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### PROVISIONAL SPECIFICATION

IMPROVEMENTS IN OR RELATING TO ELECTRODEPOSITION OF ALUMINA OVER METALS AND NON-METALS (SUITABLY MADE CONDUCTIVE) FOR DECORATIVE FINISHES.

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, RAJ 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 BALKUNJE ANANTHA SHENOI, Scientist, THIRUMANAMCHE-RI SESHADRI KRISHNAN, Senior Laboratory Assistant and VENKATARAMAN BALASUBRAMANIAN, Junior Scientific Assistant, all are Indian Nationals and are employed in the Central Electrochemical Research Institute, Karaikudi-3, India.

This invention relates to improvements in or relating to Electrodeposition of alumina over metals and non-metals (suitably made conductive).

Hitherto it has been proposed to obtain alumina coatings by spraying technique on metals other than aluminium for which the metal is anodised.

The object of this invention is to obtain adherent hydrated alumina by electrodeposition in situ over metals and non-metals (suitably made conductive) like copper, mild steel, brass, zinc, aluminium and magnesium. Subsequently, the electrodeposited alumina coating can be coloured with organic dyes or given lacquer coating or sealed.

To these ends, the invention broadly consists in electrodepositing adherent hydrated alumina in situ over metals mentioned above as anode from an aqueous aluminate solution of sodium or potassium or alkaline earth metals buffered to suitable pH with boric acid, acetic acid and citric acid having buffering action as electrolyte and by passing a direct current using stainless steel or any insoluble metal or aluminium as cathode. After electrodepositing alumina on the basis metals, it is coloured in the dye solution in the conventional manner. It can also be sealed either by lacquering or by boiling in hot water. Once a hydrated alumina is formed, in an adherent manner the layer, can be given any treatment which is prevalent for anodised aluminium.

The following examples are given to illustrate the invention :

#### Example 1

A polished brass sample was taken, degreased with trichloroethylene and treated as anode in the following electrolyte :

Sodium aluminate :	5%
Boric acid :	3%
pH :	11
Temperature :	45°C

Time : 10 minutes

Current density : 96 Amp/ft<sup>2</sup>

An alumina coating was obtained. Later it was coloured and sealed:

#### Example 2

A polished mild steel sample was taken, degreased with trichloroethylene and treated as anode in the following electrolyte :

Potassium aluminate : 7.5%

Citric acid : 5%

pH 9.5

Temperature : Room temperature

Current density : 144 Amp/ft<sup>2</sup>

Time : 10 minutes

An alumina coating was obtained. Later, it was coloured and sealed.

#### Example 3

A polished copper specimen was taken, degreased with trichloroethylene and treated as anode in the following electrolyte :

Sodium aluminate : 5%

Acetic acid : 3%

pH : 12

Temperature : Room temperature

Current density : 48 Amp/ft<sup>2</sup>

Time : 10 minutes

An alumina coating was obtained. Later, it was dyed and sealed.

#### Example 4

A polished zinc specimen was taken, degreased with trichloroethylene and treated as anode in the following electrolyte :

Sodium aluminate : 10%

Boric acid : 5%

pH : 11

Temperature : 45° C

Current density : 36 Amp/ft<sup>2</sup>

Time : 15 minutes

Price : TWO RUPEES

An alumina coating was obtained. Later, it was dyed and sealed.

#### Example 5

A polished magnesium specimen was taken, degreased with trichloroethylene and treated as anode in the following electrolyte :

Potassium aluminate :	10%
Acetic acid :	5%
pH :	11.5
Temperature :	Room temperature
Current density :	24 Amp/ft <sup>2</sup>
Time :	20 minutes

An alumina coating was obtained. Later, it was dyed and sealed.

#### Example 6

A polished aluminium specimen was taken, degreased with trichloroethylene and treated as anode in the following electrolyte :

Sodium aluminate :	7.0%
Acetic acid :	3%
pH :	12
Temperature :	40°C
Current density :	12 Amp/ft <sup>2</sup>
Time :	30 minutes

An alumina coating was obtained. Later, it was dyed and sealed.

#### Example 7

A copper plated A.B.S. plastic specimen was taken, degreased with trichloroethylene and treated as anode in the following electrolyte :

Potassium aluminate :	5%
Citric acid :	3%
pH :	12
Temperature :	Room temperature
Current density :	48 Amp/ft <sup>2</sup>
Time :	20 minutes

An adherent alumina coating was obtained. Later, it was coloured and sealed.

The following are among the main advantages of the invention :

1. The process would be capable of depositing attractive, decorative, protective coatings on a number of commonly used metals such as aluminium, copper, zinc and mild steel.
2. The process would be quick and economical to use. The chemicals involved are cheap and the plant required is, normal plating shop equipment.
3. The alumina deposited also gives abrasion resistance and corrosion resistance.

Dated this 10th day of September, 1971.

Sd/-

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## COMPLETE SPECIFICATION

### IMPROVEMENTS IN OR RELATING TO ELECTRODEPOSITION OF ALUMINA OVER METALS AND NON-METALS (SUITABLY MADE CONDUCTIVE) FOR DECORATIVE FINISHES.

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, RAJI 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, THIRUMANAMCHERI SESHADRI KRISHNAN, Senior Laboratory Assistant, and VENKATARAMAN BALASUBRAMANIAN, Junior Scientific Assistant, all of the Central Electrochemical Research Institute Karai-kudi, India, all Indian citizens.

This invention relates to improvements in or relating to electrodeposition of alumina over metals and non-metals (suitably made conductive) for decorative finishes.

Hitherto it has been proposed to obtain alumina coatings by spraying technique on metals other than aluminium for which the metal itself is anodised.

This is open to the objection that alumina coatings obtained by other known processes are not suitable for decorative purposes except the process described in the following specification.

The object of this invention is to obtain a adherent hydrated alumina by electrodeposition in situ over the basis metals like copper, mild steel, brass, zinc, aluminium and magnesium and on non-metals (suitably made conducting). Subsequently the electrodeposited alumina can be coloured with organic dyes and given lacquer coating or sealed.

According to the present invention there is provided a process for electrodepositing alumina on metals and non-metals (suitably made conducting) suitable for decorative finishes is characterised in using a bath comprising an alkali metal aluminate where any one or any of the combination of acids like boric acid, acetic acid, oxalic acid, tartaric acid and a complexing agent like sodium gluconate and sorbitol are present.

Thus adherent hydrated alumina is electrodeposited in situ over metals like copper, mild steel, brass, zinc, aluminium and magnesium and

on non-metals (suitably made conducting) as anodes from an aqueous aluminate solution of sodium or potassium adjusted to pH 8-11 with boric acid, acetic acid, oxalic acid and tartaric acid or with combination of any of the above said acids along with complexing agents like sodium gluconate or sorbitol and by passing direct current using stainless steel, graphite or aluminium as cathode.

An adherent alumina is electrodeposited over metals and non-metals (suitably made conductive) by using it as anode. The alumina coating thus obtained can be coloured using an organic dye solution used for anodising process. Post-anodising techniques like sealing and lacquering can also be done over the alumina coatings. In the present invention, an adherent alumina is electrodeposited in situ over metals like copper, mild steel, brass, zinc, aluminium and magnesium and on non-metals (suitably made conductive) from an alkali aluminate solution of pH 8 to 11.

The alumina coating is obtained by adopting the optimum pH range described in this specification. Further, the deposit is obtained within a few seconds unlike anodising process which requires more time.

Thus, the pH of an aqueous electrolyte of alkaline sodium aluminate or potassium aluminate in the concentration range of 3 to 15% is adjusted between 8 and 11 using boric acid, oxalic acid, acetic acid and tartaric acid or with combination of any of the above acids. The concentration range of acid required are between 1 and 6%. Complexing agents like sodium gluconate or sorbitol is used to keep the aluminium in solution from precipitating as aluminium hydroxide. The concentration of the complexing agents is 0.05 to 0.5%. The deposit is obtained on metals which are treated as anodes using stainless steel or aluminium or graphite as cathode. Direct current is passed through the electrodes kept in the solution using a current density of 12 to 144 amps. per sq. ft at 30 to 50°C.

The deposit obtained was subjected to accelerated corrosion test using continuous spray of 3% sodium chloride. The specimen gave better corrosion resistance than the bare metal itself and it withstood 96 hours of continuous salt spray. But the bare metal failed within 24 hours spraying.

The complete process is done within 10 minutes including sealing when compared with anodising where it takes about one hour to complete the process.

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

#### Example 1

A polished brass sample was taken, degreased with trichlorethylene and treated as anode in the following electrolyte :

Sodium aluminate :	5% w/v
Boric acid :	3% w/v
Sodium gluconate :	0.2% w/v
pH	11
Temperature :	45° C
Time :	10 minutes

Current density : 96 A/sq. ft.

An alumina coating was obtained. Later, it was coloured and sealed.

#### Example 2

A polished mild steel sample was taken, degreased with trichlorethylene and treated as anode in the following electrolyte :

Potassium aluminate :	7.5% w/v
Tartaric acid :	2.0% w/v
Boric acid :	3.0% w/v
Sorbitol :	0.5% w/v
pH :	9.5
Temperature :	Room
Current density :	144 A/sq. ft.

An alumina coating was obtained. Later, it was coloured and sealed.

#### Example 3

A polished copper specimen was taken, degreased with trichlorethylene and treated as anode in the following electrolyte :

Sodium aluminate :	5% w/v
Acetic acid :	3% w/v
Sodium gluconate :	0.5% w/v
pH :	10
Temperature :	Room
Current density :	48 A/sq. ft.
Time :	10 minutes

An alumina coating was obtained. Later, it was dyed and sealed.

#### Example 4

A polished zinc specimen was taken, degreased with trichlorethylene and treated as anode in the following electrolyte :

Sodium aluminate :	10% w/v
Boric acid :	3% w/v
Oxalic acid :	2% w/v
Sodium gluconate :	0.2% w/v
pH :	9
Temperature :	45° C
Current density :	36 A/sq. ft.
Time :	15 minutes

An alumina coating was obtained. Later, it was dyed and sealed.

#### Example 5

A polished magnesium specimen was taken, degreased with trichlorethylene and treated as anode in the following electrolyte :

Potassium aluminate :	10% w/v
Oxalic acid :	2% w/v
Boric acid :	3% w/v
Sorbitol :	0.2% w/v
pH	9

Temperature : Room  
 Current density : 24 A/sq. ft.  
 Time : 20 minutes

An alumina coating was obtained. Later, it was dyed and sealed.

#### Example 6

A polished aluminium specimen was taken, degreased with trichlorethylene and treated as anode in the following electrolyte :

Sodium aluminate : 7.0% w/v  
 Oxalic acid : 3% w/v  
 pH 11  
 Temperature : 40° C  
 Current density : 12 A/sq. ft.  
 Time : 30 minutes

An alumina coating was obtained. Later, it was dyed and sealed.

#### Example 7

A copper plated ABS plastic specimen was taken, degreased with trichlorethylene and treated as anode in the following electrolyte :

Potassium aluminate: 5% w/v  
 Tartaric acid : 3% w/v  
 pH 11  
 Temperature : Room  
 Current density : 48 A/sq. ft.  
 Time : 20 minutes

An adherent alumina coating was obtained. Later, it was coloured and sealed.

The following are among the main advantages of this invention :

1. The process would be capable of depositing attractive, decorative and protective coatings on a number of commonly used metals such as aluminium, copper, zinc, brass, magnesium and mild steel.

2. The process would be very quick. The chemicals involved are cheap and they are available in our country. The normal plating shop equipment are sufficient for this process.

3. The alumina deposited also gives corrosion resistance and abrasion resistance.

#### WE CLAIM :

1. A process of electrodepositing alumina on metals and non-metals (suitably made conducting) suitable for decorative finishes is characterised in using a bath comprising an alkali metal aluminate where any one or any of the combination of acids like boric acid, acetic acid, oxalic acid, tartaric acid and a complexing agent like sodium gluconate and sorbitol are present.

2. A process of electrodepositing alumina on metals and non-metals (suitably made conducting) as claimed in claim 1 wherein the alkali metal aluminate is used in the range of 3 to 15% w/v.

3. A process for electrodepositing alumina on metals and non-metals (suitably made conducting) as claimed in claims 1 and 2 wherein the acids used are in the concentration range of 1 to 6% w/v of the electrolyte so that the pH of the bath is adjusted between 8 and 11.

4. A process of electrodeposition of alumina over metals and non-metals (suitably made conducting) as claimed in claims 1 to 3 wherein any one of the complexing agents like sodium gluconate or sorbitol is present in the range of 0.05 to 0.5% w/v of the electrolyte.

5. A process as claimed in claims 1 to 4 wherein the current density for 12-144 A sq. ft is employed to obtain the deposit.

Dated this 11th day of August, 1972.

Sd./-

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