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## SURFACE PREPARATION

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12 Claims. (Cl. 148—6)

This invention relates to the treatment of aluminum or aluminum base alloy surfaces preparatory to applying a firmly adherent coating thereon.

The invention is primarily directed to the treatment of aluminum and aluminum base alloy surfaces for the subsequent application of paint films, but the invention is not limited thereto and is equally applicable to the preparation of such surfaces for the application of other types of coatings, such as electrochemically deposited metallic or non-metallic coatings and mechanically applied coatings, such as rolled or sprayed coatings.

It has been known to those versed in this art to roughen metallic surfaces, as by means of sand blasting or by chemical etching, prior to the application of paint coatings or plating operations. These prior processes have not proved entirely satisfactory—sometimes because of the cost of operation, sometimes because of technical shortcomings.

It is an object of this invention to provide an economical process for treating aluminum and aluminum base alloy surfaces which will provide a surface to which a subsequently applied coating will be firmly adherently bonded.

It is a further object of the invention to provide a process which will produce a surface which has markedly increased resistance to chemical corrosion, and at the same time produce an improved mechanical adhesion and bonding of a subsequently applied coating.

Other and further objects will present themselves on consideration of the specification and claims when taken in conjunction with the specific examples of the invention given herein.

The invention, in its essentials, comprises a multi-step process in which the article or surface to be subsequently provided with a metallic or non-metallic coating is first cleaned, when necessary, of any grease or foreign material in any suitable manner. The metal is thereafter treated in an alkaline solution comprising an alkali metal carbonate and a chromate to produce an oxide-coated surface. The final step of the invention preferably comprises subjecting the oxide-coated surface to the action of a solution of chromic acid, so as to strip the oxide coating and passivate the treated aluminum surface. In some instances, and particularly where a metallic coating is to be applied to the surface treated in accordance with this invention, nitric acid is substituted for the chromic acid in the final step of the invention.

In the practice of the invention, the alkali metal carbonate-chromate solution which is employed has both an etching and a coating action. For this purpose, it has been found that solutions composed of alkali metal carbonates and chromates in the ratio of about 3:1 are preferred, and a solution which has produced exceptionally good results is one made by dissolving 20 grams of sodium carbonate (anhydrous) and 5 grams of potassium dichromate in one liter of water. Alkali metal carbonate-chromate solutions of the type contemplated in the first step of this invention are recognized in the art, and it will be apparent to one skilled in the art that the amounts of carbonate and chromate may be increased or decreased within the limits known in the art so long as a satisfactory oxide coating is produced. Variations in the ratio of carbonate to chromate may also be made in a known manner. Although the specific alkali metal carbonate-chromate solution given herein has been produced by dissolving sodium carbonate and potassium dichromate in water, satisfactory carbonate-chromate solutions have also been produced by substituting sodium dichromate for potassium dichromate or by using chemically equivalent amounts of the alkali metal chromates instead of the dichromate.

Aluminum immersed in an alkali metal carbonate-chromate solution, prepared by dissolving 20 grams of sodium carbonate and 5 grams of potassium dichromate in one liter of water, for about 15 to 30 minutes at about boiling temperature produced a good example of the particular type of etched and oxide-coated surface which is contemplated by the present invention. While the alkali metal carbonate-chromate solution is preferably used at or near the boiling temperature, it can also be used at lower temperatures with a longer time of treatment. Regardless of whether these solutions are prepared from potassium dichromate or sodium dichromate or from the chromates, they are hereinafter referred to as alkali metal carbonate-chromate solutions.

After rinsing the oxide-coated article in clean water, in the preferred form of the invention, it is treated in a chromic acid solution, preferably containing about 5 per cent by weight of chomic acid, for about 15 minutes, at a temperature of approximately 180° F. This treatment substantially removes the oxide coating from the etched surface and leaves the metal uniformly etched over its entire surface. While the chromic acid solution is preferably employed hot, it also may

be used at lower temperatures, providing a longer time for action is allowed. The article or surface is finally rinsed again in clean, warm water.

When it is desired to employ the surface treatment contemplated by this invention preparatory to applying a metallic coating, as by electroplating upon the prepared surface, it has been found that the surface which has been etched and coated in the alkali metal carbonate-chromate solution should preferably be treated in a nitric acid solution containing about 5 to 70 per cent nitric acid, the time of treatment being sufficient to substantially remove the oxide coating. In a nitric acid solution, containing 35 per cent nitric acid, an immersion time of ½ to 2 minutes at room temperature was found sufficient to give the results contemplated by the invention. This treatment substantially removes the oxide coating from the etched surface and leaves the metal uniformly etched over its entire surface, the article or surface being finally rinsed in clean, warm water prior to its subsequent immersion in a suitable electroplating bath.

Coatings, such as metallic electroplates, were found to tenaciously adhere to my prepared surface. Non-metallic coatings, such as paint films, and particularly synthetic resin coatings, applied over the surface treated according to my method, were found superior, from the standpoint of chemical resistance to corrosion and mechanical bonding adherence, to those previously obtained by known processes.

To illustrate the advantages of my improved method of preparing aluminum surfaces for subsequent coating treatments, I have made a variety of tests, among which I may cite the following:

Aluminum alloy sheet 0.064 inch in thickness was treated with an alkali metal carbonate-chromate solution of my preferred composition and then treated with a hot 5 per cent solution of chromic acid. After rinsing and drying, four coats of synthetic resin enamel were applied, with baking of the enamel after the application of each successive coat. The test specimen thus prepared was then immersed in a solution containing 10 per cent sulfuric acid at a temperature of 45° C. for a period of two weeks. As a result of this immersion, there was no appreciable penetration of the coating or attack of the metal underneath the coating. Finally the test piece was bent at an angle of 90° over a rod having a diameter equal to twice the thickness of the metal specimen. The adherence of the synthetic resin enamel to the metal was excellent and there was inappreciable flaking of the coating at the bend.

There may be compared with this example a similar aluminum test piece the surface of which was sand blasted and to which thereafter four coats of the same synthetic resin enamel were applied and baked in the same manner. During the immersion in sulfuric acid, considerable corrosion of the metal occurred beneath the coat, as evidenced by the formation of blisters on the surface. When the sample was given a 90° bend, there was substantial flaking of the enamel coating from the surface adjacent the bend.

Still another comparison was made between a test piece of the same metal which had been oxide-coated in an alkali metal carbonate-chromate solution, according to the first step of my invention, and thereafter impregnated with a dichromate solution in accordance with a well known process. When this sample was immersed in a hot sulfuric acid solution, there was no visible evidence of corrosion underneath the enamel film, but when it was given the 90° bend test there was substantial flaking of the enamel from the metal at the bend, indicating that the adherence was distinctly inferior to that produced on metal coated following the preferred surface preparation of this invention.

In an electroplating test, a sample of aluminum was treated in an alkali metal carbonate-chromate solution of the preferred composition, followed by treating the oxide-coated specimen in a 35 per cent nitric acid solution for ½ to 2 minutes, held at room temperature. After rinsing and drying, the sample was nickel plated in a well known manner, and the plated product was readily buffed and found to be entirely satisfactory when subjected to the customary bend test.

Although the invention has been essentially described in terms of an alkali metal carbonate-chromate surface treatment, followed by a chromic acid or nitric acid oxide coating-removing step prior to the application of metallic or non-metallic coatings, it will be apparent to those skilled in this art that modifications of the process are possible. For example, it has been found that the oxide coating produced in the first step of the invention is readily removed in solutions containing mixtures of chromic acid and nitric acid, with the inherent advantage that the stripped surface is passivated before application of the final metallic or non-metallic coating.

The term "aluminum" as used herein and in the appended claims embraces aluminum and aluminum base alloys including the usual impurities found in aluminum ingot of commercial grade or such impurities as are picked up in the course of the usual handling operations incident to ordinary melting practice.

It will be understood, of course, that while this invention has been described with reference to certain preferred embodiments thereof, various modifications and changes may be made therein within the spirit of the invention as set forth in the appended claims.

What I claim is:

1. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in an alkali metal carbonate-chromate aqueous solution to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating with an aqueous of at least one of the acids selected from the group consisting of chromic and nitric acids.

2. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in an alkali metal carbonate-chromate aqueous solution to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating and passivating the surface with an aqueous solution of at least one of the acids selected from the group consisting of chromic and nitric acids.

3. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in a hot alkali metal carbonate-chromate aqueous solution to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating with an aqueous solution of chromic acid.

4. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in a hot alkali metal carbonate-chromate aqueous solution to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating with an aqueous solution of nitric acid.

5. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in a hot alkali metal carbonate-chromate aqueous solution to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating with a hot aqueous solution of chromic acid.

6. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in a hot alkali metal carbonate-chromate aqueous solution to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating with a hot aqueous solution of chromic acid containing about 5 per cent by weight of chromic acid.

7. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in a solution made by dissolving about 20 grams of sodium carbonate and about 5 grams of alkali metal dichromate in one liter of water to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating with an aqueous solution of at least one of the acids selected from the group consisting of chromic and nitric acids.

8. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in a hot alkali metal carbonate-chromate aqueous solution to produce a substantial oxide coatng on the surface, and thereafter removing the oxide coating with a nitric acid solution containing between about 5 to 70 per cent by weight of nitric acid.

9. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in a hot alkali metal carbonate-chromate aqueous solution to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating with a nitric acid solution containing about 35 per cent by weight of nitric acid.

10. The method of treatng an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface for about 15 to 30 minutes in a boiling soluton made by dissolving about 20 grams of sodium carbinate and about 5 grams of potassium dichromate in one liter of water to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating from the surface by treating the same for about 15 minutes with a chromic acid solution containing about 5 per cent by weight of chromic acid at a temperature of about 180° F.

11. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aliminum surface for about 15 to 30 minutes in a boiling solution made by dissolving about 20 grams of sodium carbonate and about 5 grams of potassium dichromate in one liter of water to produce a substantial oxide coating on the surface, and thereafter removing the oxide coating with a nitric acid solution containing about 5 to 70 per cent by weight of nitric acid at room temperature.

12. The method of treating an aluminum surface preparatory to applying an adherent coating thereon, which comprises treating the aluminum surface in a hot alkali metal aqueous solution to produce a substantial oxide coating on the surface, said alkali metal solution containing alkali metal carbonates and dichromates in the radio of about 3:1, and thereafter removing the oxide coating with an aqueous solution of at least one of the acids selected from the group consisting of chromic and nitric acids.

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CERTIFICATE OF CORRECTION.

Patent No. 2,171,546. September 5, 1939.

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, second column, line 55, claim 1, before the word "of" insert solution; page 3, second column, line 9, claim 10, for "treatng" read treating; line 14, same claim, for "carbinate" read carbonate; line 39, claim 12, for "radio" read ratio; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 3rd day of October, A. D. 1939.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.