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(54) PROCESS AND APPARATUS FOR INTEGRALLY COLOUR
 ANODISING ALUMINIUM

(71) We, KAISER ALUMINUM & CHEMICAL CORPORATION, a corporation organised under the laws of the State of Delaware, United States of America, of 300 Lakeside Drive, Oakland, California 94604, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

It is now well known to integrally colour anodise aluminium and aluminium alloy products such as sheets, extrusions, castings and the like for architectural and other purposes where the integrally coloured coatings provide a high aesthetic appeal. The basic process was first described by Deal et al in U.S. Reissue Patent No. 25—556 assigned to the present assignee. The electrolyte described by Deal et al is an aqueous solution of sulfosalicylic acid with small amounts of sulphuric acid or metal sulphates included therewith. Subsequently, it has been found that many compounds, both organic and inorganic, can be utilised with small amounts of sulphuric acid and/or metal sulphates in aqueous solution to form integrally coloured anodic oxide coatings. Examples of these compounds include sulfophthalic acid, sulforesorcinic acid, lignosulfonic acid, oxalic acid, maleic acid, succinic and combinations thereof, and polyacid species such as dichromate, molybdate, tungstate and vanadate. All of these electrolytes are characterised by the production of anodic oxide coatings which have a resistance which increases during the anodising process at a substantial rate and which the colour density thereof is primarily voltage dependent. This resistance characteristic of the integrally coloured anodic oxide coatings is to be distinguished from the resistance characteristics of clear or colourless anodising in sulphuric acid electrolytes and the like which increases up to a certain value and remains substantially at this value or increases just slightly during the remainder of the anodizing process. The oxide coatings

produced in chromic acid electrolytes do exhibit a rapidly increasing resistance during the formation thereof but the color density of these coatings, which is very low, is not primarily voltage dependent. Moreover, chromic acid anodizing is normally not used to anodize aluminium for architectural purposes because of the very limited color range obtainable.

In the process described by Deal et al, the aluminium workpiece is submerged in the electrolyte and subjected, as the anode, to electrolysis. Although many electrical programs can be employed, only two are presently being commercially used to any extent. The most efficient is a two-step process comprising subjecting the workpiece to a substantially constant current density until a particular voltage is reached and then maintaining this voltage at a substantially constant level until the desired total current quantity has passed through the workpiece. The elapsed time of the constant current density phase is commonly termed "time to peak"—that is, the time to reach peak voltage. The other method comprises maintaining the substantially constant current density until the desired total current quantity has passed through the workpiece.

A multitude of variables affect the color of the anodic oxide coating. These variables include workpiece composition and surface characteristics, bath composition, bath temperature, current density, peak voltage and total current quantity passed through the workpiece. In the past color control from batch to batch has been quite difficult due to the problems of controlling all the variables. This is particularly true over long periods of time due to the changes which occur within the controllable limits of bath composition. To minimize the color variance, the aluminium industry has placed stringent controls on the composition and thermal treatment of the aluminium workpiece. Similar controls have been placed on the initial and operating compositions of the electrolyte bath. In addition, most anodizing installations have cation

exchange devices for the removal of dissolved aluminium and periodically analyze the bath to determine the need of adding other components—e.g., sulfuric acid and sulfosalicylic acid. Moreover, most anodizers attempt to control the temperature of the bath within $\pm 2^\circ\text{F}$. by suitable refrigerating and heating means. However, due to the heat generation at the anode-electrolyte interface, temperature gradients exist within the bath. The anodizers agitate the bath either mechanically or with air to minimize these gradients; however, agitation does not ensure a constant temperature at the interface of the electrolyte and the surface of the workpiece—the critical place for color generation. Several anodizers have installed automatic control devices for maintaining the proper current density and peak voltage to eliminate human error in the operation of the anodizing program. Despite the above precautions, the control of color is still technically a difficult task and the reject rate remains quite high. Even with automatic control devices most, if not all, anodisers must visually inspect the workpiece to determine whether the colour thereof matches the standard colour sought. This is particularly noticeable over long periods of time due to the buildup of decomposition products in the bath. As the decomposition products build up in the bath, the colour of the oxide coating will not match the standard colour sought. Due to the cost of the electrolyte, frequency replacement would be economically prohibitive.

It is an object of the present invention to provide a process and apparatus for controlling the anodising process wherein the effect of variations of workpiece composition and surface characteristics, bath composition and temperature and the like on the ultimate colour of the anodic oxide coating are minimized.

This invention relates to a process and apparatus for controlling the integral colour anodising programme wherein reproducibility of the colour of the oxide coating from anodising load to anodising load is substantially improved. The invention is predicated upon the discovery of controlling the voltage across the electrolytic cell with respect to time in accordance with a preselected voltage-time relationship to obtain a particular colour and thereby obtaining integrally coloured anodic oxide coatings that will match from anodising load to anodising load despite variations in the workpiece composition and surface characteristics, the bath composition and temperature and the like.

Moreover, the control process of the present invention eliminates the need for determining the surface area of the workpiece because the control program is based upon voltage which is not surface area dependent. The prior art color anodizing technique required surface area determinations because the control was based

on current density (amp/ft² of workpiece surface). Furthermore, in the prior art it was believed that for reproducible colours the total current quantity should not vary from batch to batch because the thickness of the oxide coating is dependent upon the total current quantity and thickness of the coating affected the color.

The present invention was developed from the belief that the color generation of integral color anodizing processes is primarily dependent upon a dark band of anodic oxide immediately adjacent to the metallic substrate and below a relatively colorless oxide layer which is exposed to the atmosphere. A slight golden or brownish hue permeates the entire oxide coating, but this hue has very little effect on the colored appearance of the anodized workpiece. The colored appearance of the workpiece is a function of the color density and the thickness of the dark-band layer; however, the color density is primarily the color determinant. The dark-band formation occurs only after a threshold voltage is reached, the level of which varies depending upon the bath composition, temperature, etc. The color density, i.e., the lightness or darkness per unit thickness, of the dark band is primarily a function of voltage—the higher the voltage the higher the color density (i.e., the darker becomes the oxide layer). Moreover, although the total current quantity passed through the workpiece after the threshold voltage is reached determines the thickness of the dark-band layer, and thus to a certain extent the color of the workpiece, the effect of the total current quantity during that period is not as great as the effect of the voltage. Therefore, by controlling the voltage with respect to time, the color density is very reproducible and, although the current density and total current quantity will change from load to load, the change is of insufficient magnitude to noticeably change the colored appearance of the anodized workpiece.

With the prior art programs of constant current density and constant voltage or constant current density alone, the color anodizing of complicated workpieces was extremely difficult due to the difficulty in determining surface area. A trial-and-error procedure was usually employed wherein the surface area of the workpiece was merely estimated and then standard programs were employed which were designed to produce the desired color. If the desired color for the workpiece was not obtained, the process was repeated on another workpiece and variables were changed such as peak voltage, current density, total anodizing time, and the like in an attempt to match the standard colour sought. If complicated workpieces having different shapes and/or surface areas were to be anodised to the same desired colour, the entire trial and error procedure would have to be repeated.

The control process of the present invention avoids this problem allowing aluminium workpieces of all shapes and sizes to be anodized by the same control programme to give identical coloured oxide coatings.

This control of the present invention is advantageously obtained by developing a voltage-time relationship for a given bath composition and temperature and particular workpiece composition to give an anodic oxide coating with the desired colour; transferring said relationship as data to a data storage means; retrieving said voltage-time data in the form of a signal and controlling the voltage impressed across the electrolytic cell with respect to time by means responsive to said signal.

According to one aspect of the present invention there is provided in the process of integrally colour anodizing an aluminium workpiece in an electrolytic cell, wherein the workpiece is subjected as an anode to anodization in an acidic aqueous electrolyte, which produces an anodic oxide surface characterized by resistance which increases at a substantial rate during the formation thereof and by the colour density thereon which is primarily voltage dependent, the improvement in the control of the colour of the oxide coating comprising:

- (a) developing a voltage-time relationship to control the voltage during the time the workpiece is being anodized for a given bath composition and workpiece composition to give a desired colour,
- (b) providing a voltage source to said cell,
- (c) generating a signal representing a reference voltage at a particular time in accordance with said voltage-time relationship,
- (d) sensing the voltage provided by said source,
- (e) generating a signal representing the sensed voltage,
- (f) comparing by an instrumentality the sensed voltage signal with the reference voltage signal, and
- (g) adjusting the voltage source by means responsive to said instrumentality to provide a voltage corresponding to the reference voltage.

According to another aspect of the present invention there is provided apparatus for integrally colour anodizing an aluminium workpiece, comprising an electrolytic cell in which the work-piece is subjected as an anode to anodization in an acidic aqueous electrolyte to produce an anodic oxide surface characterized by resistance which increases at a substantial rate during the formation thereof and by the colour density thereon which is primarily voltage dependent, means to control of the colour of the oxide coating comprising:

- (a) means for developing a voltage-time relationship to control the voltage dur-

ing the time the workpiece is being anodized for a given bath composition and workpiece composition to give a desired colour,

- (b) a voltage source for said cell,
- (c) means for generating a signal representing a reference voltage at a particular time in accordance with said voltage-time relationship,
- (d) means for sensing the voltage provided by said source,
- (e) means for generating a signal representing the sensed voltage,
- (f) an instrumentality for comparing the sensed voltage signal with the reference voltage signal, and
- (g) control means responsive to said instrumentality for adjusting the voltage source to provide a voltage corresponding to the reference voltage.

Reference is made to the accompanying drawing to illustrate various features of the invention.

Figure 1 illustrates typical voltage-time and current density-time relationships characteristics of a prior art two-step, constant current density—constant voltage, integral colour anodising programme.

Figure 2 illustrates typical voltage-time, current density-time relationships characteristics of a prior art single-step, constant density, integral colour anodising programme.

Figure 3 is a schematic diagram of a control system for controlling the integral colour anodising in accordance with the present invention.

Figure 4 is a diagrammatic view of an advantageous device for storing and retrieving voltage-time data.

In accordance with the invention, a voltage-time relationship is developed for a particular aluminium alloy by anodizing an aluminium workpiece having the alloy composition, in accordance with known constant density-constant voltage or constant current density programs for the particular alloy and bath composition to obtain the anodic oxide coating of the desired color and texture. During anodizing, the voltage with respect to time is noted, either continually by automatic means, or periodically by manually noting the voltage at particular intervals of time.

Having developed the voltage-time relationship, it is transferred or programmed into a data storage means. Although any suitable data storage means, including both digital and analog, may be employed, the function generating device shown in Figure 4 has been found particularly convenient. It is manufactured and sold under the trademark Data-Trak by Research Incorporated. A similar device is described and claimed in U.S. Patent Specification No. 3,329,804 assigned to the same company.

In the device of Figure 4, the voltage-time

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data is scribed on a Mylar (Mylar is a registered trade mark) card 1 coated with a conductive material. The scribing removes the conductive material from the card, dividing the card into two electrically isolated conductive planes. In operation the two isolated planes are separately charged by oppositely phased AC voltages, by circuitry 2 establishing an electrostatic voltage gradient across the scribed voltage-time curve. A probe 3 adjacent to, but spacially disposed from, the card scans the card at a particular time based speed. This is achieved by advancing the card 1 relative to the probe 3 in the direction opposite to that identified by the arrow labelled "Time". The probe picks up from the electrostatic field an error signal having a magnitude proportional to the probe's deviation from the centre of the programme line. A servo system comprising a servo amplifier 4 and a servo motor 5 responds to an error signal to correct the probe's deviation by rotating the shaft of an output potentiometer 6 which is mechanically connected to the probe by cable 7 and pulleys 8, 9 and 10. The rotational movement of the potentiometer shaft varies the position of the wiper 11 thus varying the output of the potentiometer which is directly related to the voltage-time curve scribed on the conductive surfaces as measured from a base co-ordinate. The output signal may then be conveniently employed as a reference signal for controlling the voltage impressed across the electrolytic cell because it is directly related to the voltage-time data stored on the card 1 measured from a base-co-ordinate.

Referring particularly to Figure 3, a system is shown for controlling the voltage across the cell. The system comprises a direct current source 20, a voltmeter 21, a programmer or function generating device 22, a comparator 23 and a control means 24 responsive to any error signal produced by the comparator 23 in conjunction with an electrolytic cell 30 with an anode 31 and cathode 32. The DC source 20 normally comprises a rectifier and a suitable transforming device for converting alternating current to a relatively stable DC output. The voltmeter 21 senses the voltage across the anode and cathode of the anodizing cell. The comparator compares the measured cell voltage with the desired voltage represented by the reference signal from the programming or function generating device 22 and generates an error signal if the cell voltage does not correspond to the reference signal from function generating device 22. Should an error signal exist, the controller 24 will adjust the DC source to provide the programmed voltage across the cell and reduce the error signal.

In an alternative system similar to the one shown in Figure 3, the voltmeter measures the voltage between the anode and an auxiliary electrode spaced a short distance from the

anode. In this manner several anodizing tanks can be controlled by the same voltage-time program if the anode-electrode spacing is kept the same in each tank because the voltage drops in the bus bars, splines and the like are not included in the voltage-time program.

Other suitable control systems can be employed; for example, that shown in U.S. Patent Specification No. 3,121,054.

The following examples are given to more fully illustrate the advantages of the control process of the present invention.

A 5005 aluminium alloy sheet was cut up to provide specimens for the integral color anodizing tests. Three separate aqueous electrolytic baths were prepared having the compositions set forth in Table I.

TABLE I
Bath Composition—Grams/Liter
Sulfosalicylic

	Acid	H ₂ SO ₄	Al
Bath No. 1	64.6	5.8	1.7
Bath No. 2	61.9	5.6	1.9
Bath No. 3	67.3	6.0	1.5

Example 1

To develop the required voltage-time relationship, a 5005 alloy specimen was subjected to anodizing in Bath No. 1 at a temperature of 25°C in accordance with a program known to give a light-amber color to this alloy. The program comprised subjecting the specimen to a constant current density of until a voltage of 62 volts was obtained and then maintaining this voltage at a substantially constant level until a total current quantity of 10 amperes passed through the specimen and the anodic oxide coating was 0.88 mils thick. During the anodizing the cell voltage was noted at short periodic intervals. The voltage-time relationship obtained was scribed on a Data-Trak card.

Example 2

Another 5005 specimen was subjected to anodization in Bath No. 2 at 20°C. The anodizing program was the voltage-time relationship previously developed and stored on the Data-Trak card in Example 1. The anodizing was controlled by placing the previously scribed card into a model 5300 Data-Trak programmer, the output potentiometer of which controlled the cell voltage. The color of the anodic oxide coating was a light amber which acceptably "matched" the first 5005 specimen anodized to obtain the voltage-time relationship. The oxide coating thickness was 0.70 mil.

Example 3

A separate 5005 specimen was subjected to anodization in Bath No. 2 at a temperature of 25°C and controlled by the same Data-Trak card used in Example 1. The color of the anodic oxide coating "matched" both pre-

viously anodized samples. The oxide coating was 0.79 mil thick.

Example 4

Another of the 5005 specimens was subjected to anodization in Bath No. 2 at a temperature of 30°C and controlled by the same Data-Trak card used in previous examples. The color of the anodic oxide coating "matched" the previously anodized samples. The oxide thickness was 0.84 mil.

Example 5

The test described in Example 2 was repeated using Bath No. 3 and resulting color of the anodized specimen "matched" the other specimens. The oxide thickness was 0.78 mil.

Example 6

The test described in Example 3 was repeated using Bath No. 3 and again the anodic oxide coating "matched" the other specimens. The oxide thickness was 0.82 mil.

Example 7

The test described in Example 4 was repeated using Bath No. 3 and again the anodic oxide coating matched the others. The oxide thickness was 0.93 mil.

The instrumental color determinations of each of the above-mentioned specimens are set forth in Table II. The Green Reflectance values generally indicate the lightness and darkness of the specimen—the higher the value the lighter the color. An untrained eye usually cannot distinguish colors having a difference of Green Reflectance values of less than 2 reflectance units. For most architectural applications a variance of up to 3 units is acceptable.

The Green Reflectance values are obtained by measuring the reflectance of green light from the sample (a standard green filter is placed between the light source and the specimen), utilizing a standard tristimulus colorimeter. This system of color measurement is commonly termed the CIE system.

TABLE II

Green Reflectance, %	Example No.						
	1	2	3	4	5	6	7
	17	16.7	17.2	18.4	16.4	17.4	17.8

It is patent from the above examples and the data in Table II that wide variances in bath composition and temperature can be tolerated by employing the control process of the present invention with little or no perceptible differences in the color of the oxide coatings. Variations in workpiece composition and surface characteristics are similarly minimized. To appreciate the consistent results obtained with the present invention, it should be noted that, if the prior art techniques were employed, the temperature and composition variations shown in the above examples would produce differences in Green Reflectance values of from 5 to 15 units, i.e. the specimens would not "match". The term "aluminium" as used herein includes pulse aluminium and aluminium base alloys containing more than 50% aluminium.

WHAT WE CLAIM IS:—

1. In the process of integrally color anodizing an aluminium workpiece in an electrolytic cell, wherein the workpiece is subjected as an anode to anodization in an acidic aqueous electrolyte, which produces an anodic oxide surface characterized by resistance which increases at a substantial rate during the formation thereof and by the color density thereon which is primarily voltage dependent, the improvement in the control of the color of the oxide coating comprising:

- developing a voltage-time relationship to control the voltage during the time the workpiece is being anodized for a given bath composition and workpiece composition to give a desired color,
- providing a voltage source to said cell,
- generating a signal representing a reference voltage at a particular time in accordance with said voltage-time relationship,
- sensing the voltage provided by said source,
- generating a signal representing the sensed voltage,
- comparing by an instrumentality the sensed voltage signal with the reference voltage signal, and
- adjusting the voltage source by means responsive to said instrumentality to provide a voltage corresponding to the reference voltage.

2. A process according to Claim 1 which comprises continuously generating the reference voltage which varies in accordance with said predetermined voltage-relationship when comparing said reference voltage and said reference voltage deriving an error signal representing their difference, and, when adjusting the voltage source, adjusting said voltage in the sense to reduce said error signal.

3. A process according to Claim 1 or 2 wherein the voltage-time relationship is stored



viously anodized samples. The oxide coating was 0.79 mil thick.

Example 4

Another of the 5005 specimens was subjected to anodization in Bath No. 2 at a temperature of 30°C and controlled by the same Data-Trak card used in previous examples. The color of the anodic oxide coating "matched" the previously anodized samples. The oxide thickness was 0.84 mil.

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The test described in Example 3 was repeated using Bath No. 3 and again the anodic oxide coating "matched" the other specimens. The oxide thickness was 0.82 mil.

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WHAT WE CLAIM IS:—

1. In the process of integrally color anodizing an aluminium workpiece in an electrolytic cell, wherein the workpiece is subjected as an anode to anodization in an acidic aqueous electrolyte, which produces an anodic oxide surface characterized by resistance which increases at a substantial rate during the formation thereof and by the color density thereon which is primarily voltage dependent, the improvement in the control of the color of the oxide coating comprising:

- (a) developing a voltage-time relationship to control the voltage during the time the workpiece is being anodized for a given bath composition and workpiece composition to give a desired color,
- (b) providing a voltage source to said cell,
- (c) generating a signal representing a reference voltage at a particular time in accordance with said voltage-time relationship,
- (d) sensing the voltage provided by said source,
- (e) generating a signal representing the sensed voltage,
- (f) comparing by an instrumentality the sensed voltage signal with the reference voltage signal, and
- (g) adjusting the voltage source by means responsive to said instrumentality to provide a voltage corresponding to the reference voltage.

2. A process according to Claim 1 which comprises continuously generating the reference voltage which varies in accordance with said predetermined voltage-relationship when comparing said reference voltage and said reference voltage deriving an error signal representing their difference, and, when adjusting the voltage source, adjusting said voltage in the sense to reduce said error signal.

3. A process according to Claim 1 or 2 wherein the voltage-time relationship is stored

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as data in a data storage means, and wherein the reference voltage is generated by retrieving the voltage-time data from the storage means.

5 4. A process according to Claim 3, wherein the voltage-time relationship is stored by scribing the voltage-time data on a conductive surface as a voltage-time curve thereby dividing the conductive surface into two electrically isolated conductive surfaces, and wherein the reference voltage is generated by advancing said conductive surface relative to a sensor responsive to the division of said conductive surface.

10 5. A process according to Claim 4 in which said sensor is a probe arranged to follow said scribed voltage-time curve when advanced relative thereto, the speed of advance in one direction of the conductive surface relative to the probe representing the time scale of the voltage-time curve and the displacement of the probe in a direction other than the direction of advance being utilised to generate said reference voltage signal.

15 6. A process according to Claim 3 wherein the voltage-time relationship is stored as data on a plate having a conductive surface by scribing thereon the voltage-time curve, thereby dividing said conductive surface into two electrically isolated conductive surfaces, and wherein the reference voltage is generated using a probe which follows said scribed voltage-time curve whereby the reference voltage is in a linear relationship with the voltage-time curve scribed on said conductive surfaces as measured from a base co-ordinate.

20 7. A process according to any one of the preceding claims in which the voltage of the voltage source is controlled to increase from an initial value to a desired peak value and is then maintained substantially constant at said peak value.

25 8. A method according to any one of the preceding claims wherein the electrolyte comprises an aqueous solution of 0.1 to 2.0% sulphuric acid and a compound selected from the group consisting between about 5 and 30% by weight of an organic acid selected from the group consisting of Sulfosalicylic acid, sulfophthalic acid, lignosulfonic acid, sulforesorcinol, oxalic acid, maleic acid, succinic acid and combinations thereof; and between 0.03 to 0.50 gram-mol per litre of a polyacid species selected from the group consisting of dichromates, molybdates, tungstates and vanadates.

30 9. Apparatus for integrally color anodizing an aluminium workpiece, comprising an electrolytic cell in which the workpiece is subjected as an anode to anodization in an acidic aqueous electrolyte to produce an anodic oxide surface characterized by resistance which increases at a substantial rate during the formation thereof and by the color density thereon which is primarily voltage dependent,

means to control of the color of the oxide coating comprising:

(a) means for developing a voltage-time relationship to control the voltage during the time the workpiece is being anodized for a given bath composition and workpiece composition to give a desired color,

(b) a voltage source for said cell,

(c) means for generating a signal representing a reference voltage at a particular time in accordance with said voltage-time relationship,

(d) means for sensing the voltage provided by said source,

(e) means for generating a signal representing the sensed voltage,

(f) an instrumentality for comparing the sensed voltage signal with the reference voltage signal, and

(g) control means responsive to said instrumentality for adjusting the voltage source to provide a voltage corresponding to the reference voltage.

10. Apparatus according to Claim 9 in which the reference signal generating means comprises data storage means having said predetermined voltage-time characteristic stored as data therein and means for reading said stored data.

11. Apparatus according to Claim 10 in which said data storage means comprises a surface carrying said characteristic as a function of two co-ordinates and the reading means comprises a sensor operable to identify said characteristic and to follow said characteristic and means for advancing said surface relative to said sensor in the direction of one co-ordinate representing time whereby displacement of the sensor in the direction of the other co-ordinate represents the magnitude of the reference signal.

12. Apparatus according to Claim 10 in which the data storage means comprises a conductive surface having the predetermined voltage-time characteristic scribed thereon and dividing said surface into two electrically isolated conductive surfaces.

13. Apparatus according to Claim 12 in which the reading means comprises a probe, means for displacing the probe relative to the surface, servo-means responsive to any departure of said probe from said scribed characteristic to cause the probe to follow the characteristic and means responsive to one component of displacement of the probe to generate said signal.

14. Apparatus according to Claim 12 in which the reading means comprises a probe operable to follow the scribed voltage-time characteristic and means associated with the probe to control the voltage of the voltage source in a linear relationship with the scribed characteristic as measured from a base co-ordinate.



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5 15. Apparatus according to any one of Claims 9 to 13 including comparator means responsive to any difference between said reference signal and said sensed voltage to generate an error signal and means responsive to said error signal to modify said sensed voltage in the sense to reduce said error signal.

10 16. A process for integrally color anodising an aluminium workpiece in accordance with Claim 1 substantially as herein described with reference to Figs. 3 and 4 of the accompanying drawings.

17. Apparatus for integrally color anodising an aluminium workpiece constructed, arranged and adapted to operate substantially as herein described with reference to Figs. 3 and 4 of the accompanying drawings. 15

For and on behalf of the Applicants
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