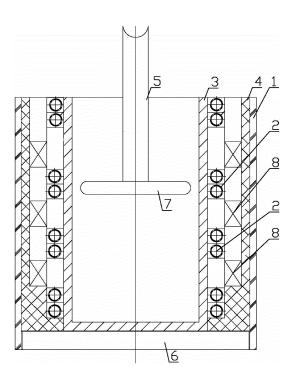
(19)	Europäisches Patentamt European Patent Office Office européen des brevets		(11) EP 2 743 359 A1						
(12)) EUROPEAN PATENT APPLICATION published in accordance with Art. 153(4) EPC								
(43)	Date of publication: 18.06.2014 Bulletin 2014/25	(51)	Int Cl.: C22B 21/06 ^(2006.01) C22B 9/02 ^(2006.01)						
(21)	Application number: 11870784.3	(86)	International application number: PCT/CN2011/083992						
(22)	Date of filing: 14.12.2011	(87)	International publication number: WO 2013/020339 (14.02.2013 Gazette 2013/07)						
(84)	Designated Contracting States: AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR	•	NURGUL, Imin Urumqi Xinjiang 830013 (CN)						
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(54) METHOD FOR PURIFYING HIGH-PURITY ALUMINIUM BY DIRECTIONAL SOLIDIFICATION AND SMELTING FURNACE THEREFOR

(57) Provided is a method for preparing high-purity aluminum by directional solidification, comprising the steps of: providing 4N to 5N aluminum as raw material, heating the same to a temperature of 670 °C to 730 °C, maintaining the temperature for 7 minutes to 80 minutes, cooling the bottom of chamber (3) to allow the aluminum liquid crystallizing in a direction from the bottom to top of the chamber (3) for 1 hour to 8 hours to obtain a crystalline ingot, during the crystallization process of a finished product of crystalline ingot, stirring and heating the aluminum liquid, maintaining a particular temperature gradient of the aluminum liquid, and removing a portion of the crystalline ingot from one end of the ingot, the remaining portion being the high-purity aluminum. Also provided is a smelting furnace, comprising a shell (1), a heating device (2), a chamber (3), a temperature measurement device, a stirring device and a cooling device (6).



Printed by Jouve, 75001 PARIS (FR)

Description

Technical field

[0001] The present invention belongs to the technical field of smelting purification of high-purity aluminium. It relates to a method for preparing high-purity aluminium by directional solidification and to a smelting furnace therefor, particularly to a method for preparing high-purity aluminium (that is, 5N to 6N aluminium) by directional solidification and to a smelting furnace therefor.

Background of the invention

[0002] The 5N to 6N aluminium is useful and needed in the electronics industry, especially in the high-tech fields such as the fields of photical storage media, semiconductor devices, superconducting cables, and so on. The purity of the so-called high-purity aluminium can be defined in two ways. The purity is either directly defined by the aluminium content or the industry standard grade, such as 99.95%, 99.99%, AL99.993A%; or is defined by "digital + N" or "digital + N + digital ". For example, 4N represents a purity of 99.99%, 4N6 represents a purity of 99.996%. Unless particularly specified, the aluminium purity described hereinafter in the present description is normally measured and specified in accordance with the specification in the China Non-ferrous Metals Industry Standard "fine aluminium ingots for remelting YS/T665-2009".

[0003] At present, the common purifying methods are as follows: three-layer liquid electrolysis refining method, organic solution electrolysis method and segregation method. These methods are employed in most of the countries in the world to prepare high-purity aluminium in a variety of purities. The purity of the aluminium extracted with the three-layer liquid electrolytic refining method is usually from 4N to 4N8, but the power consumption is usually greater than 13000kwh/t, which is about 4 to 5 times of that of the segregation method. On one hand, it has been very difficult to reduce the cost, on the other hand, the environment is seriously polluted by the harmful gases such as hydrogen fluoride, carbon monoxide, sulfur dioxide, etc., and the waste electrolyte generated during the electrolysis process. Therefore, it is not feasible to promote the application of the threelayer liquid electrolysis refining method. Due to the higher energy consumption, lower output and complexity of the process, the organic solution electrolysis method is generally used for production of a small amount of ultra-high purity aluminium of over 7N, and thus it is not suitable for an industrial production.

[0004] The segregation method, which is a kind of physical purification methods, is more and more widely used because it requires a low workload and brings in no chemical reaction pollution. The segregation method may be implemented by a variety of processes including fractional crystallization process, zone melting process,

and directional solidification process. The fractional crystallization process has been widely applied in the industry of aluminium purification. The purification effect of the process depends on the purity of the primary aluminium (the starting materials) used in the process, and the aluminium raw material having a purity of 99.5% to 99.95% is normally used and purified in the process to prepare 3N5 to 4N5 aluminium. But, the production efficiency is low, and the residual aluminium liquid exerts pollutions

¹⁰ to the resulted high purity aluminium. Moreover, the processes and equipments are complex, and the purity grade of the obtained high purity aluminium is limited. The zone melting process can produce 5N5 to 6N aluminium, and is mainly used for further purification of high-purity alu-

¹⁵ minium produced by the three-layer liquid electrolysis refining method or the segregation method. However, the equipments to carry out the zone melting process are complex too, the production efficiency is low, and the energy consumption is relatively high. So, it is not suitable ²⁰ for industrial applications.

[0005] Directional solidification methods can further be classified into pipe-cooling solidification method, bottom-cooling method, sidewall-cooling method, vertical gradient-cooling method, and lateral solidification method, etc.

²⁵ But, these conventionally existed directional solidification methods have the following defects: the energy consumption and cost of the methods are high, the yields of the methods are low, and the product prepared has a low purity and small size, which make it difficult for an indus-³⁰ trial mass production.

Summary of the invention

[0006] In order to solve the problems and to overcome
the defects, such as the low productivity, high energy consumption and high cost in traditional directional so-lidification purification of high-purity aluminium, the present invention is to provide a method for preparing high-purity aluminum by directional solidification with a
⁴⁰ high efficiency and productivity, low energy consumption, and low cost. An aluminium product of high purity can be prepared by the method of the present invention.
[0007] The technical solution adopted to solve the

technical problems faced by the present invention is a method for preparing high-purity aluminium by directional solidification, comprising the following steps:

Step one (step of selecting material): providing 4N to 5N aluminum as raw material, and cleaning the surface of the aluminum raw material;

Step two (step of melting): feeding the aluminum raw material from Step one into a chamber of a smelting furnace wherein the aluminum raw material is heated to a temperature of 670 °C to 730 °C, so that the aluminum raw material is completely melted to form an aluminum liquid;

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Step three (step of maintaining temperature): maintaining the aluminum liquid from Step two at the temperature of 670 °C to 730 °C for 7 minutes to 80 minutes;

Step four (step of purifying by solidification): cooling the bottom of the chamber to allow the aluminum liquid crystallizing in a direction from the bottom to top of the chamber for 1 hour to 8 hours to obtain a crystalline ingot, from which a finished crystalline ingot product (hereinafter also referred to as "finished product") is to be prepared by removing a portion of the ingot from the ingot end where the crystallization is lastly completed, and wherein at least during the crystallization process for forming the ingot part which corresponds to the finished product, a mechanical stirring and/or electromagnetic stirring is applied to the aluminum liquid when the aluminum liquid is heated, maintaining the crystal plane of the aluminum liquid at a temperature of 655 °C to 665 °C and the liquid surface of the aluminum liquid at a temperature of 695 °C to 705 °C, and the temperature of the aluminum liquid increase gradually from the crystal plane to the liquid surface;

Step five (step of obtaining a finished product): removing a portion of the crystalline ingot from the ingot end where the crystallization is lastly completed, wherein the ingot portion to be removed depends on the desired purity of the crystalline ingot product and ranges from 15% to 70% of the thickness of the entire crystalline ingot, and the remaining part of the crystalline ingot is the finished product, i.e. a high-purity aluminum product with the desired purity.

[0008] The term "the ingot part corresponding to the finished product" refers to the crystalline ingot portion to be formed as the finished high-purity aluminium product, i.e. the remaining part after removal of a portion constituting 15% to 70% of the entire crystalline ingot in Step five. In other words, the remaining part is the crystalline ingot part formed by crystallization of the aluminium liquid from the beginning till 30% to 85% of the crystallization completed. During the crystallization process of this part of the crystalline ingot, stirring and heating is needed for the aluminium liquid, and a temperature gradient of the aluminium liquid is maintained. During the subsequent crystallization process, stirring and heating is unnecessary but is tolerable (because this portion of the crystal ingot will be removed later and thus will not be a part of the finished high-purity aluminium product).

[0009] In the method for preparing high-purity aluminium by directional solidification of the present invention, the crystallization process is stable and controllable, the energy consumption and costs are reduced and the productivity and efficiency are improved by selecting an appropriate crystallization temperature gradient, crystallization speed and other parameters. Moreover, the aluminium liquid is stirred during the crystallization process to reduce the thickness of the impurity-enriched layer at the interface of the crystallization. The stirring makes the impurity at the interface of the crystallization dissociate from the crystalline interface and diffuse upwardly into

- the upper portion of the aluminium liquid. These measures of the present invention ensure that the finished product of the crystalline ingot has a high purity and the proportion of the ingot part which corresponds to the fin-
- ¹⁰ ished product in relation to the entire crystalline ingot is large. Thus, finished products of aluminium with large sizes and high purities can be obtained. The purities of thus prepared products may be up to 5N to 6N.

[0010] Preferably, in Step one, the surface of the above-mentioned aluminium raw material is cleaned by a physical cleaning process, followed by a chemical cleaning process to remove the oxide film on the surface of the aluminium raw material.

[0011] Preferably, in Step four, the aluminium liquid is mechanically stirred by a dried and preheated stirring blade, and the distance between the stirring blade and the crystal plane is 10 mm to 50 mm; and/or an electromagnetic stirring is applied to the aluminum liquid wherein the distance between the layer stirred by electromag-

netic stirring and the crystal plane is 10 mm to 50 mm.
[0012] With respect to the problems of low efficiency, high energy consumption and high cost of the method of preparing high-purity aluminium by traditional directional solidification methods, the present invention also provides a smelting furnace to prepare a high-purity aluminium product with large productivity, low energy consumption, and low cost through directional solidification.

[0013] The technical solution to solve the above-mentioned problems is a smelting furnace comprising a shell,

³⁵ a heating device, a chamber, a temperature measuring device, a stirring device and a cooling device. The smelting furnace is useful for implementing the method of the present invention for preparing high-purity aluminium by directional solidification. In the melting furnace:

40 **[0014]** The chamber is installed in the shell;

[0015] The heating device is installed between the shell and the chamber, and when one heating device is presented, it is arranged at the upper part of the chamber, when more than one heating devices are presented, they

⁴⁵ are arranged at an interval in a direction from the upper to the lower part of the chamber;

[0016] Cooling device is arranged under the chamber at the bottom of the furnace;

[0017] Stirring device comprises a mechanical stirring device and/or an electromagnetic stirring device;

[0018] Temperature measurement device comprises a hearth temperature sensoring device and a chamber temperature sensoring device, the hearth temperature sensoring device is arranged between the cooling device
⁵⁵ and the shell, and the chamber temperature sensoring device is used for measuring the chamber temperature at different positions along the height direction of the chamber.

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[0019] The smelting furnace of the present invention has a compact arrangement and a rational structure. It is simple to use and is useful for implementing the abovementioned method for preparing high-purity aluminium by directional solidification. The smelting furnace can be used to prepare high-purity aluminium by the coordinative combination of the heating device, the cooling device, the temperature measuring device, and the stirring device. Therefore, the power consumption and cost are low, while the product yield and purity are high.

[0020] Preferably, the stirring device comprises a mechanical stirring device; wherein the blade of the mechanical stirring device is arranged in the lower part of the mechanical stirring device, and is able to ascend or descend along the height direction of the stirring device.

[0021] Preferably, the stirring device comprises an electromagnetic stirring device, which is arranged between the shell and the heating device, and is arranged in a dislocation manner with the heating device in the height direction of the smelting furnace.

[0022] Preferably, a thermal insulation layer is arranged inside the shell and outside the heating device. [0023] Further preferably, the stirring device comprises an electromagnetic stirring device, which is arranged between the thermal insulation layer and the heating device, and is arranged in a dislocation manner with the heating device along the height direction of the smelting furnace.

[0024] Preferably, the chamber temperature sensoring device comprises several temperature sensors distributed at the outside of the chamber along the height direction of the chamber.

[0025] Preferably, the heating device is an electric heating device.

[0026] The present invention is especially suitable for large-scale preparation of high-purity aluminium with a purity of 5N to 6N.

Description of the accompanying drawings

[0027] Figure 1 is a schematic diagram of the structure of the smelting furnace used in the method for preparing high-purity aluminium by directional solidification according to an example of the present invention.

[0028] The reference numerals presented in the figure are as follows: 1. a shell; 2. a heating device; 3. a chamber; 4. a thermal insulation layer; 5. a mechanical stirring device; 6. a cooling device; 7. a stirring blade; 8. an electromagnetic stirring device.

[0029] For the convenience of illustration, the positional relationship, such as upper part, lower part, top, bottom, inside, outside, of the components of the smelting furnace is described with reference to the arrangement of the components shown in the accompanying drawings of the description.

Specific embodiments

[0030] In order to enable those skilled in the art to have a better understanding of the technical solutions of the present invention, the present invention is described in further detail below with reference to the accompanying drawings and specific embodiments.

Example 1:

[0031] The present example provides a method for preparing high-purity aluminium by directional solidification, comprising the following steps:

[0032] Step one (step of selecting material): providing 4N to 5N aluminium as raw material, and cleaning the surface of the aluminium raw material. The dust, impurities, oxide films etc. on the surface of the aluminium raw material are removed through dusting, cleaning, chemical etching etc., to improve the purity of the raw material,

20 reduce the amount of impurities to be introduced into the smelting process, and thus improve the purity of the product as much as possible.

[0033] Preferably, the above-mentioned step of cleaning the surface of the aluminium raw material includes:

the surface of the aluminum raw material is cleaned by a physical cleaning process (e.g., dusting, cleaning and other non-chemical means), and then a chemical cleaning process (e.g., chemical etching, etc.) is applied to remove the oxide film from the surface of the aluminium raw material.

[0034] Step two (step of melting): feeding the aluminium raw material from the Step one into a chamber of a smelting furnace wherein the aluminium raw material is heated to a temperature of 670 °C to 730 °C, so that the aluminum raw material is completely melted to form an aluminium liquid. That is to say, the aluminium raw material is melted in the chamber for purification to form the aluminium into a molten state.

[0035] Step three (step of maintaining temperature): maintaining the aluminium liquid from Step two at the temperature of 670 °C to 730 °C for 7 minutes to 80 minutes. That is to say, the melted aluminium raw material is thermally insulated for a period of time, so that the melting can be thoroughly, and the temperature and com-

⁴⁵ position, etc. of the aluminium liquid can be more uniform and stable.

[0036] Step four (step of purifying by solidification): cooling the bottom of the chamber to allow the aluminium liquid crystallizing in a direction from the bottom to top of the chamber for 1 hour to 8 hours to obtain a crystalline ingot, from which a finished product is to be prepared by removing a portion of the ingot from the ingot end where the crystallization is final completed, and wherein at least during the crystallization process for forming the ingot 55 part which corresponds to the finished product, a mechanical stirring and/or electromagnetic stirring is applied to the aluminium liquid, and at the same time the aluminium liquid is heated, maintaining the crystal plane of the

aluminium liquid i.e., the lower surface of the aluminium liquid, at a temperature of 655 °C (because high-purity metal liquid has a larger degree of suppercooling, the liquid temperature can be below the freezing point) to 665 °C, and maintaining the liquid surface of the aluminium liquid ,i.e., the upper surface of the aluminium liquid, at a temperature of 695 °C to 705 °C, and temperature of the aluminium liquid increasing gradually from the crystal plane to the liquid surface. Cooling the bottom of the chamber triggers crystallization of the aluminium liquid at the bottom of the chamber, and the crystal plane is gradually moved upwardly. As a result, a crystalline ingot is formed. Preferably, during the crystallization process for forming the ingot part which corresponds to the finished product (i.e., the crystalline ingot part obtained by the crystallization from the beginning till 30% to 85% of the crystallization completed), the aluminium liquid is stirred to reduce the thickness of the impurity-enriched layer at the interface of the crystallization; and meanwhile, the aluminium liquid is heated to maintain an appropriate temperature gradient of the aluminium liquid. For example, the temperature of the lowermost layer of the aluminium liquid is kept at 655 °C to 665 °C (i.e., a temperature close to the melting point of aluminium), and the uppermost layer of the aluminium liquid is kept at 695 °C to 705 °C, thus ensuring the crystallization process to proceed stably. The temperature gradient can be maintained by adjusting the degree of cooling and heating. Because the accuracy of temperature control is limited, the temperature of the lowermost layer and the uppermost layer of the aluminium liquid may fluctuate within a certain range. If the temperature can be precisely controlled, it is also feasible to maintain the temperature at a precise point. With the proceeding of crystallization, the thickness of the aluminium liquid layer reduces. The temperature of the uppermost layer of the aluminium liquid may be gradually decreased to be close to the temperature of the crystallization surface. The temperature of the crystallization surface cannot deviate from the solidification point too much. With the attenuation of thickness of the aluminium liquid layer, it is very difficult to maintain a big temperature gradient. So, the temperature of the uppermost layer of the aluminium liquid may be lowered. After the crystallization process for forming the ingot part which corresponds to the finished product is completed (i.e., when the crystallization begins to form the ingot portion which is to be removed later in Step five), stirring of the aluminium liquid may be stopped and the maintenance of the temperature gradient is no longer needed. In fact, when the crystallization proceeds to the final stage, the aluminium liquid is very thin, and thus it is difficult to continue stirring and maintaining the temperature gradient.

[0037] Preferably, the aluminium liquid is stirred by a dried and preheated stirring blade during the mechanical stirring, and the distance between the stirring blade and the crystal plane is controlled at 10 mm to 50 mm; and if the aluminium liquid is stirred by electromagnetic stirring,

the distance between the layer where the electromagnetic stirring is applied and the crystal plane is controlled at 10 mm to 50 mm. That is to say, the mechanical stirring is carried out by a stirring blade, and the stirring blade is also dried and preheated (the preheating temperature may be or approximate to the temperature of the aluminium liquid) to avoid an adverse impact on the crystallization process. When implementing mechanical stirring and/or electromagnetic stirring, the distances between

10 the stirring layer (i.e. stirring blade for mechanical stirring) and the crystal plane should be maintained at 10 mm to 50 mm in order to achieve the optimal effect of stirring. Because the crystal plane is gradually going upwardly, the above-mentioned distance may be adjusted through

¹⁵ elevating or descending the stirring blade and/or selectively turning on the electromagnetic stirring coils at different positions (or elevating or descending the electromagnetic stirring coils).

[0038] Step five (step of obtaining a finished product):
removing a portion of the crystalline ingot from the ingot end where the crystallization is lastly completed, wherein the ingot portion to be removed depends on the desired purity of the crystalline ingot product to be obtained and ranges from 15% to 70% of the thickness of the entire
crystalline ingot. The remaining portion of the crystalline ingot is the finished product crystalline ingot product, i.e., a high-purity aluminium product with the desired purity (5N to 6N). According to the principle of directional solidification, it is known that in the crystal ingot derived from this method, the portion formed at an earlier stage

- o from this method, the portion formed at an earlier stage of the crystallization process has a higher purity, and the portion formed at a later stage of the crystallization process has a lower purity due to an enrichment of impurities. Therefore, in this step, to meet the purity requirements,
- the ingot portion formed at the later stage of the crystal-lization process is truncated (this portion can be used in other fields) from the crystal ingot. The remaining part (i.e. a finished product of the crystalline ingot) is the desired finished product of high-purity aluminium. Of
 course, the purity of the finished product can also be determined, and if the purity does not meet the require
 - ments, the crystalline ingot can either be further truncated, or recycled as raw materials for the present purifying method of directional solidification, so as to be re-purified.
- ⁴⁵ [0039] Obviously, it should be known that the process for preparing high-purity aluminium by directional solidification of the present example can be carried out in vacuum or under a protective atmosphere, in order to reduce the oxidation of the aluminium liquid.

50 [0040] In the method for preparing high-purity aluminium by directional solidification of the present example, the crystallization process is stable and controllable, the energy consumption and costs are reduced and the productivity and efficiency are improved by selecting an appropriate crystallization temperature gradient, crystallization speed and other parameters. Moreover, the aluminium liquid is stirred during the crystallization process to reduce the thickness of the impurity-enriched layer at the interface of the crystallization. The stirring makes the impurity at the interface of the crystallization dissociate from the crystalline interface and diffuse upwardly into the upper portion of the aluminium liquid. These measures of the present invention ensure that the finished product of the crystalline ingot has a high purity and the proportion of the ingot part which corresponds to the finished product in relation to the entire crystalline ingot is large. Thus, finished products of aluminium with large sizes and high purities can be obtained. The purities of thus prepared products may be up to 5N to 6N.

Example 2:

[0041] The present example provides a method for preparing high-purity aluminium by directional solidification, comprising the following steps:

Step one, providing 4N aluminium as raw material, and physically cleaning the surface of the aluminium ²⁰ raw material;

Step two, feeding the aluminium raw material into the chamber of a smelting furnace, wherein the aluminium raw material is heated to a temperature of 670 °C so that the aluminium raw material is completely melted to form an aluminium liquid;

Step three, maintaining the aluminium liquid at a temperature of 670 °C for 80 minutes.

Step four, cooling the bottom of the chamber to allow the aluminium liquid crystallizing in a direction from the bottom to top of the chamber for 1 hour to obtain a crystalline ingot; during the crystallization process for forming the ingot part which corresponds to a finished product, a mechanical stirring and electromagnetic stirring being applied to the aluminium liguid, and at the same time the aluminium liquid being heated, maintaining the crystal plane of the aluminum liquid at a temperature of 655 °C to 660 °C and the liquid surface of the aluminum liquid at a temperature of 700 °C to 705 °C, and temperature of the aluminium liquid increasing gradually from the crystal plane to the liquid surface, wherein, the mechanical stirring is implemented through a dried and preheated stirring blade, and the distance between the stirring blade and the crystal plane is controlled at 10 mm, while the distance between the layer stirred by the electromagnetic stirring and the crystal plane is controlled at 10 mm to 20 mm (because the adjustment of the layer stirred by electromagnetic stirring is mainly achieved by turning on different electromagnetic stirring coils, and the arrangement density of the electromagnetic stirring coils is limited, it is difficult to maintain a precise distance between the stirring layer and the crystal plane, instead, the distance is usually in a certain range);

Step five, removing 15% of the thickness of the entire crystalline ingot from the ingot end where the crystallization is lastly completed; the remaining part of the crystalline ingot is a finished product of the crystalline ingot, i.e., a high-purity finished product of 5N aluminium.

Example 3:

- ¹⁰ **[0042]** The present example provides a method for preparing high-purity aluminium by directional solidification, and it differs from example 2 in the following aspects:
 - In step one, 5N aluminium is provided as raw material, and after physical cleaning of the surface of the aluminium raw material, chemical cleaning is subsequently carried out to remove the oxide film from the surface of the aluminium raw material.
 - In step two, the aluminium raw material is heated to a temperature of 730 °C.

In step three, the aluminium liquid is maintained at the temperature of 730 $^\circ\text{C}$ for 7 minutes.

In step four, a crystalline ingot is obtained after 8 hours of cooling; during the crystallization process for forming the ingot part which corresponds to a finished product, only a mechanical stirring is applied to the aluminium liquid, and the distance between the stirring blade and the crystal plane is 50 mm. The temperature of the crystal plane of the aluminium liquid is maintained at 660 °C and the temperature of the liquid surface of the aluminium liquid is maintained at 695 °C to 700 °C.

In step five, 70% of the thickness of the entire crystalline ingot is removed from the ingot end where the crystallization is lastly completed. A high-purity finished product, 6N aluminium is thus obtained.

Example 4:

[0043] The present example provides a method for pre ⁴⁵ paring high-purity aluminium by directional solidification, and it differs from example 2 in the following aspects:

In step one, 4N5 aluminium is provided as raw material.

In step two, the aluminium raw material is heated to a temperature of 700 °C.

In step three, the aluminium liquid is maintained at the temperature of 710 °C for 40 minutes.

In step four, a crystalline ingot is obtained after 4 hours of cooling; during the crystallization process

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for forming the ingot part which corresponds to a finished product, only an electromagnetic stirring is applied to the aluminium liquid, the distance between the layer stirred by electromagnetic stirring and the crystal plane is 30 mm (electromagnetic stirring coils which is able to ascend or descend may be employed). The temperature of the crystal plane of the aluminium liquid is maintained at 660 °C to 665 °C and the temperature of the liquid surface of the aluminium liquid is maintained at 698 °C to 702 °C.

In step five, 40% of the thickness of the entire crystalline ingot is removed from the ingot end where the crystallization is lastly completed. A high-purity finished product, 5N4 aluminium, is thus obtained.

Example 5:

[0044] The present example provides a method for preparing high-purity aluminium by directional solidification, and it differs from example 2 in the following aspects:

In step one, 4N8 aluminium is provided as raw material.

In step two, the aluminium raw material is heated to a temperature of 705 °C.

In step three, the aluminium liquid is maintained at a temperature of 705 °C for 60 minutes.

In step four, a crystalline ingot is obtained after 6 hours of cooling; during the crystallization process for forming the ingot part which corresponds to the finished product, the distance between the stirring blade and the crystal plane is 30 mm, and the distance between the layer stirred by electromagnetic stirring and the crystal plane is 40 mm to 50 mm. The temperature of the crystal plane of the aluminium liquid is maintained at 658 °C to 662 °C and the temperature of the liquid surface of the aluminium liquid is maintained at 700 °C.

In step five, 50% of the thickness of the entire crystalline ingot is removed from the ingot end where the crystallization is lastly completed. A high-purity finished product, 5N6 aluminium is thus obtained.

Example 6:

[0045] As shown in Figure 1, the present example provides a smelting furnace used in the above-mentioned method for preparing high-purity aluminium by directional solidification, comprising: shell 1, heating device 2, chamber 3, a temperature measuring device (not shown in the figure), stirring device 5 and cooling device 6. [0046] In the smelting furnace, chamber 3 is mounted in the shell 1; the aluminium liquid is crystallized inside the chamber.

[0047] At least one heating device 2 is arranged in the smelting furnace, which is arranged between the shell 1 and chamber 3. When one heating device 2 is presented,

⁵ it is arranged in a position at the upper part of the chamber 3, and when several heating devices 2 are presented, they are arranged at an interval in a direction from the upper to the lower part of the chamber 3. That is to say, in order to maintain the above-mentioned temperature

¹⁰ gradient in the crystallization process, if there is only one heating device 2, it should be arranged in the upper part of chamber 3, and if there is a plurality of heating devices 2, they are arranged at an interval in a direction from the upper to the lower part of the chamber 3 (i.e. preferably

¹⁵ arranged in the upper part of chamber 3), and there should be a space between adjacent heating devices 2. During the process of crystallization, by adjusting the temperatures of cooling device 6 and heating device(s) 2, the above-said temperature gradient can be main²⁰ tained.

[0048] Preferably, the heating device 2 is electric heating device 2.

[0049] The cooling device 6 is arranged under the chamber 3 at the bottom of the furnace for cooling the bottom of chamber 3, so that the aluminium melt can crystallize along a direction from the bottom to top of the chamber.

[0050] Preferably, a thermal insulation layer 4 is arranged inside the shell 1 and outside the heating device 2 for a purpose of improving the thermal insulation effect of chamber 3, reducing power consumption, and accurately maintaining temperature.

[0051] The stirring device includes mechanical stirring device 5 and/or electromagnetic stirring device 8; they are used for stirring the aluminium melt.

[0052] Preferably, when the stirring device comprises mechanical stirring device 5, stirring blade 7 of mechanical stirring device 5 is arranged in the lower part of mechanical stirring device 5, and is able to ascend or de-

40 scend along the height direction of the stirring device. That is to say, the upper end of stirring blade 7 can be connected to a bracket (not shown in the figure) and the bracket is connected to a lifting device (not shown in the figure), so that the stirring blade 7 is able to ascend or

⁴⁵ descend in order to maintain the above-mentioned distance between the stirring blade and the crystal plane. There are various configurations of the mechanical stirring device 5, which is not necessarily described in detail herein.

50 [0053] Preferably, when the stirring device comprises electromagnetic stirring device 8, the electromagnetic stirring device 8 is arranged between shell 1 and heating device 2 or between insulation layer 4 and heating device 2, and is set in a dislocation manner with heating device 55
 51 in the height direction of the smelting furnace. That is to say, when insulation layer 4 is not presented, electromagnetic stirring device 8 (mainly electromagnetic stirring coils) is arranged between shell 1 and heating device

2, and when insulation layer 4 is presented, electromagnetic stirring device 8 is arranged between insulation layer 4 and heating device 2. Electromagnetic stirring device 8 is always set at a position different from the position of heating device 2 along the height direction of the smelting furnace. In this way of arrangement, electromagnetic stirring device 8 is apart and far from heating device 2, and thus is not affected by heating device 2. Since the electromagnetic stirring device is arranged at a different position in height from the heating device 2, the electromagnetic fields generated thereby will not pass through heating device 2 and can be applied directly onto the aluminium liquid. Thus, an excellent stirring effect can be achieved. In the crystallization process, with the upward move of the crystal plane, different electromagnetic stirring devices 8 (i.e., electromagnetic stirring coils) are turned on sequentially, the above-mentioned distance between the stirring layer and the crystal plane can be maintained. Of course, if only one electromagnetic stirring device 8 is presented, and if it can ascend and descend like stirring blade 7, the above-said distance between the stirring layer and the crystal plane can also be maintained.

[0054] The temperature measuring device comprises a hearth temperature sensoring device and a chamber temperature sensoring device. The hearth temperature sensoring device is installed between cooling device 6 and shell 1, and the chamber temperature sensoring device is used for measuring the temperature of chamber 3 at different positions in the height direction.

[0055] Preferably, the chamber temperature sensoring device comprises several temperature sensors (e.g., thermocouples) distributed at the outside of the chamber 3 along the height direction for measuring the temperature of chamber 3 at different heights. Of course, it is ³⁵ feasible if only one chamber temperature sensoring device is presented, as long as it is able to ascend or descend along the height direction.

[0056] During the crystallization process, heating device 2 and cooling device 6 can be adjusted according 40 to the temperature measurement results of the temperature measuring device, so as to maintain the above-said temperature gradient of the aluminium liquid.

[0057] In the smelting furnace of the present example, heating device 2, thermal insulation layer 4, mechanical 45 stirring device 5, cooling device 6, electromagnetic stirring device 8, the temperature measuring device can all employ the known devices in the filed. The smelting furnace may also include other known structures, for example, a device for maintaining a vacuum or providing a 50 protective atmosphere, etc.

[0058] The smelting furnace of the present invention has a compact arrangement and a rational structure. It is simple to use and is useful for implementing the abovementioned method for preparing high-purity aluminium by directional solidification. The smelting furnace can be used to prepare high-purity aluminium by the coordinative combination of the heating device, the cooling device, the temperature measuring device, and the stirring device. Therefore, the power consumption and cost are low, while the product yield and purity are high.

[0059] It is understood that the above-illustrated embodiments are exemplified only to illustrate the principle of the present invention. However, the present invention is not limited thereto. For those skilled in the art, without departing from the spirit and substance of the present invention, various modifications and improvements can 10 be made, and these variations and modifications also fall

within the protection scope of the present invention.

Claims

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 A method for preparing high-purity aluminum by directional solidification, characterized in that it comprises the following steps:

> step one, providing 4N to 5N aluminum as raw material, and cleaning the surface of the aluminum raw material;

step two, feeding the aluminum raw material from the step one into a chamber of a smelting furnace wherein the aluminum raw material is heated to a temperature of 670 °C to 730 °C, so that the aluminum raw material is completely melted to form an aluminum liquid;

step three, maintaining the aluminum liquid from step two at the temperature of 670 °C to 730 °C for 7 minutes to 80 minutes;

step four, cooling the bottom of the chamber to allow the aluminum liquid crystallizing in a direction from the bottom to top of the chamber for 1 hour to 8 hours to obtain a crystalline ingot, from which a finished crystalline ingot product is to be prepared by removing of a portion of the ingot from the ingot end where the crystallization is lastly completed, and wherein at least during the crystallization process for forming the ingot part which corresponds to the finished crystalline ingot product, a mechanical stirring and/or electromagnetic stirring is applied to the aluminum liquid, and at the same time the aluminum liquid is heated, maintaining the temperature of the crystal plane of the aluminum liquid at 655 °C to 665 °C and the temperature of the liquid surface of the aluminum liquid at 695 °C to 705 °C, and temperature of the aluminum liquid increasing gradually from the crystal plane to the liquid surface:

step five, removing a portion of the crystalline ingot from the ingot end where the crystallization is lastly completed, wherein the ingot portion to be removed depends on the desired purity of the crystalline ingot product to be obtained and ranges from 15% to 70% of the thickness of the entire crystalline ingot, the remaining portion of

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the crystalline ingot being the finished crystalline ingot product, i.e. a high-purity aluminum product with the desired purity.

2. The method for preparing high-purity aluminum by directional solidification as claimed in claim 1, characterized in that in step one, cleaning the surface of the aluminum raw material includes:

the surface of the aluminum raw material being cleaned by a physical cleaning process, and then

a chemical cleaning process being applied to remove the oxide film from the surface of the aluminum raw material.

3. The method for preparing high-purity aluminum by directional solidification as claimed in claim 1 or claim 2, **characterized in that**, in step four,

a mechanical stirring is applied to the aluminum liquid by a dried and preheated stirring blade, and the distance between the stirring blade and the crystal plane is 10 mm to 50 mm; and/or

an electromagnetic stirring is applied to the aluminum liquid wherein the distance between the layer stirred by electromagnetic stirring and the crystal plane is 10 mm to 50 mm.

4. A smelting furnace useful for implementing the method for preparing high-purity aluminum by directional solidification as claimed in any one of claims 1 to 3, **characterized in that** it comprises a shell, a heating device, a chamber, a temperature measurement device, a stirring device and a cooling device; wherein, the chamber is installed in the shell;

the heating device is arranged between the shell and the chamber, and when one heating device is presented, it is arranged in a position at the upper part of the chamber, when a plurality of heating devices are presented, they are arranged at an interval in a direction from the upper to the lower part of the chamber;

the cooling device is arranged under the chamber at the bottom of the furnace;

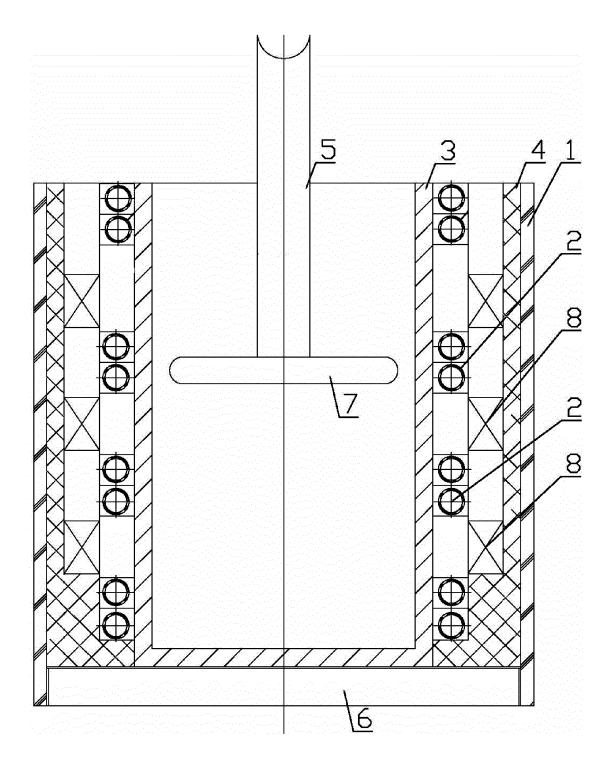
the stirring device comprises a mechanical stirring device and/or an electromagnetic stirring device; and

the temperature measurement device comprises a hearth temperature sensoring device and a chamber temperature sensoring device, the hearth temperature sensoring device is arranged between the cooling device and the shell, and the chamber temperature sensoring device is used for measuring the chamber temperature at different positions along the height direction of the chamber.

5. The smelting furnace useful for implementing the method for preparing high-purity aluminum by direc-

tional solidification as claimed in claim 4, **characterized in that** the stirring device comprises a mechanical stirring device; wherein,

- the stirring blade of the mechanical stirring device is arranged in the lower part of the mechanical stirring device, and is able to ascend or descend along the height direction of the stirring device.
- 6. The smelting furnace useful for implementing the method for preparing high-purity aluminum by directional solidification as claimed in claim 4 or claim 5, characterized in that the stirring device comprises an electromagnetic stirring device, which is arranged between the shell and the heating device, and is arranged in a dislocation manner with the heating device along the height direction of the smelting furnace.
 - 7. The smelting furnace useful for implementing the method for preparing high-purity aluminum by directional solidification as claimed claim 4 or claim 5, characterized in that a thermal insulation layer is arranged inside the shell and outside the heating device.
 - 8. The smelting furnace useful for implementing the method for preparing high-purity aluminum by directional solidification as claimed in claim 7, characterized in that the stirring device comprises an electromagnetic stirring device, the electromagnetic stirring device which is arranged between the thermal insulation layer and the heating device and is arranged in a dislocation manner with the heating device along the height direction of the smelting furnace.
 - **9.** The smelting furnace useful for implementing the method for preparing high-purity aluminum by directional solidification as claimed in claim 4 or claim 5, **characterized in that** the chamber temperature sensoring device comprises several temperature sensors distributed at the outside of the chamber along the height direction of the chamber.
 - **10.** The smelting furnace useful for implementing the method for preparing high-purity aluminum by directional solidification as claimed in claim 4 or claim 5, **characterized in that** the heating device is an electric heating device.



INTERNATIONAL SEARCH REPORT

International application No.

		Terre	CN2011/083992		
A. CLAS	SIFICATION OF SUBJECT MATTER				
According		extra sheet			
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)					
IPC: C22B Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched					
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)					
WPI, EPODOC, CNKI, CN-PAT: direct+, unidirect+, segreg+, solid+, al, alumin?um, stir+, agitate+, puri+, clean+					
C. DOCL	IMENTS CONSIDERED TO BE RELEVANT				
Category*	Citation of document, with indication, where a		Relevant to claim N		
Y	JP5-295462A (NIKKEI GIKEN KK et al.) 09 Nov.1993(09.11.1993) Column 3, paragraphs 0011-0015, fig.1, embodiment 1		1-3		
Х	Column 3, paragraphs 0011-0015, fig.1, embc		4-10		
Y	ZHANG Jiao et al., The Growth of High Purit Solidification, JOURNAL OF SHANGHAI JL	1-3			
•	Vol.39 No.11, page 1788, section 1, Testing		1.10		
A	CN85201157U(DALIAN POLYTECHNIC CC the whole document	DLLEGE)11 Jun.1986(11.06.1986) see	1-10		
A	ZHANG Jiao et al., Effects of Development in Purification Technology of High Purity Alumin	1-10			
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А	JP57160567A(SUMITOMO LIGHT METAL) the whole document	IND CO)02 Oct.1982(02.10.1982)see	1-10		
A	CN101463428A(UNIV SHANGHAI JIAOTO whole document	1-10			
🔲 Furth	her documents are listed in the continuation of Box C.	See patent family annex.			
* Spec	special eacegories of check documents.				
	ment defining the general state of the art which is not dered to be of particular relevance	or priority date and not in conflict cited to understand the principle o			
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	ailing address of the ISA	Authorized officer			
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Haidian District, Beijing 100088, China Facsimile No. (86-10)62019451		Telephone No. (86-10) 62084745			
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CN85201157U	11.06.1986	none	
JP57160567A	02.10.1982	none	
CN101463428A	24.06.2009	CN101463428B	30.06.2010
	y annex) (July 2009)		

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5 Continuation of: second sheet. A. CLASSIFICATION OF SUBJECT MATTER C22B 21/06(2006.01)i C22B 9/02(2006.01)i 10 11 15 20 26 30 30 11	
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