

## UNITED STATES PATENT OFFICE

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## METHOD OF COATING ZINC BASE ALLOYS

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5 Claims. (Cl. 148—6.16)

1

This invention relates to chemical treatment of metal surfaces, and has reference more particularly to the provision of an improved method of producing a protective, corrosion-resistant coating on the surface of metal of the class consisting of zinc and zinc base alloys. This application is a continuation of an application, Serial No. 782,032, filed October 24, 1947, in the names of Willett C. Magruder, Barclay F. Smith, and Edward A. Kruszynski, now abandoned.

The invention is particularly concerned with the production of a visible and iridescent coating on zinc and zinc base alloys, for the purpose of retarding or preventing corrosion and erosion of the metal surfaces upon exposure to the atmosphere or to moisture such as water, salt spray, and the like. The present coating method is especially suitable for zinc base die casting alloys such as those containing aluminum and magnesium, or aluminum, magnesium and copper, and for objects having metal surfaces of zinc or zinc base alloys, as zinc dipped and zinc plated objects.

According to the method of the present invention, there is produced on the surfaces of objects formed of zinc or zinc base alloys, and on objects surfaced with zinc or zinc base alloys, a visible, colored and iridescent coating of a protective, corrosion-resistant character, wherein the coating is in the nature of a thin film such as will not alter in any material degree, the dimensions of the object on which the coating is formed, nor obstruct or fill up small passageways which may be provided in the object. In the treatment, however, the surfaces of all recesses and passageways in the object, will be coated uniformly and to the same film-like extent as appears on the external or exposed surfaces of the object.

It is a further purpose of the present invention to afford a coating solution or bath which will produce upon the surfaces of metals of the class hereinabove specified, an insoluble film-like coating characterized by uniformity in thickness, color and iridescence, and wherein the production of the film coating is attained economically and in a minimum of time.

The presently improved process is based upon immersion of the metal surface to be coated, in an aqueous solution or bath consisting of chromium nitrate acidulated by organic and inorganic acids controlled as to the concentrations thereof in the solution, such as to determine and maintain the solution at a pH value between 0.8 and 1.3. Advantageously, the acidifying agents em-

2

ployed in the chromium nitrate bath, consist of phosphoric acid as the inorganic agent, and oxalic acid as the organic agent. We have found that a visible and iridescent, corrosion-resistant film of uniform character and varying from gray to silver in color, is produced on the surface of metals in the class hereinabove specified, by immersion of the metal in an aqueous solution of this nature, containing per liter of water, between 70 and 130 grams of chromium nitrate, about 13 grams of oxalic acid and about 5 ml. of phosphoric acid, and maintaining the metal immersed in such solution for a comparatively short time, as between 5 seconds and 20 seconds, until the desired color of the film (between gray and silver) appears. In this solution, the concentration of oxalic and phosphoric acids are relatively closely controlled to the proportions indicated, such as will determine and maintain the solution at a pH value within the range of 0.8 to 1.3. We have found that the use of the acids specified, in concentrations controlled to afford acidulation of the solution within the pH range given, is quite important to a successful action of the solution in producing the desired uniform protective film on metal surfaces of zinc and its alloys.

In practice of the invention according to one mode of carrying out the process, an aqueous solution is made up to contain the following ingredients per liter of water:

Chromium nitrate ( $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ), 100 grams  
Oxalic acid  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ,  $13\frac{1}{3}$  grams  
Phosphoric acid ( $\text{H}_3\text{PO}_4$ ) (90%), 5 ml.

Die castings of zinc base alloy for example, when immersed for about ten seconds in such a solution which is preferably and advantageously heated and maintained at a temperature of about 160 degrees F., have produced on the surfaces thereof, a chemically bound, insoluble and protective film which is uniformly iridescent and gray or silver in color, and of a nature which characterizes the coated castings by a marked resistance to corrosion upon exposure of the coated metal to the atmosphere or moisture. Moreover, the silver color of the coating film, as well as its iridescence, presents a pleasing, decorative appearance, and affords additionally, ready identification of objects so coated, in differentiation from uncoated objects.

In carrying out the present process, before immersion in the coating solution, the zinc or zinc base metal objects as die-castings and the like, are cleaned of dirt, oil and grease deposits on the surfaces to be coated. The surface clean-



3

ing step may be carried out in any suitable way, as by immersion of the metal objects in an alkaline bath, or in a solution containing a solvent such as carbon tetrachloride, trichlorethylene or the like, followed by immersion in an alkaline bath. After cleaning, the zinc or zinc base metal surfaces are treated by immersion in the coating solution of the character and composition herein specified. While the coating process is not critically dependent upon the temperature of the solution, it is facilitated to some appreciable degree by maintaining the solution at a temperature of about 160 degrees F. More importantly, however, such heated condition of the bath is of great advantage in connection with a following drying stage presently to be indicated.

Upon completion of the coating stage, the coated metal objects are rinsed preferably in hot water, hot water here being employed in order to avoid any material cooling of the coated metal which comes from the coating solution in a heated state consequent to the temperature of the solution (about 160 degrees F.). Moreover, the use of hot water materially facilitates the rinse operation, by reducing the time required to effect a thorough rinse of the coated metal.

Following the rinse step, the hot, coated metal is dried in any suitable manner, as by subjecting the metal to hot air under natural or forced draft, and at a temperature ranging up to 200 degrees F. or higher. Since at the outset of the drying step, the coated metal is in a heated condition consequent to the approximately 160 degrees F. temperature of the coating bath and the hot water rinse, drying of the coated metal and fixation of the coating film thereon are effected in a relatively short time. When so dried, the coating film is set so completely that it does not require aging before handling or subsection of the coated metal to abrasion.

While a specific formula of coating bath has been given and certain possible variations therein indicated, it will be understood that we have not attempted to indicate all the variations permissible within the general principles set out in the foregoing description.

We claim:

4

1. The method of producing a visible, colored protective coating on a surface of metal of the class consisting of zinc and a zinc base alloy which comprises immersing the metal surface for from 5 to 20 seconds in a solution consisting of water, from 70 to 130 grams of chromium nitrate per liter of water, and acidifying agents consisting of oxalic acid and phosphoric acid in proportions of two to three parts oxalic acid to one part phosphoric acid, to render and maintain the solution at a pH value between .08 and 1.3.

2. The method according to claim 1 wherein the said acidifying agents are proportioned in the solution as follows: about 5 ml. of phosphoric acid per liter of solution and about 13 grams of oxalic acid per liter of solution.

3. The method of producing a visible, colored protective coating on a surface of metal of the class consisting of zinc and a zinc base alloy, which comprises immersing the metal surface for from 5 to 20 seconds in a heated solution containing per liter of solution from 70 to 130 grams of chromium nitrate, about 13 grams of oxalic acid, about 5 ml. of phosphoric acid, and the balance water.

4. The method of producing a visible, colored protective coating on a surface of metal consisting of zinc and a zinc base alloy which comprises immersing the metal surface for about 10 seconds in an acidulated solution containing per liter of solution about 100 grams of chromium nitrate, about 13½ grams of oxalic acid, about 5 ml. of phosphoric acid, and the balance water.

5. The method according to claim 4 wherein said solution is maintained at a temperature of about 160 degrees F.

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#### REFERENCES CITED

The following references are of record in the file of this patent:

#### UNITED STATES PATENTS

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