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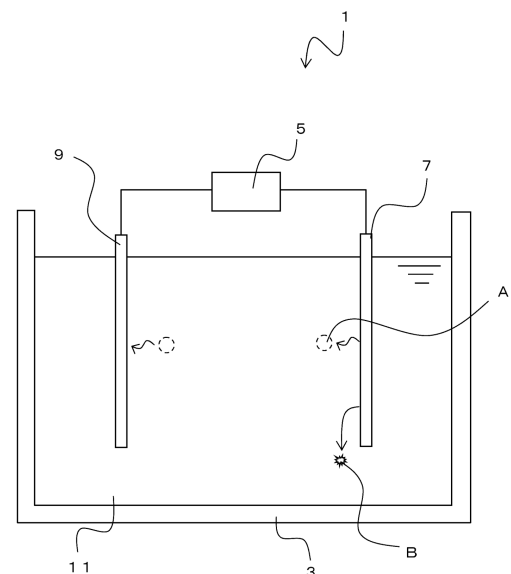
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(54) **METHOD FOR PRODUCING ALUMINUM**

(57) This method for producing aluminum is for obtaining, from an aluminum alloy material, aluminum having higher purity than said aluminum material, the method being characterized by: immersing, in an electrolytic solution (11), a cathode electrode (9) and an anode electrode (7) including an aluminum alloy material containing 0.1 - 24 mass% of Si; causing a current to flow at a current density of 0.1 - 25 mA/cm² on the surface of the anode electrode (7) where the electrolytic solution (11) and the aluminum alloy material come in contact with each other; and depositing aluminum on the cathode electrode.

FIG. 1



Description

TECHNICAL FIELD

- 5 **[0001]** The present invention relates to a method for producing aluminum in which aluminum with higher purity can be obtained from, for example, a casting alloy or the like.

BACKGROUND OF THE INVENTION

- 10 **[0002]** Unlike iron and steel materials, widely used aluminum cannot be smelted from raw ore using thermal reduction reaction with carbon, and thus it is generally necessary to use electricity in the smelting processes, such as in Bayer process or Hall-Hérault process. For this reason, production of virgin aluminum metal emits a large amount of carbon dioxide, imposing heavy burden on environment.

- 15 **[0003]** Thus, it is preferable to reuse used aluminum products through recycling. For example, attempts have been made to easily produce highly purified aluminum material from aluminum alloy scrap (Patent Document 1, for example).

PRIOR ART

[0004]

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[Patent Document 1] International Patent Publication No. 2020/196013
(WO2020/196013)

SUMMARY OF THE INVENTION

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(PROBLEMS TO BE SOLVED BY THE INVENTION)

- 30 **[0005]** The term "aluminum" includes a wide variety of alloys that are used depending on applications. For example, in a case of aluminum cans, A3000 series (Al-Mn series) wrought materials are used, and there is no problem if such materials are directly recycled back into aluminum cans. However, when converting such materials into other alloys, components thereof may cause a problem. In such the case, it is required to reduce an amount of the recycled alloy and to mix a certain amount of virgin metal. For this reason, recycling into the same alloy series is most efficient.

- 35 **[0006]** However, although there has been a large amount of scrap of aluminum alloys that have been used for automobile engine parts etc., the amount of reused aluminum alloys is in decline due to widespread use of electric vehicles and the like in the recent years. This makes it difficult to reuse the aluminum alloys within the same field. In particular, metals in the above field are often cast products (castings, die-castings) containing more alloy components than wrought materials, which makes it difficult to be converted into products in other fields. For example, an AC2A alloy (JIS) used as a casting material contains 4.0 mass% or more and 6.0 mass% or less of silicon (Si) and the content of Si and the like is much higher compared to the wrought materials. This makes it extremely difficult to reuse such the alloy for other

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- [0007]** The present invention was made in view of such problems. It is an object of the present invention to provide a method for producing aluminum, in which aluminum base metal or a used aluminum alloy, particularly an aluminum alloy with high Si content, is used as aluminum raw material such that aluminum having higher purity than the aluminum raw material can be produced.

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(MEANS FOR SOLVING PROBLEMS)

- 50 **[0008]** To achieve the above object, an aspect of the present invention is a method for producing aluminum from an aluminum raw material, in which aluminum with higher purity than the aluminum raw material can be obtained. The method includes immersing an anode electrode, which includes the aluminum raw material containing 0.1 mass% or more and 24 mass% or less of Si, and a cathode electrode in an electrolytic solution, and applying a current to flow at a current density of 0.1 mA/cm² or more and 25 mA/cm² or less on a surface of the anode electrode where the aluminum raw material comes into contact with the electrolytic solution, thereby depositing aluminum on the cathode electrode.

- 55 **[0009]** The aluminum raw material of the anode electrode may further contain 0.1 mass% or more and 5 mass% or more of copper (Cu).

- [0010]** The aluminum raw material of the anode electrode may further contain 0.15 mass% or more and 1.8 mass% or less of iron (Fe).

- [0011]** It is preferable to apply the current to the anode electrode while stirring the electrolytic solution around the anode

electrode.

[0012] It is preferable to make a surface area of a portion of the anode electrode where the aluminum raw material is in contact with the electrolytic solution larger than a surface area of a portion of the cathode electrode that is in contact with the electrolytic solution.

[0013] It is preferable that the electrolytic solution includes dialkyl sulfone and aluminum halide.

[0014] It is preferable that a molar ratio of the dialkyl sulfone to the aluminum halide in the electrolytic solution is 1.5 mol or more and 5 mol or less of the aluminum halide per 10 mol of the dialkyl sulfone.

[0015] It is preferable that the electrolytic solution further includes at least one nitrogen-containing compound selected from a group consisting of ammonium halide, a hydrogen halide salt of primary amine, a hydrogen halide salt of secondary amine, a hydrogen halide salt of tertiary amine, and a quaternary ammonium salt represented by a general formula of $R^1R^2R^3R^4N^+X^-$ where R^1 to R^4 are the same or different alkyl groups and X is a counter anion for a quaternary ammonium cation.

[0016] It is preferable that, while the current is flowing through the anode electrode, the aluminum is deposited on the cathode electrode such that an arithmetic mean height Sa of the anode electrode is $2.3\text{ }\mu\text{m}$ or more and $10\text{ }\mu\text{m}$ or less.

[0017] It is preferable that the current density is 0.1 mA/cm^2 or more and 20 mA/cm^2 or less.

[0018] Also, the above-mentioned features may be combined with each other in any combination.

(EFFECTS OF THE INVENTION)

[0019] The present invention can provide a method for producing aluminum from an aluminum raw material such as a used aluminum alloy, particularly an aluminum alloy with high Si content, so that aluminum with higher purity can be produced.

BRIEF DESCRIPTION OF DRAWINGS

[0020]

FIG. 1 is a schematic view showing an aluminum producing device used in a method for producing aluminum according to an embodiment of the present invention.

FIG. 2A is a scanning electron microscope (SEM) image of a cross section in proximity of a surface of an anode electrode after electrolysis when a current density at the anode electrode is 10 mA/cm^2 .

FIG. 2B is an SEM image of the cross section in proximity of the surface of the anode electrode after electrolysis when the current density at the anode electrode is 20 mA/cm^2 .

FIG. 2C is an SEM image of the cross section in proximity of the surface of the anode electrode after electrolysis when the current density at the anode electrode is 40 mA/cm^2 .

FIG. 2D is an SEM image of the cross section in proximity of the surface of the anode electrode after electrolysis when the current density at the anode electrode is 80 mA/cm^2 .

FIG. 3 is a view showing a correlation between the current density at the anode electrode and surface roughness of the anode electrode surface.

FIG. 4A is an SEM image of a surface of an electrodeposited film deposited on the cathode electrode when high-purity aluminum is used as the anode electrode.

FIG. 4B is an SEM image of a cross section of the electrodeposited film deposited on the cathode electrode when high-purity aluminum is used as the anode electrode.

FIG. 5A is an SEM image of a surface of an electrodeposited film deposited on the cathode electrode when an AC2A alloy is used as the anode electrode and the electrolysis is performed with the high current density.

FIG. 5B is an SEM image of a cross section of the electrodeposited film deposited on the cathode electrode when the AC2A alloy is used as the anode electrode and the electrolysis is performed with the high current density.

FIG. 6A is an SEM image of a surface of an electrodeposited film deposited on the cathode electrode when an AC2A alloy is used as the anode electrode and the electrolysis is performed with the low current density.

FIG. 6B is an SEM image of a cross section of the electrodeposited film deposited on the cathode electrode when the AC2A alloy is used as the anode electrode and the electrolysis is performed with the low current density.

FIG. 7 is a view showing concentration of impurities in the electrodeposited film deposited on the cathode electrode.

DETAILED DESCRIPTION

[0021] A method for producing aluminum from an aluminum alloy to obtain high-purity aluminum according to an embodiment of the present invention includes immersing an anode electrode, which includes an aluminum alloy containing 0.1 mass% or more and 24 mass% or less of Si, and a cathode electrode in an electrolytic solution, and

applying a current to flow at a current density of 0.1 mA/cm² or more and 25 mA/cm² or less on a surface of the anode electrode where the aluminum material comes into contact with the electrolytic solution, thereby depositing aluminum on the cathode electrode.

[0022] Hereinafter, a method for producing aluminum according to the embodiment of the present invention will be described with reference to the accompanying drawings. FIG. 1 is a schematic view of an aluminum producing device 1 used for the method for producing aluminum according to the embodiment of the present invention. In the descriptions hereafter, concentration of each element can be measured by ICP emission spectrometry.

[0023] The aluminum producing device 1 used for the method for producing aluminum according to the present embodiment includes an electrolysis tank 3, a DC power supply 5, an anode electrode 7, a cathode electrode 9, and an electrolytic solution 11. The anode electrode 7 includes an aluminum alloy extracted from scrap or the like. And, the anode electrode 7 and the cathode electrode 9 on which highly purified aluminum is to be deposited are immersed in the electrolytic solution 11, and the DC power supply 5 applies a DC voltage between the anode electrode 7 and the cathode electrode 9. At this time, while the DC voltage is applied between the anode electrode 7 and the cathode electrode 9, a current flowing between the anode electrode 7 and the cathode electrode 9 is controlled, or a surface area of a portion of the aluminum alloy contained in the anode electrode 7 that is in contact with the electrolytic solution 11 is adjusted so that the current density at the portion of the surface of the aluminum alloy of the anode electrode 7 that is in contact with the electrolytic solution 11 falls within a predetermined range. By applying the current to flow through the anode electrode 7 in this way, an electrodeposited film of high-purity aluminum is deposited on the cathode electrode 9.

[0024] In the present description, the current density at a portion of the surface of the aluminum alloy contained in the anode electrode that is in contact with the electrolytic solution will be referred to as the "current density at the anode electrode". Also, in the present description, the current density at the portion of the surface of the aluminum alloy contained in the anode electrode that is in contact with the electrolytic solution is a value obtained by dividing the current between the anode electrode and the DC power supply by an area of the portion of the surface of the aluminum alloy contained in the anode electrode that is in contact with the electrolytic solution.

[0025] The electrolysis tank 3 provided in the aluminum producing device 1 stores the electrolytic solution 11 in which aluminum can be dissolved, and the anode electrode 7 and the cathode electrode 9 are immersed in the electrolytic solution 11. The anode electrode 7 and the cathode electrode 9 are connected with each other via the DC power supply 5. The anode electrode 7 is made of an aluminum alloy with aluminum purity lower than a target purity, and contains 0.1 mass% or more and 24 mass% or less of Si. Furthermore, the aluminum alloy may also contain 0.1 mass% or more and 5 mass% or less of Cu and 0.15 mass% or more and 1.8 mass% or less of Fe. If the content of Si exceeds the above-mentioned upper limit, Si may become a factor that hinders elution of aluminum during the electrolysis, slowing down a rate of deposition of the targeted high-purity aluminum and causing a decrease in throughput. On the other hand, if the contents of Cu and Fe exceed the above-mentioned upper limits, Cu and Fe tend to be eluted into the electrolytic solution 11. This makes it harder to obtain the targeted high-purity aluminum. The anode electrode 7 can be obtained by molding a used material, such as an AC2A alloy, into a shape of an electrode by casting or the like. The anode electrode 7 may contain at least 0.1 mass% or more and 24 mass% or less of Si and may further contain elements, except for magnesium (Mg), that are more noble than aluminum.

[0026] The cathode electrode 9 is made of aluminum, for example, and pure aluminum or an aluminum alloy specified by JIS standard for wrought materials can be applied. That is, the cathode electrode 9 has a lower content of at least Si and Cu than the aluminum alloy contained in the anode electrode 7. By making the cathode electrode 9 from aluminum, the entire cathode electrode on which aluminum is electrodeposited can be used as high-purity aluminum base metal. Thus, it is preferable that the cathode electrode 9 is made of aluminum having the purity that is equal to or higher than the target purity. Other than aluminum, metals such as titanium or stainless steel may be used for the cathode electrode 9. Titanium or stainless steel has a dense oxide film on a surface thereof. Thus, when the cathode electrode made of titanium or stainless steel is used and aluminum is electrodeposited on the surface thereof, the electrodeposited aluminum can be easily peeled off from the cathode electrode 9.

[0027] The DC power supply 5 that applies DC voltage between the anode electrode 7 and the cathode electrode 9 may be any known DC power supply device. However, to reduce an amount of impurities eluted into the electrolytic solution 11, which will be described below, it is preferable that the DC power supply is provided with a current control mechanism that controls the output current. Also, to control the current density at the anode electrode 7, a current detector (not shown) for measuring the current flowing between the anode electrode 7 and the DC power supply 5 may be provided near the anode electrode 7. The current density at the anode electrode can be calculated from the surface area of the anode electrode 7 that is in contact with the electrolytic solution 11 and the electric current detected by the current detector. For this reason, it is preferable that the output current of the DC power supply 5 can be adjusted by the current control mechanism of the DC power supply 5 such that the current density at the anode electrode falls within a predetermined range. Also, an area of the aluminum alloy used for the anode electrode 7 that is in contact with the electrolytic solution 11 may be changed depending on the current flowing through the anode electrode 7. For example, the current density at the anode electrode may be adjusted by changing an area of the anode electrode exposed above a liquid surface of the electrolytic solution 11, thereby

changing the contact area of the anode electrode 7 with the electrolytic solution 11.

[0028] The electrolytic solution 11 in which the anode electrode 7 and the cathode electrode 9 are to be immersed is a non-aqueous system (non-aqueous electrolytic solution) containing, for example, (1) dialkyl sulfone and (2) aluminum halide. By using such the electrolytic solution 11, high-purity aluminum can be formed on the surface of the cathode electrode 9 at a high film-forming rate. Furthermore, it is preferable that the electrolytic solution 11 further includes (3) at least one nitrogen-containing compound selected from a group consisting of ammonium halide, a hydrogen halide salt of primary amine, a hydrogen halide salt of secondary amine, a hydrogen halide salt of tertiary amine, and a quaternary ammonium salt represented by a general formula of $R^1R^2R^3R^4N \cdot X$ where R^1 to R^4 are the same or different alkyl groups and X is a counter anion for a quaternary ammonium cation. The above-mentioned electrolytic solution 11 is industrially advantageous compared to electrolytic solutions using ionic liquids in terms of easy availability of reagents and low cost.

[0029] Examples of the dialkyl sulfone to be contained in the above-mentioned electrolytic solution 11 include those having an alkyl group with one to six carbon atoms (which may be linear or branched), such as dimethyl sulfone, diethyl sulfone, dipropyl sulfone, dihexyl sulfone, and methyl ethyl sulfone. From viewpoints of good electrical conductivity and easy availability, dimethyl sulfone can be preferably used.

[0030] Examples of the aluminum halide include aluminum chloride and aluminum bromide. The aluminum halide is preferably anhydrous. An anhydrous compound does not include water molecules that cause a decrease in electrodeposition efficiency, and thus the electrodeposition efficiency is not to be decreased.

[0031] Examples of the ammonium halide that can be used as the nitrogen-containing compound include ammonium chloride and ammonium bromide. Also, examples of the primary amine to tertiary amine in the hydrogen halide salts of primary to tertiary amine include those in which the alkyl group has one to six carbon atoms (which may be linear or branched), such as methylamine, dimethylamine, trimethylamine, ethylamine, diethylamine, triethylamine, propylamine, dipropylamine, tripropylamine, hexylamine, and methylethylamine. Examples of the hydrogen halide include hydrogen chloride and hydrogen bromide. Examples of the alkyl groups represented by R^1 to R^4 in the quaternary ammonium salt represented by the general formula: $R^1R^2R^3R^4N \cdot X$ where R^1 to R^4 are the same or different alkyl groups and X is a counter anion for a quaternary ammonium cation are those having one to six carbon atoms (which may be linear or branched), such as a methyl group, ethyl group, propyl group, and hexyl group. Examples of X include halide ions such as chloride ions, bromide ions, and iodide ions, as well as BF_4^- and PF_6^- . Specific examples of the compounds include tetramethylammonium chloride, tetramethylammonium bromide, tetramethylammonium iodide, and tetramethylammonium boron tetrafluoride. Preferable examples of the nitrogen-containing compound include tertiary amine hydrochlorides, such as trimethylamine hydrochloride, in viewpoints of facilitating the formation of high-purity aluminum at the high film-forming rate.

[0032] A compounding ratio of the dialkyl sulfone and the aluminum halide is, for example, preferably 1.5 mol or more and 5 mol or less of the aluminum halide per 10 mol of the dialkyl sulfone. Furthermore, an upper limit of the compounding ratio is more preferably 4.0 mol or less, 3.0 mol or less, or 2.5 mol or less in order, and is most preferably 1.5 mol or more and 2.0 mol or less. If a compounding amount of the aluminum halide is less than 1.5 mol per 10 mol of the dialkyl sulfone, the aluminum formed may be darkened (a phenomenon called "burning") or the film-formation efficiency may be deteriorated. On the other hand, if the compounding amount of the aluminum halide exceeds 5.0 mol per 10 mol of the dialkyl sulfone, liquid resistance of the electrolytic solution 11 becomes too high, which may cause the electrolytic solution 11 to generate heat and decompose.

[0033] Also, if a composition ratio of the aluminum halide to the dialkyl sulfone is too high, concentration of ions that serve as aluminum source may become too high, which may hinder transfer of aluminum ions from the anode electrode side. On the other hand, if the composition ratio of the aluminum halide to the dialkyl sulfone is too low, the number of ions contributing to electrical conduction decreases, which may deteriorate the efficiency of progress of the electrolysis.

[0034] On the other hand, the nitrogen-containing compound that is to be compounded together with the dialkyl sulfone and the aluminum halide is preferably 0.01 mol or more and 2.0 mol or less, or more preferably 0.05 mol or more and 1.5 mol or less, per 10 mol of the dialkyl sulfone. If the compounding amount of the nitrogen-containing compound is below 0.01 mol per 10 mol of the dialkyl sulfone, it may be difficult to obtain the effects of compounding thereof, such as an effect of improving the film-forming rate through an improvement in the electrical conductivity of the electrolytic solution 11, and effects of improvement in purity and ductility of aluminum. Also, if the compounding amount of the nitrogen-containing compound exceeds 2.0 mol per 10 mol of the dialkyl sulfone, the composition of the electrolytic solution 11 may be essentially changed, which may hinder aluminum from depositing.

[0035] The electrolysis of aluminum using the above-mentioned electrolytic solution 11 may be carried out under conditions where a temperature of the electrolytic solution 11 is 80°C or higher and 120°C or lower and the applied current density is 0.1 mA/cm² or more and 25 mA/cm² or less. It is more preferable to be carried out under conditions where the applied current density is 0.1 mA/cm² or more and 20 mA/cm² or less. A lower limit of the temperature of the electrolytic solution 11 is to be decided taking into consideration a melting point of the electrolytic solution 11, and is preferably 85°C or higher, or more preferably 95°C or higher (if the temperature falls below the melting point of the electrolytic solution 11, a plating solution solidifies and plating is no longer possible). When the temperature of the electrolytic solution 11 is 120°C or

lower, active reaction generated between aluminum and the electrolytic solution can be suppressed, which makes it harder for a large amount of impurities to be incorporated into aluminum. To efficiently obtain an electrodeposited film of high-purity aluminum by electrolysis, it is necessary to efficiently transfer metal ions, which are to be deposited on the cathode electrode, from the anode electrode to the surface of the cathode electrode. Here viscosity of the electrolytic solution decreases by increasing the temperature and, as a result, transport efficiency of substances increases, thereby improving electrical conductivity. Thus, considering the electrical conductivity, the temperature of the electrolytic solution is to be high. However, if the temperature of the electrolytic solution becomes too high, the surface of the cathode electrode becomes activated, and deposition of impurities is more likely to proceed. This may result in deterioration of purity. For such reasons, the temperature of the electrolytic solution 11 during electrolysis is preferably 85°C or higher, or more preferably 95 °C or higher, and the upper limit thereof is preferably 120°C or lower.

[0036] Also, the anode current density of 0.1 mA/cm² or more can suppress a decrease in the film-forming efficiency. On the other hand, the anode current density of 25 mA/cm² or less can facilitate achievement of the high-purity aluminum, as will be described below. Furthermore preferably, the anode current density of 20 mA/cm² or less can more easily achieve high purity. There is no particular limitation for the electrolytic solution 11 to be used.

[0037] Next, a theory for obtaining high-purity aluminum in the aluminum producing device 1 according to the embodiment of the present invention will be described with reference to FIG. 1. When the anode electrode 7 and the cathode electrode 9 are immersed in the electrolytic solution and a DC current is applied to flow, aluminum loses its electrons on the surface of the anode electrode 7 and is eluted into the electrolytic solution 11 as aluminum ions (A in FIG. 1). On the surface of the cathode electrode 9, electrons are supplied to the aluminum ions and aluminum metal is deposited.

[0038] To efficiently supply the aluminum ions to the cathode electrode 9, it is preferable to stir the electrolytic solution 11 while the current is flowing through the anode electrode 7. Thus, it is preferable that a release outlet for releasing inert gas such as nitrogen gas is provided at a lower portion of the electrolysis tank 3 and bubbles of the inert gas are released from such the release outlet into the electrolytic solution 11 (the cathode electrode 9) (so-called bubbling). Convection also occurs in the electrolytic solution 11 due to flow of the bubbles, thereby stirring the electrolytic solution 11. Alternatively, a pump or a stirring device may be used to provide liquid current, or the electrodes themselves may be swung. Also, although a pair of one each of the anode electrode 7 and the cathode electrode 9 is disposed in the illustrated example, a plurality of the anode electrodes 7 and the cathode electrodes 9 may also be disposed alternately.

[0039] Here, the inventors of the present invention have found that (1) when metal ions are eluted from the anode electrode 7, the eluted metal ions vary depending on the current density at the anode electrode 7. For example, since Cu has a higher standard electrode potential than aluminum, aluminum is ionized preferentially at the anode electrode 7 in an equilibrium state. However, in reality, ionization of Cu also progresses when Cu is dissolved in aluminum, or the current density is equal to or more than a predetermined value and concentration of the aluminum ions on the surface of the anode electrode is increased. In contrast, it has been found that keeping the current density at the anode electrode 7 at 25 mA/cm² or less can suppress elution of elements that are more noble than aluminum, such as Cu. Thus, in the aluminum producing device 1 according to the present embodiment of the present invention, the DC power supply 5 having the current adjustment mechanism is used. The current adjustment mechanism of the DC power supply 5 can change the current flowing from the DC power supply 5 to the anode electrode 7, so that the current through the anode electrode 7 can be adjusted to achieve the above-mentioned current density even when the surface shape of the anode electrode 7 changes and the area of the surface in contact with the electrolytic solution 11 changes.

[0040] The inventors have also found (2) conditions under which, when aluminum ions are eluted from the anode electrode 7, segregated portions of Cu, Si, and the like (crystallized portions of Cu and Si) that have been present in the material constituting the anode electrode 7 come out to the surface of the anode electrode 7 and fall off. It has been found that high-purity aluminum can be efficiently produced if Cu and Si are not eluted into the electrolytic solution 11 and different element components such as Cu and Si that are crystallized on the surface of the anode electrode 7 fall off in a solid phase (B in FIG. 1) as above. That is, by setting the current density at the anode electrode 7 equal to or less than the predetermined value, it has been found that, in a case in which Cu and Si are not completely uniformly dispersed in the material constituting the anode electrode 7 but a Cu phase, Si phase, or an intermetallic compound phase including the same (a phase with a maximum diameter of 10 μm or more, for example) is present, such the phase falls off from the anode electrode 7 and can be removed without being eluted.

[0041] To allow the impurities contained in aluminum of the anode electrode 7 to fall off from the anode electrode 7 efficiently without being eluted into the electrolytic solution 11, it is preferable to apply a DC voltage between the anode electrode 7 and the cathode electrode 9 while, for example, stirring the electrolytic solution 11 around the anode electrode 7. For example, instead of or in addition to the above-mentioned bubbling directed toward the cathode electrode 9 or the like, stirring the electrolytic solution by bubbling in proximity of the anode electrode 7 or by using a stirring device or the like may be performed, or the anode electrode 7 can be swung or rotated. Also, the surface of the anode electrode 7 may be physically rubbed with another insulating member during electrolysis to allow the impurities to fall off.

[0042] Also, to allow the impurities to efficiently fall off from the anode electrode 7, a process for coarsening the impurity

phase may be performed during manufacturing of the anode electrode 7 to facilitate falling off of impurities. This makes it easier for the impurities to fall off. For example, when forming the anode electrode by melting and casting an aluminum material containing Si or Cu, such as an aluminum alloy or aluminum base metal extracted from scrap, it is possible to form the Cu phase, Si phase, or intermetallic compound phase including the same, to have a size of 10 μm or more (or even 20 μm or more) by reducing the cooling rate after solidification.

[0043] To reduce the current density at the anode electrode 7, it is preferable to make the surface area of the anode electrode (the area in contact with the electrolytic solution 11, the same applies hereafter) sufficiently larger (1.5 times or more, for example) than the surface area of the portion of the cathode electrode 9 that is in contact with the electrolytic solution 11. Although there is an upper limit to the current density at the anode electrode 7 as mentioned above, the deposition rate of aluminum deposited on the cathode electrode 9 increase as the current flowing through the anode electrode 7 increases. Thus, it is possible to improve efficiency of electrodeposition of high-purity aluminum by making the surface area of the portion of the aluminum alloy of the anode electrode 7 that comes into contact with the electrolytic solution larger than the surface area of the portion of the cathode electrode 9 that comes into contact with the electrolytic solution 11.

[0044] Also, the anode electrode 7 of the present embodiment may be configured by a mesh cage formed of a metal material of an element that is more noble than aluminum, with an aluminum alloy extracted from scrap and processed into granules housed inside the cage. By using such the anode electrode 7, it is possible to increase the current flowing through the anode electrode 7 while keeping the current density at the anode electrode 7 at or below the above-mentioned upper limit value, thereby increasing the deposition rate of aluminum deposited on the cathode electrode 9.

[0045] Also, in the electrolytic solution 11, particles of the impurities (sludge) falling off from the anode electrode 7 are precipitated below the anode electrode 7. Thus, a step of collecting such the sludge at the time of electrolysis or after the electrolysis step may be included. For example, the sludge can be collected by filtering using filter paper, a filter, an anode bag, or a cathode bag, so that the electrolytic solution 11 can be reused. The filter paper, filter, anode bag, or cathode bag used may have a nominal aperture size of 0.1 μm to 100 μm . A lower limit thereof is preferably 1 μm or more. In a case of the aluminum material in which crystal grains of the Cu phase, Si phase, or intermetallic compound phase including the same are increased, the lower limit is preferably 10 μm or more, or more preferably 20 μm or more.

[0046] As described above, according to the present embodiment, high-purity aluminum can be efficiently obtained, particularly from an aluminum alloy containing large amounts of Si and Cu, with reduced amounts of such impure elements. For example, the total content of Si, Fe, and Cu in the high-purity aluminum that can be obtained from the present embodiment may be 0.1 mass% or less. Also, an amount of CO_2 emitted by the electrolysis of the present embodiment to obtain 1 kg of high-purity aluminum can be reduced by approximately 45% compared to the amount of CO_2 emitted when obtaining 1 kg of virgin metal from ore using conventional smelting method.

[0047] Generally, a three-layer electrolysis method is known for purification of aluminum to a high degree. However, it is difficult to increase purity of used materials of casting alloys by the three-layer electrolysis method since casting alloys in particular have high impurity concentration and the anode composition cannot be maintained. Thus, it is difficult to apply the three-layer electrolysis method. Furthermore, Si, which is a typical alloy element, has a density similar to that of aluminum and is lighter than a molten salt used. Thus, Si may be mixed into refined aluminum and thus separation of Si is difficult.

[0048] In contrast, the method for producing aluminum according to the present embodiment can efficiently remove the impurity elements including Si, and thus the obtained aluminum can be further purified to a higher degree through the three-layer electrolysis method. That is, aluminum obtained by the present embodiment is effectively used as an aluminum raw material (an anode-side raw material) in the three-layer electrolysis method.

[0049] As above, according to the method for producing aluminum according to the present embodiment, aluminum having higher purity can be obtained from an aluminum alloy or aluminum base metal containing 0.1 mass% or more and 24 mass% or less of Si. Thus, recycled casting alloys, for example, can be highly purified to be converted for other purposes. Also, it is easier to convert recycled alloys, such as automobile engine parts including, in particular, a large amount of Cu, into products for other purposes, thereby making it possible to utilize used alloys more effectively. Also, it is possible to produce high-purity aluminum from aluminum base metal.

[0050] Also, by stirring the liquid near the anode electrode 7, sludge can efficiently fall off from the anode. Also, by making the surface area of the anode electrode 7 that comes into contact with the electrolytic solution larger than the surface area of the cathode electrode, it is possible to suppress the current density at the anode while efficiently depositing high-purity aluminum onto the cathode.

[0051] Also, by using the electrolytic solution including dialkyl sulfone, aluminum halide, and nitrogen-containing compound, it is possible to carry out the operation at a lower temperature than when a molten salt or the like is used, and handling is safe and easy.

[0052] Also, by collecting the sludge fallen off from the anode electrode 7, contamination and the like can be suppressed, and the aluminum alloy can be highly purified efficiently.

[0053] At this time, by collecting the sludge with filtering paper or the like with appropriate aperture size, a sludge

collection rate can be increased.

(EXAMPLES)

[0054] Using a device having the similar configuration to the aluminum producing device shown in FIG. 1, electrolysis of an aluminum alloy used as an anode electrode and deposition of high-purity aluminum onto a cathode electrode were carried out. As the anode electrode, an AC2A aluminum alloy (Si: 4.0 mass% or more and 6.0 mass% or less, Cu: 3.0 mass% or more and 4.5 mass% or less, Fe: 0.8 mass% or less) was used. FIG. 2A to FIG. 2D are SEM images of a cross section in proximity of a surface of the anode electrode, showing the difference in the anode electrode surface after electrolysis when the current density at the anode electrode is changed. In each of the SEM images, the current density at the anode electrode is 10 mA/cm² in FIG. 2A, 20 mA/cm² in FIG. 2B, 40 mA/cm² in FIG. 2C, and 80 mA/cm² in FIG. 2D. A cumulative amount of current per unit volume of an electrolytic solution is in a range of 9.2 to 10.6 Ah/L.

[0055] As mentioned above, by applying an electric current through the anode electrode, aluminum of a parent material of the AC2A aluminum alloy is eluted. Here, segregations (crystallized products) of Cu, Si, Fe, and the like or intermetallic compounds (hereinafter, simply referred to as impurities) are partially present in each of structures of the AC2A aluminum alloy of the anode electrode. Such impurities remain on the surface when aluminum is eluted when the current density at the anode electrode is 10 mA/cm² (FIG. 2A) or 20 mA/cm² (FIG. 2B). Thus, when elution of aluminum progresses, such impurities eventually fall off from the surface of the anode electrode to be sludge.

[0056] On the other hand, when the current density at the anode electrode is 40 mA/cm² (FIG. 2C) or 80 mA/cm² (FIG. 2D), the aluminum surface and the impurity surface almost coincide with each other. This indicates that the impurities are partially eluted together with aluminum. As above, when the current density increases, Cu, which has a higher standard electrode potential than aluminum, for example, ionizes together with aluminum.

[0057] FIG. 3 shows results of measuring surface roughness of the anode electrode surface after electrolysis. When the current density at the anode electrode is 25 mA/cm² or less, i.e. in the cases in which the current density is 10 mA/cm² and 20 mA/cm², both an arithmetic mean height Sa and the maximum height Sz are relatively high, in inverse proportion to the current density at the anode electrode. On the other hand, when the current density at the anode electrode is more than 25 mA/cm² i.e., when the current density is 40 mA/cm² or 80 mA/cm², the surface roughness has a smaller value. This is because impurities contained in the aluminum alloy used as the anode electrode remained on the anode electrode surface at the time of high purification of aluminum when the current density was 25 mA/cm² or less. By setting the current density at 25 mA/cm² or less in this way, the surface roughness (the arithmetic mean height Sa and the maximum height Sz) of the anode electrode surface can be controlled, thereby suppressing elution of the impurities.

[0058] It is preferable that aluminum is deposited on the cathode electrode such that the arithmetic mean height Sa is 2.3 μm or more and 10 μm or less during electrolysis (any time from the start of electrolysis until the scheduled end time of electrolysis at which a predetermined time has passed) or at the scheduled end time of electrolysis. For example, while a current is flowing through the anode electrode, elements contained in the aluminum alloy is eluted from the anode electrode into the electrolytic solution, and thus, during such the time, it is preferable to deposit aluminum onto the cathode electrode while controlling the current through the anode electrode or adjusting the area of the anode electrode exposed above the liquid surface such that the arithmetic mean height Sa is 2.3 μm or more and 10 μm or less.

[0059] When the arithmetic mean height Sa of a portion of the aluminum alloy of the anode electrode surface that is in contact with the electrolytic solution is 2.3 μm or more, elution of Cu or the like is reduced. When the arithmetic mean height Sa is 10 μm or less, the current is concentrated onto tip portions of the small roughness appearing on the surface of the anode electrode, and this suppresses the rise of the anode current density, thereby reducing the elution of Cu or the like. The arithmetic mean height Sa and the maximum height Sz were measured using a laser microscope at five locations of 100 μm by 100 μm square area with n = 5 each, and average values thereof were calculated. The laser microscope used was a pinhole confocal optical system (VK-X100 manufactured by Keyence corporation was used) with a wavelength of 658 nm, a 50x objective lens, a display vertical resolution of 5 nm, and a display horizontal resolution of 10 nm.

[0060] To perform electrolysis while maintaining the surface roughness within a predetermined range, an electrolysis test may be carried out in advance under various conditions using an anode electrode produced with the same composition as the anode electrode that is to be actually used (the aluminum alloy or aluminum base metal obtained from scrap, for example), and the electrolysis conditions can be set using information, which is obtained from the electrolysis test, on correlation between each condition and the surface roughness. For example, by applying a DC voltage between an anode electrode and a cathode electrode in the same electrolytic solution while varying the current flowing through the anode electrode to perform electrolysis and measuring the roughness over elapsed time, information on correlation between the elapsed time and change in the surface roughness for each current can be obtained. Also, by varying the current and the surface area of the anode electrode that is in contact with the electrolytic solution, the information on correlation between the current density and the surface roughness can be obtained. For example, as mentioned above, after a predetermined time has elapsed since the start of electrolysis, coarse impurities remain on the surface of aluminum, increasing the surface roughness (Sa and Sz), and then falling off and exposure of the coarse impurities are repeated thereafter. Thus, it

is preferable to maintain such the state while performing the electrolysis.

[0061] Such the electrolysis conditions are set based on the above-mentioned correlation information. Also, if it is determined by the correlation information that the surface roughness at any time from the start of electrolysis until the end of electrolysis after the predetermined time has elapsed or at the scheduled end time of electrolysis would exceed the above-mentioned predetermined range, the current density may be reduced before the surface roughness exceeds the range so as to suppress further increase in the surface roughness. That is, the current may be reduced based on the correlation information obtained in advance after a predetermined period of time since the start of electrolysis. Note that when setting electrolysis information by obtaining the correlation information based on the surface roughness after the predetermined period of time since the start of electrolysis, the timing for changing the current value and an amount of reduction of the current value can be set by measuring in advance the correlation information between the surface roughness and the current density when the current density is changed after the predetermined time has elapsed since the start of electrolysis. Also, conversely, as the electrolysis progresses, the surface roughness increases and thus the surface area of the anode electrode increases substantially. For this reason, the current may be increased so as to be within the range of set current density, taking into account the increase in the surface area of the anode electrode. That is, based on the above-mentioned correlation information, the current may be increased after the predetermined time has elapsed since the start of electrolysis.

[0062] Next, electrodeposited films deposited on the surface of the cathode electrode were observed. FIG. 4A is an SEM image of a surface of an electrodeposited film and FIG. 4B is an SEM image of a cross section of the electrodeposited film, which were the results of cases where high-purity aluminum (99.99%) was used as the anode electrode and the anode current density and the cathode current density were set to 80 mA/cm². If the high-purity aluminum is used as the anode electrode, there is almost no contamination with impurities and a high-purity electrodeposited film can be obtained even at a high current density.

[0063] On the other hand, FIG. 5A and FIG. 5B are SEM images of a surface and cross section of an electrodeposited film, respectively, when an AC2A alloy is used as the anode electrode and the anode current density and the cathode current density were varied 10 mA/cm² to 80 mA/cm². When the AC2A alloy was used as the anode electrode and the current density exceeded 40 mA/cm², particles appeared to be Cu were observed on a part of the cross section. This indicates that Cu ions were reduced on the surface of the cathode electrode.

[0064] In contrast, FIG. 6A is an SEM image of a surface of an electrodeposited film and FIG. 6B is an SEM image of a cross section of the electrodeposited film, which were the results of cases where the AC2A alloy was used as the anode electrode and the anode current density and the cathode current density were kept constant at 10 mA/cm². When the AC2A alloy was used as the anode electrode and the current density was 25 mA/cm² or less, no Cu was observed on the cross section and it was possible to obtain the electrodeposited film of approximately the same form as in the cases in which the high-purity aluminum was used as the anode electrode (FIG. 4A and FIG. 4B).

[0065] FIG. 7 shows the results of component analysis of the electrodeposited films, showing the difference between the case where the current was varied 10 mA/cm² to 80 mA/cm² and the case where the current was kept constant at 10 mA/cm². By setting the current density at the anode electrode to a predetermined value or less, it was possible to reduce concentration of impurities of the obtained electrodeposited film.

[0066] Also, the concentration of impurities of the electrodeposited films were evaluated in more detail by changing the electrolysis conditions. Table 1 shows the various conditions and evaluation results for each working example.

[Table 1]

No.	Electrolytic Solution			Current Density (mA/cm ²)	Electrodeposited Film Impurities (ppm)				Evaluation			
	Composition Ratio	Temperature (°C)	Electrical Conductivity (mS/m)		Si	Cu	Fe	Total	Si	Cu	Fe	Total Impurities
1	10:3.8	95	443	10	23	193	81	816	A	A	A	A
2	10:3.8	95	443	25	11.5	386	98	855	B	A	A	A
3	10:3.8	120	700	25	65	1201	193	1119	B	B	B	B
4	10:2	120	805	25	0	363	126	859	A	A	B	A
5	10:2	95	720	25	9	408	8	786	A	A	A	A
6	10:3.8	95	443	10-80	71	3169	711	4470	B	C	C	C

[0067] The electrolytic solutions used for the electrolysis in No. 1 to No. 6 all contained dialkyl sulfone, aluminum chloride, and additives of ammonium chloride and tetramethylammonium chloride. The electrolytic solutions used for the electrolysis in the examples No. 1 to No. 3 and No. 6 contained 0.2 mol of aluminum chloride as well as 1.0 mol of tetramethylammonium chloride per 10 mol of dialkyl sulfone. On the other hand, the electrolytic solutions used in the examples No. 4 and No. 5 contained 0.1 mol of aluminum chloride as well as 0.5 mol of tetramethylammonium chloride per 10 mol of dialkyl sulfone.

[0068] In the table, "Composition Ratio" of the electrolytic solution is a molar ratio of dialkyl sulfone to aluminum halide (aluminum chloride). The electrical conductivity varies depending mainly on the composition ratio of dialkyl sulfone to aluminum halide and the temperature.

[0069] The current density at the anode electrode for each of examples No. 1 to No. 6 is as shown in the table, where the current density in No. 6 was varied within a range of 10 mA/cm² to 80 mA/cm².

[0070] The concentration of impurities in the electrodeposited films was evaluated as below.

[0071] For Si, the concentration of 50 ppm or less was rated A (excellent), the concentration of more than 50 ppm and 150 ppm or less was rated B (good), and the concentration of more than 150 ppm was rated C (bad). Also, for Cu, the concentration of 1000 ppm or less was rated A (excellent), the concentration of more than 1000 ppm and 1500 ppm or less was rated B (good), and the concentration of more than 1500 ppm was rated C (bad). Also, for Fe, the concentration of 100 ppm or less was rated A (excellent), the concentration of more than 100 ppm and 200 ppm or less was rated B (good), and the concentration of more than 200 ppm was rated C (bad). Also, for the total impurity concentration of Si+Cu+Fe, the concentration of 1000 ppm or less was rated A (excellent), the concentration of more than 1000 ppm and 2000 ppm or less was rated B (good), and the concentration of more than 2000 ppm was rated C (bad). In all cases, impurity contents are expressed as mass ratios.

[0072] From the results, when considering Si only, all tests were passed (achieving evaluation B or higher). On the other hand, in No. 6 where the current density reached 80 mA/cm² exceeding 25 mA/cm², although the Si content was 150 ppm or less and evaluated as good, the contents of Cu and Fe exceeded 1500 ppm and 200 ppm, respectively. Thus, Cu and Fe were evaluated as fail (evaluation C), and, as a result, the impurity concentration as a whole failed. That is, it is found that the current density has a greater effect on Cu and Fe than on Si.

[0073] In addition, it is also found that, for the same composition ratio, the electrical conductivity tends to increase as the temperature rises.

[0074] Considering the results of Table 1 based on the above, from a comparison between No. 1, No. 2 and No. 6, the impurity concentration can be reduced by decreasing the current density. Also, from a comparison between No. 2 and No. 3 and a comparison between No. 4 and No. 5, it is found that the impurity concentration tends to increase as the temperature rises. Also, from a comparison between No. 2 and No. 5 and a comparison between No. 3 and No. 4, it is found that the impurity concentration tends to increase as the composition ratio of the aluminum halide becomes too high.

[0075] It is preferable that the temperature of the electrolytic solution is 80°C or more and 120°C or less, more preferably 85°C or more and 110°C or less, or furthermore preferably 95°C or more and 110°C or less.

[0076] Although the embodiments of the present invention have been described referring to the attached drawings, the technical scope of the present invention is not limited to the embodiments described above. It is obvious that persons skilled in the art can think out various examples of changes or modifications within the scope of the technical idea disclosed in the claims, and it will be understood that they naturally belong to the technical scope of the present invention.

(DESCRIPTION OF NOTATIONS)

[0077]

- 1 aluminum producing device
- 3 electrolysis tank
- 5 DC power supply
- 7 anode electrode
- 9 cathode electrode
- 11 electrolytic solution

Claims

1. A method for producing aluminum from an aluminum raw material, in which aluminum with higher purity than the aluminum raw material can be obtained, the method comprising:

immersing an anode electrode, which includes the aluminum raw material containing 0.1 mass% or more and 24 mass% or less of Si, and a cathode electrode in an electrolytic solution; and

applying a current to flow at a current density of 0.1 mA/cm² or more and 25 mA/cm² or less on a surface of the anode electrode where the aluminum raw material comes into contact with the electrolytic solution, thereby depositing aluminum on the cathode electrode.

2. The method for producing aluminum according to claim 1, wherein the aluminum raw material of the anode electrode further contains 0.1 mass% or more and 5 mass% or less of Cu.
3. The method for producing aluminum according to claim 1, wherein the aluminum raw material of the anode electrode further contains 0.15 mass% or more and 1.8 mass% or less of Fe.
4. The method for producing aluminum according to claim 1, wherein the current is applied to the anode electrode while stirring the electrolytic solution around the anode electrode.
5. The method for producing aluminum according to claim 1, wherein a surface area of a portion of the anode electrode where the aluminum material is in contact with the electrolytic solution is made larger than a surface area of a portion of the cathode electrode that is in contact with the electrolytic solution.
6. The method for producing aluminum according to claim 1, wherein the electrolytic solution includes dialkyl sulfone and aluminum halide.
7. The method for producing aluminum according to claim 6, wherein a molar ratio of the dialkyl sulfone to the aluminum halide in the electrolytic solution is 1.5 mol or more and 5 mol or less of the aluminum halide per 10 mol of the dialkyl sulfone.
8. The method for producing aluminum according to claim 7, wherein the electrolytic solution further includes at least one nitrogen-containing compound selected from a group consisting of ammonium halide, a hydrogen halide salt of primary amine, a hydrogen halide salt of secondary amine, a hydrogen halide salt of tertiary amine, and a quaternary ammonium salt represented by a general formula of R¹R²R³R⁴N⁺X⁻ where R¹ to R⁴ are the same or different alkyl groups and X is a counter anion for a quaternary ammonium cation.
9. The method for producing aluminum according to claim 1, wherein while the electric current is flowing through the anode electrode, the aluminum is deposited on the cathode electrode such that an arithmetic mean height Sa of the anode electrode is 2.3 μm or more and 10 μm or less.
10. The method for producing aluminum according to any one of claims 1 to 9, wherein the current density is 0.1 mA/cm² or more and 20 mA/cm² or less.

FIG. 1

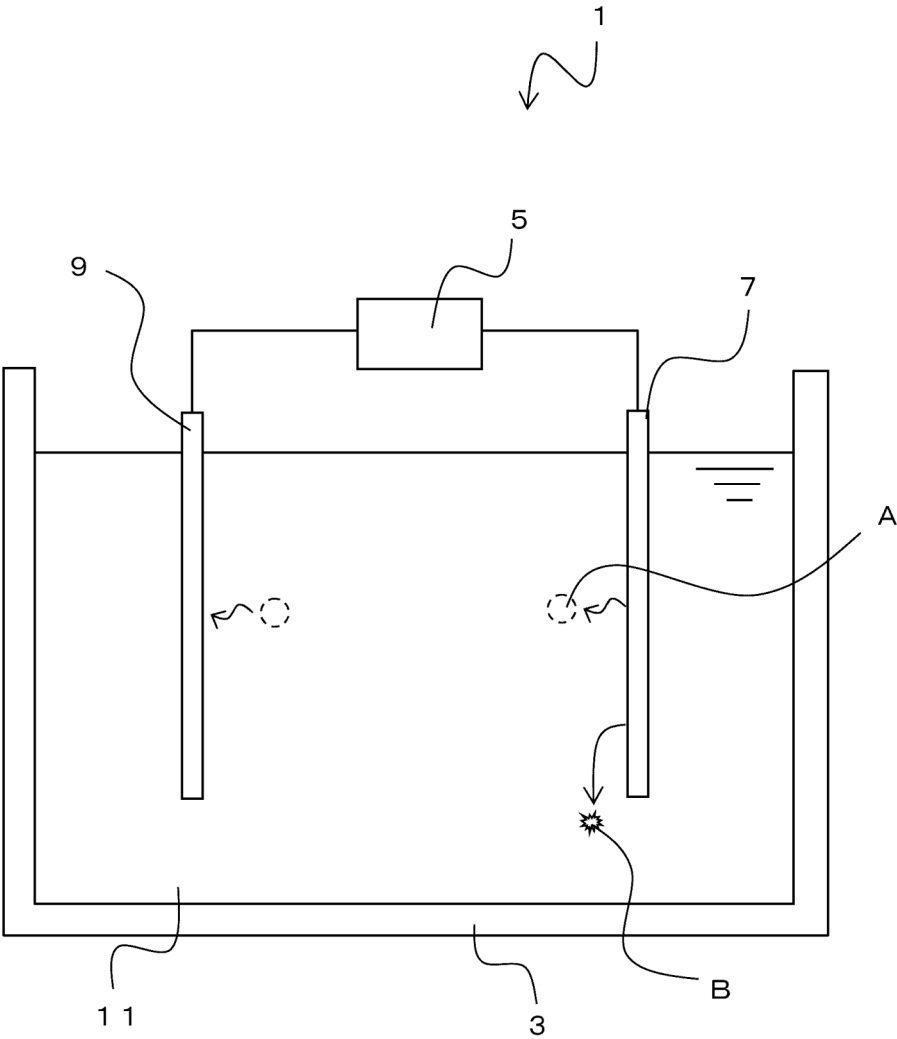


FIG. 2A

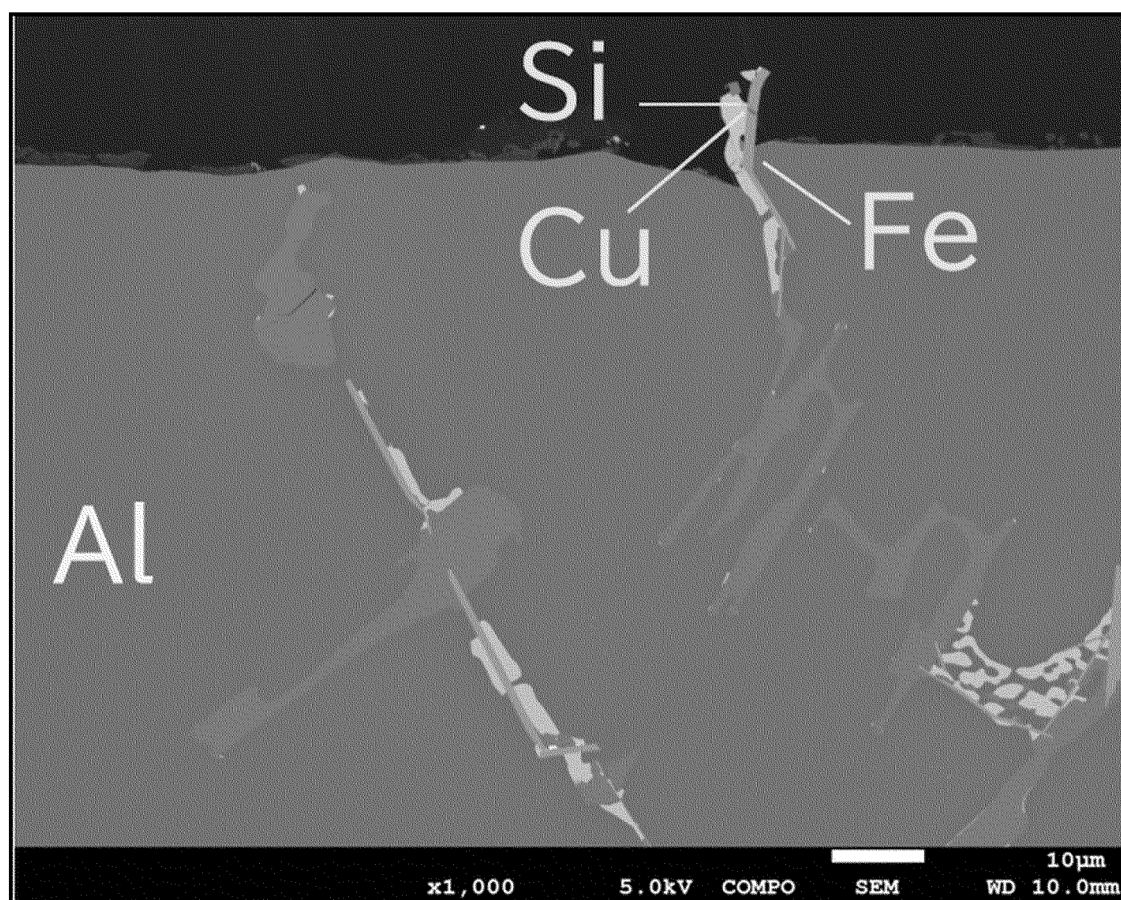


FIG. 2B

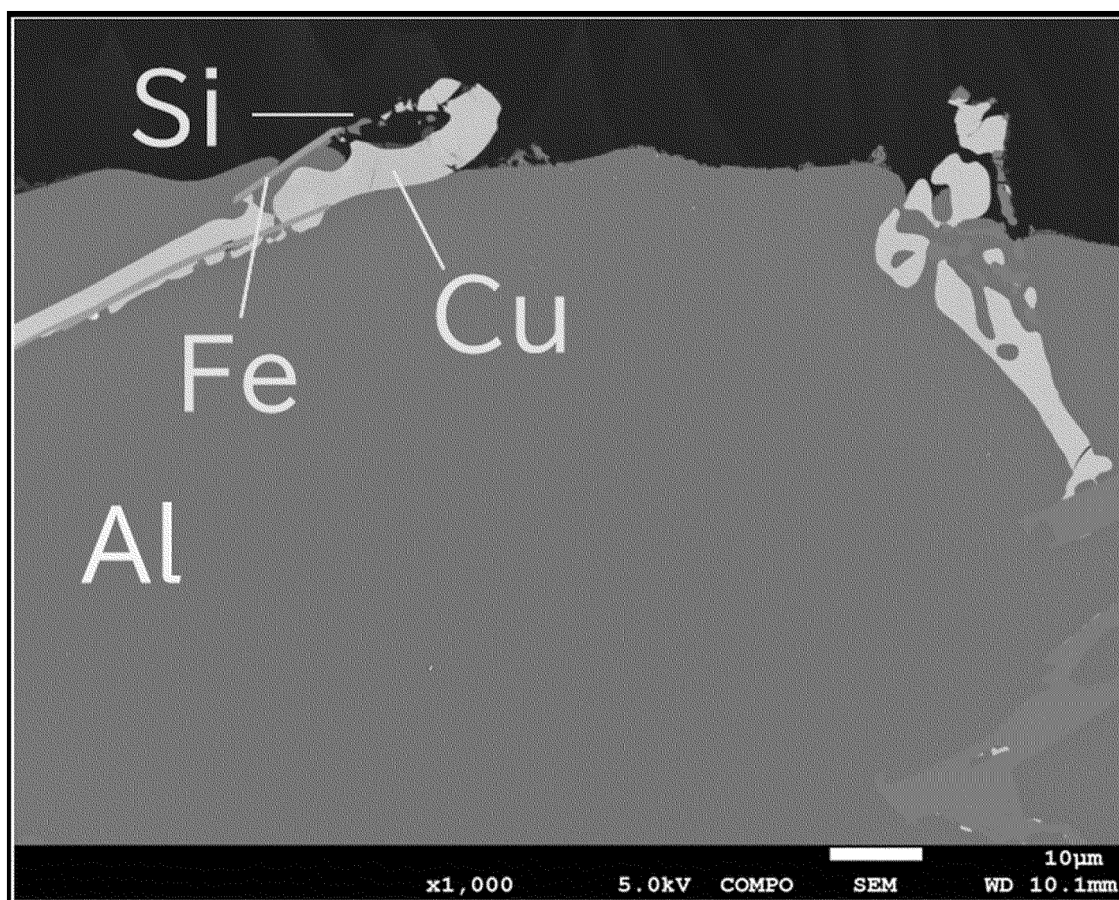


FIG. 2C

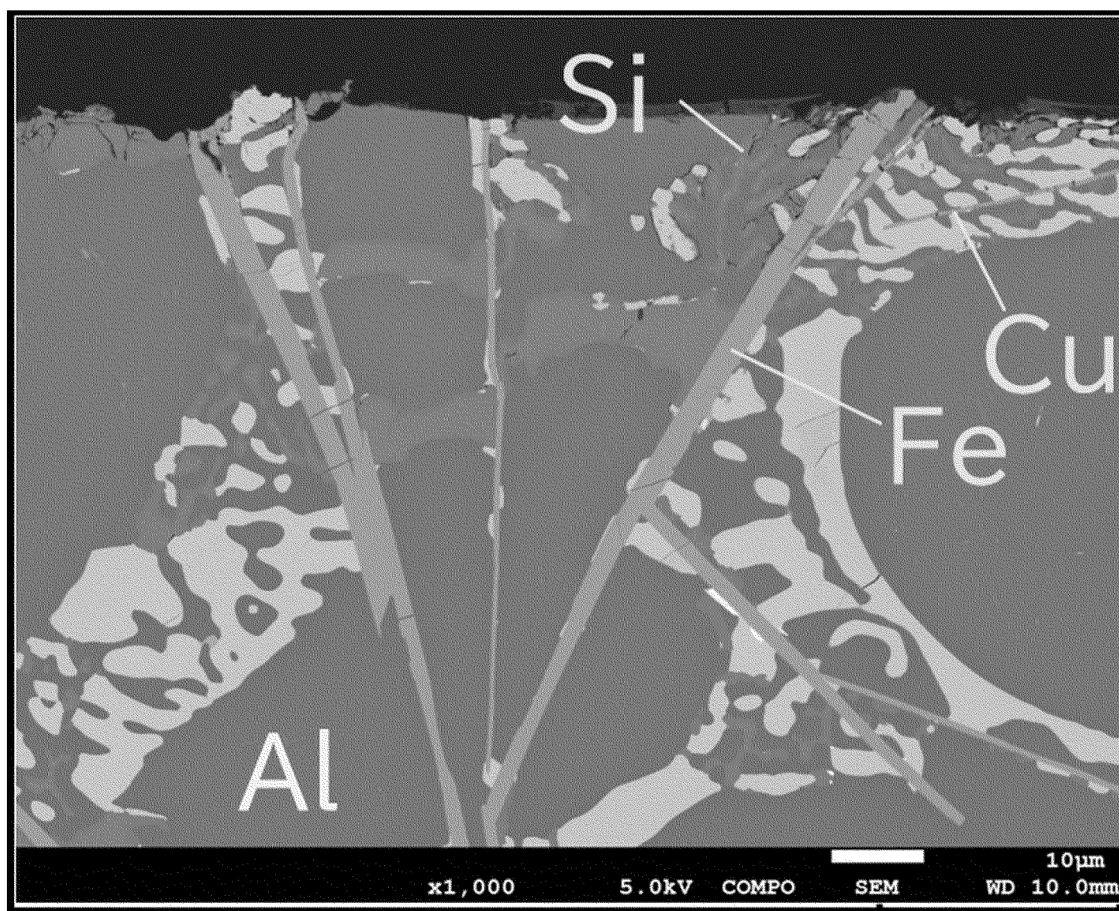


FIG. 2D

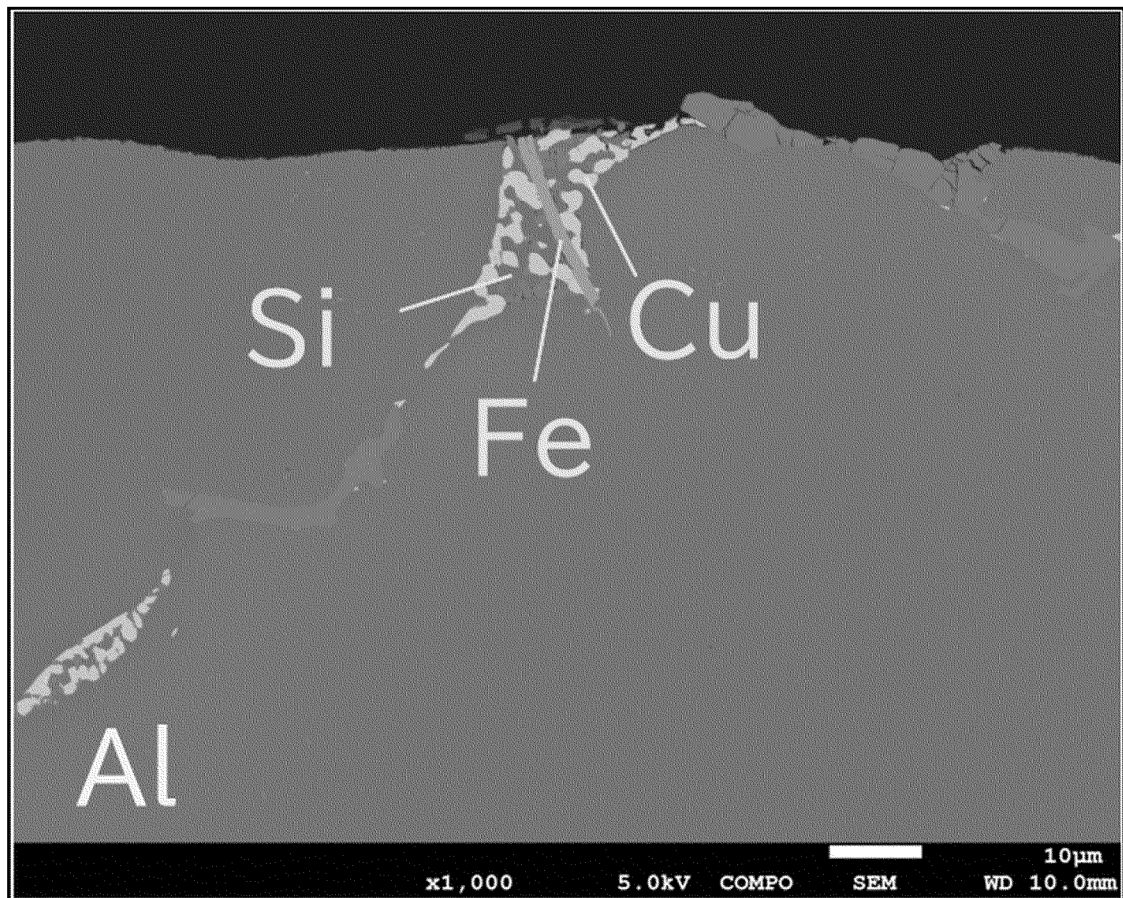


FIG. 3

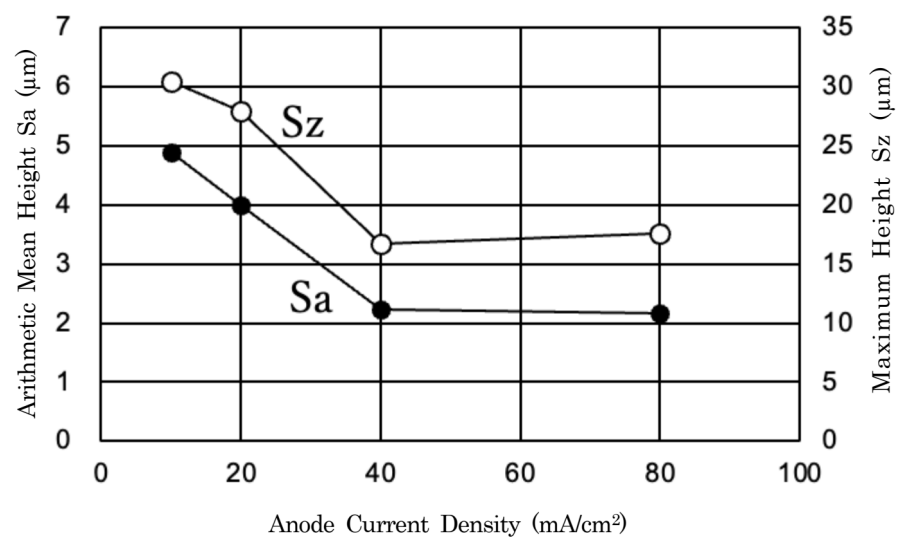


FIG. 4A

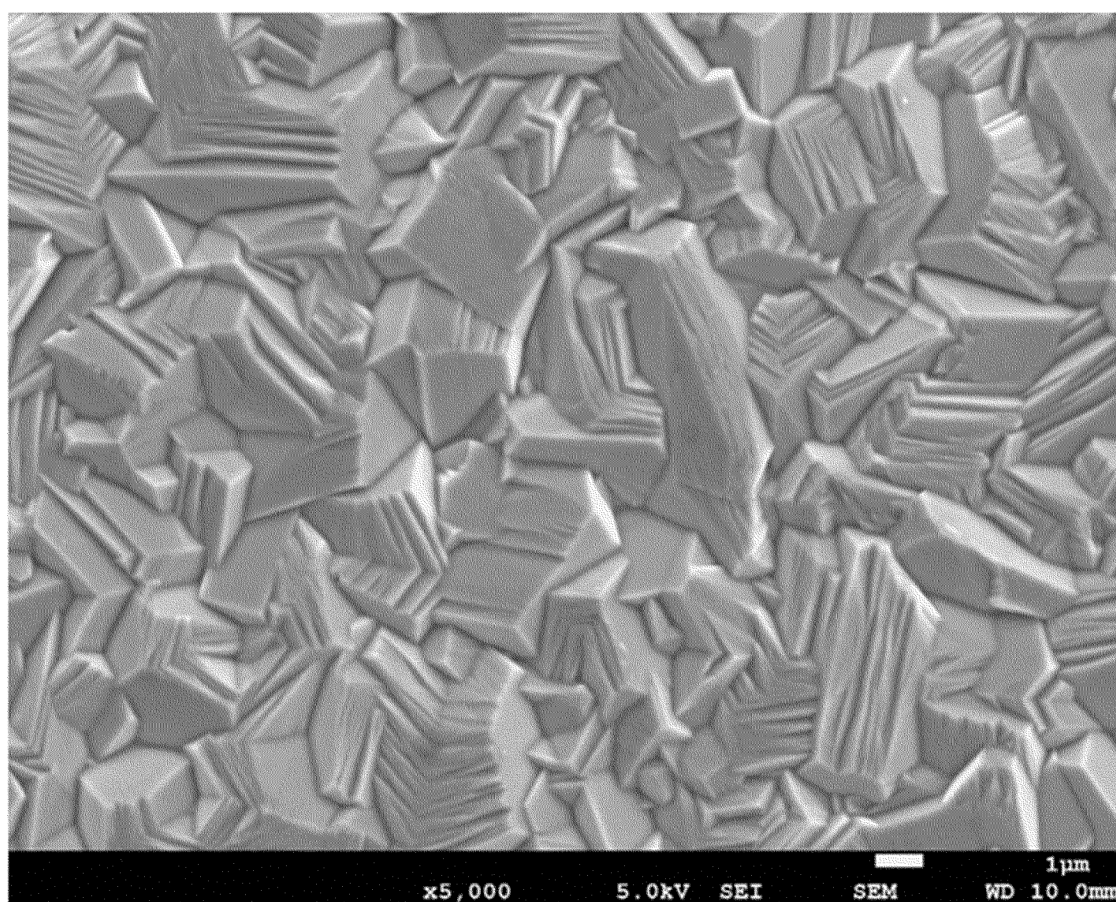


FIG. 4B

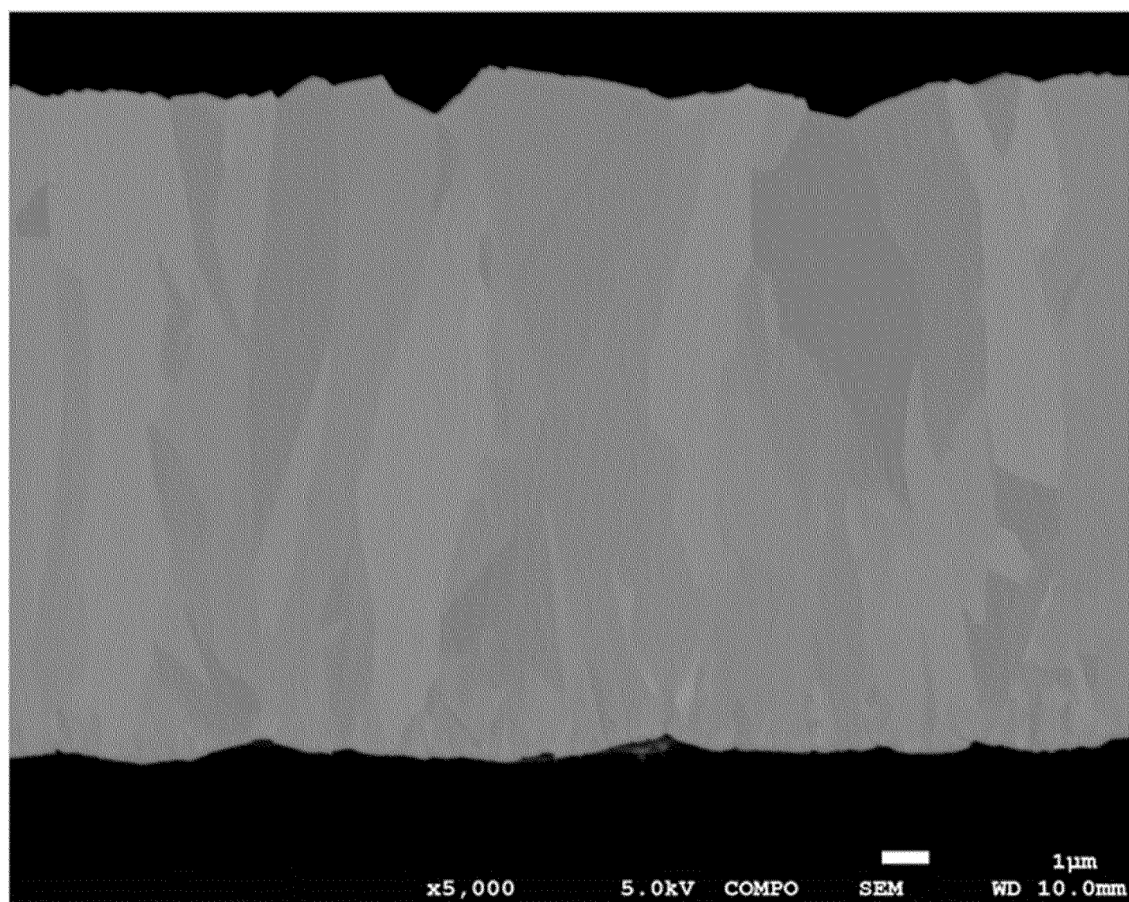


FIG. 5A

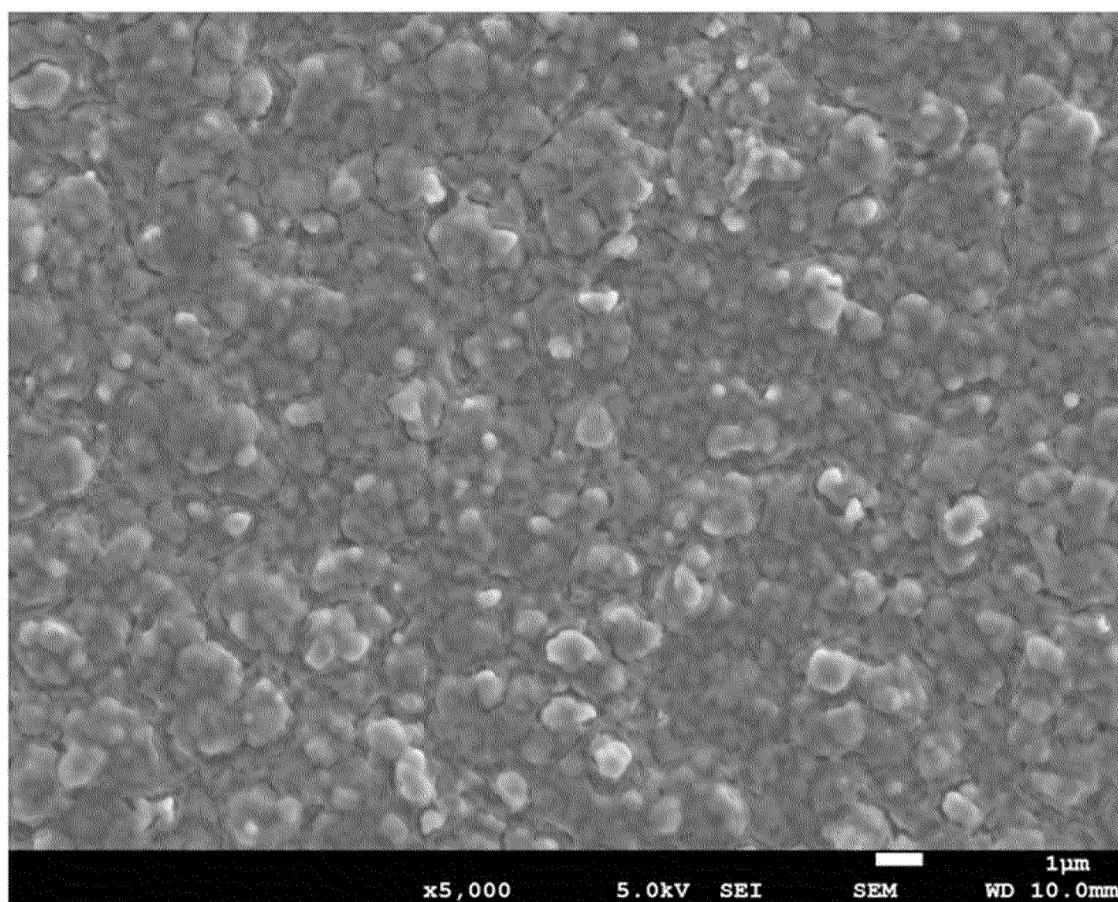


FIG. 5B

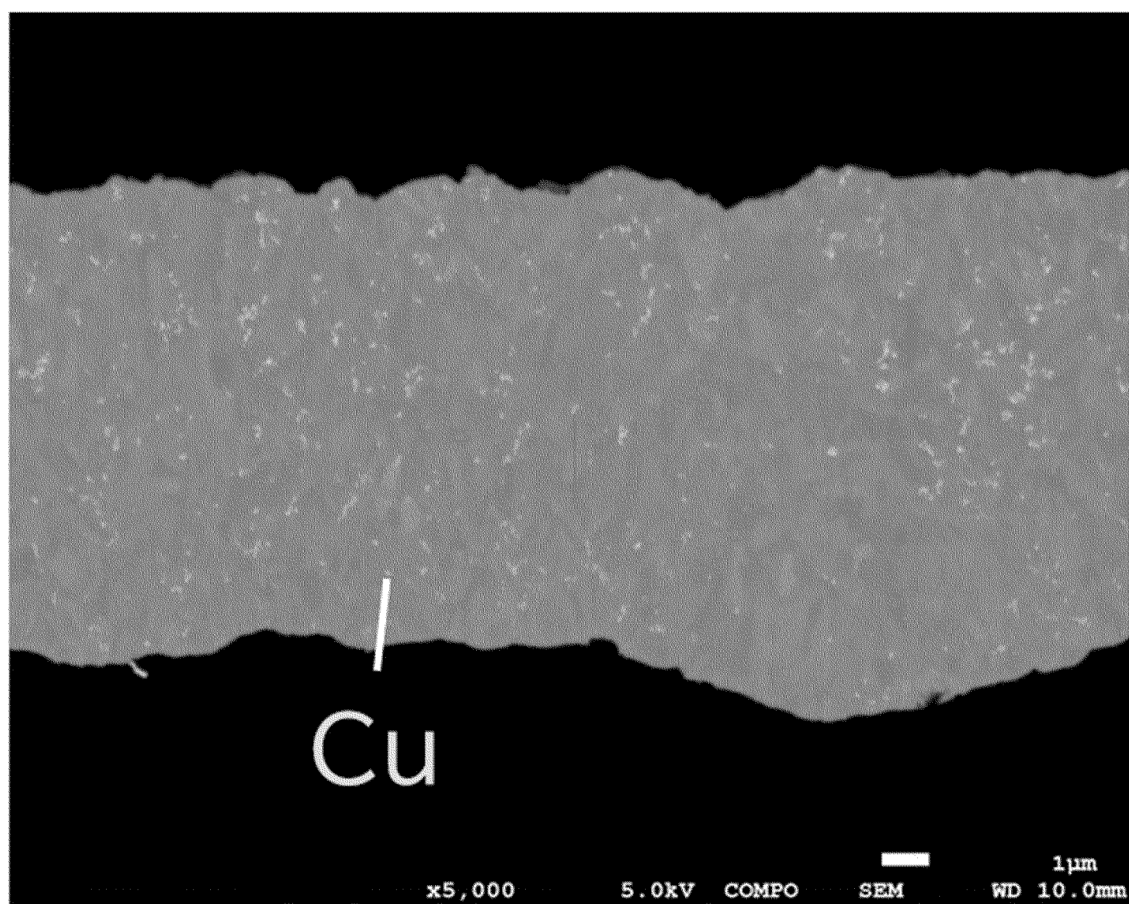


FIG. 6A

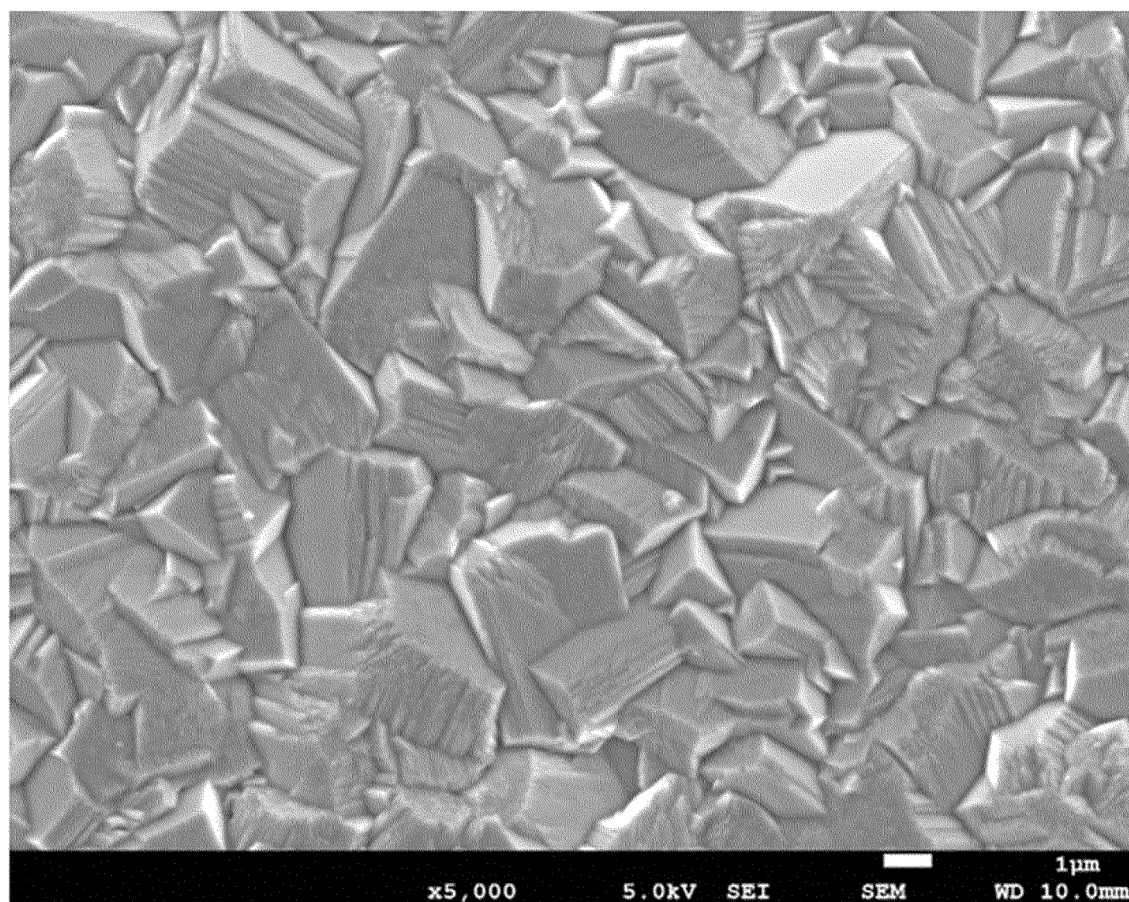


FIG. 6B

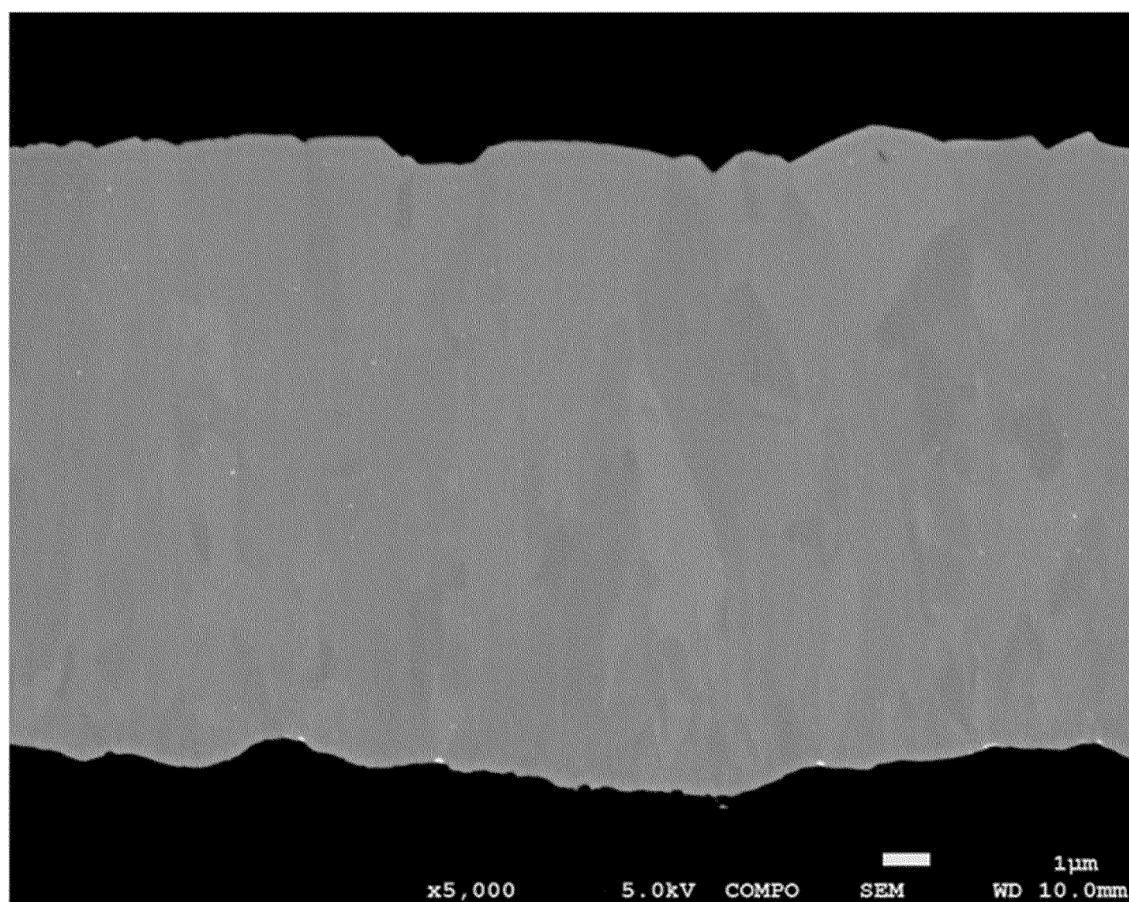
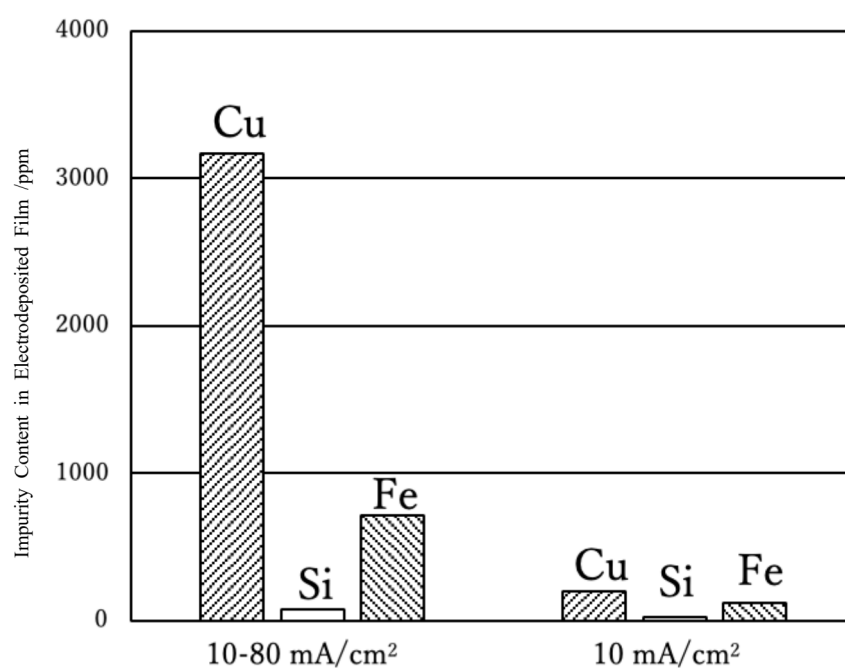


FIG. 7



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2024/044583

A. CLASSIFICATION OF SUBJECT MATTER

C25C 3/24(2006.01)i; **C25C 1/02**(2006.01)i; **C25C 7/02**(2006.01)i
FI: C25C3/24; C25C7/02 306; C25C7/02 305; C25C1/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C25C3/24; C25C1/02; C25C7/02

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan 1922-1996
Published unexamined utility model applications of Japan 1971-2025
Registered utility model specifications of Japan 1996-2025
Published registered utility model applications of Japan 1994-2025

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	LU, Xin et al., 溶融塩電解によるアルミニウムスクラップのアップサイクル, までりあ, 01 April 2023, vol. 62, no. 4, pp. 221-228, doi.org/10.2320/materia.62.221, jim.or.jp/journal/m/pdf3/62/04/221.pdf, (Upcycling of Aluminum Scrap by Molten Salt Electrolysis, Materia Japan) entire text, all drawings	1-10
A	WO 2020/196013 A1 (UACJ CORP.) 01 October 2020 (2020-10-01) claims, paragraph [0032]	1-10
A	WO 2023/210748 A1 (TOHOKU UNIVERSITY) 02 November 2023 (2023-11-02) entire text, all drawings	1-10
A	JP 64-17886 A (KOURA, Nobuyuki) 20 January 1989 (1989-01-20) entire text	1-10
A	JP 55-113891 A (ALUMINUM CO OF AMERICA) 02 September 1980 (1980-09-02) entire text	1-10

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

* Special categories of cited documents:

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“Y” document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

“&” document member of the same patent family

Date of the actual completion of the international search

05 March 2025

Date of mailing of the international search report

18 March 2025

Name and mailing address of the ISA/JP

Japan Patent Office (ISA/JP)
3-4-3 Kasumigaseki, Chiyoda-ku, Tokyo 100-8915
Japan

Authorized officer

Telephone No.

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/JP2024/044583

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Patent document cited in search report			Publication date (day/month/year)	Patent family member(s)			Publication date (day/month/year)
WO	2020/196013	A1	01 October 2020	US	2022/0002892	A1	
				claims, paragraph [0071]			
				CN	113557313	A	
				JP	7503048	B2	
WO	2023/210748	A1	02 November 2023	(Family: none)			
JP	64-17886	A	20 January 1989	(Family: none)			
JP	55-113891	A	02 September 1980	US	4214955	A	
				GB	2039300	A	
				DE	3000210	A	
				FR	2445863	A	
				NO	794321	L	
				AU	5349679	A	
				SE	7910698	A	
				CA	1151098	A	
				IT	8047506	A	
				SE	7910698	L	

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

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