

Government of India, The Patent Office, 214, Acharya Jagadish Bose Road, Calcutta-17. Complete specification No.143404 dated 7th May 1976. Application and Previsional specification No.915/Cal/1975. Acceptance of the complete specification advertised on 19th November 1977.

Index at acceptance - 7004-05 [LVIII(5)],
142 [LXVI(6)].

International classification - B 0 1 K 1/00.

**"IMPROVEMENTS IN OR RELATING TO DECORATIVE ANODISING
OF ALUMINIUM AND ITS ALLOYS IN ALKALINE ELECTROLYTES
USING ALTERNATING CURRENT".**

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
inventions:

This is an invention by Balkumje Anantha Shenoi, Scientist
and Venkataraman Balasubramanian, Senior Scientific Assistant and
Subbiah John, Senior Technical Assistant, all employed at Central
Electrochemical Research Institute, Karaikudi-623006, Tamil Nadu,
India and Indian Nationals.

PRIC Rs-2.00

This invention relates to improvements in or relating to decorative anodising of aluminium and its alloys in alkaline electrolytes using alternating current.

Hitherto alternating current was not used for the decorative anodising of aluminium and its alloys using alkaline electrolytes.

It is well known that porous anodic oxide coatings suitable for decorative purposes were grown on aluminium and its alloys in electrolytes which have some solvent action on the alumina. The two of the most important commercial processes widely used are based on sulphuric and chromic acids. Alumina will also dissolve in alkaline solutions to give aluminate ions and an anodic film can form in alkaline electrolytes.

The main object of the present invention is to use alternating current in the place of direct current for the production of porous anodic oxide coating an aluminium and its alloys which will be ultimately coloured with organic dyestuffs for decorative purposes.

The main finding underlying the invention consists in anodising aluminium and its alloys using A.C. in an aqueous bath comprising of 2.5 - 7.5% W/V Borax and sufficient amount of sodium hydroxide to bring the pH 9.0 - 10.5 at a temperature of 60-75°C for 45-60 minutes at voltages of 20-30V to produce current densities of the order of 1 to 2A/dm².

The new result flowing from the new finding is that porous anodic coating suitable for dyeing which will ultimately produce pleasing decorative appearance can be developed.

EXAMPLE 1

Polished and degreased 2S aluminium alloy (99% Aluminium) plates were cleaned in 10% sodium hydroxide and washed in tap water. The specimens were then acid cleaned in 10% nitric acid for desmutting and washed in tap water and rinsed in distilled or deionised water. The plates were then anodised under the following conditions:

Borax (sodium tetraborate - $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$)	= 5% W/V
Sodium hydroxide	to adjust the pH to 10
Temperature	- $70 \pm 2^\circ\text{C}$
Current	Alternating current 50 cps
Voltage	20-25V
Current density	$2\text{A}/\text{dm}^2$
Time	60 minutes
Thickness	8 microns

After anodising the plates were washed in tap water and then in distilled water. The specimens were then coloured by dipping in organic dyes for 15 minutes at a temperature of $60 - 65^\circ\text{C}$. The plates were then sealed using the conventional methods. Due to the electropolishing effect of the alkaline electrolyte, the plates had a pleasing decorative appearance.

EXAMPLE 2

3S Aluminium plates (containing 1.2% manganese) were cleaned as in example 1 and alternating current anodising carried out under the conditions:

Borax	5% W/V
Sodium hydroxide	to adjust the pH to 10
Temperature	$65 \pm 2^\circ\text{C}$
Voltage	22V
Current density	$1.5\text{A}/\text{dm}^2$
Time	50 minutes
Thickness	6 microns

After anodising, the plates were washed and dipped in dye solution. The plates got coloured and dye absorption was excellent. The plates

were then sealed using the conventional sealing solutions. Even after sealing the colour of the plates had a good decorative appearance.

EXAMPLE 3

Hindalium plates (containing 4.0-4.9% magnesium, 0.5-1.0% silicon, 0.1% copper) were cleaned as in example 1 and A.C anodising carried out under the following conditions:

Borax	5% W/V
sodium hydroxide to adjust the pH to 10	
Temperature	65 ± 2°C
Voltage	23V
Current density	2A/dm ²
Time	60 minutes
Thickness	8 microns

After anodising the plates were coloured by dipping in acid violet, red, green and golden yellow dyes. The plates got coloured and dye absorption was very good. The plates were then sealed using conventional sealing solutions. The plates did not bleed and appearance was very good. The plates had a pleasing decorative finish.

EXAMPLE 4

2S aluminium plates were cleaned as in example 1 and anodising carried out under the following conditions:

Borax	2.5% W/V
Sodium hydroxide	to adjust the pH to 10.0
Temperature	70 ± 2°C
Current	Alternating current 50 cps
Voltage	20-30V
Current	1A/dm ²
Time	60 minutes
Thickness	5 microns

After anodising the plates were washed thoroughly in tap water and then in distilled water. The specimens were then dipped Golden Colour dye bath. The plates took the dye very nicely and then sealed in conventional sealants. The plates had a decorative appearance.

EXAMPLE 5

3S aluminium plates were cleaned as in example 2 and anodising carried out under the following conditions:

Borax	7.5% W/V
Sodium hydroxide	to adjust the pH to 10.0
Temperature	$70 \pm 2^{\circ}\text{C}$
Current	Alternating current 50 cps
Voltage	20 - 30V
Current density	$1\text{A}/\text{dm}^2$
Time	60 minutes
Thickness	4 microns

After anodising the plates were washed and coloured by dipping in violet dye bath which was kept at 60°C for 15 minutes. The plates took the dye and after sealing the appearance was good.

The following are the main advantages of this invention:

1. The process of anodising with alternating current offers the opportunity of anodising in a tank of electrolyte with no cathode plates or other encumbrances. Agitation is accomplished by pumping or recirculating the electrolyte through an external heat exchanger for heating purposes. Hence the entire volume of the electrolyte can be effectively utilised for anodising more area of the work can be processed with maximum current loading.
2. The cost of electrical equipment for A.C. anodising is much cheaper than D.C. since all that is required is a step down transformer and voltage regulator.
3. The electrolyte involved is relatively innocuous and inexpensive.
4. Owing to the natural cleaning power of A.C. where large volumes of hydrogen and oxygen are alternately evolved; the chemical pretreatment of the aluminium alloys containing copper and silicon can be minimised to avoid the revelation of metallurgical defect.
5. The electrolyte is nontoxic and less corrosive in comparison with the other anodising electrolytes.
6. Due to the electropolishing effect of this electrolyte, the plates have a decorative appearance.

Dated this 29th day of April 1973.

-Sd-

ASST. Prof. Officer
Council of Scientific &

143404

THE PATENTS ACT 1970

COMPLETE SPECIFICATION

(See Section 10)

**"IMPROVEMENTS IN OR RELATING TO PROCESS FOR
ELECTROLYTIC DECORATIVE 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 particularly describes and
ascertains the nature of this invention and the manner in which
it is to be performed :-

This is an invention by Balkunje Anantha Shenoi, Scientist
and Venkataraman Balasubramanian, Senior Scientific Assistant and
Subbiah John, Senior Technical Assistant, all employed at
Central Electrochemical Research Institute, Karaikudi-623006,
Tamil Nadu, India and Indian Nationals.

This invention relates to improvements in or relating to a process decorative anodising of aluminium and its alloys in alkaline electrolyte using alternating current.

Hitherto alternating current was not used for the decorative anodising of aluminium and its alloys using alkaline electrolytes.

It is well-known that porous anodic oxide coating suitable for decorative purposes were grown on aluminium and its alloys in electrolytes which have some solvent action on the alumina. The two of the most important commercial processes widely used are based on sulphuric and chromic acids. Alumina will also dissolve in alkaline solutions to give aluminate ions and an anodic film can form in alkaline electrolytes.

The main object of the present invention is to use alternating current in the place of directcurrent for the production of porous anodic oxide coating on aluminium and its alloys which will be ultimately coloured with organic dyestuffs and inorganic pigments for decorative applications. The use of alternating current for anodising eliminates the costly rectifier needed for anodising and what is actually needed for AC anodising is a step-down transformer and a voltage regulator.

According to the inventor the improved process for decorative electro-lytic anodising of aluminium and its alloys in an aqueous alkaline solution is characterised in that alternating current is used

The main finding underlying the invention consists in anodising aluminium and its alloys using AC in an aqueous bath comprising 2.5-7.5% w/v Borax (Sodium Tetra borate) and sufficient amount of sodium hydroxide to bring the pH 9.0-10.5 at a temperature of 60-75°C for 45-60 minutes at voltages of 20-30V to produce current densities of the order of 1 to 2A/dm². The result flowing from the new finding is that porous anodic oxide coating suitable for dyeing which will ultimately produce pleasing decorative appearance can be developed.

The present invention consists of a process for decorative anodising aluminium and its alloys using alternating current in alkaline electrolyte which comprises polishing (electropolishing, chemical polishing or mechanical polishing), degreasing, cleaning and anodising aluminium and its alloys using alternating current in an aqueous bath comprising 2.5-7.5% w/v borax (sodium tetra borate, Na₂B₄O₇·10H₂O) and sufficient amount of sodium hydroxide to adjust the pH to 9.0-10.5 which bath operates at a temperature of 60-75°C for 45-60 min. at voltages of 20-30V to give current densities of the order of 1 to 2 A/dm².

The following typical examples are given to illustrate the invention and not to limit the scope of the invention:

EXAMPLE 1

Polished and degreased 2S aluminium alloy (99% aluminium) plates were cleaned in 10% sodium hydroxide and washed in tap water. The specimens were then acid cleaned in 10% nitric acid for desmutting and washed in tap water and rinsed in distilled or deionised water. The plates were then anodised under the following conditions:

Borax (sodium tetraborate: Na ₂ B ₄ O ₇ ·10H ₂ O):	5% w/v
Sodium hydroxide to adjust the pH to	10.5
Temperature:	60°C
Current:	Alternating current 50 cps
Voltage	20-25V
Current density	2A/dm ²
Time	60 minutes
Thickness	8 microns

After anodising the plates were washed in tap water and then in distilled water. The specimens were then coloured by dipping in organic dyes for 15 minutes at a temperature of 60-65°C. The plates were then sealed using the conventional methods. Due to the electropolishing effect of the alkaline electrolyte, the plates had a pleasing decorative appearance.

EXAMPLE 2

38 aluminium plates (containing 1.2% manganese) were cleaned as in Example 1 and alternating current anodising carried out under the conditions:

Borax	5% w/v
Sodium hydroxides	to adjust the pH to 10
Temperature:	65 ± 2°C
Voltage:	22V
Current density:	1.5 A/dm ²
Time:	50 minutes
Thickness:	6 microns

After anodising, the plates were washed and dipped in dye solution. The plates got coloured and dye absorption was excellent. The plates were then sealed using the conventional sealing solutions. Even after sealing the colour of the plates had a good decorative appearance.

EXAMPLE 3

Hindalium plates (containing 4.0-4.9% magnesium 0.5-1.0% silicon, 0.1% copper) were cleaned as in Example 1 and AC anodising carried out under the following conditions:

Borax:	5% w/v
Sodium hydroxide	to adjust the pH to 10.5
Temperature:	65 ± 2°C
Voltage:	23V
Current density	2 A/dm ²
Time:	60 minutes
Thickness:	8 microns

After anodising the plates were coloured by dipping in acid violet, red, green and golden yellow dyes. The plates got coloured and dye absorption was very good. The plates were then sealed using conventional sealing solutions. The colour did not bleed and appearance was very good. The plates had a pleasing decorative finish.

EXAMPLE 4

2S aluminium plates were cleaned as in Example 1 and anodising carried out under the following conditions:

Borax:	2.5% w/v
Sodium hydroxide:	to adjust the pH to 10.0
Temperature:	$70 \pm 2^{\circ}\text{C}$
Current	Alternating current 50 cps
Voltage:	20-30V
Currents	$1\text{A}/\text{dm}^2$
Time	60 minutes
Thickness:	5 microns

After anodising the plates were washed thoroughly in tap water and then in distilled water. The specimens were then dipped in golden colour dye bath. The dye absorption of the plate was excellent and sealing carried out in conventional sealants.

EXAMPLE 5

3S aluminium plates were cleaned as in Example 2 and anodising carried out under the following conditions:

Borax:	7.5% w/v
Sodium hydroxide:	to adjust the pH to 10.0
Temperature:	75°C
Current	Alternating current 50 cps
voltage	20-30V
Current density	$1\text{A}/\text{dm}^2$
Time	60 minutes
Thickness	4 microns

After anodising the plates were washed and coloured by dipping in violet dye bath which was kept at 60°C for 15 minutes. The dye absorption of the plate was good. The plate was then sealed in conventional sealants.

The following are the main advantages of this invention:

1. In this process of anodising with alternating current, both the electrodes are of aluminium and are anodised simultaneously in a tank of electrolyte with no cathode plates. Agitation is effected by pumping and recirculating the electrolyte through a heat exchanger for heating. Since the entire volume of the electrolyte can be effectively utilised for anodising more area of the work can be

2. The cost of electrical equipment for AC anodising is much cheaper than DC since all that is required is a step-down transformer and voltage regulator.
3. The electrolyte involved is relatively innocuous and inexpensive.
4. Owing to the natural cleaning power of AC where large volumes of hydrogen and oxygen are alternately evolved, the chemical pretreatment of the aluminium alloys containing copper and silicon can be minimised to avoid the revelation of metallurgical defect.
5. The electrolyte is non-toxic and less corrosive in comparison with the other anodising electrolytes.
6. Due to the electropolishing effect of this electrolyte, the plates have a decorative appearance.

This invention consists in the use of alternating current in the place of direct current for the production of porous anodic oxide coating on aluminium and its alloys in alkaline media and ultimately colouring the oxide coating with organic dyestuffs or inorganic pigments for decorative applications. The electrolyte is an aqueous bath comprising 2.5-7.5% w/v sodium tetra borate (Borax) and sufficient amount of sodium hydroxide to keep the pH at 9.0-10.5. Anodising is carried out at a temperature of 60-75°C for 45-60 min. at voltages of 20-30V to give current densities of the order of 1-2A/dm².

We Claim:

1. An improved process for decorative electrolytic anodising of aluminium and its alloys in an aqueous alkaline solution characterised in that alternating current is used;
2. A process as claimed in claim 1 wherein the alkaline solution consists of 2.5-7.5% w/v borax (sodium tetraborate) and sufficient amount of sodium hydroxide to adjust the pH to 9.0-10.5.
3. A process as claimed in claim 1 wherein the alkaline anodising bath operates at a temperature of 60-75°C for 45-60 min. at voltages of 20-30V to give current densities of the order of 1 to 2A/dm².

Dated this 2nd day of May, 1976.

Sd. xxx
(I.M.S.MIAK)
SCIENTIST 'E' PATENI
Council of Scientific & Industrial Res.