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(54) **METHOD FOR OBTAINING ALUMINIUM FROM ALUMINA-CONTAINING RAW MATERIAL.**

(57) The proposed method provides for one-stage treatment of an alumina-containing raw-material with a mixture of alkali metal salts of a fluorine-containing oxygen-free acid and hydrochloric acid taken at the ratio of respectively from 5:1 to 1:7.5 parts by weight. The mixture of the salts is introduced at the quantity of 40-85 % of the total weight, and treatment is carried out at a temperature of 850-1100 °C up to obtaining a reactional mass consisting of non-mixing fluoride-silicate and chloride-aluminate alkaline melts. The chloride-aluminate alkaline melt is separated from the obtained mass and introduced to the melt of alkali metal chlorides, and the obtained mass is subjected to electrolysis up to obtaining the desired product. The invention may be used in the fields of technology connected with the obtaining and use of aluminium.

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Technical Field

The present invention relates to the field of nonferrous metallurgy and in particular deals with a method of producing aluminum from aluminous raw material.

Background Art

At present, the main ores for production of aluminum are high-grade low-silicon bauxites from which alumina is extracted by the Bayer method, with subsequent reduction of aluminum by electrolysis of cryolite-aluminous melts.

In spite of a number of improvements, this method of producing aluminum by electrolysis of cryolite-aluminous melts remains energy intensive and doesn't correspond to modern requirements of environmental protection. The problem of the source of raw materials of the alumina and aluminum industry becomes pressing in a number of countries with a developed aluminum production industry and possessing restricted resources of bauxitic ores or without such resources at all. A great number of investigations in these countries is aimed at providing less energy intensive methods of producing aluminum among which the most promising is a widely known chlorination of aluminous ores and electrolytic reduction of the obtained aluminum chloride.

Aluminum is third in abundance in the Earth's crust among all the elements and first among metals (G.V.Voitkevich, O.A.Bessonov "Khimicheskaya evolutsia zemli", Moscow,"Nedra" Publishing House,1986,p.127) and in the main aluminum forms with oxygen a complex anion $(AlO_4)^{5-}$ in a quaternary coordination and forms alumo-oxygen tetrahedrons approaching in size silicon-oxygen tetrahedrons $(SiO_4)^{4-}$. Connected with this is mainly paragenetic interrelation of aluminum with silicon in the Earth's crust and wide occurence of aluminous ores - kaolin clays, disthene-sillimanite-andalusite slates, etc. In case of providing technology mined economically, countries leading in aluminum production won't be dependent upon import of bauxites and chloride method of electrolysis will allow to reduce energy consumption for aluminum production by 35-45%.

Also known in the art is a method of producing aluminum (US,A,4,108,741) from an electrolyte of the following composition, % by mass: 60-87 $AlCl_3$, 14-30 NaCl, 5-10 KCl, 0.1-3.0 magnesium or calcium chloride. The method of producing aluminum from such an electrolyte stipulates conduction of electrolysis at an anode current density of 0.5-200 A/dm^2, a cathode current density of. 0.5-200 A/dm^2 and a voltage of an electrolytic cell of about 2.7-11.4 V.After electrolysis there is formed a spongy precipitate containing from 30 to 60% by mass of aluminum and slag of a complex composition. Aluminum is separated from the slag by melting the precipitate to give an aluminum phase and a phase of a molten slag.

Disadvantages of this method consist in the following.

1. Used in this method is a scarce reactive - purified and dehydrated aluminum chloride.
2. Use is made of a hygroscopic substance $AlCl_3$ found in the open air in the form of $AlCl_3 \cdot 6H_2O$ (aluminum chloride hexahydrate - ACH) and, therefore, at reduced temperatures of electrolysis in the electrolytic cell in the composition of a precipitate formed are 40-70% by mass of an undesirable by-product (aluminum oxide).
3. A process of separation of aluminum from the phase of a molten slag is conducted not in a standard electrolytic cell but on special equipment which in fact requires additional material and energy expenditures.

Also known in the art is a method of producing aluminum from luminous raw material, including treatment of this raw material, separation of aluminum chloride from the reaction mass, introduction of aluminum chloride into a melt of chlorides of alkali metals and subsequent electrolysis of the formed mass to give the end product - aluminum (GB,A,2,135,663).

In this method there is used as aluminous raw material kaolin clay comprising, % by mass:

moisture	22.0
Al_2O_3, total	35.0 (dry basis)
Al_2O_3, accessible	32.2
Fe_2O_3, total	1.15 (dry basis)
Fe_2O_3, accessible	1.08.

Treatment of this raw material is conducted in several stages. Firstly, the raw material is dried and dehydrated, and then, it is repeatedly treated with 20-26% by mass of hydrochloric acid at a temperature of

60-110 °C to give, after settling and filtering, a pregnant liquor containing up to 17.7% by mass $AlCl_3$ and up to 0.46% by mass of iron. The removal of aluminum makes up 92% of the initial mass of the raw material.

Gaseous chlorine is blown through the pregnant liquor to converse all Fe^{2+} into Fe^{3+}, and iron is withdrawn from the liquor making use of organic liquid ion-exchanger (a mixture of decyl alcohol, kerosine, secondary or tertiary high-molecular amine). Purified liquor is subjected to evaporation and crystallization to give $AlCl_3 \cdot 6H_2O$ - aluminum chloride hexahydrate (ACH) which is calcined at 450-1,000 °C, preferably at 600-750 °C, to produce active alumina possessing a high reactivity, a high content of residual chlorine and a low content of residual hydrogen. The obtained product is chlorinated by recycle gaseous Cl_2 at a pressure of from 0.01 to 1.5 MPa, preferably from 0.1 to 0.5 MPa, and at a temperature of from 500 °C to 950 °C, preferably 550-750 °C, in the presence of gaseous and solid reductants.

As a result of this multistage treatment, anhydrous but hygroscopic aluminum chloride is introduced into a melt containing chlorides of calcium or magnesium, sodium or lithium.

The obtained mass of the following composition; % by mass:

$AlCl_3$	2-15
$NaCl_2$ or $MgCl_2$	15-17
NaCl or LiCl	15-83

is subjected to electrolysis in a mono- or bi-polar electrolytic cell at a temperature of 700 °C, current density of 0.5-1 A/cm^2 with electrode spacing of about 1.5 cm. At the cathode, aluminum is deposited and it is siphoned out of the cell for washing; at the non-consumable carbon anode, chlorine is discharged and it is collected with cell off-gases.

This known method of producing aluminum from aluminous raw material has the following disadvantages.

1. The method stipulates a multistage treatment with the use of special acid-resisting equipment capable to withstand a pressure of up to 1.5 MPa and temperatures of up to 950 °C, as well as the use of such noxious substances as hydrochloric acid, gaseous chlorine, etc.
2. To withdraw iron from the pregnant liquor, use in made of scarce gaseous chlorine and organic liquid ion-exchanger (a mixture of decyl alcohol, kerosine, secondary or tertiary high-molecular amine).
3. Aluminum chloride produced according to this method is hygroscopic; therefore, it is unsuitable for storage and transportation and requires immediate use in the electrolytic process. For this reason,aluminum chloride production must be disposed alongside electrolytic baths.
4. Anhydrous, and yet hygroscopic aluminum chloride in the process of dissolving in the electrolytic melt changes composition of the latter leading to an increase of current utilization factor and, accordingly, to increased energy consumption in the process for aluminum production. Liberation of gaseous chlorine at the anode also promotes changing of the electrolytic melt composition.

Disclosure of the Invention

The present invention is based on the problem to provide a method of producing aluminum from aluminous raw material by way of simplifying the technology.

This problem is solved by that in a method including treatment of this raw material, separation from the obtained reaction mass of a product containing aluminum chloride, its introduction into a melt of alkali metals chlorides, and then electrolysis of the obtained mass to produce the end product, in which, according to the invention, treatment of the aluminous rag material is performed in a single stage with a mixture of alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids taken, respectively, in a ratio of from 5:1 to 1:7.5 parts by mass, in doing so the aforesaid mixture of salts is introduced in amount of 40-85% of the total mass and the process of treatment is conducted at a temperature of 850-1,100 °C till formation of the reaction mass consisting of immiscible fluoride-silicate and chloride-aluminate alkaline melts, as a product containing aluminum chloride from this reaction mass separated is chloride-aluminate alkaline malt, and electrolysis is conducted at a temperature of 720-800 °C.

In doing so, as aluminous raw material it is desirable to use disthene-sillimanite-andalusite concentrate or fluoride-silicate melt.

It is recommended to conduct the process of treating aluminous raw material with a mixture of alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids at a temperature of 870-950 °C.

The method of the present invention allows to produce aluminum from aluminous raw material according to a simplified technology as it stipulates a single-stage treatment of aluminous raw material to obtain a product containing aluminum chloride without employment of a special acid-resisting equipment.

In the process of treatment, iron is concentrated in the fluoride-silicate melt and effective purification of the chloride-aluminate alkaline melt takes place which doesn't require employment of a special equipment and additional consumption of scarce components.

The product produced by the method of the present invention and containing aluminum chloride (chloride-aluminate alkaline melts) is not hygroscopic; in this connection,it can be stored and transported which allows to obviate the need to locate production of the product comprising aluminum chloride in the vicinity of electrolytie baths.

Chloride-aluminate alkaline melts approach in composition electrolytic melt of alkali metals chlorides; therefore, their direct introduction changes electrolyte composition at a minimum. The latter results in a high current utilization factor and, correspondingly, in reduced energy consumption in the process of aluminum production. Minimal change of the electrolyte composition is also enhanced by the reaction of chloride with sodium and potassium oxides and inconsiderable discharge of chlorine gas into the atmosphere.

Besides, instead of noxious gaseous and liquid substances (hydrochloric acid, chlorine gas, etc), the method of the invention incorporates the use of more safe solid alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids. This fact also allows to simplify significantly equipment for the process of treatment of aluminous raw material.

The method of the invention is realized as follows.

Aluminous raw material is subjected to treatment with a mixture of alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids.

To do this, using standard equipment, aluminous material is added with a mixture of alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids taken in a ratio of from 5:1 to 1:7.5 parts by mass, respectively, and the aforesaid mixture of salts is taken in amount of 40-85% of the total mass; the process of treatment being conducted in an electric furnace at 770-1,100 °C. preferably at 870-950 °C, until an immiscible fluoride-silicate (I) melt and chloride-aluminate alkaline (II) melt containing 9-14% by mass of aluminum chloride are obtained.

In this case, as an alkali metal salt of oxygen-free fluoroacid, use can be made of, for example, K_2SiF_6, Na_2SiF_6, Na_3AlF_6, NaF, and as aluminous raw material use can be made of, in particular, dehydrated kaolin concentrate of the following composition, % by mass:

SiO_2	53 - 60
Al_2O_3	37 - 44
Fe_2O_3	0.5 - 1.2
TiO_2	0.4 - 0.8
Na_2O	0.1 - 0.3
K_2O	1.2 - 1.8,

or disthene-sillimanite-andalusite concentrate of the following composition, % by mass:

SiO_2	36 - 42
Al_2O_3	55 - 61
Fe_2O_3	0.6 - 1.5
TiO_2	0.4 - 1.2
Na_2O	0.2 - 0.8
K_2O	0.1 - 0.6,

as well as anorthosites, synnerites, nepheline syenites, mineral part of coal ash and other aluminous raw material.

In case use is made of a mixture of alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids, ratio of these salts being respectively more than 5:1 or less than 1:7.5; the aforesaid process gives a homogeneous melt.

If the quantity of the mixture of alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids is less than 40% or more than 85% of the total reaction mass, the process also results in formation of a homogeneous melt.

Conducting the process at a temperature below 850 °C gives a reaction mass consisting of lens-like or ball-like inclusions of fluoride-silicate melt in the chloride-aluminate alkaline melt or analogous inclusions of chloride-aluminate alkaline melt in the fluoride-silicate melt. Such a mixture is of limited utility for mechanical separation of the formed immiscible melts.

Conducting the process at a temperature above 1,100 °C results in the fact that considerable amounts (more than 3.70% of the total reaction mass) of fluorides of alkali metals and other volatile components volatize.

Besides, a need is generated for increasing energy consumption to attain and maintain such high-temperature conditions.

Performing the process of the aforementioned treatment of aluminous raw material with a mixture of alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids at a temperature of 870-950 °C is preferable, and this results in formation of a well-defined two-layer texture of immiscible melts: fluoride-silicate melt (I) and chloride-aluminate alkaline melt (II). Such texture is suitable for mechanical separation of immiscible melts in the process of and after treatment of aluminous raw material.

In doing so, the fluoride-silicate melt (I) comprises, % by mass:

Al_2O_3	17 - 32
SiO_2	18 - 50
NaCl	1 - 3
K_2SiF_6	4 - 43
KF	0 - 4
Na_2SiF_6 or NaF	0 - 21
Na_2O	0 - 7,

and chloride-aluminate alkaline melt (II) comprises, % by mass:

$AlCl_3$	9 - 40
NaCl	8 - 71
KCl	8 - 48
Na_2SiF_6	0.5 - 3
NaF or KF	5 - 32
Na_2O	3 - 6.

The obtained chloride-aluminate alkaline melt (II) is separated from the reaction mass and introduced into a melt of alkali metals chlorides, after which the obtained mass comprising components in the following ratio, % by mass:

$AlCl_3$	0.5 - 5.0
NaCl	45.0 - 75.0
KCl	20.0 - 45.0
NaF or KF	1 - 10.0
Na_2O or K_2O	0 - 4.0

is subjected to electrolysis in a graphite cell while the fluoride-silicate melt (I) is directed for a multi-stage treatment using it as an aluminous raw material. In doing so, aluminum extraction makes up 88-93% of the mass thereof in the initial aluminous raw material. Siliceous concentrate remained after repeated treatment may be used in various branches of industry (for production of silicate materials, for manufacture of package glass, as an adsorbent for oil products and for other purposes), besides, it is ecologically safe product.

Electrolysis is conducted in a graphite mono- or bi-polar electrolytic cell at a temperature of 720-800 °C (preferably at 740-780 °C), current density of 0.5-3 A/cm^2 with electrode spacing of 0.5-1 cm till formation of the end product - aluminum at the cathode.

In case of conducting electrolysis at a temperature above 800 °C, evaporation of the melt increases, anode corrodes at the air-melt boundary and, in doing so, electrolyte is severely contaminated with graphite.

In case the temperature of conducting electrolysis is below 720 °C, it is difficult to maintain thermal balance in the cell due to considerable viscosity of the melt, its sticking to the graphite anode, decreased area of the active anode zone or complete setting of the electrolytic melt,

The obtained product - aluminum is removed from the cell, collected for casting and analyzed. In this case, the degree of purity of aluminum is from 98.7-99,3%, The obtained aluminum is directed for further utilization in various branches of industry.

Chlorine gas discharged at the anode reacts with oxides of sodium and potassium,concentration of which is decreased practically to zero where, in doing so, formed are sodium and potassium chlorides which are fed to the electrolytic cell, Thus, minimum discharge of gaseous chlorine into the atmosphere takes place; this fact ensuring inconsiderable changes in the electrolyte composition and enhancing to maintain stable electric conductivity of the electrolyte.

For a better understanding of the present invention, given below are the following examples not limiting the scope of the invention.

Example 1

The method of the invention is realized as follows.

An amount of 30 g of dehydrated kaolin concentrate are treated with a mixture of potassium chloride (50 g) and malladrite - Na_2SiF_6 (20 g) in an electric furnace at a temperature of 900 °C in the course of 1.5 hours, As a result of melting, there are produced two immiscible melts of a two-layer texture: 44,75 g (45%) of fluoride-silicate melt (I) of the following composition, % by mass:*

Al_2O_3	16.63
SiO_2	45.84
NaCl	1.81
K_2SiF_6	32.82
NaF	1.22
Na_2O	1.68,

and 54.69 g (55%) of chloride-aluminate alkaline melt (II) of the following composition, % by mass:

$AlCl_3$	22.13
NaCl	7.74
KCl	42.28
Na_2SiF_6	0.94
KF	22.59
Na_2O	4.32.

After melting, weight losses amount up to 0.56% of the initial reaction mass.

The obtained chloride-aluminate alkaline melt (II) is separated from the reaction mass and the remained fluoride-silicate melt (I) (44.75 g) is again directed for treatment as an initial aluminous raw material. Aluminum extraction degree makes up 93% of its mass in the initial dehydrated kaolin concentrate.

Chloride-aluminate alkaline melt (II) is added (1/4 part by mass) into a graphite cell filled (3/4 parts by mass) with electrolytic melt of alkali metals chlorides, NaCl to KCl ratio being equal to 2.5:1.

Initial composition of the electrolyte, % by mass:

$AlCl_3$	5.47
NaCl	57.98
KCl	29.10
Na_2SiF_6	0.26
KF	5.51
K_2O	1.68.

*Notice: In all Examples data of the chemical analysis are calculated for 100%.

Extraction of aluminum is performed at a temperature of 760 °C and current density of 1.5 A/cm^2. After 4 hours of electrolysis, 1/4 part of the electrolyte is discharged and a new portion of the melt II is added.

Final composition of the electrolyte, % by mass:

$AlCl_3$	0.61
NaCl	61.76
KCl	31.49
Na_2SiF_6	0.13
KF	6.01.

The discharged melt is utilized again for treatment of dehydrated kaolin concentrate.

As a result of electrolysis, at the bottom of the graphite cell there is formed a layer of aluminum which is removed from the cell, analyzed (purity degree makes up 99.3%) and collected for casting.

In the process of electrolysis, chlorine generated at the anode reacts with potassium oxide to give, as a result of this reaction, potassium chloride which is fed to the electrolytic cell. Thus, practically eliminated is the discharge of gaseous chlorine into the atmosphere.

Example 2

The method of the invention is realized in a manner similar to that of Example 1. 15 g of disthene concentrate are treated with a mixture of sodium chloride (75 g) and hieratite - K_2SiF_6 (10 g) in an electric furnace at a temperature of 950 °C in the course of 1,5 hours. As a result of melting, there are obtained two immiscible melts of a two-layer texture: 19.88 g (20%) of fluoride-silicate melt (I) of the following composition, % by mass:

Al_2O_3	31.17
SiO_2	42.02
NaCl	2.31
K_2SiF_6	4.16
NaF	20.34,

and 79.50 g (80%) of chloride-aluminate alkaline melt (II) of the following composition, % by mass:

$AlCl_3$	10.35
NaCl	71.14
KCl	7.91
Na_2SiF_6	0.56
NaF	6.10
Na_2O	3.94.

After melting, weight losses amount up to 0.62% of the initial reaction mass.

The obtained chloride-aluminate alkaline melt (II) is separated from the reaction mass and the remained fluoride-silicate melt (I) (19,88 g) is again directed for treatment as an initial aluminous raw material. Extraction degree of aluminum makes up 90% of its mass in the initial disthene concentrate.

Chloride-aluminate alkaline melt (II) is introduced (50% by mass) into a graphite cell filled (50% by mass) with electrolytic melt of alkali metals chlorides, NaCl to KCl ratio being equal to 1.5:1.

Initial composition of the electrolyte, % by mass:

$AlCl_3$	5.20
NaCl	65.89
KCl	23.85
Na_2SiF_6	0.34
NaF	2.86
Na_2O	1.86.

Extraction of aluminum is performed at a temperature of 720 °C and current density of 3 A/cm^2. After 4 hours of electrolysis, 50% of the electrolyte are removed and a new portion of the melt II is introduced.

Final composition of the electrolyte, % by mass:

$AlCl_3$	0.53
NaCl	71.52
KCl	24.59
Na_2SiF_6	0.13
NaF	3.23.

The removed melt is utilized again for treatment of disthene concentrate.

As a result of electrolysis, at the bottom of the graphite cell there is formed a layer of aluminum which is discharged from the cell, analyzed (purity degree makes up 98.9%) and collected for casting.

Chlorine generated in the process of electrolysis reacts with sodium oxide where the latter is transformed into sodium chloride which is fed to the electrolytic cell. In this case, practically eliminated is the discharge of gaseous chlorine into the atmosphere.

Example 3

The method of the invention is realized in a manner similar to that of Example 1. In this case, 60 g of dehydrated kaolin concentrate are treated with a mixture of potassium chloride (20 g) and cryolite - Na_3AlF_6 (20 g) in an electric furnace at a temperature of 1,100 °C in the course of 1 hour.

As a result of melting,there are obtained two immiscible melts of a two-layer texture: 81.86 g (85%) of fluoride-silicate melt (I) of the following composition, % by mass:

Al_2O_3	31.62
SiO_2	36.23
NaCl	3.39
K_2SiF_6	23.00
Na_2O	5.76,

and 14.44 g (15%) of chloride-aluminate alkaline melt (II) of the following composition, % by mass:

$AlCl_3$	40.13
NaCl	12.34
KCl	26.73
Na_2SiF_6	2.99
KF	11.51
Na_2O	6.30.

After melting, weight losses amount up to 3,70% of the initial reaction mass.

The obtained chloride-aluminate alkaline melt (II) is separated from the reaction mass and the remained fluoride-silicate melt (I) (81.86 g) is again directed for treatment as an initial aluminous raw material. Extraction degree of aluminum makes up 92% of its mass in the initial kaolin concentrate.

Chloride-aluminate alkaline melt (II) (12.5% by mass) is introduced into a graphite cell filled (87.5% by mass) with electrolytic melt of alkali metals chlorides, NaCl to KCl ratio being equal to 3:1.

Initial composition of the electrolyte, % by mass:

$AlCl_3$	5.06
NaCl	68.92
KCl	23.03
Na_2SiF_6	0.33
KF	1.44
K_2O	1.22.

Extraction of aluminum is performed at a temperature of 800 °C and current density of 0.5 A/cm^2. After 4 hours of electrolysis, 1/8 part of the electrolytic melt is removed, and a new portion of the melt II is introduced.

Discharged melt is utilized again for treatment of dehydrated kaolin concentrate.

Final composition of the electrolyte, % by mass:

$AlCl_3$	0.58
NaCl	72.70
KCl	24.82
Na_2SiF_6	0.13
KF	1.77.

As a result of electrolysis, at the bottom of the graphite cell there is formed a layer of aluminum,which is discharged from the cell, analyzed (purity degree makes up 99.1%) and collected for casting.

Chlorine discharged at the anode reacts with potassium oxide, concentration of which reduces from 1.22% to zero, in this case, potassium chloride is formed, and it is fed to the electrolytic cell, Thus, minimal discharge of gaseous chlorine into the atmosphere takes place.

Example 4

The method of the invention is realized in a manner similar to that of Example 1, but in this case, 40 g of disthene concentrate are treated with a mixture of sodium chloride (10 g) and hieratite - K_2SiF_6 (50 g) in an electric furnace at a temperature of 800 °C in the course of 2 hours.

As a result of melting, there are obtained two immiscible melts of a two-layer texture: 84,50 g (85%) of fluoride-silicate melt (I) of the following composition, % by mass:

Al_2O_3	27.23
SiO_2	17.75
NaCl	0.69
K_2SiF_6	43.28
Na_2SiF_6	11.05,

and 14,91 g (15%) of chloride-aluminate alkaline melt (II) of the following composition, % by mass:

$AlCl_3$	32.11
NaCl	9.39
KCl	17.58
Na_2SiF_6	2.78
KF	31.79
Na_2O	6.35.

After melting, weight losses amount up to 0.59% of the initial reaction mass.

The obtained chloride-aluminate alkaline melt (II) is separated from the reaction mass and the remained fluoride-silicate melt (I) (84.50 g) is again directed for treatment as an initial aluminous raw material. Extraction degree of aluminum makes up 88% of its mass in the initial disthene concentrate.

Chloride-aluminate alkaline melt (II) (1/6 part by mass) is introduced into a graphite cell filled (5/6 part by mass) with electrolytic melt of alkali metals chlorides, NaCl to KCl ratio being equal to 5:1.

Initial composition of the electrolyte, % by mass:

$AlCl_3$	5.33
NaCl	65.59
KCl	23.70
Na_2SiF_6	0.38
NaF	3.95
Na_2O	1.05.

Extraction of aluminum is performed at a temperature of 740 °C and current density of 2 A/cm^2. After 4 hours of electrolysis, 1/6 part of the electrolyte is removed and a new portion of the melt (II) is introduced.

Final composition of the electrolyte, % by mass:

$AlCl_3$	0.59
NaCl	70.33
KCl	24.58
Na_2SiF_6	0.13
NaF	4.37.

Discharged melt is utilized again for treatment of disthene concentrate.

As a result of electrolysis, at the bottom of the graphite cell there is formed a layer of aluminum, which is discharged from the cell, analyzed (purity degree makes up 98.7%) and collected for casting.

In the process of electrolysis, chlorine generated at the anode reacts with sodium oxide. As a result of this process, sodium chloride is formed and it is fed to the electrolytic cell.Thus, practically eliminated is discharge of gaseous chlorine into the atmosphere.

Example 5

The method of the invention is realized in a manner similar to that of Example 1, but in this case, 15 g of dehydrated kaolin concentrate are treated with a mixture of potassium chloride (65 g) and villiaumite - NaF (20 g) in an electric furnace at a temperature of 870 °C in the course of 2 hours.

As a result of melting, there are obtained two immiscible melts of a two-layer texture: 19,90 g (20%) of fluoride-silicate melt (I) of the following composition, % by mass:

Al_2O_3	16.75
SiO_2	39.85
NaCl	1.24
K_2SiF_6	30.99
KF	3.85
Na_2O	7.32,

and 79.59 g (80%) of chloride-aluminate alkaline melt (II) of the following composition, % by mass:

$AlCl_3$	8.97
NaCl	16.42
KCl	47.69
Na_2SiF_6	0.45
KF	20.13
Na_2O	6.34.

After melting, weight losses amount up to 0.51% of the initial reaction mass.

The obtained chloride-aluminate alkaline melt (II) is separated from the reaction mass and the remained fluoride-silicate melt (I) (19.90 g) is again directed for treatment as an initial aluminous raw material. Extraction degree of aluminum makes up 92% of its mass in the initial dehydrated kaolin concentrate.

Chloride-aluminate alkaline melt (II) (55% by mass) is introduced into a graphite cell filled (45% by mass) with electrolytic melt of alkali metals chlorides, NaCl to KCl ratio being equal to 1.5:1.

Initial composition of the electrolyte, % by mass:

$AlCl_3$	4.94
NaCl	41.63
KCl	38,94
Na_2SiF_6	0.21
KF	10.27
K_2O	4.01.

Extraction of aluminum is performed at a temperature of 780 °C and current density of 1,A/cm^2. After 4 hours of electrolysis, 55% of the electrolyte are removed and a new portion of the melt II is introduced.

Final composition of the electrolyte, % by mass:

$AlCl_3$	0.53
NaCl	45.78
KCl	42.88
Na_2SiF_6	0.08
KF	10.73.

Discharged melt is again utilized for treatment of dehydrated kaolin concentrate.

As a result of electrolysis, at the bottom of the graphite cell there is formed a layer of aluminum, which is removed from the cell, analyzed (purity,degree makes up 99.2%) and collected for casting.

Chlorine generated in the process of electrolysis reacts with potassium oxide to transform the latter to potassium chloride which is fed to the electrolytic cell. In this case, practically eliminated is the discharge of gaseous chlorine into the atmosphere.

Industrial Applicability

The invention can find application in various branches of industry dealing with production and utilization of aluminum.

Claims

1. A method of producing aluminum from aluminous raw material, including treatment of this raw material, separation of a product containing aluminum chloride from the obtained reaction mass, introduction of this product into a melt of alkali metals chlorides and subsequent electrolysis of the formed mass until formation of the end product, **characterized** in that the treatment of aluminous raw material is conducted at a single stage by way of introduction thereto of a mixture of alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids taken in a ratio of from 5:1 to 1:7.5 parts by mass, respectively, in doing so, the aforesaid mixture of salts is introduced in amount of 40-85% of the total mass and the process of treatment is conducted at a temperature of 850-1,100 °C until formation of a reaction mass consisting of immiscible fluoride-silicate and chloride-aluminate alkaline melts; as a product containing aluminum chloride separated from this reaction mass is chloride-aluminate alkaline melt, the electrolysis being conducted at a temperature of 720-800 °C.

2. A method of producing aluminum according to Claim 1, **characterized** in that as an aluminous raw material use is made of disthene-sillimanite-andalusite concentrate.

3. A method of producing aluminum according to Claim 1, **characterized** in that as an aluminous raw material use is made of fluoride-silicate melt.

4. A method of producing aluminum according to Claim 1, **characterized** in that the process of treatment of aluminous raw material with a mixture of alkali metals salts of fluorine-containing oxygen-free and hydrochloric acids is conducted at a temperature of 870-950 °C,

INTERNATIONAL SEARCH REPORT

International application No.
PCT/RU 92/00149

A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl. 5 : C25C 3/06, C22B 3/06

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)

Int.Cl. 5 : C25C 3/06, C22B 4/02, 21/00, 3/06, 3/10

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)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 4465566 (LOUTFY RAOUF O. ET AL) 14 August 1984 (14.08.84), the abstract	1-4
A	US, A, 4415412 (VANDEGRIFT GEORGE F. ET AL), 15 November 1983 (15.11.83), the abstract	1-4
A	GB, A, 2135663 (ATLANTIC RICHFIELD COMPANY), 5 September 1984 (05.09.84), the abstract (cited in the description)	1-4

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

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"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	Date of mailing of the international search report
17 March 1993 (17.03.93)	14 April 1993 (14.04.93)
Name and mailing address of the ISA/RU	Authorized officer
Facsimile No.	Telephone No.

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