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(54) STREAMLINED PROCESS FOR PRODUCING ALUMINUM-SCANDIUM ALLOY

(57) An alloy product is produced by an aluminothermic reduction process and an alloying process with one or more other metals or master alloy, where the reduction process and the alloying process are performed in a single stage. The final alloy product may have a scandium concentration that is greater than 0% and less than about 2%. According to another aspect of the present disclosure, a first melt is produced at a first melt temperature, a melting and alloying step is performed at a second melt temperature, less than the first melt temperature, and the temperature of the first melt is not substantially less than the second melt temperature before the melting and alloying step.

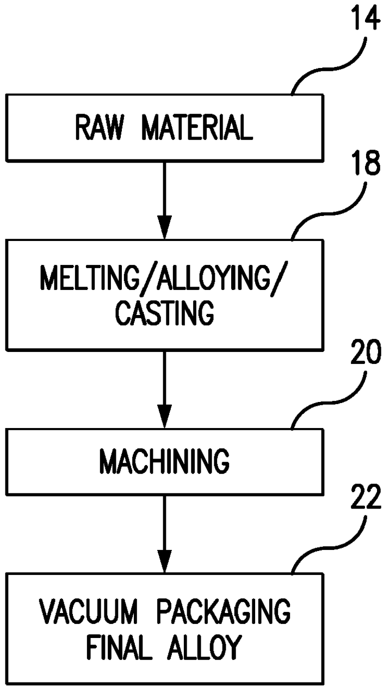

FIG. 1

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Description

Field of the Invention

[0001] The present disclosure relates to a system and a method for producing an aluminum (Al)-scandium (Sc) alloy. The present disclosure also relates to a system and a method for producing a product including an Al-Sc alloy, and to a product made by such systems and methods.

Brief description of the related art

[0002] Methods of producing Al-Sc alloys are described in United States Patent Application Publications Nos. 2019/0218645 (published July 18, 2019), 2019/0218644 (published July 18, 2019), and 2018/0087129 (the ’129 publication). The present disclosure overcomes disadvantages of the prior art to a great extent.

Summary of the Disclosure

[0003] An According to one aspect of the present disclosure, a method of producing an Al-Sc final alloy product includes an aluminothermic Sc-reduction process and an alloying process with one or more other metals or master alloys. According to this aspect of the present disclosure, the aluminothermic Sc-reduction process and the alloying process are performed together in a single stage, without generating or remelting an Al-Sc master alloy with the one or more other metals or master alloys.

[0004] The method may include, if desired, casting an Al-Sc alloy, after the alloying process, then machining a cast Al-Sc alloy, and then packaging the Al-Sc final alloy product.

[0005] If desired, the aluminothermic Sc reduction process includes reduction of a Sc halide, such as scandium fluoride (ScF_3). Indeed, halides in combination with other salts such as sodium fluoride (NaF), sodium chloride, potassium chloride (KCl), calcium chloride, and ammonium bifluoride may be preferred because they enable the reduction of Sc at a relatively lower temperature and a shorter reaction time.

[0006] The present disclosure also relates to a method of producing a final alloy product, where the method includes melting and alloying material in a single stage, without remelting an alloy produced by an aluminothermic reduction process. The final alloy product may include an Al-Sc alloy and may have, for example, a Sc concentration of less than about 2%.

[0007] The present disclosure also relates to a method of producing an Al-Sc alloy, including: a primary melting step which includes producing a first melt by performing an aluminothermic Sc-reduction process and simultaneously melting and alloying a first metal or master alloy having a first volatilization temperature; and a melting and alloying step which includes adding, to the first melt, a second metal or master alloy having a second volatilization temperature.

[0008] According to this aspect of the present disclosure, the second volatilization temperature is less than the first volatilization temperature, and the primary melting step is performed before the melting and alloying step. The first melt is produced at a first melt temperature, and the melting and alloying step is performed at a second melt temperature, the second melt temperature being less than the first melt temperature, and the temperature of the first melt is not substantially less than the second melt temperature between the primary melting step and the melting and alloying step.

Brief Description of the Drawings

[0009] The invention will be described based on figures. It will be understood that the embodiments and aspects of the invention described in the figures are only examples and do not limit the protective scope of the claims in any way. The invention is defined by the claims and their equivalents. It will be understood that features of one aspect or embodiment of the invention can be combined with a feature of a different aspect or aspects of other embodiments of the invention, in which:

Fig. 1 is a flow chart for an example of a method of making a final alloy in accordance with the present disclosure;

Fig. 2 is a flow chart for melting and alloying processes within the method of Fig. 1; and

Fig. 3 is a flow chart for a comparative process.

Detailed Description of the Disclosure

[0010] The technical problem is solved by the independent claims. The dependent claims cover further specific embodiments of the invention.

[0011] Scandium (Sc) is an effective alloying strengthener for aluminum (Al) and Al alloys. It imparts substantial improvement in strength even with small additions between 0.1% to 1%, or 50 MPa per 0.1%. The strengthening property of Sc comes from its ability to act as a grain refiner and to form nano-sized precipitates during specific heat treatment processes that are evenly distributed and coherently bound to an Al matrix. Compared to other microalloying elements, Sc minimizes losses in the desirable lightweight property and ductility of Al.

[0012] In particular, among Al alloy families, Sc has the most pronounced strengthening effect on the 5000 series (Al-Mg). The Al-Mg combination is a soft alloy and has relatively low strength, often below usable limits in demanding applications. With Sc, strength improves while the desired mechanical properties are preserved, or even enhanced. Sc additions of 0.25% can increase yield strength by 150% while maintaining ductility. Other benefits are a significant enhancement in formability, improved thermal stability, improved resistance to recrystallization, and enhanced resistance against fatigue-crack growth.

[0013] The development and use of high-performance Sc-containing alloys is projected to ramp up in the future. These alloys are particularly suitable for automotive and air transportation applications, due to their ability to reduce the weight of critical moving parts. Sc-containing alloy is also a promising material for aerospace applications, including bulkheads, heat shields, running gears, and fuel and exhaust systems.

[0014] Other applications for Sc-containing alloys are in high-strength extrusions for body-frame and crash-management systems. Due to its enhanced corrosion resistance, Sc-containing alloys are also suitable for use in marine transportation applications and heat-exchanger tubes in desalination plants. Al-Sc welding wire imparts high part-strength and high fatigue-resistance aside from other benefits such as improved processing, reliability, and high weld quality, applicable to additively manufactured parts.

[0015] However, this expected commercial growth may be inhibited by scarcity of Sc sources. Sc is rarely found in concentrated form and there are no known commercially usable Sc deposits. Sc may be recovered as a by-product of other mineral refining processes such as rare earth and in acid wastes from titanium dioxide pigment production. Other sources involve small refining circuits which may be directly acquired by end users preventing availability of Sc in the open market. This lack of a secure source of Sc is further aggravated because the price of Sc may be effectively controlled by governmental authorities or businesses in a few countries.

[0016] Another challenge is the current high production cost of Sc-Al alloy. Alloying pure Sc metal with Al alloys requires high reaction temperature and a long dissolution time. It is possible to use a master alloy containing up to 10% Sc. However, preparation of the master alloy may entail separate melting and casting steps which translate to additional processing cost and production cycle time.

[0017] A known method of producing Al-Sc master alloy is through the aluminothermic reduction of Sc from its oxide or halide compounds. Scandium oxide (Sc_2O_3) is thermodynamically stable, hence difficult to be reduced to metallic Sc even in the presence of reductants such as calcium (Ca) and magnesium (Mg). In addition to this, the known method requires high temperature and a long reaction time.

[0018] The use of Sc halides such as scandium chloride ($ScCl_3$) and scandium fluoride (ScF_3) in combination with other salts such as sodium fluoride (NaF), sodium chloride (NaCl), potassium chloride (KCl), calcium chloride ($CaCl_2$), and ammonium bifluoride (NH_4HF) is preferred because it permits the reduction of Sc at a lower reduction temperature and a shorter reaction time.

[0019] In the known method, the reduced Sc diffuses into the Al melt to form the master alloy with concentrations up to 10% Sc. The molten mixture is allowed to cool to a temperature in the range of from 690 °C to 750 °C, and then cast. However, Al-Sc master alloy has no commercial application other than as a raw material for producing high strength Al-Sc final alloys with lower concentrations of Sc.

[0020] In other words, the Al-Sc master alloy typically has to be remelted with other master alloys and diluted with pure Al to achieve the desired concentration in the final product. Alloying is done between 660 °C and 1,000 °C depending on the characteristic melting properties of the master alloys being added. The melt is allowed to cool to the desired pouring temperature before it is cast into molds. This two-stage practice leads to long production time, high cost, and high material losses.

[0021] Referring now to Figs. 1 and 2, a streamlined system or method in accordance with the present disclosure can produce a final alloy product in a single stage by combining an aluminothermic Sc reduction process and an alloying process with other metals in the single stage. A method performed in accordance with the present disclosure may benefit from a similarity in (1) the temperature required for the Sc reduction reaction to occur and (2) the temperature required to melt the other master alloys.

[0022] In operation, the raw materials may be weighed and prepared depending on the composition of the final alloy. Then the aluminothermic reduction of Sc is performed by melting the Sc compound which can be an oxide or a halide (but is preferably ScF_3), together with an alkali halide salt as flux and pure aluminum metal.

[0023] This primary melting step may be performed, if desired, simultaneously with melting and alloying of other metals or master alloys which have high volatilization temperatures. The primary melting step, and optionally the simultaneous melting and alloying of the high volatilization-temperature metals or master alloys, may be performed at a temperature in the range of from about 650 °C to about 850 °C under vacuum, or in the range of from about 700°C to about 1,000 °C under atmospheric pressure.

[0024] Subsequently, the primary melt is cooled to a temperature suitable for the addition of other metals or master alloys which have lower volatilization temperatures. Any slags that are formed are skimmed off and removed. Vacuum can be induced within a vacuum-induction furnace to remove gases such as oxygen (O), hydrogen (H), and nitrogen (N) from the melt. The final alloy melt is then poured and cast into molds.

[0025] If desired, the remelting and alloying of other metals or master alloys with a high volatilization temperature in the range of from 800 °C to 850 °C under vacuum, or at 900 °C to 1,000 °C under atmospheric pressure, may be performed for 1-2 hours. The melt is then cooled to the temperature suitable for addition of the other metals or master alloys with low volatilization temperature. Any slags that form are skimmed off and removed. Vacuum can be induced to remove gases such as O, H, and N from the melt. The melt is then poured and cast into molds.

[0026] A system or process constructed or performed in accordance with the present disclosure may have several advantages over known processes, including: (1) high reduction efficiency of Sc of up to 90%, (2) lower metal losses from volatilization due to lower reaction time and bypassing the double-step alloy production, (3) lower operating cost, (4) shorter cycle time, and (5) flexibility of the process to produce Al alloys with a wide range of Sc concentrations. The wide range may be, for example, a range of from about 0.05% to about 2% Sc.

[0027] A system or process constructed or performed in accordance with the present disclosure may be advantageously applied, if desired, to the production of other rare-earth-based metal alloys and master alloys, including Al alloys including rare-earth elements such as yttrium (Y), cerium (Ce), neodymium (Nd), lanthanum (La) and transition metals such as titanium (Ti), zirconium (Zr), and manganese (Mn).

[0028] As illustrated in Fig. 1, an example of a method of making a final alloy product includes a preliminary step 14 of providing raw materials for forming the final alloy. The raw materials provided in step 14 may include one or more suitable Sc compounds which may be an oxide or a halide of Sc, together with an alkali halide salt as flux, pure aluminum metal, the high volatilization-temperature metals or master alloys, and the low volatilization-temperature metals or metal alloys.

[0029] The Sc source may advantageously be a Sc halide, preferably ScF_3, and the flux may advantageously be NaF only. NaF is less expensive and less hygroscopic than KF. If desired, the present disclosure may be employed without requiring a cover flux. Production of a final alloy containing Sc higher than its eutectic composition of about 0.35-0.65 wt% is also part of the present disclosure. KCl may be added to the flux to further lower the melting point of a NaF-ScF_3 flux system and ensure efficient reduction of Sc at such higher concentration. The presence of KCl decreases the volatilization temperature of Al. Therefore, reduction and alloying can also be performed under atmospheric pressure. Sc recovery with and without KCl were 80% and 55%, respectively.

[0030] After the preliminary step 14, the raw materials are melted, alloyed, and cast in suitable molds (step 18). The cast/molded alloy may then be machined as desired (step 20) and then packaged (step 22) for subsequent use or for distribution to customers.

[0031] The packaged product may be configured for a variety of suitable purposes, including in an automotive, air transportation, or aerospace (including for a bulkhead, heat shield, running gear, and fuel and exhaust system) application, or for a body-frame or crash-management system. The packaged product may be configured, if desired, for use in marine transportation. The packaged product may be, for example, a heat-exchanger tube for a desalination plant, or Al-Sc welding wire.

[0032] Referring now to Fig. 2, the melting and alloying processes within step 18 may include the primary melting step which includes the aluminothermic Sc reduction process and the simultaneous remelting and alloying of other metals or master alloys which have high volatilization temperatures all of which are performed at a first, high temperature (collectively, step 30). Melting and alloying by addition of the other metals or master alloys which have low volatilization temperatures may be performed after step 30, at a second, lower temperature (step 32).

[0033] An advantageous feature of the process illustrated in Fig. 2 is that the temperature of the melted alloy created by the primary melting step 30 is not decreased substantially below the second temperature before the subsequent step 32 commences. No casting (cooling) has to occur between steps 30 and 32. As a result of this advantageous feature, the total amount of time and energy needed to produce the final alloy can be reduced.

[0034] The processes illustrated in Figs. 1 and 2 may be contrasted with the process illustrated in Fig. 3. In the Fig. 3 process, raw materials for producing a master alloy are melted, alloyed, and cast (cooled) (step 44), machined (step 46), vacuum packaged (step 48), and then subsequently unpacked (step 50) before the master alloy is melted again and alloyed with other raw material (step 52) to form a final alloy with a Sc concentration less than that of the master alloy. An advantageous feature of the present disclosure is that a final alloy may be produced with a small, desired Sc concentration, less than that of a master alloy, without performing steps 44, 46, 48, 50 shown in Fig. 3.

[0035] In one aspect of the present disclosure, ScF_3 may be used as the Sc source. Among other reasons, it may be more efficient to reduce ScF_3 than to reduce Sc_2O_3. Two tests were conducted to compare the reduction efficiency of

Sc_2O_3 to that of ScF_3. The tests are described in the following, and the results are in Table 1. The tests showed that the reduction efficiency of Sc_2O_3 was only 45% compared to a reduction efficiency of 96% for ScF_3.

[0036] In both tests, about 25 grams of Al granules were melted with the respective Sc source and flux, and the amount of Sc source was based on the target concentration in the alloy. For both tests, the amount of NaF flux was 5% excess of the stoichiometric requirement according to the following reactions: $3NaF + ScF_3 \rightarrow Na_3ScF_6$; $Na_3ScF_6 + Al \rightarrow Na_3AlF_6 + Sc$; and $3Al + Sc \rightarrow Al_3Sc$. For Test 1, the amount of KCl was 75% of the total weight of flux added. In each test, the temperature was in the range of from 850 °C to 930 °C, and the melting time was 20 minutes.

Table 1

	Sc compound	Flux	Target concentration, %	Actual Sc assay in the alloy, %	% Sc reduction
Test 1	Sc_2O_3	NaF + KCl	1.05%	0.47%	45%
Test 2	ScF_3	NaF	1.23%	1.18%	96%

[0037] Another disadvantage associated with Sc_2O_3 is that, compared to the reduction of Sc with NaF, more and/or multiple flux compounds may be needed to reduce Sc_2O_3. The more such flux compounds are added, the more slags are generated, and this may cause higher loss of metals to slags. Moreover, the use of flux may be generally undesirable because it may require pre-fusing flux before adding it to molten Al which would require additional steps and process cost. If desired, a process in accordance with the present disclosure may be performed without pre-fusion of flux.

[0038] Typically, ScF_3 is prepared by directly reacting Sc_2O_3 with hydrofluoric acid or contacting it with hot hydrogen fluoride gas at high temperature, both of which present challenges in safety and operations due to their high toxicity. To significantly minimize the associated risks, a process in accordance with the present disclosure involves preparing ScF_3 by dissolving Sc_2O_3 in hydrochloric acid, and then stoichiometrically precipitating using NaF.

[0039] If desired, a method in accordance with the present disclosure may involve the removal of O, N, and H. If desired, a suitable vacuum-induction furnace (not illustrated in the drawings) may be used to extract such gases. If desired, the present disclosure may be implemented without generating any gases, especially no toxic gases.

[0040] According to one aspect of the present disclosure, a target chemical composition of a final alloy includes Sc, magnesium (Mg), and zirconium (Zr) in the amounts shown in Table 2.

Table 2

Element	Weight %
Al	Balance
Sc	0.20-0.40
Mg	4.00-6.00
Zr	0.10-0.30

[0041] As used herein, the word "about" qualifies the associated value by plus or minus 10%. For example, in the present disclosure, "about" 100 units means greater than or equal to 90 units and less than or equal to 110 units.

[0042] The present disclosure is not limited to the examples described herein or illustrated in the drawings. Except to the extent a feature is recited in the following claims, the present disclosure relates to a variety of systems, methods, and products in addition to the ones described herein

Claims

1. A method of producing an aluminum-scandium final alloy product, comprising:

- an aluminothermic scandium-reduction process; and
- an alloying process with one or more other metals or master alloys;

wherein the aluminothermic scandium-reduction process and the alloying process are performed in a single stage, without generating or remelting an Al-Sc master alloy with the one or more other metals or master alloys.

2. The method of claim 1, wherein the aluminothermic scandium-reduction process includes reduction of a scandium halide.

3. The method of claim 2, wherein the scandium halide includes scandium fluoride (ScF_3).

4. The method of claim 1, wherein the aluminothermic scandium-reduction process is performed in the presence of a flux including an alkali halide salt.

5. The method of claim 4, wherein the alkali halide salt includes sodium fluoride (NaF).

6. The method of claim 1, further comprising casting an aluminum-scandium alloy,
wherein the casting occurs after the alloying process.

7. The method of claim 6, further comprising machining a cast aluminum-scandium alloy, wherein the machining occurs after the casting.

8. The method of claim 7, further comprising packaging the aluminum-scandium final alloy product, wherein the packaging occurs after the machining.

9. A method of producing a final alloy product from material, comprising:

- melting the material; and
- alloying the material;
wherein the melting and alloying are performed in a single stage;
wherein the method does not include remelting an alloy produced by an aluminothermic reduction process.

10. The method of claim 9, wherein the final alloy product is an aluminum-scandium final alloy product, and wherein the aluminum-scandium final alloy product has a scandium concentration of less than 2%.

11. The method of claim 10, comprising an aluminothermic scandium-reduction process.

12. The method of claim 11, wherein the aluminothermic scandium-reduction process includes reduction of a scandium halide.

13. The method of claim 12, wherein the scandium halide includes scandium fluoride (ScF_3).

14. The method of claim 11, wherein the aluminothermic scandium-reduction process is performed in the presence of a flux including an alkali halide salt.

15. The method of claim 14, wherein the alkali halide salt includes sodium fluoride (NaF).

16. A method for producing a scandium-aluminum alloy, comprising:

- a first step which includes producing a first melt by performing an aluminothermic scandium-reduction process and simultaneously melting and alloying a first metal or master alloy having a first volatilization temperature; and
- a second step which includes adding, to the first melt, a second metal or master alloy having a second volatilization temperature, wherein the second volatilization temperature is less than the first volatilization temperature, and wherein the first step is performed before the second step;

wherein the first melt is produced at a first melt temperature, and the second step is performed at a second melt temperature, wherein the second melt temperature is less than the first melt temperature, and wherein the temperature of the first melt is not substantially less than the second melt temperature between the first step and the second step.

17. The method of claim 16, wherein the first step includes an aluminothermic scandium-reduction process.

18. The method of claim 17, wherein the aluminothermic scandium-reduction process includes reduction of a scandium halide.

19. The method of claim 18, wherein the scandium halide includes scandium fluoride (ScF_3).

20. The method of claim 17, wherein the aluminothermic scandium-reduction process is performed in the presence of a flux including an alkali halide salt.

21. The method of claim 20, wherein the alkali halide salt includes sodium fluoride (NaF).

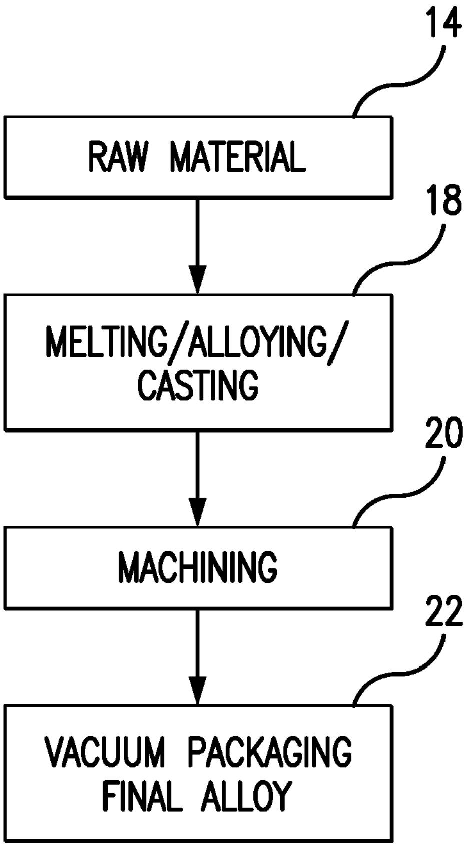
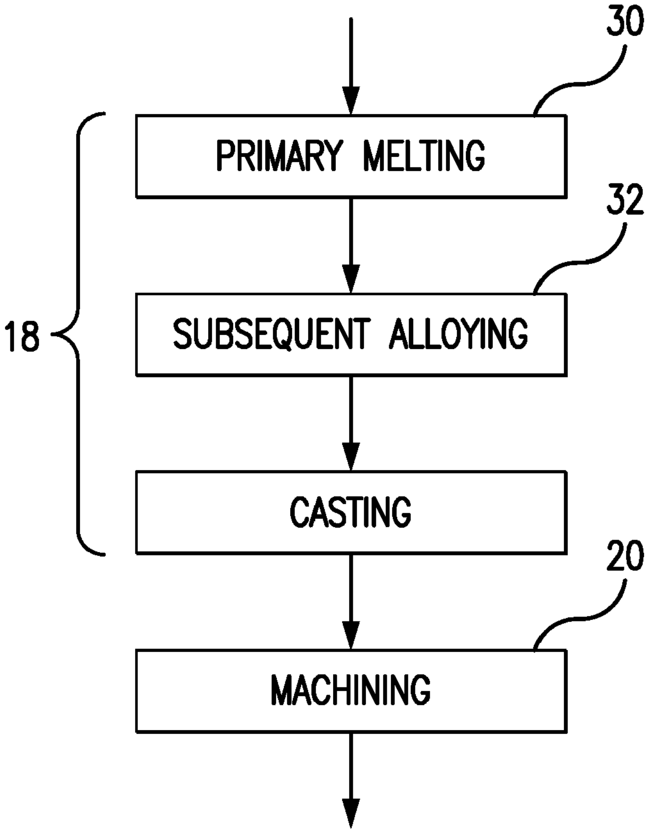

14
RAW MATERIAL
18
MELTING/ALLOYING/ CASTING
20
MACHINING
22
VACUUM PACKAGING FINAL ALLOY

FIG. 1


30
PRIMARY MELTING
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SUBSEQUENT ALLOYING
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CASTING
20
MACHINING

FIG. 2

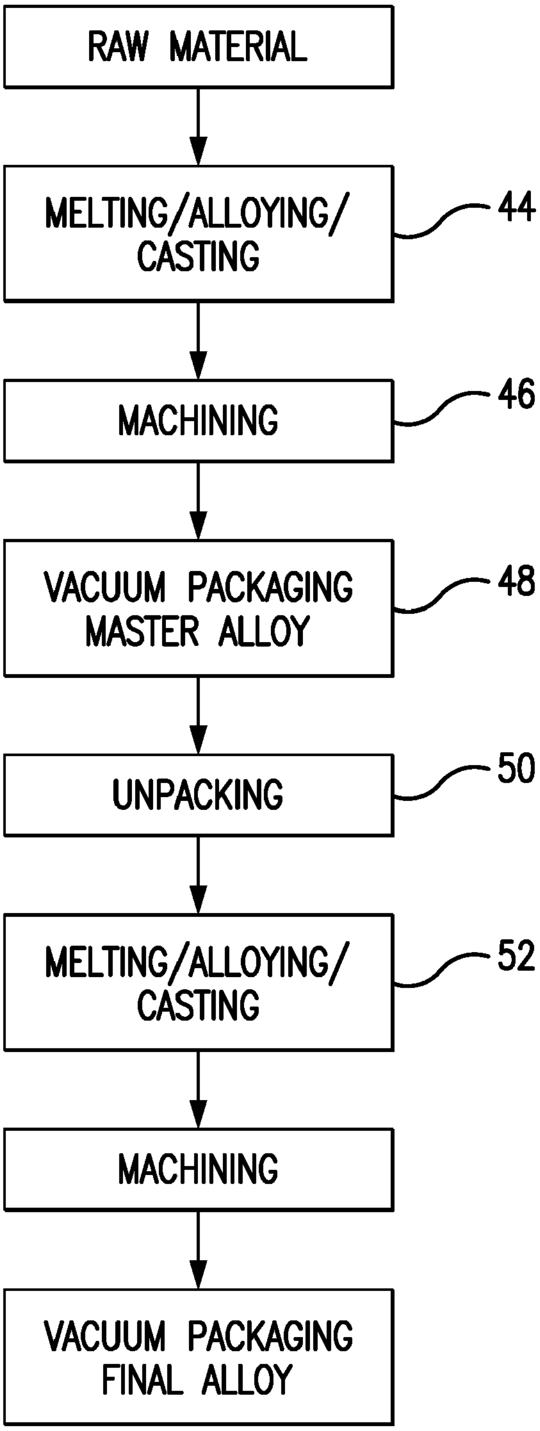
RAW MATERIAL
MELTING/ALLOYING/ CASTING
44
MACHINING
46
VACUUM PACKAGING MASTER ALLOY
48
UNPACKING
50
MELTING/ALLOYING/ CASTING
52
MACHINING
VACUUM PACKAGING FINAL ALLOY

FIG. 3

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EUROPEAN SEARCH REPORT

Application Number
EP 23 19 4282

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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The present search report has been drawn up for all claims

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EPO FORM 1503 03.82 (P04C01)

Place of search	Date of completion of the search	Examiner
The Hague	30 January 2024	Kim Lee, Bij Na

CATEGORY OF CITED DOCUMENTS

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EUROPEAN SEARCH REPORT

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DOCUMENTS CONSIDERED TO BE RELEVANT

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	30 January 2024	Kim Lee, Bij Na

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D : document cited in the application
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&: member of the same patent family, corresponding document

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DOCUMENTS CONSIDERED TO BE RELEVANT

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Place of search	Date of completion of the search	Examiner
The Hague	30 January 2024	Kim Lee, Bij Na

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