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## METHOD OF SEALING ANODIZED ALUMINUM SURFACES AND ARTICLE PRODUCED THEREBY

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This invention relates to the treatment of oxide layers formed on aluminum, magnesium, and aluminum or magnesium base alloys which will hereinafter be grouped and referred to by the term "aluminum." It relates more specifically to the method for sealing oxide coatings formed on aluminum surfaces to improve the characteristics thereof.

For many purposes, an oxide layer is formed as a protective layer on the aluminum by making the aluminum the anode in an electrolytic cell having an electrolyte formed of about 2 to 70 percent by weight sulphuric acid or other acids or acid salts of the type chromic acid, oxalic acid, sulfamic acid and the like. Any suitable metal, such as lead, may form the cathode. A voltage of about 5 to 20 volts is impressed upon the cell while the electrolyte is held at about 40° C. until an oxide coating of the desired thickness is formed.

In another method, the aluminum may be immersed in a solution, generally alkaline in nature, and the oxide film is formed thereon by chemical reaction. There are a number of other less commercial processes for producing oxide layers on aluminum.

The oxide layer formed under these conditions consists generally of anhydrous alumina ( $Al_2O_3$ ). Especially when formed by anodic oxidation, the layer of aluminum oxide is considered to be relatively hard, porous, highly absorbent and of substantial thickness, depending upon the nature of the metal or alloy, the composition and character of the electrolyte, and the intensity of the electrical current. The oxide coating may contain other modifying components depending upon the process used and the base metal upon which the oxide layer is formed. The introduction of oxide coatings on the surfaces of aluminum is intended to improve resistance to corrosion, resistance to abrasion, and absorption of coloring when treated with suitable tinctorial agents, such as organic dyes and inorganic pigments to provide permanent color in the surfaces of the aluminum.

It has been found that the characteristics of the oxide film can be markedly improved by a process, hereinafter referred to as "sealing" for rendering the film impervious and less porous. Sealing has been carried out commercially by immersing the anodized aluminum in water maintained between 80° C. and its boiling point. Sealing is believed to consist primarily in the conversion of the substantially porous and pervious oxide film of anhydrous alumina to a hydrated product, such as aluminum monohydrate  $Al_2O_3 \cdot H_2O$  with consequent swelling or volume increase of the oxide particles partially to close or seal the pores.

Sealing by the processes heretofore employed is superficial in nature. Measurements have shown that an oxide layer of 15 microns may be converted into the monohydrate to a depth of only 3 to 5 microns by the treatment with hot water depending upon the length of the sealing operation. Further to improve corrosion resistance, the sealing bath has been modified by additions of chromic acid, or boric acid, or metal salts such as sodium dichromate, zinc dichromate, ammonium dichromate, alone or in combination with organic acids, metal fluorides, metal borates and the like, or by the addition of nickel or cobalt sulfates and the like. The modified processes offer some improvement but optimum conversion is not achieved and the sealed surface is ineffective for resistance against attack by strongly acidic or alkaline medium.

It is an object of this invention to provide a method of

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treating oxide coating formed on aluminum to improve the corrosion resistance thereof especially to alkaline or acidic medium.

Another object is to provide an improvement in the method for sealing anodized aluminum surfaces, and it is a related object to produce an improved seal on aluminum oxide surfaces.

A further object is to provide a method for sealing anodized aluminum surfaces at low temperature.

In accordance with the practice of this invention, the improved sealing relation is achieved by the treatment of the formed aluminum oxide layer with a dilute solution of a halogenated phenyl or polyphenyl compound. It is preferred to make use of pentachlorodiphenyl as the halogenated compound but other substances such as partially chlorinated phenyl, diphenyl, terphenyls and the like may be used. It has been found sufficient if the compound is present in solution in amounts ranging from 1 to 10 percent by weight. It is preferred to carry out the treatment at room temperature, but it will be understood that elevated temperatures may be used depending upon the solvent system.

Dilution of the halogenated phenyl or polyphenyl compounds may be effected in suitable organic solvents capable of fostering rapid and complete wetting out of the aluminum oxide surface layer so that full penetration may be achieved. It is preferred to make use of a solvent or solvent system which is non-inflammable, non-toxic, and has a slow evaporation rate, and for such purpose use may be made of the halogenated solvents such as perchloroethylene, trichloroethylene, dichloroethylene, ethylene, chlorohydrin, trichlorobenzene and the like. The solvent system may be further improved by the addition of small amounts of chlorinated paraffin such as in amounts ranging from 0.2 to 2.0 percent by weight.

It has been found sufficient to immerse the aluminum having the oxide surface thereon for 5 to 30 minutes in the bath, but it will be understood that substantial sealing can be effected in less time and that no harmful results will be secured if immersion exceeds 30 minutes. After immersion, the sealed aluminum oxide surfaces may be dried at room conditions but it is preferred to accelerate drying by exposure to elevated temperatures, such for example as drying in an air oven for 10 to 30 minutes at 100 to 120° C. depending upon the mass.

The following are specific examples given by way of illustration but not by way of limitation of the practice of this invention.

### Example 1

A piece of anodically anodized aluminum is immersed for a few minutes in a bath formed of 1 to 5 percent by weight pentachlorodiphenyl dissolved in perchloroethylene maintained at room temperature. After immersion, the piece is dried by heating for about 30 minutes at a temperature of 100 to 120° C.

### Example 2

A piece of aluminum having an anodized surface is immersed at room temperature into a bath formed of 2 percent by weight hexochloroterphenyl and 0.5 percent by weight chlorinated paraffin dissolved in trichloroethylene. After immersion for about 10 to 20 minutes, the piece is removed, rinsed, and then dried at elevated temperature of about 100° C.

### Example 3

A piece of aluminum having an oxide layer formed thereon is immersed in a bath formed of 2 to 3 percent by weight pentachlorodiphenyl and 1.0 percent chlorinated paraffin dissolved in perchloroethylene. The bath is maintained at room temperature or slightly above. After immersion for about 10 minutes, the piece is allowed to dry.

It will be understood that changes may be made in the details of composition, treatment and drying without departing from the spirit of the invention, especially as defined in the following claims.

I claim:

1. In the method of sealing an anodized oxide coating formed on aluminum, the step of coating the oxide coat-

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ing with a dilute solution consisting essentially of chlorinated diphenyl as the substance which remains upon elimination of the diluent, and then drying the treated coating at elevated temperature up to about 120° C.

2. In the method of sealing an anodized oxide coating formed on aluminum, the step of coating the oxide coating with a solution the solids of which consist essentially of 1 to 10 percent of pentachlorodiphenyl.

3. In the method of sealing an anodized oxide coating on aluminum, the step of coating the oxide coating with a solution the solids of which consist essentially of 1 to 10 percent by weight of chlorinated diphenyl and 0.2 to 2.0 percent by weight of chlorinated paraffin.

4. In the method of sealing an anodized oxide coating on aluminum, the steps of coating the oxide coating with a solution the solids of which consist essentially of 1 to 10 percent by weight of chlorinated diphenyl and 0.2 to 2.0 percent by weight of chlorinated paraffin, and drying the treated coating at a temperature of about 100° C.-120° C.

5. In the method of sealing an anodized oxide coating on aluminum, the steps of coating the oxide coating in a bath the solids of which consist essentially of 1 to 10 percent by weight pentachlorodiphenyl and 0.2 to 2.0 percent by weight chlorinated paraffin, maintaining the bath at a temperature corresponding to room conditions, and then drying the coating at elevated temperatures.

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6. Aluminum having an anodized surface coated with a material consisting essentially of pentachlorodiphenyl thereon as a sealer.

7. Aluminum having an anodized surface coated with a material consisting essentially of pentachlorodiphenyl and chlorinated paraffin thereon as a sealer.

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