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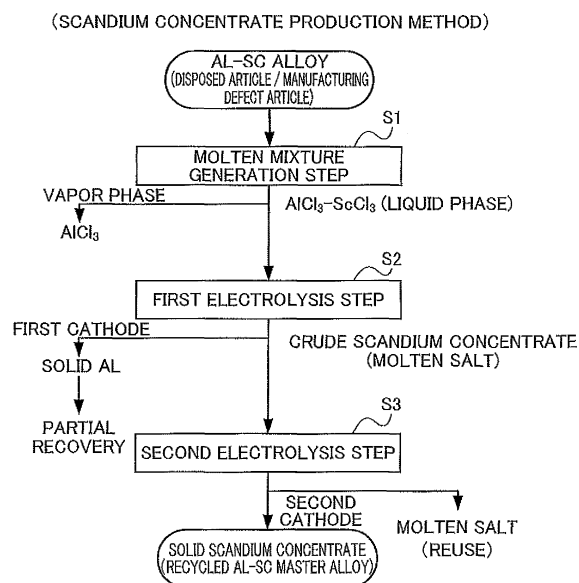
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(54) **SCANDIUM CONCENTRATE PRODUCTION METHOD**

(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 using a first cathode 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 aluminum is produced around the first cathode; and a second electrolysis step (S3) in which, after a molten aluminum production step, the molten mixture is subjected to second electrolysis using a second electrode at a potential that makes it possible to recover scandium and a scandium concentrate is produced around a second cathode.

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 an 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, 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 molten mixture generation step of bringing chlorine into contact with an alloy containing aluminum and scandium, and melting to generate a molten mixture of aluminum chloride and scandium chloride; a first electrolysis step of subjecting the molten mixture to first electrolysis using a first cathode at a potential between a metalation potential of aluminum and a metalation potential of scandium to generate aluminum at the periphery of the first cathode; and a second electrolysis step of subjecting the molten mixture after the molten aluminum generation step to second electrolysis using a second electrode at a potential capable of recovering scandium to generate a scandium concentrate at the periphery of the second cathode.

[0013] In addition, according to a second aspect of the present invention, in the scandium concentrate produc-

tion 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 of no higher than 500°C, the first electrolysis step is a step of generating solid aluminum at the periphery of the first cathode, and the second electrolysis step is a step of generating a solid scandium concentrate at the periphery of the second cathode.

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 concentration 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 the configuration of 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. This 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 S2 of subjecting the molten mixture to first electrolysis using a first cathode at a potential between the metalation potential of aluminum and the metalation potential of scandium to generate aluminum at the periphery of the first cathode; and a second electrolysis step S3 of subjecting the molten mixture,

after the molten aluminum generation step S2, to a second electrolysis using a second electrode at a potential capable of recovering scandium to generate a scandium concentrate on the periphery of the second cathode.

(Molten Mixture Generation Step S1)

[0018] Upon producing scandium concentrate, it has been considered to use a molten salt electrolysis method, which heats an ionic solid to high temperature to melt, and then electrolyzes this. However, due to the melting point of aluminum being 660.5°C, with the molten salt electrolysis method, the molten salt must be heated to a temperature higher than this melting point, and thus an abundance of energy is required. Therefore, the present invention first brings chlorine gas into contact with an Al-Sc alloy that is the raw material (disposed article, manufacturing defect, etc.) to obtain chlorides of the Al-Sc alloy having a low melting point compared to the Al-Sc alloy. Next, the chlorides of Al-Sc alloy are melted. It is possible to sufficiently dissolve at a temperature on the order of 450°C if chlorides of Al-Sc alloy.

[0019] Salts or eutectic salts used upon melting the above-mentioned mixture require consideration in both aspects of the viewpoint of the melting point or eutectic temperature, and the viewpoint of the metalation potentials of metals that are the separation targets; however, the former viewpoint will be explained first. The stability is high at a temperature about 10°C higher than the temperature that can melt the chlorides of Al-Sc alloy, and is not particularly limited so long as not being a temperature at which a change in the salt composition arises due to volatilization. More specifically, the melting point or eutectic temperature are preferably 360°C to 500°C, more preferably 380°C to 450°C, and even more preferably 390°C to 400°C. If the melting point or eutectic temperature is too low, it is not preferable because a change in the salt composition due to volatilization can occur when heating the salt or eutectic salt to an extent that can melt the chlorides of Al-Sc alloy. If the melting point or eutectic temperature is too high, it is not preferable because it will heat the salt or eutectic salt up to an unnecessarily high temperature.

[0020] Next, the latter viewpoint will be explained. 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 the molten salt. With the present invention, since aluminum and scandium coexist, since it is configured so that only aluminum is generated in the first electrolysis, and scandium is first generated in the second electrolysis, 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, and it is more preferably at least 1.0 V.

[0021] In an Ag⁺/Ag electrode system at 450°C of a

LiCl-KCl eutectic salt, the metalation potential of Al^{3+} is -1.04 V, and the metalation potential of Sc^{3+} is -1.83 V. The difference between the two is about 0.8 V, and is sufficient to suppress both the aluminum and scandium from 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 does not arise so long as being chloride-based salts.

[0022] Based on the above-mentioned points, it is possible to efficiently recover scandium concentrate by using as the LiCl-KCl eutectic salt (eutectic temperature: 354°C, difference in metalation potentials between Al^{3+} and Sc^{3+} : approx. 0.8 V). 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. It is also unnecessary to heat more than necessary upon electrolysis. Then, both aluminum and scandium are suppressed from being generated upon the first electrolysis.

[0023] The above-mentioned chlorides of Al-Sc alloy are dissolved in the molten salt heated to an extent capable of sufficiently melting the chlorides of 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 chlorides of Al-Sc alloy 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)

[0024] 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 using a first cathode at a potential between the metalation potential of aluminum and the metalation potential of scandium to generate solid aluminum at the periphery of this first cathode in a dendrite form (arborescent crystal).

[0025] 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 first cathode can be exemplified.

[0026] 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 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.

[0027] The temperature of the molten salt is not particularly limited so long as being able to melt the chlorides of Al-Sc alloy and being lower than the melting point of aluminum; however, from the point of curbing the energy cost to a minimum, the point of suppressing the generated aluminum from becoming liquid, etc., it is preferably 360°C to 500°C, and more preferably 380°C to 450°C.

[0028] Chlorine gas is generated at the anode during the first electrolysis. In order to raise the current efficiency, it is preferable to quickly remove the generated chlorine gas from the anode.

[0029] Accompanying causing aluminum to precipitate in the first electrolysis, the scandium concentration contained in the molten salt rises. In other words, it is possible to adjust the scandium quality of the scandium concentrate precipitating in the second electrolysis by adjusting the amount of aluminum precipitating in the first electrolysis.

(Second Electrolysis Step S3)

[0030] Next, the second electrolysis step S3 will be explained. The second electrolysis step S3 subjects the molten mixture to the second electrolysis using a second cathode differing from the first cathode at a potential capable of recovering scandium, after the molten aluminum generation step S2. By doing this, solid scandium concentrate is generated in dendrite form (arborescent crystal) at the periphery of the first cathode.

[0031] The material of the electrode is not particularly limited, and it is acceptable to use the same as the electrode used in the first electrolysis; however, it is necessary for at least the first cathode and second cathode to be two different cathode bars.

[0032] It is required for the potential in the second electrolysis to generate scandium on the cathode in dendrite form along with aluminum, more specifically, to be no more than the metalation potential of scandium. If not in this range, it is not preferable because solid scandium concentrate will not satisfactorily generate at the cathode. The potential in the second electrolysis is sufficient so long as being no more than -1.83 V; however, when considering the stability of operation, it is preferably no more than -2.0 V, and more preferable no more than -2.2 V.

EXAMPLES

[0033] 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>

[0034] FIG. 2 is a schematic view illustrating the configuration of an electrolysis apparatus 1 used in the present examples. The electrolysis apparatus 1 includes:

the two of a small and large quartz container 21, 22 that enclose the Al-Sc alloy along with molten salt; a quartz tube 3 with one side open, and accommodating the quartz containers 21, 22 from this opening; a rubber stopper 4 that seals this quartz tube 3; a reference electrode (silver) 5, anode (graphite) 6 and first or second cathode (nickel) 71, 72 inserted inside of the large quartz container 21; a gas substitution unit 8 that substitutes the inside of the quartz tube 3 with gas; a thermocouple 9 that is inserted inside of the large quartz container 21; 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.

[0035] The LiCl-KCl eutectic salt (mole ratio of KCl to KCl = 59:41) was placed in the large quartz container 21 in which the small quartz container 22 was stored in advance, and this quartz container 21 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 was installed at the position shown in FIG. 2, followed by sufficiently substituting 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 450°C, and held for 30 minutes under an argon gas flow. Then, it was confirmed visually that the LiCl-KCl eutectic salt had melted to form the molten salt 12.

[0036] 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, 30 g of this chloride was added, and 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.

[0037] Next, the reference electrode (silver) 5, anode (graphite) 6 and first cathode (nickel) 71 were immersed to the positions shown in FIG. 2, and the first electrolysis was performed while holding at the potential of -1.15 V for the potential of the Ag⁺/Ag reference electrode 5. Solid aluminum of dendrite form was thereby recovered from the first cathode 71.

[0038] After the first electrolysis, electric current is temporarily stopped, and the solid aluminum is recovered along with the first cathode 71.

[0039] Next, the first cathode 71 was replaced with the second cathode 72, and the second electrolysis was performing while maintaining a potential of -1.95 V for the potential of the Ag⁺/Ag reference electrode 5. Both aluminum and scandium were thereby precipitated from the first cathode 71, a result of which solid scandium concentrate 13 of dendrite form was obtained.

[0040] After the second electrolysis, the inside of the quartz tube 3 was cooled to room temperature. Then, the salt 12 and scandium concentrate 13 that solidified from cooling were retrieved.

[0041] For each of the solid aluminum recovered in the first electrolysis and the solid scandium concentrate 13 recovered in the second electrolysis, analysis was performed using an X-ray fluorescence spectrometer (XRF) and an ICP mass spectrometer. The weight of aluminum metal contained in the aluminum recovered in the first electrolysis was 3.5 g, and the purity exceeded 98%. In addition, the weight of the scandium concentrate 13 received in the second electrolysis was 2.0 g, and the purity was approximately 30%. 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 a high-quality Al-Sc master alloy, respectively.

EXPLANATION OF REFERENCE NUMERALS

[0042]

1	electrolysis apparatus
21	large quartz container
22	small quartz container
3	quartz tube
4	rubber stopper
5	reference electrode
6	anode
71	first cathode
72	second 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 bringing chlorine into contact with an alloy containing aluminum and scandium, and melting to generate a molten mixture of aluminum chloride and scandium chloride;

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

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

2. A scandium concentrate production method accord-

ing to claim 1,
wherein the molten mixture generation step is a step
of melting the alloy brought into contact with the chlo-
rine into a melt of a chloride-based salt or eutectic
salt having a melting point or eutectic temperature 5
of no higher than 500°C,
wherein the first electrolysis step is a step of gener-
ating solid aluminum at the periphery of the first cath-
ode, and
wherein the second electrolysis step is a step of gener- 10
ating a solid scandium concentrate at the periphery
of the second cathode.

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FIG. 1

(SCANDIUM CONCENTRATE PRODUCTION METHOD)

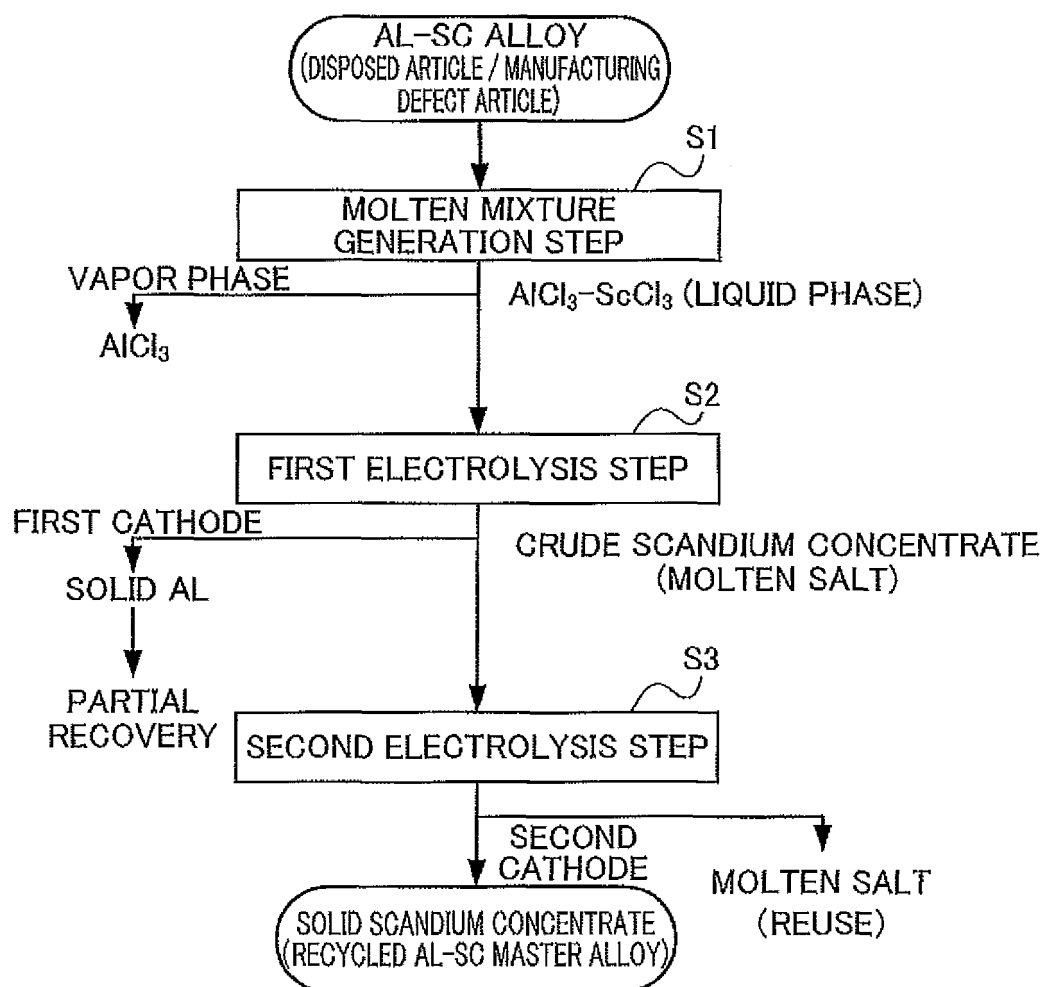
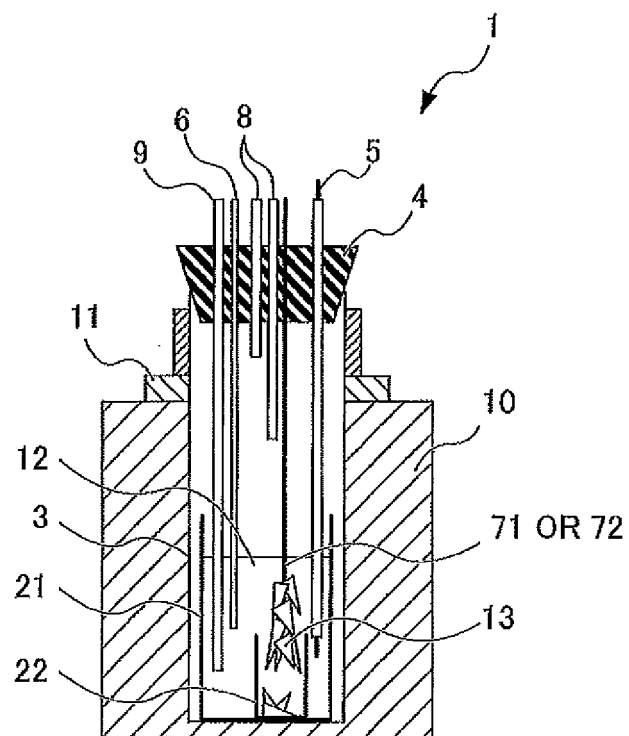


FIG. 2



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2015/051827

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.

* 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
25 February 2015 (25.02.15)

Date of mailing of the international search report
10 March 2015 (10.03.15)

Name and mailing address of the ISA/
Japan Patent Office
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Patent documents cited in the description

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