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"IMPROVEMENTS IN OR RELATING TO MILKY WHITE ANODISING OF ALUMINIUM AND ITS ALLOYS".

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, Rafi Marg, New Delhi-1, India, an Indian registered body incorporated under the registration of Societies Act (Act XXI of 1860).

The following specification describes the nature of this invention.

This is an invention by RAIKUNJE ANANTHA SHENOI, Scientist and SUBBIAH JOHN, Senior Technical Assistant, both are Indian Nationals and employed in the Central Electrochemical Research Institute, Karaikudi-623006, Tamil Nadu, India.

PRICE : TWO RUPEES

145154

This invention relates to improvements in or relating to milky white anodising of aluminium and its alloys.

Hitherto it has been proposed to anodise aluminium and its alloys to a nearly milky white finish in the following ways :

1. Inorganic pigmentation of the anodic oxide coating produced by the conventional anodising processes.
2. Emetal process employs an oxalic acid solution containing salts of thallium, zirconium or titanium.
3. Addition of 3% of zirconium in the form of a sulphate complex to the sulphuric acid solution gives a white coating.
4. Using titanium complex compounds with a mixture of sulphuric acid and oxalic acid, a white coating is obtained.
5. Opaque coatings can also be obtained in a mixture of chromic acid, boric acid and/or oxalic acid.
6. Anodising in the conventional solutions and post-treating with aqueous solutions of aluminium nitrate, nitric acid, phosphoric acid etc. to give a white coating.
7. Anodising in a mixture of sodium carbonate and sodium phosphate to produce anopaque white coating on super purity aluminium.

The drawbacks associated with the hitherto known processes are that -

1. The first process is diffusion controlled and hence control is very difficult to produce a white coating.
2. Opaque coatings produced by the second, third and fourth processes require costlier addition agents and moreover, these are not available in India.
3. The coating obtained by the fifth process is yellowish grey and moreover, the disposal of chromic acid is also a problem.
4. The specimens treated by the process given in (6) always possess the silvery white appearance of a cleaned metallic surface instead of a milky white colour of a pigmented surface.
5. Seventh process is not suitable for aluminium alloys because it produces a greyish coating.

The main object of the present invention is to obviate these disadvantages by using a much simpler process for producing a nearly milky white coating which involves abundantly available chemicals and which can be obtained from local market at a comparatively lower cost than that are necessary for the processes hitherto mentioned.

The main findings underlying the invention consists in anodising of aluminium and its alloys in an aqueous bath comprising the following:

1. an alkali metal carbonate 0.1 to 10% more preferably 0.5 to 2.5%
2. a suitable complexing agent in amounts of 0.1 to 15% more preferably 0.25 to 5% and
3. an accelerator 0.05% to 1% more preferably 0.1 to 0.5%

at a temperature of 30 to 80°C more preferably between 45 and 55°C for 5 to 45 minutes more preferably 20 to 30

The new result flowing from the new finding is that an opaque coating with a milky white appearance suitable for decorative, protective and architectural applications on aluminium and its alloys can be produced.

The novel feature of this process is that this coating is best suited for multicolouring, i.e. in preparing nameplates. The oxide coating produced is porous and absorptive and hence it can be coloured with organic dyes and inorganic pigments. The coating also requires a final sealing treatment for improved corrosion resistance.

The following typical examples are given to illustrate how the invention is carried out in actual practice but not to limit the scope of this invention:

EXAMPLE 1

2S aluminium (minimum 99% Al) plates were polished, degreased and cleaned in 10% sodium hydroxide and washed in water. This was followed by acid dipping in 10% nitric acid for desmutting. Then the specimens were washed in tap water and rinsed in distilled or deionised water and anodising carried out under the following conditions:

Sodium carbonate	1.0%
Glycerol	5%
Sodium fluoride	0.5%
Temperature	45°C
Current density	1-2 A/dm^2
Voltage density	10-30V
Time	25 minutes

After anodising the plates were washed and dried. The plates had an opaque milky white finish.

EXAMPLE 2

3S aluminium plates (containing 1.2% Manganese, rest aluminium) were cleaned as in Ex. 1 and anodised under the following conditions:

Sodium carbonate	2.5%
Polyethylene glycol or Polypropylene glycol :	1%
Sodium fluoride	0.3%
Temperature	55°C
Current density	1-2 A/dm^2
Voltage	10-20V
Time:	30 minutes

The plates were beautifully white anodised and appearance was very good.

EXAMPLE 3

26S aluminium alloy (4.25% copper, rest aluminium)plates were cleaned as in Ex. 1 and anodised under the following conditions:

Potassium carbonate	2%
Glycerine	5%
Potassium fluoride	0.1%
Temperature	50-55°C
Voltage	10-30V
Current density	1-2 A/dm^2
Time	30 minutes

The plates were anodised to a whitish finish and appearance was good.

EXAMPLE 4

Super purity aluminium plates were cleaned as in Ex. 1 and anodised under the following conditions:

Sodium carbonate	5%
Ethyl alcohol	5%
Sodium fluoride	0.05%
Wetting agent	0.05%
Temperature	40° C
Voltage	10-25V
Current density	1-2 A/dm^2
Time	20 minutes

The anodised plates were of milky white colour.

EXAMPLE 5

2S aluminium plates were cleaned as in Ex. 1 and anodised under the following conditions:

Sodium carbonate:	2.5%
EDTA	0.5%
Sodium fluoride	0.1%
Temperature:	60°C
Voltage	5-20V
Current density	1-2 A/dm^2
Time:	20 minutes

The plates were anodised to a milky white finish.

EXAMPLE 6

3S aluminium plates were cleaned as in Ex. 1 and anodised under the following conditions:

Potassium carbonate	4%
Sodium gluconate	1%
Potassium fluoride	0.2%
Temperature	50°C
Voltage	10-25V
Current density	0.6-2 A/dm^2
Time	25 minutes

The coating obtained is of milky white colour.

The main advantages of this present invention are -

1. The chemicals involved in this process are cheap and are available indigenously.
2. This process is best suited for multicolouring, i.e. in preparing nameplates.

Dated this 24th day of December, 1975.

Sd/-
Asstt. Patents Officer,
Council of Scientific & Industrial Research

145154

THE PATENTS ACT. 1970

COMPLETE SPECIFICATION

(Section—10)

IMPROVEMENTS IN OR RELATING TO MILKY WHITE ANODISING OF ALUMINIUM AND ITS ALLOYS.

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH,
Rafi Marg, New Delhi-1, India, an Indian
Registered body incorporated under the Registration of Societies Act (Act XXI of 1860).

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed :—

This invention relates to improvements in or relating to milky white anodising of aluminium and its alloys.

Hitherto it has been proposed to anodise aluminium and its alloys to a nearly milky white finish in the following ways:

I. Inorganic pigmentation of the anodic oxide coating produced by the conventional anodising processes.

II. Ematal anodising process employs an oxalic acid solution containing salts of thallium, zirconium or titanium.

III. Addition of 3% of zirconium in the form of a sulphate complex to the sulphuric acid solution gives a white anodic coating.

IV. Using titanium complex compounds with a mixture of sulphuric acid and oxalic acid, a white oxide coating is obtained.

V. Opaque coatings can also be obtained in a mixture of chromic acid, boric acid and/or oxalic acid.

VI. Anodising in the conventional solutions and post treating with solutions of aluminium nitrate, nitric acid, phosphoric acid etc. to give a white coating.

VII. Anodising in a mixture of sodium carbonate and sodium phosphate produces an opaque white coating on super purity aluminium.

The draw backs associated with the hitherto know processes are that

a) The first process is diffusion controlled and the coating is only superficial. The control is very difficult to obtain reproducible white coating.

b) Opaque coatings produced by the II, III and IV processes require costlier addition agents and more over these are not indigenously available.

c) The coating obtained by the V process is yellowish grey and more over the disposal of chromic acid is also a problem.

d) The articles treated by the VI process always possesses the silvery white appearance of a cleaned metallic surface instead of a milky white colour of a pigmented surface. Hence this process is not commercially used.

e) The last mentioned process is not suitable for aluminium alloys because it gives a greyish coating on most of the alloys.

The main object of the present invention is to obviate these disadvantages by using a much simpler process for producing milky white coating which involves abundantly available chemicals and which can be obtained from local market at a comparatively lower cost than that are necessary for the processes hitherto mentioned.

The main finding underlying the invention consists of anodising of aluminium and its alloys in an aqueous bath comprising 0.5 to 2.5% of an alkali metal carbonate, 0.25 to 5% of a suitable complexing agent and 0.1 to 0.5% of an accelerator at temperature between 55 to 65°C for 20 to 30 minutes to produce a milky white oxide coating at a current density of 0.5 - 2.5 A/dm^2.

The new result flowing from the new finding is that an opaque oxide coating with a milky white appearance suitable for

decorative, protective and architectural applications on aluminium and its alloys can be produced.

The present invention consists of process for producing a white opaque oxide coatings on aluminium and its alloys which comprises the steps of polishing, degreasing, alkaline cleaning desmutting and anodising in an aqueous alkaline solution containing sodium and potassium carbonate and having a complexing agent from the group consisting of glycerine, polyethylene glycol polypropylene glycol, EDTA, ethyl alcohol and an accelerator selected from the group consisting of fluoride and silico fluoride and adjusting the anodising time so that the oxide coating formed on the surface is milky white at a current density of 0.5 - 2.5 A/dm^2

The novel feature of this process is that the oxide coating so produced is suitable for multicolouring i.e., in preparing name plates. The oxide coating so obtained is porous and absorbtive and hence it can be coloured with organic dyes and inorganic pigments.

The following typical examples are given to illustrate how the invention is carried out in actual practice but not to limit the scope of this invention.

EXAMPLE-I

2S aluminium (miniumum 99% Al) plates were polished, degreased and cleaned in 10% sodium hydroxide and washed in water This was followed by acid dipping in 10% nitric acid for desmutting. Then the specimens were washed in tap water and rinsed in distilled or deionised water and anodising carried out under the following conditions:-

Sodium carbonate	1.0%
Glycerol	5%
Sodium fluoride	0.5%
Temperature	65°C
Current density	1.0 A/dm^2
Voltage	10-30V
Time	25 minutes

After anodising the plates were washed and dried. The plates had an opaque milky white finish.

EXAMPLE-2

3S aluminium plates (containing 1.2% Manganese, rest aluminium) were cleaned as in Ex.1 and anodised under the following conditions:

Sodium carbonate	2.5%
Polyethylene glycol or Polypropylene glycol	1%
Sodium silico fluoride	0.3%
Temperature	55°C
Current density	2.0 A/dm^2
Voltage	10-20 V
Time	30 minutes

The plates were beautifully white anodised and appearance was very good.

EXAMPLE-3

26S aluminium alloy (4.25% copper, rest aluminium) plates were cleaned as in Ex.1 and anodised under the following conditions:

Potassium carbonate	2%
Glycerine	5%
Potassium fluoride	0.1%
Temperature	60 - 65°C
Voltage	10 - 30 V
Current density	0.5 A/dm^2
Time	30 minutes

The plates were anodised to a whitish finish and appearance was good.

EXAMPLE-4

Super purity aluminium plates were cleaned as in Ex.1 and anodised under the following conditions:

Sodium carbonate	0.5%
Ethyl alcohol	0.25%
Sodium fluoride	0.25%
Temperature	60°C
Voltage	10 - 25 V
Current density	2.5 A/dm^2
Time	20 minutes

The anodised plates were of milky white colour.

EXAMPLE-5

2S aluminium plates were cleaned as in Ex.1 and anodised under the following conditions:

Sodium carbonate	2.5%
EDTA	0.5%
Sodium fluoride	0.1%
Temperature	60°C
Voltage	5 - 20V
Current density	1.0 A/dm^2
Time	20 minutes

The coating obtained is of milky white colour.

The main advantages of this present invention are -

1. The chemicals involved in this process are cheap and are available indigenously.
2. This process is best suited for multicolouring, i.e., in preparing nameplates.

A process of forming white opaque oxide coating on aluminium which involves the anodising of cleaned aluminium articles in an aqueous solution consisting of 0.5 to 2.5% sodium and potassium carbonate and 0.25 to 5% of a complexing agent from the group consisting of glycerine, polyethylene glycol, polypropylene glycol, EDTA, ethyl alcohol and containing 0.1 to 0.5% of accelerator from the group consisting of fluoride, silicofluoride at temps. of 55 to 65°C for 20 - 30 min. to produce a milky white opaque oxide coating at a CD of 0.5 - 2.5 A/dm^2.

1. A process for producing white opaque oxide coatings on aluminium and its alloys which comprises the steps of polishing, degreasing alkaline cleaning, desmutting and anodising in an aqueous alkaline solution containing 0.5 to 2.5% of an alkali metal carbonate such as sodium and potassium carbonate, 0.25 to 5.0% of a suitable complexing agent from the group consisting of glycerine, polyethylene glycol, polypropylene glycol, EDTA, ethyl alcohol and 0.1 to 0.5% of accelerator slected from the group consisting of fluoride and silico fluoride at temperatures between 55 to 65°C for 20 to 30 minutes to produce a milky white oxide coating at a current density of 0.5 - 2.5 A/dm^2

Dated this 22nd day of December 1976

R Bhaskar Rai

PATENTS OFFICER,
COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH