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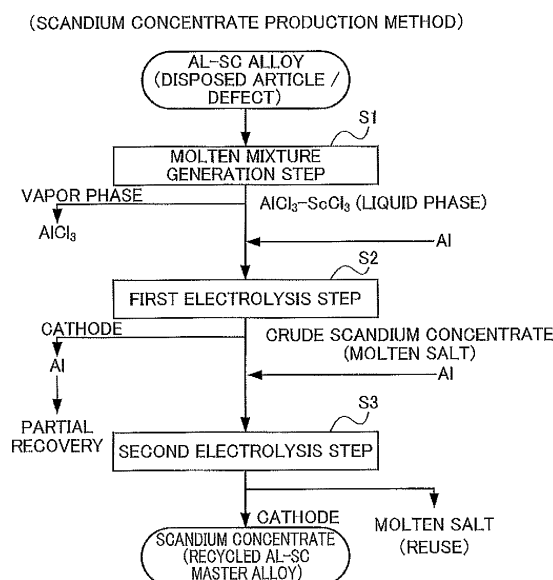
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(54) **SCANDIUM CONCENTRATE PRODUCTION METHOD**

(57) The present invention effectively recovers a scandium concentrate from an Al-Sc alloy. This scandium concentrate production method includes: a molten mixture production step (S1) in which an alloy that contains aluminum and scandium is brought into contact with chlorine and melted in order to produce a molten mixture of aluminum chloride and scandium chloride; a first electrolysis step (S2) in which the molten mixture is subjected to first electrolysis at a potential that is between the potential that causes aluminum to form a metal and the potential that causes scandium to form a metal and molten aluminum is produced; and a second electrolysis step (S3) in which, after the step in which molten aluminum is produced, the molten mixture is subjected to second electrolysis at a potential that makes it possible to recover scandium and a scandium concentrate is produced.

FIG. 1



Description

TECHNICAL FIELD

[0001] The present invention relates to a scandium concentrate production method, and in more detail, relates to a method of reusing an alloy containing scandium and aluminum as aluminum and scandium concentrates.

BACKGROUND ART

[0002] Aluminum scandium alloys containing aluminum and scandium (hereinafter also referred to as "Al-Sc alloy") have a characteristic of being light weight and high strength, and in addition to sports articles, have been used in fields requiring shock resistance. Additionally, in the future, applications as a structural material for aircraft, electric vehicles, high-speed rail, etc. are also expected. However, since the production volume of scandium is very small, scandium is extremely high cost. For this reason, it is not easy to broadly apply scandium industrially.

[0003] In recent years, the technology for recovering scandium that accompanies nickel oxide ore in a very small amount has progressed, and it is becoming possible to stably obtain larger amounts of scandium than before. However, to recover scandium from nickel oxide ore, since multiple processes such as ion exchange, solvent extraction, neutral precipitation and calcination are required, the matter of scandium being high cost does not change even if using this technology.

[0004] However, since scandium is easily oxidized but has a high melting point, it is not possible to obtain Al-Sc alloy by simply melting scandium and aluminum. Therefore, generally a technique has been adopted to add, to molten aluminum, scandium oxide while reducing with metals such as calcium to obtain a master alloy having a scandium quality on the order of 1-2%, and then diluting this with aluminum to obtain the intended Al-Sc alloy. In addition, it has also been proposed to produce a scandium master alloy with halogenated scandium as the raw material (refer to Patent Document 1).

[0005] Patent Document 1: Japanese Unexamined Patent Application, Publication No. 2003-171724

DISCLOSURE OF THE INVENTION

Problems to be Solved by the Invention

[0006] By performing processing in the reverse direction to the processing described in Patent Document 1, it has been considered to separate scandium from Al-Sc alloy. However, in addition to the stability of halogenated scandium, when considering the risks from using hazardous chlorine, and further the industrial facilities and cost, it is not easy to practically realize scandium recovery technology by performing processing in the reverse direction to the processing described in Patent Document 1.

[0007] On the other hand, since the expectations for scandium are rising, in the future, the production volume of Al-Sc alloy will increase, and eventually, the disposal of structures made using Al-Sc alloy, and defective articles produced in the manufacturing process of these structures, etc. are also expected to increase.

[0008] The scandium quality of these disposed articles, etc. is far higher than the scandium quality of nickel oxide ores, etc., and recovering scandium from the disposed articles, etc. and reusing is expected to be an effective means. However, even if considering the scandium quality of disposed articles, etc. to be high, an element contained in Al-Sc alloy is aluminum, and the content of scandium is a very small amount comparing with the content of aluminum; therefore, it is not possible to effectively recover scandium concentrate by simply melting Al-Sc alloy.

[0009] The present invention has been made in order to solve the above such problems, and the object thereof is to effectively recover scandium concentrate from Al-Sc alloy. When explained in further detail, the Al-Sc alloy in structures, etc. is widely used by concentrating to 0.1 to 1% Sc. As mentioned above, the Sc concentration of the Al-Sc master alloy is 1 to 2%. The present invention has an object of efficiently recovering scandium concentration of a degree that can be used as is as an Al-Sc master alloy from scrap articles of Al-Sc alloy having a Sc concentration on the order of 0.1 to 1%.

[0010] As a result of accumulating intensive research to solve the above-mentioned problems, the present inventors found that the above-mentioned object could be achieved by bringing chlorine into contact with an alloy containing aluminum and scandium, and melting, followed by subjecting the molten mixture to electrolysis at predetermined conditions, thereby arriving at completion of the present invention.

Means for Solving the Problems

[0011] More specifically, the present invention provides the following matters.

[0012] A first aspect of the present invention is a scandium concentrate production method, including: a molted mixture generation step of generating a molten mixture of aluminum chloride and scandium chloride by bringing chlorine into contact with an alloy containing aluminum and scandium, and melting; a first electrolysis step of subjecting the molten mixture to first electrolysis at a potential between the metalation potential of aluminum and the metalation potential of scandium to generate molten aluminum; and a second electrolysis step of subjecting the molten mixture, after the molten aluminum generation step, to second electrolysis at a potential capable of recovering scandium to generate a scandium concentrate.

[0013] In addition, according to a second aspect of the present invention, in the scandium concentrate production method as described in the first aspect, the molten mixture generation step is a step of melting the alloy

brought into contact with the chlorine into a melt of a chloride-based salt or eutectic salt having a melting point or eutectic temperature higher than 500°C.

Effects of the Invention

[0014] According to the present invention, it is possible to effectively recover a scandium concentrate from Al-Sc alloy. This scandium concentrate can be used as is as a high-quality Al-Sc master alloy. In addition, by passing through solvent extraction, etc., it is also possible to recover scandium very efficiently compared to a case of recovering from nickel oxide ore.

BRIEF DESCRIPTION OF THE DRAWINGS

[0015]

FIG. 1 is a schematic view showing a scandium concentrate production method according to the present invention; and

FIG. 2 is a schematic view illustrating an electrolysis apparatus 1 used in the present examples.

PREFERRED MODE FOR CARRYING OUT THE INVENTION

[0016] Hereinafter, a specific embodiment of the present invention will be explained in detail; however, the present invention is not to be limited in any way to the following embodiment, and can be realized by applying appropriate modifications within the scope of the object of the present invention. It should be noted that places where explanation would be redundant, the explanation may be omitted as appropriate, but is not to limit the gist of the invention.

<Scandium Concentrate Production Method>

[0017] FIG. 1 is a schematic view showing a scandium concentrate production method according to the present invention. The present method includes: a molten mixture generation step S1 of generating a molten mixture of aluminum chloride and scandium chloride by bringing chlorine into contact with an alloy containing aluminum and scandium and melting; a first electrolysis step of subjecting the molten mixture to first electrolysis at a potential between the metalation potential of aluminum and the metalation potential of scandium; and a second electrolysis step of subjecting the molten mixture, after the molten aluminum generation step S2, to second electrolysis at a potential capable of recovering scandium to generate a scandium concentrate.

(Molten Mixture Generation Step S1)

[0018] The molten mixture generation step S1 brings chlorine gas into contact with an Al-Sc alloy serving as

a raw material (disposed articles, manufacturing defect articles, etc.), to obtain a mixture of aluminum chloride and scandium chloride. The contact temperature with chlorine gas is not particularly limited.

[0019] Next, the above-mentioned mixture is melted. The present invention uses a molten salt electrolysis method to melt an ionic solid by bringing to high temperature, and then electrolyzing this. In the view of the melting point of aluminum being 660.5°C, and heating the molten salt to a temperature higher than this melting point, it is necessary for the molten salt to have a melting point or eutectic temperature higher than 500°C. In the present invention, the vapor pressure of the chloride increases by the activity of the salt rising accompanying an increase in temperature, when the melting point or eutectic temperature is remarkably low compared to the melting point of aluminum, due to heating up to a temperature higher than the melting point of aluminum upon electrolysis, and thus the volatilization of salt will occur. The salt composition gradually changes by way of this volatilization, and the electrolytic voltage continually changes accompanying this; therefore, it is difficult to appropriately control electrolysis. For this reason, it is not preferable to use a molten salt having a melting point or eutectic temperature that is remarkably low compared to the melting point of aluminum. For example, the eutectic temperature of the LiCl-KCl eutectic salt is 350°C, and it is not preferable because the eutectic temperature is remarkably low compared to the melting point of aluminum.

[0020] In particular, it is preferable to use a molten salt having a melting point or eutectic temperature close to the melting point of aluminum, in the point of not requiring to heat more than necessary upon electrolysis.

[0021] Additionally, the molten salt requires there to be a difference of at least 0.8 V between the metalation potential of aluminum and the metalation potential of scandium. The electrolytic potentials of elements differ according to the type and composition of molten salt. Due to aluminum and scandium coexisting, the present invention configures so that only aluminum is generated in the first electrolysis, and scandium is first generated in the second electrolysis; therefore, the molten salt requires to have at least a certain difference between the metalation potential of aluminum and the metalation potential of scandium. A greater difference is preferable, it is more preferably at least 1.0 V, and even more preferably at least 1.2 V.

[0022] In an Ag⁺/Ag electrode system at 450°C of a LiCl-KCl eutectic salt, the metalation potential of Al³⁺ is -1.04 V, and the metalation potential of Sc³⁺ is -1.83 V. The difference between the two is about 0.8 V, and thus is sufficient to suppress both aluminum and scandium generating in the first electrolysis. Additionally, although detailed data for the metalation potential is not known, even if the type of metal element constituting the salt differs, a great difference in the metalation potentials will not arise so long as being chloride-based salts.

[0023] Based on the above-mentioned points, it is pos-

sible to efficiently recover scandium concentrate by using NaCl-KCl eutectic salt (eutectic temperature: 660°C) as the eutectic salt. When making these into a molten salt, the stability rises and changes in composition are suppressed, even if heated to temperatures higher than the melting point of aluminum. In addition, it is also unnecessary to heat more than necessary upon electrolysis. Then, aluminum and scandium are suppressed from both being generated upon the first electrolysis.

[0024] The heating temperature of the molten salt is sufficient so long as an extent able to sufficiently melt the Al-Sc alloy. The liquidus temperature of the Al-Sc alloy will differ according to the scandium concentration contained in the Al-Sc alloy. The liquidus temperature of the Al-Sc alloy is determined by referencing a published phase diagram of Al-Sc alloy. For example, in the case of the scandium concentration being on the order of 0.2 to 0.4%, the liquidus temperature of the Al-Sc alloy is on the order of 660°C, which is the eutectic temperature. On the other hand, the liquidus temperature rises as the scandium concentration contained in the Al-Sc alloy increases, and the liquidus temperature in the case of the scandium concentration being 1% is about 730°C, and the liquidus temperature in the case of being 2% exceeds 800°C.

[0025] The above-mentioned mixture, i.e. mixture of aluminum chloride and scandium chloride, is dissolved in the molten salt heated to an extent capable of sufficiently melting the Al-Sc alloy. Since the saturated vapor pressure of aluminum chloride and the saturated vapor pressure of scandium chloride differ, when dissolving the above-mentioned mixture in the above-mentioned molten salt, while a part of the aluminum chloride (AlCl_3) volatilizes, the remaining aluminum chloride and scandium chloride (ScCl_3) easily melt into the eutectic salt to form a uniform melt.

(First Electrolysis Step S2)

[0026] Next, the first electrolysis step S2 will be explained. The first electrolysis step S2 subjects the molten mixture obtained in the molten mixture generation step S1 to the first electrolysis at a potential between the metalation potential of aluminum and the metalation potential of scandium to generate molten aluminum.

[0027] Although the type of electrode is not particularly limited, for example, establishing silver as the reference electrode, graphite as the anode, and nickel as the cathode can be exemplified.

[0028] The potential in the first electrolysis is required to be between the metalation potential of aluminum and the metalation potential of scandium, and in more detail, no more than the metalation potential of aluminum and at least the metalation potential of scandium. If not in this range, it is not preferable because not only molten aluminum, but also scandium can also generate at the cathode. In particular, the potential in the first electrolysis is preferably closer to the metalation potential of aluminum,

and specifically, is preferably within the range of -1.50 V to -1.04 V, and is more preferably within the range of -1.30 V to -1.10 V.

[0029] The temperature of the molten salt is not particularly limited so long as higher than the melting point of aluminum.

[0030] In the first electrolysis, it is preferable to store the aluminum in a storage container along with molten salt in advance. The interior of the storage container is heated up to a temperature higher than the melting point of aluminum; therefore, the stored aluminum is used as an aluminum electrode (cathode) through the above-mentioned nickel. Then, by passing through the first electrolysis, new aluminum produced in the electrolysis of the Al-Sc alloy melts into the aluminum that had already melted. As a result thereof, the concentration of scandium contained in the molten salt rises.

(Second Electrolysis Step S3)

[0031] Next, the second electrolysis step S3 will be explained. The second electrolysis step S3 subjects the molten mixture to the second electrolysis at a potential capable of recovering scandium, after the molten aluminum generation step S2. By doing this, the liquid scandium dissolves in the liquid aluminum obtained in the first electrolysis, and a scandium concentrate is generated.

[0032] Although not essential, it is preferable to extract part of the aluminum obtained in the first electrolysis out of the storage container, while tilting the storage container before performing the second electrolysis. By doing this, it is possible to improve the scandium quality of the scandium concentrate obtained by performing the second electrolysis.

[0033] The type of electrode is not particularly limited, and the same one as the electrode used in the first electrolysis can be used.

[0034] The potential in the second electrolysis is required to be capable of recovering scandium, and be no more than the metalation potential of scandium. If not in this range, it is not preferable because liquid scandium will not dissolve in the liquid aluminum obtained in the first electrolysis at the cathode. Although the potential in the second electrolysis is sufficient so long as no more than -1.83 V, when considering the stability of the operation, it is preferably no more than -2.0 V.

[0035] Upon performing the second electrolysis, the amount of aluminum stored in advance inside the storage container can be arbitrarily set according to the target scandium quality for the scandium concentrate. The temperature of the molten salt is not particularly limited so long as higher than the temperature of the liquidus of the Al-Sc alloy contained in the molten salt; however, based on the stability of the operation, etc., it is preferably at least 5°C higher than the temperature of the liquidus of the Al-Sc alloy, and more preferably at least 10°C higher.

[0036] The scandium concentrate of the present invention may be reused as is as a high-grade Al-Sc master

alloy, or may be reused after remelting and making into a master alloy. In addition, by recovering scandium from a scandium concentrate by a known method such as solvent extraction, it is possible to very efficiently recover scandium compared to a case of recovering from nickel oxide ore.

EXAMPLES

[0037] Hereinafter, the present invention will be explained in further detail by way of examples; however, the present invention is not to be subjected to any limitations in these descriptions.

<Example>

[0038] FIG. 2 is a schematic view illustrating the configuration of the electrolysis apparatus 1 used in the present Examples. The electrolysis apparatus 1 includes: a quartz container 2 that encloses the Al-Sc alloy along with molten salt; a quartz tube 3 with one side open, and accommodating the quartz container 2 from this opening; a rubber stopper 4 that seals this quartz tube 3; a reference electrode (silver) 5, anode (graphite) 6 and cathode (nickel) 7 inserted inside of the quartz container 2; a gas substitution unit 8 that substitutes the inside of the quartz tube 3 with argon gas; a thermocouple 9 that is inserted inside of the quartz container 2; an electric furnace 10 that keeps the temperature inside of the quartz tube 3 at a predetermined temperature; and an insulation board 11 that keeps the adiabaticity of the inside of the quartz tube 3.

[0039] Into the quartz container 2, 20 g of aluminum and the NaCl-KCl eutectic salt (mole ratio of NaCl to KCl = 1:1) was placed, and this quartz container 2 was stored inside of the quartz tube 3. Then, the opening of the quartz tube 3 is sealed by the rubber stopper 4, and the thermocouple 9 is installed at the position shown in FIG. 2, followed by sufficiently replacing the air with argon through the gas substitution unit 8 in order to keep the internal atmosphere of the quartz tube 3 inert. Then, the inside of the quartz tube 3 was heated to 750°C, and held for 30 minutes under an argon gas flow. Then, it was confirmed visually that the aluminum and NaCl-KCl eutectic salt had melted to form the molten salt.

[0040] Independently from this, chlorine gas was brought into contact with an Al-Sc alloy having a scandium concentration of 1%, whereby the alloy was chlorinated. The scandium quality of the chloride thereby obtained was approximately 10%. Into the above-mentioned molten salt, 15 g of this chloride was charged, and in order to keep the interior atmosphere of the quartz tube 3 inert, chlorine gas was supplied for 15 minutes at the flowrate of 0.1 L/min into the quartz tube 3 through the gas substitution unit 8.

[0041] Next, the reference electrode (silver) 5, the anode (graphite) 6 and the cathode (nickel) 7 were immersed to the positions shown in FIG. 2, and the first

electrolysis was performed while holding at the potential of -1.25 V for the potential of the Ag⁺/Ag reference electrode 5. Aluminum was thereby recovered from the bottom of the quartz container 2 through the cathode 7.

[0042] After the first electrolysis, electric current was temporarily stopped, the pressure inside of the quartz tube 3 was reduced, and about 10 g of the aluminum produced in the first electrolysis was removed by vacuum.

[0043] Next, the inside of the quartz tube 3 was heated to 880°C, and the second electrolysis was performed. The aluminum remaining at the bottom of the quartz container 2 and the aluminum and scandium eluted from the Al-Sc alloy contained in the molten salt thereby mixed, and a scandium concentrate was obtained.

[0044] After the second electrolysis, the inside of the quartz tube 3 was cooled to room temperature. Then, the salt 12 (mixture of aluminum and NaCl-KCl eutectic salt) and scandium concentrate 13 that solidified from cooling were retrieved.

[0045] For each of the aluminum recovered in the first electrolysis and the scandium concentrate 13 recovered in the second electrolysis, analysis was performed using an X-ray fluorescence spectrometer (XRF) and an ICP mass spectrometer. The purity of aluminum metal contained in the aluminum recovered in the first electrolysis exceeded 98%, and the scandium concentrate 13 recovered in the second electrolysis was about 3.5%. Based on the above, it was confirmed that the aluminum recovered in the first electrolysis and the scandium concentrate 13 recovered in the second electrolysis can be reused as is as aluminum and as high-quality Al-Sc master alloy, respectively.

EXPLANATION OF REFERENCE NUMERALS

[0046]

- 1 electrolysis apparatus
- 2 quartz container
- 3 quartz tube
- 4 rubber stopper
- 5 reference electrode
- 6 anode
- 7 cathode
- 8 gas substitution unit
- 9 thermocouple
- 10 electric furnace
- 11 insulation board

Claims

1. A scandium concentrate production method comprising:

a molten mixture generation step of generating a molten mixture of aluminum chloride and scan-

dium chloride by bringing chlorine into contact with an alloy containing aluminum and scandium, and melting;

a first electrolysis step of subjecting the molten mixture to first electrolysis at a potential between the metalation potential of aluminum and the metalation potential of scandium to generate molten aluminum; and

a second electrolysis step of subjecting the molten mixture, after the molten aluminum generation step, to second electrolysis at a potential capable of recovering scandium to generate a scandium concentrate.

2. The scandium concentrate production method according to claim 1, wherein the molten mixture generation step is a step of melting the alloy brought into contact with the chlorine into a melt of a chloride-based salt or eutectic salt having a melting point or eutectic temperature higher than 500°C.

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FIG. 1

(SCANDIUM CONCENTRATE PRODUCTION METHOD)

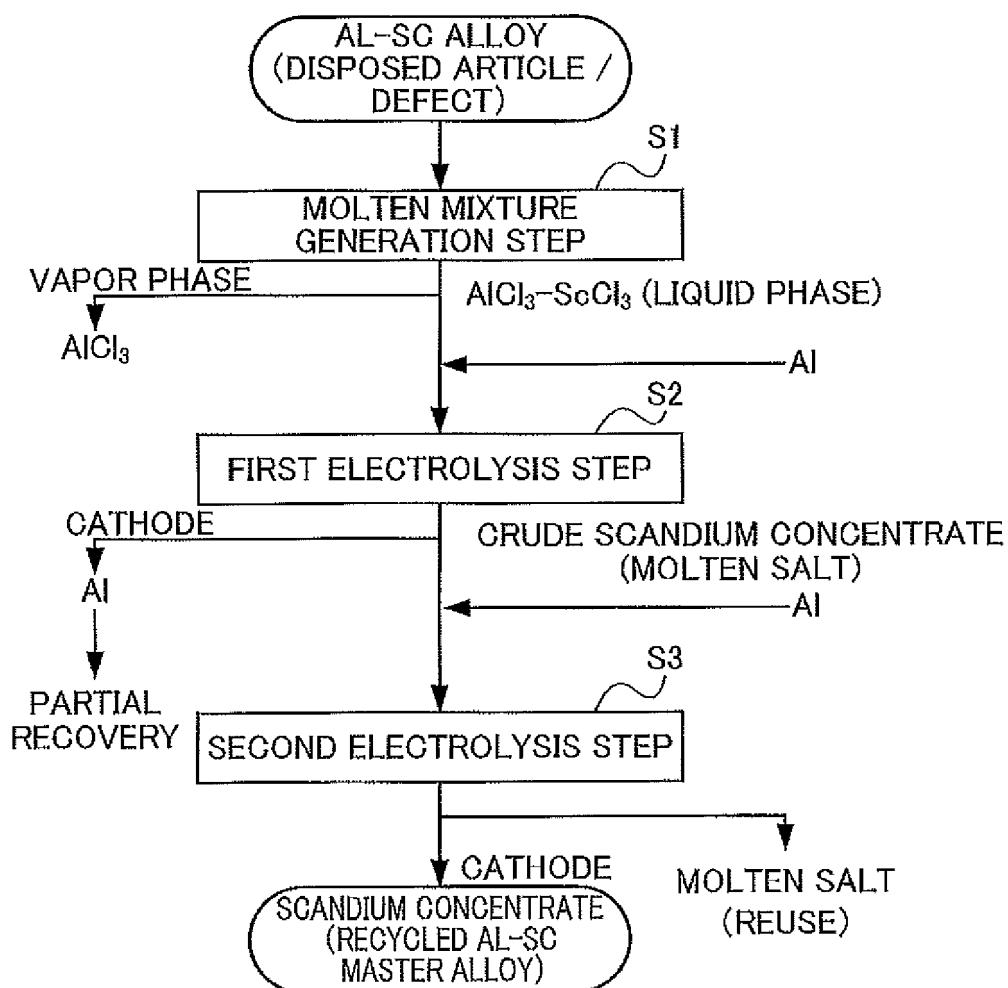
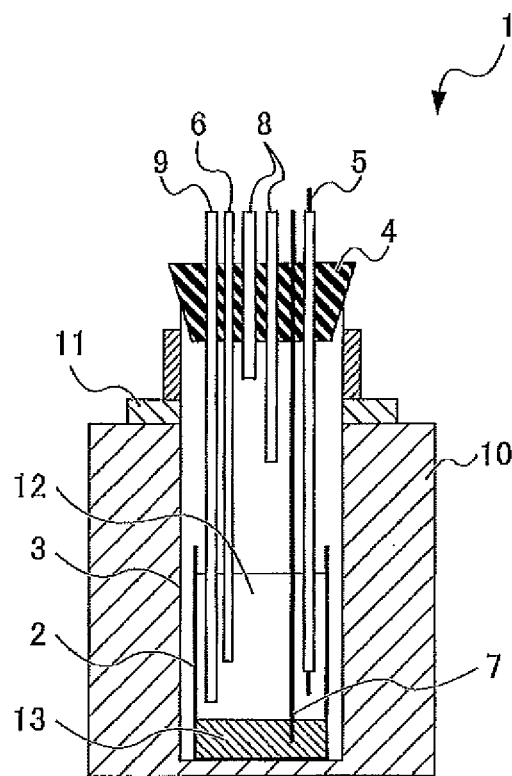


FIG. 2



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2015/051828

A. CLASSIFICATION OF SUBJECT MATTER

C25C3/36(2006.01)i, C22B7/00(2006.01)i, C22B59/00(2006.01)i, C22C21/00(2006.01)i, C25C3/06(2006.01)i, C25C3/34(2006.01)i

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)

C25C3/36, C22B7/00, C22B59/00, C22C21/00, C25C3/06, C25C3/34

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

Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2015

Kokai Jitsuyo Shinan Koho 1971-2015 Toroku Jitsuyo Shinan Koho 1994-2015

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	JP 2007-254822 A (Pacific Metals Co., Ltd.), 04 October 2007 (04.10.2007), (Family: none)	1-2
A	JP 2012-136766 A (Kyoto University), 19 July 2012 (19.07.2012), (Family: none)	1-2

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

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Patent documents cited in the description

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