PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Improvements in or relating to Aluminium Alloys

We, KAISER ALUMINUM & CHEMICAL CORPORATION, a corporation organised and existing under the laws of the State of Delaware, United States of America, of 300 Lakeside Drive, Oakland, California, 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: —

This invention relates to aluminium alloy and a method for treating it which particularly adapts the alloy to accept an inherently coloured anodic oxide coating.

The process for producing inherently coloured anodic oxide coatings on aluminium and aluminium alloys is now familiar. The coatings are produced by immersing the aluminium article to be anodised as the anode in an electric circuit which includes the anode, an inert cathode, and an electrolyte containing sulphuric acid and any of certain substituted aromatic sulphonic acids. The aromatic sulphonic acids having particular utility are sulphosalicylic acid and sulphophthalic acid although others have been found to be operative. Although sulphuric acid is the most useful material to provide sulphate ions, the anodising process may be carried out with heavy metal sulphates dissolved in the electrolyte instead of or in addition to sulphuric acid, the most common of which is ferric sulphate. The anodic oxide coatings obtained by this process range in colour from gold to black and may be made with almost any intermediate shade of bronze or brown. Such colour anodising processes have gained wide acceptance for producing aluminium for architectural and other decorative uses.

The process of producing inherently coloured anodised aluminium articles has created problems which were not known to the anodising art when ony colourless coatings were produced. Chief among these problems is reliable reproduction of colours and other appearance factors. For example, an architect may choose coloured aluminium for a structure from small samples, after which an anodiser must make thousands of square feet of anodised aluminium in the form of large panels, and these panels must match the sample as well as each other in order to be acceptable for use in the structure. During the course of making a large number of such articles the electrolyte compostion or other processing variables may change, and such changes may affect the appearance of the product. Maintaining processing variables and electrolyte composition constant, although not a simple matter, is a commercially feasible one from the anodiser's point of view and attention to these details can mitigate changes in the appearance of the product.

However, one element that is beyond the anodiser's control is the colour response an alloy has to a colour-anodising treatment that results from metallurgical considerations. For example, two specimens of a given aluminium alloy may have slightly differently coloured anodic oxide coatings produced on them by identical anodising processes if, for example, one of these alloys has an impurity-level composition different from the other, or even if two alloys with identical composition were subjected to different thermal treatments during the course of their production. For instance, differences in the homogenising temperature of the rolling ingots, or differences in time held at the homogenising temperature, or differences in the rate of cooling ingots

[Price 4s. 6d.]

after homogenising have been known to produce significant differences in colour response when those ingots were finally rolled into sheets and subjected to colour anodising. Moreover, appearance differences arising from slight differences in impurity-level may be exaggerated by variations in thermal treatment.

In making sheet material for colour anodising, alloys designated by the aluminium Association of America classification system as 5005 have demonstrated desirable properties with respect to their ability to produce coloured anodic oxides, and their ability to be formed into smooth sheets with physical properties adequate for architectural uses. The Aluminum Association of America composition designations for 5005 alloy are:

Si	Fe	Cu	Mn	Mg	Cr	Zn	Others (each)	Others (Total)	Balance
0.4	0.7	0.2	0.2	0.5—1.1	0.1	0.25	0.05	0.15	Aluminium

Aluminum Association of America designations which do not show a range indicate maximum permissible composition. It has been found that composition variations within the above given ranges are not permissible for alloys used in colour anodising in that certain of the components cause large colour response changes so that the difference between maximum and minimum compositions create alloys which, when subjected to identical anodising treatments, will produce colours too different from one another to be acceptable. Further, it has been found that the presence of too much of some of the impurity elements, e.g., Mn, Cr, Fe, Si causes the alloy to be extremely sensitive to its thermal history with regard to colour response. That is, certain impurity elements will cause the alloy to respond to colour anodising substantially differently if thermally treated in one way than if thermally treated in a different way.

This invention includes the discovery of composition limits for aluminium alloys used for producing inherently coloured anodic oxide coatings, and thermal treatments for those alloys which create a sheet material that is not sensitive either to reasonable variations in compositions or in thermal history. Therefore, this invention provides aluminium which is easy to anodise with reproducible and predictable appearance and aluminium which has these anodising response properties locked in by mill practice so that anodisers, who are usually poorly equipped to carry out metallurgical processes, can simply anodise the material in the as-received thermally-treated condition and rely on the appearance-response that will be produced from the anodising process.

The invention includes constituting an aluminium alloy which consists of aluminium, from 0.06—0.15% silicon, from 0.45—0.65% iron, from 0.03—0.10% copper, up to 0.07% manganese, from 0.7—0.9% magnesium, from 0.05 to 0.10% chromium, up to 0.25% zinc and up to 0.15% miscellaneous impurities, balance aluminium. All compositions stated in the specification and appended claims are on a weight basis. The alloy thus constituted is cast as an ingot and heated to homogenising temperature, preferably between 1100 and 1150° F where it is maintained until most of the soluble elements enter solid solution, preferably for a period of at least 7 hours. The objective of this treatment is to produce a metallurgical structure in which the primary alloying ingredients and impurities are uniformly dispersed or dissolved in the aluminium. The alloy is then cooled slowly to a temperature lower than 950° F, the cooling being at an average rate of 30° F per hour or slower. After the temperature of 950° F is reached the ingot can be cooled to room temperature at substantially any rate and it may then be scalped or otherwise prepared for rolling. Before rolling into the form of sheet, the ingot is reheated to a temperature below 950° F preferably from 850—950° F and maintained at that temperature for about 1 hour. Rolling may then be performed in the usual manner to produce sheet or plate of any desired thickness.

It is presently believed that milkiness or cloudiness in anodic oxide coatings is caused by particles of precipitated alloying elements or combinations of alloying elements. In normal thermal treatments where an alloy is homogenised and removed from an oven to air cool, the cooling rate from homogenising temperature is so rapid that the alloy quickly reaches a temperature at which diffusion of atoms or molecules within the alloy is very slow. As a result, precipitation is in the form of many small particles, and these many small particles will appear in a later produced anodic oxide coating where they will scatter light to give the effect of a cloudy or milky coating. Among the first particles to precipitate as the temperature of an alloy drops below the homogenising temperature are the iron-containing particles which are usually combinations of iron with aluminium, manganese, chromium and other ingredients of the alloy.

In the process of the present invention the time spent between homogenising temperature where all of the soluble ingredients are either in solid solution or dissolving, and 950° F, where the diffusion rate for iron-bearing particles becomes too slow to permit significant growth of particles of precipitate, is prolonged

enough the permit the iron-containing precipitate particles to form as relatively large particules. The same total amount of precipitation occurs whether in small particles or large particles and the volume of a particle is proportional to the third power of its diameter, and it is therefore evident that if particles of precipitate are permitted to grow significantly larger, an alloy will contain many fewer particles. It has been found that there is much less scattering of light when few large particles are in an anodic oxide film than when many small particles are, and as a result an anodic oxide film containing fewer and larger particles will appear clear and rich even though the same amount of precipitate is present within the film.

Illustrations of the practice and benefits of this invention are given in the following examples, which compare the practice of the invention (Example 2) with the former practice (Example 1):

EXAMPLE 1:

Over an extended period, about 500 lots of 5005 alloy were cast and rolled. Metal compositions of these lots were within the following limits, expressed in per cent:

Si	0.15—0.30
Fe	0.50—0.70
Cu	0.03—0.13
Mn	0.10 max.
Mg	0.70—0.90
Cr	0.10 max.
Zn	0.25 max.
Others (each)	0.05 max.
Others (total)	0.15 max.
Balance aluminium	

All ingots were homogenised by holding for one to three hours at 1000—1025° F and then cooling in air to room temperature, without special control of the rate of cooling, and then scalped. For hot rolling, the scalping ingots were then reheated to 875—950° F for one hour minimum and rolled. Cold rolling practice and intermediate anneals varied according to the final temper desired and generally followed established plant practice. After etching in 5% by weight aqueous caustic soda solution for 10 minutes at 130° F, sheet samples from each lot were anodised in an aqueous bath containing 90—100 g/l of sulphosalicylic acid and 4.5—5.5 g/l of sulphuric acid at 77° F with a beginning anode current density of 24 amp/sq. ft. When the anodising cell voltage reached 50 volts, anodising was continued at that voltage until 10.5 ampere-hours per square foot of electricity had passed through the sheet. The anodised sheets were then immersed for 20 minutes at 195—205° F in an aqueous bath containing 1.0 g/l of sodium lignosulphonate and 0.5 g/l of nickel acetate at pH 5.0±0.2. The colour of each anodised sheet was measured according to the CIE system of colour measurement using a Photovol Tristimulus Reflectionmeter Model 610. Green reflectance, an indication of the lightness or darkness of the surface, was measured by the percentage of incident green light reflected by the surface. Yellowness factor, as defined by the term

$$\frac{\text{Amber Reflectance} - \text{Blue Reflectance}}{\text{Green Reflectance}} \times 100,$$

as also measured. These measurements provide a practical method of comparing colour differences on integrally coloured anodised surfaces. The various lots of sheet in this example showed a green reflectance range of 12 to 24 and a yellowness factor range of 16 to 43. In architectural applications of integrally coloured anodised aluminium, a colour range of this magnitude would be considered objectionable.

EXAMPLE 2:

Over an extended period, about 50 lots of 5005 alloy were cast and rolled. Metal compositions of these lots were within the following limits, expresed in per cent:

Si	0.06—0.15
Fe	0.47—0.61
Cu	0.05—0.08
Mn	0.01—0.07
Mg	0.75—0.87
Cr	0.05—0.08
Zn	0.25 max.
Others (each)	0.05 max.
Others (total)	0.15 max.
Balance aluminium	

All ingots were homogenised by holding for not less than nine hours at 1100—1150° F and cooled at an average rate of 30° F per hour, or slower, to 950° F. The ingots were then air-cooled to room temperature and scalped. For hot-rolling the ingots were reheated to 850—950° F, held for at least one hour, and then rolled. Cold rolling and annealing practice were as in Example 1. All sheet samples from this example were then etched, anodised and sealed as in Example 1 and the colour factors measured. The green reflectances ranged from 19 to 27 inclusive and the yellowness factors from 22 to 29 inclusive. This represents a substantially smaller colour range than in Example 1 and would be the preferred material for architectural use.

In addition to the narrower range of anodised colour shown by the material of this invention there is an added benefit of noticeably more glossy pleasant appearing anodic coatings. Moreover, clear anodic coatings produced by sulphuric acid anodising of the

material of this invention also show a similar pleasant gloss, in contrast to the matte appearance of similarly anodised material of Example 1. Furthermore, by extended etching of the material of this invention, followed by sulphuric acid anodising, a desirable white appearance can be imparted to the surface.

The alloy constituted, thermally treated and rolled as set forth above has the following properties. It is a 5005 alloy with desirable properties of strength, fabricability, good surface characteristics, and good anodising characteristics. The alloy is not unduly sensitive to variations in thermal history. That is, two ingots having any thermal histories within the ranges characteristic of this invention will have substantially the same response to a colour anodising treatment. The material constituted as above is not sensitive to variations in composition, within the stated limits. That is, two ingots having any composition characteristic of this invention will have substantially the same response to a colour anodising treatment. The compositions and thermal treatments specified above lock-in the colour anodising response of the sheet so that anodisers can rely on the colour response of the alloy they receive and need only concentrate on the details of the electrolytic anodising function that they perform to insure reproducible colours in their product.

WHAT WE CLAIM IS:—

1. A method of treating an alumiunium alloy, which comprises:

a. subjecting an alloy, consisting of 0.06—0.15% of silicon, 0.45—0.65% of iron, 0.03—0.10% of copper, not more than 0.07% of manganese, 0.7—0.9% of magnesium, 0.05—0.1% of chromium, not not more than 0.25% of zinc, not more than 0.15% of other ingredients and the balance aluminium, to heat-treatment by heating it to a homogenising temperature and maintaining it at such temperature for a period sufficient to bring the majority of the soluble ingredients into solid solution, and

b. cooling the alloy from the homogenising temperature to a temperature below 950° F. at an average rate of cooling of not more than 30° F. per hour.

2. A process according to claim 1, in which the homogenising temperature is 1100°—1150° F.

3. A method according to claim 1 or 2, in which the alloy is maintained at the homogenising temperature for a period of at least 7 hours.

4. A method according to claim 1, 2 or 3, in which the alloy is further cooled to room temperature.

5. A method according to any preceding claim, in which the alloy is reheated to a temperature of 850—950° F.

6. A method according to claim 5, in which the alloy is maintained at the reheat temperature for at least 1 hour.

7. A method according to claim 1 or 2, in which the alloy is cooled from 950° F. to room temperature, scalped and heated to a temperature of 850—950° F.

8. A method according to any preceding claim, in which the alloy is rolled to form a sheet.

9. A method according to any preceding claim, in which the heat-treated and cooled alloy is subjected to colour anodising.

10. An aluminium alloy, when treated by a method as claimed in any preceding claim.

11. An aluminium alloy composition, suitable for rolling into sheet form and colour-anodising, consisting of 0.06—0.15% of silicon, 0.45—0.65% of iron, 0.03—0.10% of copper, not more than 0.07% of manganese, 0.7—0.9% of magnesium, 0.05—0.10% of chromium, not more than 0.25% of zinc, not more than 0.15% of other ingredients and the balance aluminium.

12. An aluminium alloy composition according to claim 11, substantially as hereinbefore described.

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material of this invention also show a similar pleasant gloss, in contrast to the matte appearance of similarly anodised material of Example 1. Furthermore, by extended etching of the material of this invention, followed by sulphuric acid anodising, a desirable white appearance can be imparted to the surface.

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WHAT WE CLAIM IS:—

1. A method of treating an alumiunium alloy, which comprises:

a. subjecting an alloy, consisting of 0.06—0.15% of silicon, 0.45—0.65% of iron, 0.03—0.10% of copper, not more than 0.07% of manganese, 0.7—0.9% of magnesium, 0.05—0.1% of chromium, not not more than 0.25% of zinc, not more than 0.15% of other ingredients and the balance aluminium, to heat-treatment by heating it to a homogenising temperature and maintaining it at such temperature for a period sufficient to bring the majority of the soluble ingredients into solid solution, and

b. cooling the alloy from the homogenising temperature to a temperature below 950° F. at an average rate of cooling of not more than 30° F. per hour.

2. A process according to claim 1, in which the homogenising temperature is 1100°—1150° F.

3. A method according to claim 1 or 2, in which the alloy is maintained at the homogenising temperature for a period of at least 7 hours.

4. A method according to claim 1, 2 or 3, in which the alloy is further cooled to room temperature.

5. A method according to any preceding claim, in which the alloy is reheated to a temperature of 850—950° F.

6. A method according to claim 5, in which the alloy is maintained at the reheat temperature for at least 1 hour.

7. A method according to claim 1 or 2, in which the alloy is cooled from 950° F. to room temperature, scalped and heated to a temperature of 850—950° F.

8. A method according to any preceding claim, in which the alloy is rolled to form a sheet.

9. A method according to any preceding claim, in which the heat-treated and cooled alloy is subjected to colour anodising.

10. An aluminium alloy, when treated by a method as claimed in any preceding claim.

11. An aluminium alloy composition, suitable for rolling into sheet form and colour-anodising, consisting of 0.06—0.15% of silicon, 0.45—0.65% of iron, 0.03—0.10% of copper, not more than 0.07% of manganese, 0.7—0.9% of magnesium, 0.05—0.10% of chromium, not more than 0.25% of zinc, not more than 0.15% of other ingredients and the balance aluminium.

12. An aluminium alloy composition according to claim 11, substantially as hereinbefore described.

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