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BLACKENING OF STEEL

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by

F. Pearlstein
R.F. Weightman

January 1976

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20 ABSTRACT (Continue on reverse side if necessary and identify by block number)

Chemical and electrochemical processes for blackening steel were investigated and the coatings compared for corrosion resistance, abrasion resistance, abrasion resistance, smut formation and light or infrared reflectance.

The alkaline oxidizing process is an economical means for chemically applying a black oxide coating and is valuable where dimensional change cannot be tolerated; however, minimal corrosion resistance is provided.

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phosphate coated steel provided greater corrosion resistance than the black oxide coating and excelled in its low degree of light reflectance and freedom from smut. The black oxide and manganese phosphate coatings were approximately equal in abrasion resistance and superior in this regard to the other black coatings tested. Zinc or cadmium plated steel with a chemical black chromate film and proprietary black chromium plated steel provided greatest corrosion resistance. Black nickel deposits were somewhat inferior to black chromium in most characteristics tested but are more easily and economically applied.

Black oxide coatings were applied to five stainless steel alloys and the salt spray corrosion resistance determined. Types 304, 316 and 17-7 PH stainless steel were uncorroded after 96 hours salt spray exposure; 410 and 430 were rusted within 48 hours.

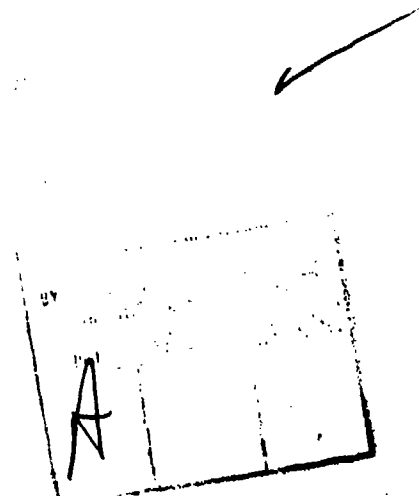
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INTRODUCTION

A number of proprietary and non-proprietary chemical and electro-chemical processes are available for applying a black finish on steel. Such finishes find many applications for military equipment where reduced light reflectance is of importance as in optical instruments or for minimizing detection of exterior surfaces.

Little information is available on the characteristics of black finishes for steel. The objective of this study was to ascertain and compare corrosion resistance, wear resistance and light reflectance for a number of representative black finishes applied to steel.

Considered for this study was the caustic blackening process which is most widely used in the metal finishing industry for blackening steel. The steel is simply immersed in a hot, alkaline solution containing chemical oxidizers; chemical action converts the steel surface to a black oxide of iron, Fe_3O_4 . The caustic blackening process produces very thin deposits and virtually no dimensional changes (usually below $0.1 \mu m$). Proprietary solutions are also available for application of black finishes to steel at ambient temperature. However, preliminary tests indicated these coatings to be unsatisfactory because of the high degree of smut present and were thus not included in this study.

Chemical chromate conversion coatings are readily applied to zinc or cadmium². The films range in color from light iridescent to yellow to olive drab and provide effective resistance to corrosion of the underlying metal. The lighter chromate films can be dyed a variety of pastel colors; the darker coatings are readily dyed black. However, solutions have been developed³ that provide a black chromate coating to zinc or cadmium in a single step; this coating was evaluated in the study described herein.

Light phosphate coatings on steel are formed by chemical conversion and are well known for their ability to provide an effective base for organic coatings⁴. The heavy zinc or manganese phosphate coatings⁵ are dark in color and are usually used as-formed except for supplementary oil or wax treatments. The manganese phosphate coatings are more resistant to wear or alkaline environments than the zinc phosphate coatings and were evaluated in this report.

Black chromium electrodeposits are applied similarly to conventional chromium, but with the catalyst (sulfate or mixed sulfate-fluorosilicate) omitted and acetic acid

¹A. J. Mitchell, Metal Finishing, 71, 41 (1973) March.

²A. Gallaccio, F. Pearlstein, M. R. D'Ambrosio, Metal Finishing, 64, 50 (1966) August.

³K. P. Bellinger, E. G. Chapdelaine, U. S. Patent 3, 219, 489 (1965).

⁴Federal Specification TT-C-490B

⁵Federal Specification MIL-P-16232D

or other agents added.^{6, 7, 8, 9} A typical black chromium deposit was evaluated as part of this study.

So-called "black nickel" coatings are electrodeposited from solutions typically comprised of nickel and zinc sulfate, ammonium sulfate and a sulfur-containing compound¹⁰; the deposits are nickel-zinc alloys, containing as much as 40 percent zinc and inclusions chiefly of sulfur compounds¹¹. Non-sulfide black nickel plating baths have also been formulated¹². A typical black nickel deposit on steel was included in this study. Another type of black nickel involves deposition of metallic nickel with carbon inclusions; this deposit can be quite thick to provide a high degree of wear resistance¹³ but was not included in these tests.

MATERIALS

Metal specimens* AISI 1010, cold rolled steel, ground to surface roughness 0.4 to 0.5 μ m (15-20 microinches), 0.8 mm thickness.

Caustic Black Oxide:

Proprietary Solutions - Code A, 0.57 kg/l (Production Bath)
Code C, 0.84 kg/l
Code D, 0.87 kg/l
Code E, 0.72 kg/l

Non-Proprietary Solution

Code B
600 g/l NaOH
207 g/l NaNO₂
150 g/l KNO₃

Manganese Phosphating Solution:

Proprietary Solution - Code F, 10 percent by volume

Black Chromate for Zinc or Cadmium:

Proprietary Solution - Code G (Zinc) & H (Cadmium)
Prepared by mixing 17 parts (volume) solution a, 4 parts (volume) solution b and 79 parts water at 66° C.

⁶L. P. Gilbert, C. C. Buhman, U. S. Patent 2,623,847

⁷K. S. Willson, U. S. Patent 3,620,935 (1971).

⁸A. K. Graham, Proc. Am. Electropl. Soc., 46, 61 (1959)

⁹T. Ishida, Y. Noda, H. Okada, J. Metal Finishing Soc., Japan, 10, 388 (1959)

¹⁰J. A. McCarthy, Metal Finishing, 62, 56 (1964) Sept.

¹¹D. Fishlock, Metal Finishing, 61, 56 (1963) September.

¹²B. A. Shenoi, K. S. Indira, Metal Finishing, 61, 65 (1963) October.

¹³Federal Specification MIL-N-55392.

*The Q-Panel Co., 15610 Industrial Parkway, Cleveland, Ohio 44135.

Black Chromium Plating Bath (production* bath) Code I:

277 g/l CrO_3
7.5 g/l BaCO_3
300 ml/l Glacial Acetic Acid

Black Nickel Plating Bath - Code J:

83 g/l $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$
32 g/l KSCN
45 g/l $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$
23 g/l $(\text{NH}_4)_2\text{SO}_4$

Zinc Plating Bath (analysis of production* bath)

Total NaCN - 95 g/l
Total Zinc - 32 g/l
NaOH - 39 g/l

Cadmium Plating Bath (analysis of production* bath)

Total NaCN - 93 g/l
Total Cadmium - 12 g/l

PROCEDURE

Steel panels were treated as follows prior to application of test deposits:

- a. Ten minutes immersion in 60 g/l sodium orthosilicate + 3 g/l anionic surfactant at 85°C; cold, 25°C, water rinse (CWR).
- b. One minute immersion in 50 percent (vol) hydrochloric acid (38 percent) at 25°C; CWR.

After applications of finishes and rinsing, the panels, except those zinc or cadmium plated and black chromated, were immersed one minute in 0.6 g/l CrO_3 at pH 2.4 at $73 \pm 2^\circ\text{C}$ and allowed to drain dry without rinsing.

Test caustic-blackening solutions were prepared, heated to operating temperature, and scrap steel immersed to "break-in" the bath. On the following day, the baths (Code B, C, D, E) were reheated ($142 \pm 2^\circ\text{C}$) and test panels prepared by immersion for 20 minutes. The panels treated with the proprietary production bath (Code A) were immersed seven minutes at 130°C .

*Used for production finishing at Bldg. 46-1, Plating Shop.

Steel panels for the manganese phosphate coating were first immersed 30 seconds in a grain refining solution 5.4 g/l FX69-267 (Code F) at 25° C and pH 8 and, without rinsing, immersed in the proprietary phosphating solution (Code F) for 20 minutes at 96° C.

Steel panels were rack-plated with 8 μ m (micrometers) thickness of zinc or cadmium which were then black chromate treated by two minutes immersion in the proprietary bath at 25° C.

Black chromium plating was applied at 900 A/m² for 3 minutes and continued at 300 A/m² for 27 additional minutes in the bath at 30° C. Austenitic stainless steel anodes were used.

Black nickel plating was applied cathodically at 6 A/m² for 45 minutes in the bath at 25° C. Bagged nickel anodes were used.

Corrosion Tests

Treated steel panels (10.2 cm x 15.3 cm) in triplicate were exposed to five percent neutral salt spray* and examined periodically for basis metal corrosion. Panels were also exposed to cyclic humidity** for evaluation of corrosion resistance at non-marine conditions.

Wear Resistance Tests

The wear or abrasion resistance of black finishes were determined using a Taber Abraser (Method 6192 of Fed. Test Method Standard No. 141); CS-10 abrasive wheels were used at 500 gram total load on each arm. The panels (10.2 cm x 10.2 cm) were examined after 100, 200, 400, 600, 800, 1000, 1500, 2000, 3000, 4000 and 5000 cycles for wear of the black finish with exposure of the underlying metal. The abrasive wheels were resurfaced at the end of each examination period. The test was terminated when the black coating had worn sufficiently to expose the underlying metal. Triplicate specimens were used for each blackening treatment.

Reflectance Tests

White light and infrared reflectance of blackened steel were determined after wiping gently with cheese cloth to remove any smut. White light reflectance was determined by directing a beam of light from a 100 watt projection lamp upon the test panels at an angle of 45 degrees in accordance with method 6121 of Federal Test Method Standard No. 141. The percent of light reflected was determined using a photocell (Weston Photronic Cell, Model 594). Infrared reflectance at one micron wavelength was also determined using a Beckman DK-2A ratio recording spectrophotometer vs MgO as a standard.

*ASTM Designation B117.

**4 hrs at 43° C (81% RH); 4 hrs at 54° C (94% RH); . . . etc.

RESULTS & DISCUSSION

All of the blackening treatments provided marked darkening of the surface, but there were considerable differences evident in the appearance. Below is a description of the panels and an evaluation of smut on the surface from observation of white tissue wiped over the surface:

<u>BLACKENING TREATMENT</u>	<u>APPEARANCE</u>	<u>SMUT</u>
Caustic Black (Code A)	Mottled Black	Moderate (black)
Caustic Black (Code B)	Lustrous Black	Slight (brown)
Caustic Black (Code C)	Lustrous Black	Slight (brown)
Caustic Black (Code D)	Lustrous Black	Very slight (brown)
Caustic Black (Code E)	Lustrous Black	Very slight (brown)
Manganese Phosphate (Code F)	Dull Black	None
Black Chromate (Code G)	Lustrous Black*	Trace
Black Chromate (Code H)	Semibright Black*	Slight to moderate (black)
Black Chromium (Code I)	Flat Black	Slight to moderate (black)
Black Nickel (Code J)	Lustrous Black*	Very slight (brown)

*Some iridescence present.

The production caustic blackened steel* (Code A), black chromated cadmium (Code H) and black chromium plated steel (Code I) had significant smut on the surface that may be deleterious for certain applications. For example, smut deposits may be transferred to optical elements and reduce light transmission. The manganese phosphate (Code F) and black chromated zinc (Code G) had little or no visible smut. All of the finishes can be considered decorative but those listed above as "lustrous" were particularly attractive.

The thicknesses of the black coatings were determined by using a magnetic gage. The thicknesses of caustic black deposits were estimated at about 0.5 μm but could not be determined with certainty because the order of thickness was below the level for which the magnetic gage can be used with a high degree of accuracy. The black chromate

*See Appendix A for a discussion of caustic blackening of stainless steels.

deposits (on zinc or cadmium plate) were about 0.7 to 1 μm thickness. The manganese phosphate coating was over 7 μm thick which is substantially thicker than any of the other black coatings tested. Black chromium deposits were less than about 0.3 μm thick as determined by the magnetic gage; about 5 μm would have been expected from information given in the Federal Specification for this deposit¹⁴. It is possible that the production solution used for black chromium plating was not in proper operating condition though deposit appearance was satisfactory. See Appendix B. Black nickel deposits were measured by magnetic gage and determined to be over 2 μm thick. However, it is not known whether the deposit, like nickel, is ferromagnetic which would result in inaccurate thickness measurements.

Corrosion Tests

The results of salt spray corrosion tests and cyclic humidity tests are shown in Table 1.

Table 1. Corrosion of Blackened Steel
Basis Metal Corrosion Ratings

Code	Salt Spray Exposure			5 Days	Cyclic Humidity	
	2 Hrs.	24 Hrs.	48 Hrs.		13 Days	30 Days
A	0	0	0	2	0	0
B	0	0	0	4	2+	1
C	0	0	0	4	2	1+
D	0+	0	0	4	3	2
E	3	0	0	4+	4	3+
F	5	2	1	5	4	3
G	5	5	5*	5	5*	5*
H	5	5	5*	5	5	5*
I	5	1	0	4	3	2
H	5	0+	0	4	2	1

Ratings: 5 = no basis metal corrosion
 4 = traces corrosion
 3 = slight corrosion
 2 = moderate corrosion
 1 = considerable corrosion
 0 = very extensive corrosion

*white salts

¹⁴ MIL-C-14538B

The caustic blackened panels had little resistance to corrosive attack when exposed to salt spray. Only Code E caustic black provided significant, though incomplete, resistance to corrosion after 2 hours of salt spray exposure. The other blackening treatments provided complete protection over the two hour exposure period. Only the zinc or cadmium plated, black chromated, surfaces were still resistant to basic metal (steel) corrosion after 24 hours of salt spray exposure. After 48 hours of exposure, though there was no evidence of rusting, some white corrosion products were visible from corrosion of the underlying zinc and cadmium. The order of corrosion resistance of the various finishes was similar when exposed to cyclic humidity as when exposed to salt spray, but the rate of attack was much slower in the former case. It can be appreciated that the corrosion resistance could be markedly improved by application of an inhibited oil or a wax; relatively thick absorbent coatings, such as the manganese phosphate, are greatly improved by such supplementary treatments.

Wear Resistance Tests

The results of Taber Abraser tests are shown in Table 2.

Table 2. Abrasion Resistance of Blackened Steel

Code	Blackening Treatment	Wear Cycles to Exposure of Underlying Metal		
		<u>1</u>	<u>2</u>	<u>3</u>
A	Caustic Black	1500	1500	1000
B	Caustic Black	3000	3000	2000
C	Caustic Black	4000	1500	3000
D	Caustic Black	2000	2000	4000
E	Caustic Black	2000	1000	2000
F	Manganese Phosphate	3000	2000	3000
G	Black Chromated Zn	600	400	600
H	Black Chromated Cd	400	200	400
I	Black Chromium	100	100	100
J	Black Nickel	200	100	100

The caustic blackened steel was surprisingly resistant to the abrasive wear of the Taber test since these coatings are not considered to be particularly wear resistant. The endpoint of the test was difficult to ascertain with these panels since the coating became burnished and more reflective. Even when white metal was evident in the wear track, reflection from the adjacent surface could mislead the observer. Of the other coatings, only the manganese phosphate coating was comparable in wear resistance to

the caustic black oxide. The manganese phosphate coatings are well known for their excellent wear resistance especially when lubricants are incorporated into the deposit. The wear resistance of both the black chromium and black nickel was surprisingly poor.

Reflectance Tests

The results of reflectance measurements are shown in Table 3.

Table 3. Reflectance of Light from Blackened Steel

Code	<u>Blackening Treatment</u>	<u>Percent Reflectance</u>	
		<u>White Light</u>	<u>Infrared</u>
-	None	13	58
A	Caustic Black	2	11
B	Caustic Black	4	11
C	Caustic Black	5	13
D	Caustic Black	4	15
E	Caustic Black	6.5	11
F	Manganese Phosphate	0	9
G	Black Chromated Zn	4	8
H	Black Chromated Cd	1.5	12
I	Black Chromium	1	7
J	Black Nickel	3	10

White light reflectance was highest for those surfaces described earlier as "lustrous". The manganese phosphate and black chromium coatings provided least reflectance to white light. There was less difference, proportionally, in infrared reflectance values; the lowest reflectances were provided by the black chromium and black-chromated zinc surfaces. Several steel panels were abrasive* blasted prior to application of Code A caustic black finish; white light and infrared reflectance from this surface was 0 and 4 percent respectively. Abrasive blasting can thus be used to advantage when minimum reflectance is required.

*180 grit aluminum oxide

CONCLUSIONS

Based upon magnetic gage determinations, the caustic blackened, black chromate and black chromium deposits are about $1 \mu\text{m}$ thickness or less. The thickness of black nickel and manganese phosphate coatings are approximately 2 and $7 \mu\text{m}$ respectively.

There are varying degrees of smut on the blackened surfaces; none is present on manganese phosphate and virtually none on black chromated zinc surfaces.

One of the proprietary (Code E) caustic black deposits on steel is somewhat superior in corrosion resistance to the others tested, but none of the black oxide coatings are capable of preventing rusting during two hours of salt spray exposure. The manganese phosphate, black chromium and black nickel deposits are protective over a two hour exposure period, but significant rusting is produced after 24 hours of salt spray exposure. Only the black chromated zinc or cadmium plated steel is capable of preventing formation of visible corrosion products after 24 hours of salt spray exposure; white corrosion products are produced after 48 hours.

The caustic black oxide coatings provide a high degree of wear resistance as determined by the Taber Abraser test. Only the manganese phosphate coating is comparable in wear resistance. The black chromate, black chromium and black nickel deposits are decidedly inferior in wear resistance.

White light reflectance is low on all blackened surfaces but lowest with manganese phosphate and black chromium deposits. The blackened surfaces reflect a higher percentage of infrared radiation than white light. Sandblasting prior to blackening results in a surface of low reflectivity.

APPENDIX A

Black Oxide Coating of Stainless Steels

A limited study was conducted on a variety of stainless steels, i. e., A. I. S. I. Types 410, 430, 304, 316, 17-7 PH. Panels (10.2 cm by 15.3 cm) were prepared for blackening by:

- a. Ten minute immersion in 60 g/l sodium orthosilicate + 3 g/l anionic surfactant at 85°C; water rinse at 25°C (CWR)
- b. One minute immersion in 50% (vol) hydrochloric acid (38%) at 25°C; CWR.

The panels (Group A) were then immersed in a proprietary stainless steel blackening solution* for twenty minutes at $128 \pm 2^\circ\text{C}$. None of the stainless steel alloys was successfully blackened by this procedure. Another group of panels (Group B) was activated in a proprietary solution** for a 2.5 minutes at 74°C and water rinsed prior to blackening. All alloys were then effectively blackened in the proprietary blackening solution. See Table I. After the blackening treatment and rinsing, panels were immersed one minute in 0.6 g/l CrO_3 at pH 2.4 at $73 \pm 2^\circ\text{C}$ and allowed to drain dry without rinsing. The panels were exposed to salt spray (ASTM B117); results are shown in Table A1.

The proprietary activation treatment was effective for providing blackening of all the stainless steel alloys tested in the proprietary caustic-oxidizer blackening solution. Depending upon the alloy, the black oxide coating produced varied from a flat black (probably owing to etching of the surface by the activator solution) to a lustrous black with iridescence. Types 304, 316 and 17-7 PH stainless steel panels were resistant to corrosion during salt spray exposure whether or not the blackening treatment was successful. The Types 410 and 430 stainless steels were considerably less resistant to corrosion, but it is interesting to note that the corrosion resistance was significantly reduced when successfully blackened.

*570 g/l Black Magic SS, The Mitchell Bradford Chemical Co., Wampus Lane, Milford, Conn. 06460.

**180 g/l Black Magic SS Activator, The Mitchell Bradford Chemical Co., Wampus Lane, Milford, Conn. 06460.

Table A1. Appearance and Corrosion Resistance of Blackened Stainless Steels

<u>Group</u>	<u>A. I. S. I. Type</u>	<u>Appearance</u>	<u>Corrosion Rating** After Salt spray Exposure</u>			
			<u>24 hrs</u>	<u>48 hrs</u>	<u>96 hrs</u>	<u>192 hrs</u>
A	410	unchanged (not blackened)	4	3	3	2
A	430	unchanged (not blackened)	4	3-1/2	3	3
A	304	unchanged (not blackened)	5	5	5	5
A	316	unchanged (not blackened)	5	5	5	5
A	17-7 PH	unchanged (not blackened)	5	5	5	5
B	410	flat black	1-1/2	0	0	0
B	430	flat black	4	2	1	0
B	304	semilustrous black	5	5	5	4
B	316	lustrous black*	5	5	5	5
B	17-7 PH	semilustrous black	5	5	4-1/2	5

*iridescence present

**Corrosion Ratings:

- 5 - No rust
- 4 - Traces of rust
- 3 - Slight rust
- 2 - Moderate rust
- 1 - Considerable rust
- 0 - Very extensive rusting

APPENDIX B

Black Chromium Plated Steel

Steel panels were prepared using a proprietary¹⁵ solution at 21°C with a cathodic current density of 3100 A/m² applied for a total of ten minutes. The coating thickness was measured using a magnetic gage and was found to average about 4 μm.

Taber Abraser test results showed these black chromium deposits capable of withstanding 300 to 400 cycles before basis metal was exposed. The abrasion resistance was higher than that of the thinner black chromium deposits tested previously but was nonetheless lower than the caustic black, manganese phosphate or black chromated zinc deposits described earlier.

The proprietary black chromium deposit was found protective to steel after 24 hours of exposure to salt spray with some small, light areas that may be the result of incipient corrosion. Several corrosion specks (<0.3 mm diameter) were visible within some of the small light-colored regions after 48 hours of salt spray exposure. The proprietary black chromium deposit was completely protective to steel over a 30 day period of exposure to cyclic humidity conditions. The proprietary black chromium deposit thus appears superior in corrosion prevention to the other coatings tested.

¹⁵J. P. Branciaroli, P. G. Stutzman, *Plating*, 56, 37 (1969)

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