Rock Island Arsenal Laboratory

TECHNICAL REPORT

CONVERSION COATING FOR CHROMIUM

62-2-2

By

R. H. Wolff

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Ordnance Management Structure Code No. 5010.11.810
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Approved by:

A. C. Hanson
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Laboratory Director

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ABSTRACT

Selected chemicals in water solution were used electrolytically in an effort to produce an oxide conversion coating upon electrodeposited chromium. It was found that some materials and solution combinations are capable of producing thin, colored, iridescent films at the anode or cathode and sometimes at both electrodes. Chromic oxide films were obtained in what appears to be the hydrous form at the anode. Such films could be produced as relatively thick gels. Unfortunately, these gels have no bond to the basis chromium and upon removal from solution, dry to a loosely adherent powder. No satisfactory method was found for producing chromic oxide supplementary coatings on chromium surfaces using aqueous solution techniques.

RECOMMENDATIONS

It is recommended that no further work be considered for production of an oxide conversion coating on chromium by aqueous solution methods. However, it is believed that further investigation for the production of a colored oxide coating be considered from another direction. Such direction might include the use of molten salts with the resultant disadvantages of higher operating temperatures. It is also possible that an alloy is needed with the chromium to provide a matrix for bonding the oxide.

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OBJECT

To develop a conversion coating on chromium that will provide the advantages of chromium in hardness and abrasion resistance, coupled with the camouflage appearance and corrosion resistance of chromium oxide. To accomplish this conversion of chromium by electrolytic action using chemicals in water solution.

INTRODUCTION

Layers of chromium metal are used extensively in Ordnance where hardness, wear and heat resistance are required. In normal application the chromium is electrodeposited. The resulting deposits are usually bright and the surfaces are not particularly receptive to the retention of paint. Black chromium may be used in certain cases but because of the structure of the coating, many of the normal properties of bright chromium are not retained. Consequently, the uses of chromium for external coatings are limited where camouflage measures are a consideration.

It has been observed that heating stainless steel in the air sometimes causes the surface to become discolored. This discolored layer varies from green to black and has been found to be very resistant to cleaning operations. The oxide is most readily removed by high current electropolishing or by abrasive buffing, either of which results in stripping some of the basis material from the surface.

As a coating for external use, this oxide film offers advantages in that it forms a dull nonreflective surface in a natural camouflage color range. The surface by visual observation is smooth but not glossy so that a possibility of good paint bonding exists.

The stainless steels derive their remarkable resistance to corrosion from the presence of chromium. The effectiveness of the chromium in establishing passivity is shown by the increased resistance to oxidizing conditions as the chromium content is increased(1).

It is believed by many investigators that passivation results from the presence of a thin oxide or hydrate film on the metal, stabilized by chromium. The rate at which steels develop passivity depends upon the chromium content. Steels that contain more than approximately 20% chromium become passive in the atmosphere without developing even a slight film of rust. Since the chromium plays such an important part in this corrosion mechanism, it would appear that a conversion surface coating of chromium based on relatively pure chromium oxide, would result in an effective corrosion barrier, and also provide characteristics different from those associated with chromium plate alone.

Available data show that there are three oxides of chromium: the monoxide or chromous oxide, CrO; the sesquioxide or chromic oxide, Cr_2O_3; and the trioxide or chromic acid anhydride, CrO_3. The monoxide is not very stable. It is a black pyrophoric powder which can be made to burst into flames if struck sharply or if heated in the air(2). It is most readily prepared from a chromium amalgam by oxidation, either with nitric acid or in the air.

Chromium trioxide is a dark red crystalline compound which is usually prepared commercially in flakes. It is also called chromic anhydride or chromic acid. The latter term, which is commonly used, is incorrect by chemical terminology. CrO_3 is very hygroscopic, readily soluble in water and melts with some decomposition, at a temperature of 387°F (197°C).

Chromic oxide is a compound most widely used and recognized as a green pigment. The melting point has been given as 4415°F (2435°C). It is a semiconductor. The color varies from light green in finely divided particles to almost black in large crystals. The color turns from green to brown when heated but is restored to green upon cooling. In crystalline form, Cr_2O_3 is as hard as corundum or next to diamond in hardness on the Moh scale. Chromic oxide is produced by heating finely divided chromium in the air or by igniting various salts such as hydrous chromic oxide, ammonium or mercurous chromate. The result is finely divided powder, the fineness of which depends upon the temperature of ignition. Chromic oxide is insoluble in water, alcohol or acetone and only slightly soluble in all acids and alkalis except hot 70% perchloric acid which changes it to CrO_3.

X-ray diffraction examination was made of a typical grey-green coating, formed by heating a stainless steel strip to approximately 1000°F in an uncontrolled atmosphere. It was established that the coating was primarily chromic oxide Cr_2O_3 with the presence of CrO and a possible complex including iron, $(CrFe)_2O_3$.

There are a number of references in the literature reporting analyses of similar films to be Cr_2O_3 and mixtures of spinel. These films were produced at various temperatures from 932°F (500°C) to 2192°F (1200°C)(3,4). In general the experiments were conducted in controlled atmospheres of oxygen. Other data relating to the composition of the oxide films were gathered from investigations to determine the oxidation resistance of highly alloyed chromium steels heated to high temperatures for extended time periods(5,6). The conditions of high temperature and oxygen atmosphere readily produce the oxide coatings.

To make practical use of an oxide coating, one approach would be to heat the object in an oven or furnace at suitable temperature and atmosphere to produce the oxide layer. The disadvantage would be restriction of the process by size of the oven. Furthermore, it is not metallurgically feasible to subject steels to the relatively high temperatures required without danger of changing the properties by heat treatment effects. On the other hand, if chromium plating is considered as a prerequisite, it would be relatively easy to duplicate the chromium tank in size and use a solution to process objects of any size that could be plated.

Much data has been assembled concerning the chromium oxides resulting from study of appropriate ferrous-chromium alloys for high temperature uses. Very little work has been done to explore the possibility of anodically forming the oxide after the manner of anodizing of metals such as aluminum. Johansen, et al(7) in an experiment regarding anodic oxidation at very low current densities, used an ammonium borate solution. This experiment, conducted under constant conditions, examined aluminum, chromium, hafnium, niobium, tantalum, vanadium and zirconium. Chromium was among the metals which did not result in forming a film under this set of conditions.

The efforts of the present work were directed toward the development of an oxide protective coating by electrolytic conversion of chromium metal in aqueous solution.

PROCEDURE AND RESULTS

Test specimens were prepared by plating 10 to 15 mils of chromium on small bars of FS 1018 steel. In size the specimens were 3/16" x 3/8" x 8". The chromium plating was accomplished from a 33 oz./gal. bath with chromium to sulfate ratio of 100 to 1. In test these specimens were used at both electrodes so that observations could be made of the chromium behavior as cathode as well as anode. Unless otherwise noted, solution volumes were made to 300 cc., a quantity adequate for test and not wasteful of chemicals.

Electrode spacings were allowed to vary from 2 to 2-1/2 inches apart and only two electrodes were used, one cathode and one anode fixed in position so the specimens could not rotate. By this arrangement it was possible to observe the ability of the film to cover the complete specimen. No special pretreatment was given the chromium specimens except to insure they were kept clean between the plating and actual use in test.

The general procedure after immersion and electrical connection of the specimens, involved slowly increasing the voltage until gas bubbles began to form on the cathode. At this point (usually 1.8 volts) the system was observed for several minutes for evidence of attack or coating. The voltage was then increased to determine whether the reactions would change in a visible manner other than acceleration of rate. If no changes were observed up to 9 volts the test specimens were removed from the solution for rinsing in distilled water and examination upon drying. After the experimental work had been started an additional current source was made available so that approximately 24 volts DC could be applied. However, none of the tests showed this increase in potential to be of any particular benefit.

Experimental solutions were used, which on the basis of present knowledge, form coatings on some metals or because of the chemistry involved could produce the conditions for forming the oxide. The solutions are grouped and listed below according to observations of specimens as (1) dissolving action, (2) no visible appearance change, (3) thin colored films, and (4) gel films.

The first two groups are based upon action in the solution. The resulting action was positive attack on the specimen with usually an orange colored film originating at the surface of the specimen and quickly coloring the whole solution. This film was very dense at the specimen surface and appeared to roll down along the specimen and slowly disperse into the solution. The film is so completely soluble that all the color stayed in the solution and upon lifting the specimen from the test the surface was rinsed clean as it was drawn from the liquid. Specimens from this group showed etching of the chromium in relatively short time.

Group 2 specimens were probably undergoing dissolving attack also, but at a slow rate so that the specimens were not different in appearance after the experiment. Coloring of the solution was masked by the initial color resulting from solution composition.

Dissolving Action

Solution (300 cc volume unless otherwise noted)	Comment
Nitric Acid (10 cc) 75°F	
Oxalic Acid 5% 75°F	
Sodium Chloride 5% 75°F	
Nickel Nitrate 5% 140°F	Cathode etched slightly
Sodium hypophosphite 10% 75°, 120°, 180°	
Sodium hydroxide 10% 180°, 6 volts	
Zinc phosphatizing solution 160° 12 volts	Cathode turns black, smutty, rubs off
Sodium perborate 5% 160° up to 18 volts	
Sodium peroxide 5% 160°	
Zinc silicofluoride 5% 75° 3-4 volts	Cathode coated with zinc
Copper sulfate 5% 75° 2 volts	Cathode coats with non-adherent copper
Chromic acid 10% 75° 10 volts	Cathode iridescent golden brown
Potassium thiosulfate 5% 75° 3-1/2 volts 150° 6 volts	Soluble green film at anode
Sodium sulfite 5% 75° 6 volts 150° 6 volts	Green anode film which strips as soluble when specimen is moved or removed from sol^{n}

Dissolving Action

Solution	Comment
Sodium aluminate 10 gm Sodium silicate 10 gm 130°F 2-6 volts	Anode: yellow dissolving film
+ Sodium dihydrogen phosphate 6 gm 130° 2 volts	Anode reaction same as above
Chromium fluoride 2 gms 75°F 10 volts	Anode: dissolving yellow film
Cobaltous chloride 4 gms 75°F 4 volts	

No Appearance Change In Specimens

(Dissolving action assumed.)

Solution	Comment
Chromic acid (pH 0.2) 10%	
Chromic acid (pH* 3.8) 10%	Anode very faint yellow
Chromic acid (pH* 6.8) 10%	
Chromic acid (pH* 9.8) 10%	
Chromic acid 10% Ammonium hydroxide (5cc) Sodium hydrosulfite 5 gms	Exothermic reaction heats to 150° and solution turned to gel. Current flows but no coatings produced.
Chromic acid 5% Sodium hydrosulfite 2 gms	Upon addition of 5 gms more hydrosulfite the sol^{n} gelled

Thin, Colored Films

Chromic acid 10% (pH* 4.3) 140° 2.2 volts	Anode: blue with bubble spots Cathode: uniform blue violet
Chromic acid (pH* 3.8) 10% 140°	Anode: faint yellow
Chromic acid (pH* 3.8) 10% 180° 2.2 volts	Anode: yellow Cathode: dark yellow w/ blue patch center
Chromic acid (pH* 3.8) 10%	Cathode test only:
(a) 180° 1.9 volts	Blue iridescent
(b) 180° 2.2 volts	Uniform yellow
(c) fresh spec. at room temperature 1.8 volts	No change
(d) temperature raised to 180°	Yellow color

*pH adjustments made by adding ammonium hydroxide.

Thin, Colored Films

Solution	Comment
Sodium hydrosulfite 10% 180° 4-6 volts	Anode: etched olive drab colored coating Cathode: black smut wipes off
10% Chromic acid heats as used at 6 volts	Anode: black Cathode: brown iridescent
Potassium permanganate (1 gm) 75° 6 volts	Anode: faint olive drab film Cathode: hvy. purple film easily scratched

Gel Films Formed

Sodium hydroxide 5% (400 cc) Sodium hydrosulfite 5 gms 150° 3 to 6 volts	Anode: green film
Sodium hydroxide 5% (300 cc) Sodium hydrosulfite 5 gms 150° 4-1/2	Anode: brilliant green
Sodium sulfide 5% Sodium hypophosphite 5% 75° 3 volts	Anode: dull green
Sodium sulfide 1 gm 75° 6 volts Add approx. 2 gms more sulfide	Anode: Dull blue grey Green gel film
Sodium hydrosulfite 10 gms Sodium hydroxide 2 gms 75° 2-6 volts	Anode: green gel poor adhesion in sol^n
Sodium sulfhydrate 10 gms 75° 6 volts	Anode: green film quickly turns orange
Sodium hydroxide (5 gm) Sodium sulfide (5 gm) 75° 2-6 volts	Anode: dissolving yellow film becomes gel film of ugly orange & green color

Gel rinses away in running water leaving faint blue iridescence.

Gel Films Formed

Solution	Comment
Sodium aluminate (12 gm) 150° 2-6 volts	Anode: orange film with scattered more intense colored dots. Film comes loose w/ gentle rubbing under stream of H_2O.
Same as above w/ Sodium fluoride (5 gms)	Anode reaction same as above.

DISCUSSION

The coatings of oxide that form on metals during oxidation differ widely in their ability to inhibit further oxidation. Such oxides can generally be divided into three main types: (1) those in which the coating has essentially no protective effect and in which the coating grows linearly with respect to time, (2) those that have a limited protective effect in that the rate of oxide growth decreases with time, and (3) those in which the coating completely protects the metal after it has grown to a definite thickness. It has been suggested that in the first type the oxide is porous. This would be the case, for example, if the volume of oxide formed from a volume of metal is less than the volume of metal. If the reverse is true, i.e., the ratio of oxide volume to metal volume is greater than unity, the oxide forms an intermediate or completely protective coating(8).

In addition to this relationship of oxide coating to metal, consideration must be given to the circumstance of oxide formation. Subjecting a metal to the conditions of anodizing in any particular electrolyte produces the following possibilities:

(a) The anode reaction products may be soluble in the electrolyte. In this case the metal is dissolved until the solution is saturated or the metal is gone.

(b) The reaction products may be almost insoluble in the electrolyte and form a strongly adherent and nonconducting film on the anode. The film growth continues until the resistance of the film prevents the current from reaching the anode (basis metal). Films formed in this manner are usually thin.

(c) The reaction products may be sparingly soluble in the electrolyte and form a strongly adherent film which is nonconducting when dry over the anode.

(d) The reaction products may be sparingly soluble and form a nonadherent film. If a suitable electrolyte of high concentration is used, electropolishing may be possible.

In relating the experimental results to these defining conditions, it may be determined that chromium produces an oxide coating in which the oxide volume produced from a volume of metal is greater than unity(9). A comparison of several commonly used metals over which the ratio of oxide volume to metal volume is more than one, may be made as follows(9):

Metal	Ratio $\frac{\text{Oxide volume}}{\text{Metal volume}}$
Aluminum	1.38
Chromium	1.97
Copper	1.71
Nickel	1.64
Zinc	1.44

From these indications a satisfactory protective oxide coating is expected to form with chromium. A consideration of the experimental electrolytic results, however, places the solution method of oxide production into the type indicated by (a) or (d). By reference to results under dissolving reactions, the chromium anode reaction products were soluble in solutions of strong inorganic acids or bases; with solutions of salts of a strong acid or a strong base; and with strong organic acid such as oxalic acid. In the case of the gel forming reactions, the chromium anode products were found to be sparingly soluble. The films were moderately adherent while wet but could be easily brushed away when dried. Increasing the voltage on these specimens resulted in growth phenomena that clearly indicated the film did not prevent the current passage to the basis metal. These films correspond closely to the hydrous oxide, $Cr_2O_3xH_2O$. This gel is slightly soluble in acid solution when first formed but becomes insoluble upon aging or heating(10). Aging is also accelerated in alkaline solution. Treatments of this sort were used in unsuccessful efforts to prevent the oxide from separating from the basis chromium. Specimens on which the green gel was produced were variously processed by soaking in ammonium hydroxide and drying in the air or by use of an oven (forced convection) or a muffle furnace. Baking temperatures were varied from 150°F (65°C) to 600°F (312°C). In all of these cases the result was a fine green powder with no bond to the basis chromium.

It was found that a range of colors could be produced by variation of voltage to the cathode in chromic acid solutions of pH approximately 3.6. The influence of voltage could be seen in the color effects caused by current density. These effects were such that a specimen might be golden yellow around the edges and have an iridescent blue center spot which changed in size with variation of the voltage (and current density). These films offered varying and usually slight resistance to abrasion and moderate resistance to attack by concentrated hydrochloric acid. The coatings appeared to be very thin and did not show evidence of becoming thicker with time. It is speculated that the colors were due to optical interference effects. It is known that similar thin films can be produced from chromate solutions upon mild steel cathodes(11).

Films such as these offer slight potential use as a superficial color layer or as a base for paint. The ease with which the color is changed with current density would present a problem in the production of uniform appearance. This problem arises from the fact that irregular variation of current density normally results from the shape and size of the work piece. Present benefits of such a thin, colored coating are not considered sufficiently rewarding in return for the additional operation.

Failure to produce a heavy chromium oxide layer in a fashion similar to the anodizing of aluminum to aluminum oxide is disappointing. The possibility does exist that only thin layers of protective chromium oxide are obtainable. This situation could occur if the bond is strong between the chromium and chromium oxide and weak or nonexistent between the growth of chromium oxide layers formed over the initial oxide layer.

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5. Metal Coatings--Production Materials
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