to obtain a black coating which did not harm the basis metal. The low temperature Class 4 process was proposed to be used in place of the present Class 3 molten dichromate process. The Class 3 process produces a black coating which affords poor resistance to corrosion for the basis metal. The high operating temperature limits application to items which will not be metallurgically damaged.

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Gilbert, L. O.	"Alkali Black Finishes" Rock Island Arsenal Lab. Report No. 41-3530, 28 March 1942.
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APPENDIX A

ROCK ISLAND ARSENAL LABORATORY REPORT #41-3530 of Alkali Black Finishes

1. The alkali black finishes are produced in strongly alkaline solutions containing varying amounts of some oxidizing agent. The chemical used in building the solution usually consists of 60-85% caustic soda, and 15-40% oxidizing agent such as sodium nitrate, sodium nitrite, a mixture of sodium and potassium nitrate or nitrite, trisodium phosphate, sulphur, manganese oxide, or sodium molybdate. The exact percent of oxidizing agent is dependent upon the material used. A very satisfactory chemical for the preparation of an alkali-black type processing bath may be produced by mixing 60 percent sodium hydroxide (caustic soda) and 40 percent sodium nitrite. Sodium nitrate may be substituted for nitrite but does not give a coating as resistant to corrosion as that produced when sodium nitrite is used. The chemical is added to water until a solution boiling at 290°F. is obtained. This mixture constitutes the solution used in the first processing tank. The second or high temperature bath is made up with the same chemical except that a larger quantity must be added in order to obtain a boiling temperature of 300°F. Parts to be processed should be thoroughly cleaned, immersed for one-half hour in the solution boiling at 290°F., removed and placed immediately in the bath operating at 300°F. for one-half hour after which they are rinsed in cold and hot water, dried in an air blast and dipped in oil. Solutions must be kept at the boiling point during processing.

2. In the case of parts where the surface has been sandblasted (or shot blasted) prior to processing, the parts after several weeks storage during humid summer months may develop a red film of rust. This rust formation may be attributed to magnetic particles of iron removed by the sandblast. They remained attached to the rough surface. These particles are converted by the solution to rust and when oiled are nearly invisible. After a short period of storage a red film forms over the parts even though covered with a rust preventive oil. This action is due to oxidation of the attached particles which are more susceptible to the action of the air than the surface. The presence of these particles of rust then promotes rusting of the entire surface. If the sandblasted parts are given a one minute phosphoric acid pickle before processing, this difficulty may be overcome. In this treatment the attached particles are

dissolved by the acid and no further difficulty with rusting is encountered. Phosphoric acid is used for this purpose in order to eliminate the possibility of contamination of the bath by rust promoting salts such as sodium sulfate which would be formed in the solution if sulfuric acid is used and the articles improperly rinsed. In use, this danger is eliminated as trisodium phosphate is one of the constituents used in some alkali black processing solution. In the nitrite type of bath, copper or brass such as brazed joints or parts having brass or copper sleeves should not be processed due to the dissolving action of the nitrite on copper. This dissolved copper later shows up as a red film on the work as removed from the bath. This film of deposited metallic copper can be wiped from the work after application of oil. However, this film may collect on inaccessible parts of the work such as in threads, blind holes and corners. This copper film, unless completely removed, may accelerate corrosion of the entire surface of the part. A bath contaminated with copper may be cleared of this contamination by processing several pounds of steel wool in each tank. A similar film of red rust is formed on parts when the temperature of the first (low temperature 290°F.) bath is maintained too high for the type of steel being processed. The temperature should be lowered, if upon removal from the low temperature bath, the parts show a greenish or red coloration. Various steels require minor readjustment of temperature and the processing time depending upon the alloying constituents of the material. (In no case should aluminum or zinc articles be placed in the solution as these materials will be violently dissolved in the solution with possible injury to the operator.)

3. The black finish obtained consists of a mixture of the oxides of iron; ferric and ferrous oxide sometimes called ferric-ferroso oxide or magnetic oxide of iron. This oxide is produced by actual conversion of the surface of the metal to the oxide. This conversion of the metal extends to a depth of .00035 to .00045 and does not have a tendency to crack, chip or peel. The coating will withstand a one-half hour 20% salt spray test at 90°F. when unoiled and forty-five to one hour and fifteen minutes exposure when oiled with a rust inhibitive oil. The salt spray resistance is reduced when treatment in the second or high temperature bath is omitted. Although the appearance of the part when removed from the first or low temperature bath is undistinguishable from that obtained after treatment in the second or high temperature bath, the higher temperature is necessary to drive the coating to maximum depth. There is no measurable increase in dimensions and the surface produced has the added advantage of having a lower coefficient

of friction. For this reason, the finish is applicable to ball or roller bearings, drills, taps, reamers and dies where pressure welding, excessive friction and wear between moving parts is severe.

4. Spring steel of the S.A.E. (or WD) 9250 type, i.e., silicomanganese, is colored brown or mahogany in the alkali-black type of bath. Some alloys, usually hardened high speed steel, will have a red color when processed only one-half hour in each bath. Prolonging the treatment in the high temperature bath results in satisfactory blackening of these articles. In general, hardened steel requires a greater length of processing time to produce the same black, protective finish, than do the softer steels. Much time may be saved in processing, if parts are preheated in boiling water, before being placed in the processing bath. In this practice, a shorter length of time is required for the solution to come to the boiling point after being chilled due to the introduction of the work. Processing time should be calculated from the moment the solution reaches the boiling point, rather than the time at which the parts were placed in the bath.