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PROCESS AND APPARATUS FOR THE ELECTROLYTIC CONTINUOUS DIRECT PRODUCTION OF REFINED ALUMINUM AND OF ALUMINUM ALLOYS

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Fig. 1

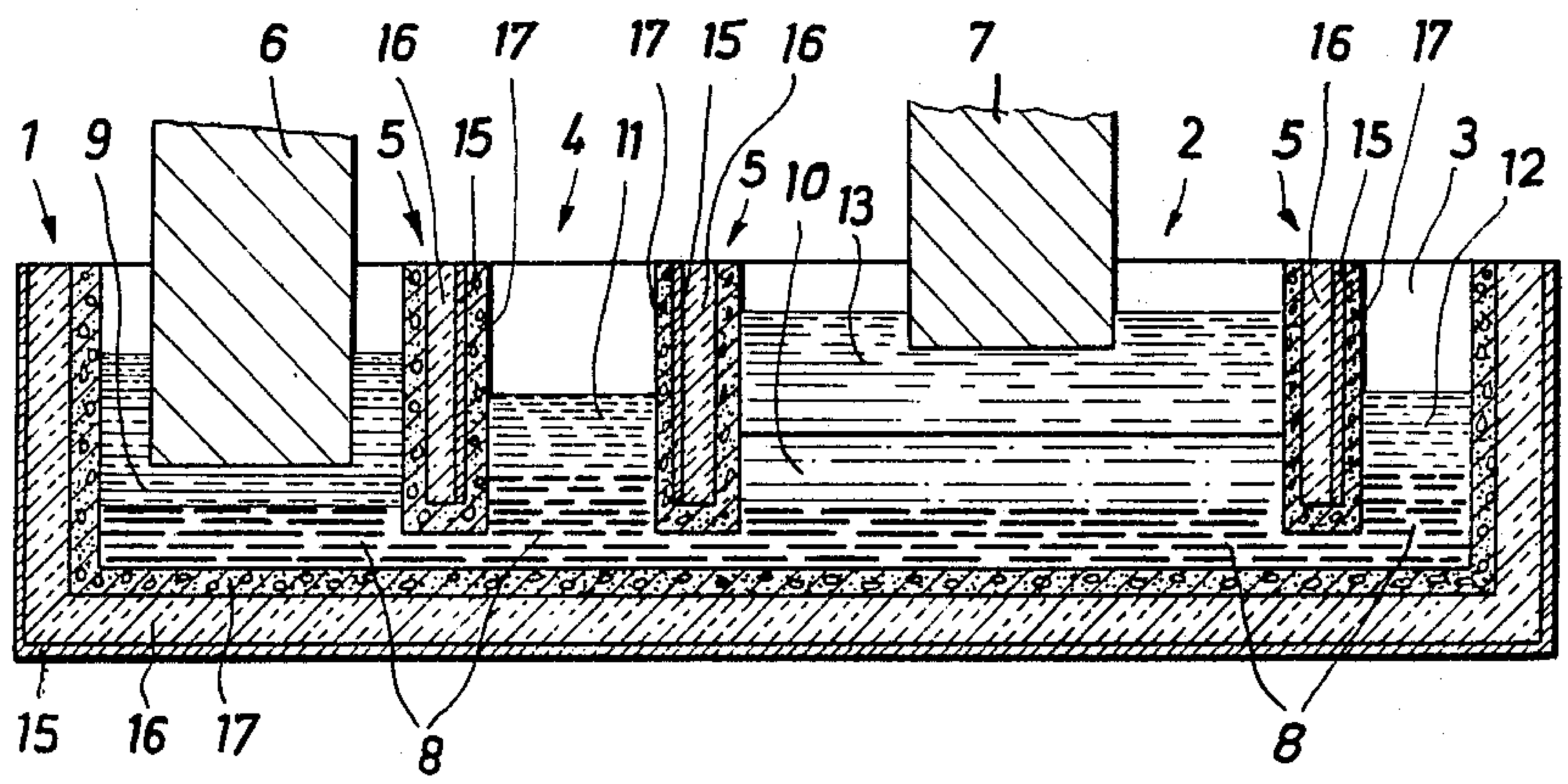
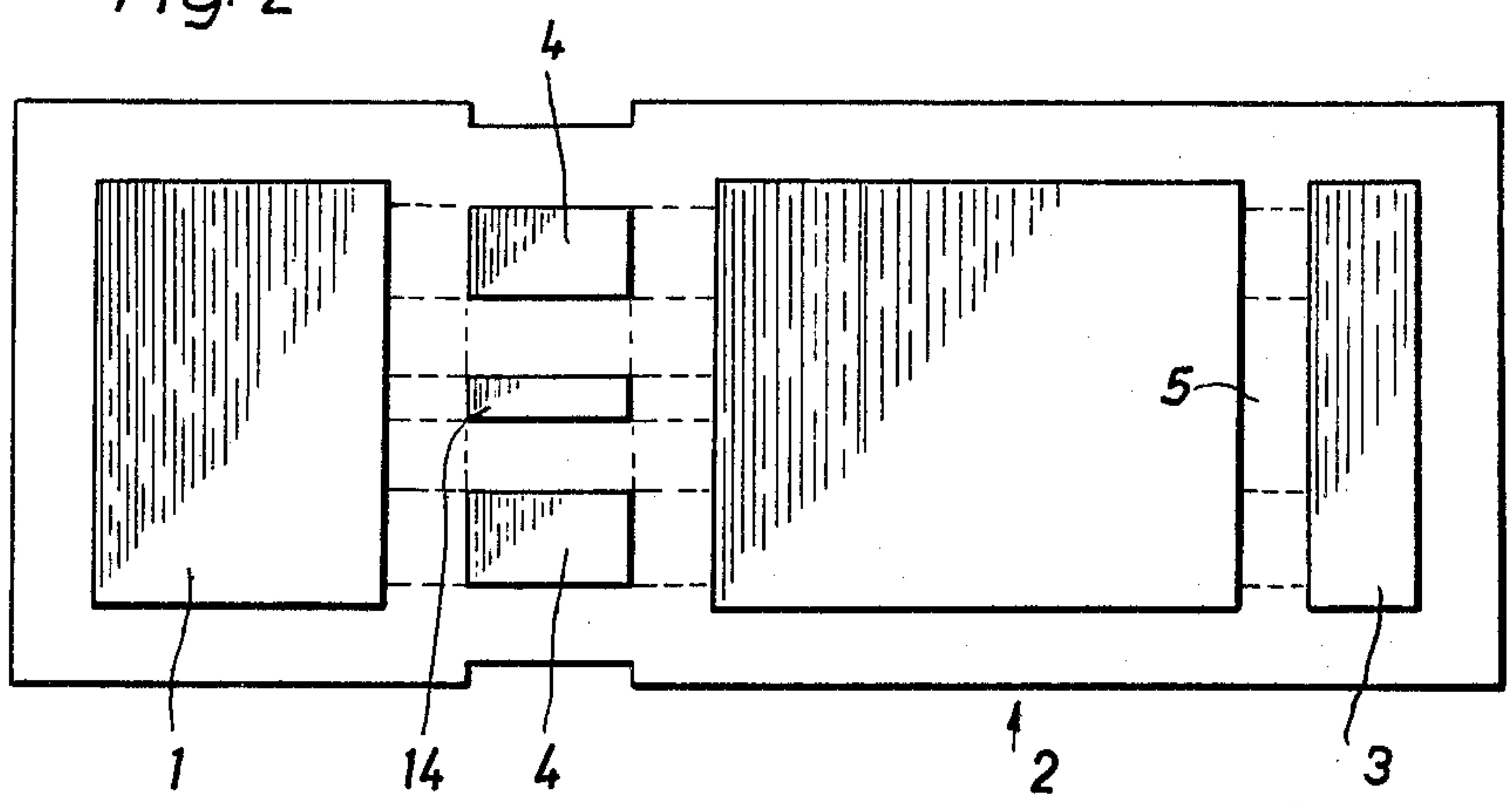


Fig. 2



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PROCESS AND APPARATUS FOR THE ELECTROLYTIC CONTINUOUS DIRECT PRODUCTION OF REFINED ALUMINUM AND OF ALUMINUM ALLOYS

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ABSTRACT OF THE DISCLOSURE

A process and apparatus for processing aluminum in which a first vessel containing liquid aluminum alloy is spaced from a second vessel containing liquid alloy via at least one channel connecting the vessels at the lower portions thereof and through which the liquid alloy can flow from one vessel to the other, there being electrodes extending downwardly into the vessels to positions above the channel, the electrodes associated with one of the vessels losing material into the alloy therein.

This invention relates to processes and apparatus for the continuous and direct production of refined aluminum and of aluminum alloys by electrolysis of a molten bath consisting of fluorides with aluminum dissolved therein.

According to the invention there may be employed a production unit consisting of two vessels that are not directly connected together but are connected by one or more channels. These two vessels and the connecting channels are filled with liquid aluminum alloy, forming the cathode in the first vessel and the anode in the second vessel. For the production process, electrodes can be used which consist of metal or carbon compounds of one or more metals.

A process is already known by which, in a first step, crude aluminum is produced and, in a second step, pure aluminum is extracted out of this crude aluminum by a second electrolysis. In such a process, impurities accumulate in the crude aluminum because purer aluminum is taken off. After a relatively short production period, a substantial part of the crude aluminum must be taken out, or the production unit has to be cut off. The crude aluminum to be taken out with its high content of iron and other impurities is of very low quality.

In another known process, an electrolytic cell for the production of crude aluminum is directly connected with an electrolytic cell working according to the three layer system. An aluminum alloy forms the bipolar connection. In such a process, the aluminum alloy cannot be kept with a constantly uniform composition. Furthermore, it is not possible to influence the circulation of the aluminum alloy, enriched with aluminum in the zone for the primary electrolysis, into the zone for the three layer electrolysis, where the aluminum alloy has less aluminum, and back into the room for the primary electrolysis. In order to reduce disturbances, it is necessary to work with a relatively big stock of aluminum alloy.

Other disadvantages of the known processes are the inevitable fluctuations of the surfaces of the baths, of the electrical conditions, as well as the necessary high voltage of the cell, the high thermal strain on the separating wall, and the high consumption of energy and material for the refined aluminum.

By the invention, the disadvantages of known processes are avoided and a technique is employed according to which the raw materials in the first vessel are decomposed electrolytically by direct current in known manner,

and the aluminum is deposited in a liquid alloy, which is used as the cathode and which is put into the production unit before the beginning of the electrolysis, the technique being further characterized in that in the second vessel, which is not directly connected but which is connected by channels with the first vessel, there is contained liquid fluoride as an electrolyte, the liquid alloy being used as the anode. The aluminum alloy constitutes a liquid unit carrying the stream of direct current. The liquid fluoride baths are kept separated. Out of the anode of the second vessel, the refined aluminum is processed electrolytically. The electrodes consist of metal, partly or completely.

The production units of the invention may be composed of various structural parts, for example, the first vessel is one part and the second vessel together with a draw-room, out of which the alloy can be taken, is another structural part. Between these two parts, one or several channels are arranged providing a connection for the liquid alloy which is in the vessels. The channels may be inclined relatively to the horizontal or vertical with respect to the opposite wall of the other vessel. They may be of different cross-sections in order to further the mixing and the circulation of the liquid bipolar alloy. Because the first vessel has no common wall separating it from the second vessel, it is impossible that undesired electric current can flow from the liquid bath of the first vessel towards the liquid bath of the second vessel. The distance between the two vessels and by this the thermal insulation of each vessel can be prearranged. Thereby it is impossible that the electrolyte of the first vessel can influence the electrolyte and the refined aluminum within the second vessel. Uniform and constant thermal conditions may be maintained despite great differences of temperatures of the baths of the two vessels.

Alloying metal, for example copper, can be fed into the liquid aluminum alloy by using, in the first vessel, electrodes consisting partly or wholly of material containing copper, such as oxide of copper, aluminum-copper alloy or copper alone. Contact pins, supplying current into the anode in the first vessel, need not be pulled out or placed in higher positions because they are produced of copper or of aluminum-copper alloy. This alloy is, for example, purified by segregation and thus, without the greater part of its impurities, can be taken out of the draw-room, designed for that purpose in the second vessel. Copper electrodes of appropriate dimensions and temperature yield only small amounts of metal to the bath. By these measures, the proportion of copper in the liquid aluminum-copper alloy can exactly be adjusted within a large range. For the same purpose, compounds of copper can be supplied to the bath in the first vessel. For example, copper oxide can be supplied to the bath. By these means, other metals can also be fed into the liquid alloy such as, for example, manganese, nickel, titanium, and these may perhaps also be fed into the bath in the second vessel.

From the second vessel or channels, aluminum can be taken out in determined quantity by distilling it out of the liquid aluminum-copper alloy under reduced pressure with appropriate apparatus. At the same time, the temperatures at the respective working zones can be influenced.

The channels can be used to heat the related production unit or to keep it at a determined temperature, without connecting the electrodes with an outer circuit.

By the new process, refined aluminum and aluminum alloy, each with determined uniform quality, can be produced even if the raw materials used for the process are not pure. For example, when processing an aluminum-copper alloy having one-third copper, the final metal being aluminum, the first vessel is filled with a bath of fluorides into which bath raw alumina or roughly prepared alumina have been brought. With the new process, it is sufficient to purify the aluminous ore, such as bauxite, so that the content of iron and titanium compounds have been brought down to values that are still six times higher than those in the alumina conventionally used for the production of aluminum. Iron, titanium and silicon are taken in by the aluminum-copper alloy and they are removed from it after segregation as far as necessary. The final metal in the second vessel is refined aluminum.

In order to feed the raw materials into the first vessel in exact quantities, pure or raw alumina beforehand is, for example, dissolved in a fluoride bath. Then this bath, saturated with alumina, is poured upon the bath in the first vessel by means of a large crucible, similar to the working process in German Patent No. 718,189. When the prepared fluoride bath, saturated with alumina, is poured, the bath which is already in the first vessel and having less alumina, is pushed upwards through a shaft or behind a separting wall which leaves free passage for the bath at its lowermost part, so that it cannot mix with the bath being poured in. The old bath flows over a spillway into a second crucible, by means of which it is poured upon the bath in another first vessel or into a cell, which produces aluminum according to the conventional process. When working in the described manner, the insulating crust on the bath in the vessel is preserved, except a narrow opening through which to pour in the new bath. To each consecutive first vessel requiring alumina in the production unit series an exact quantity of bath is given with respectively uniform quality. By this manner of working the "anodic effect" can be eliminated without the danger that the bath becomes over-saturated with alumina. Operating the furnaces is facilitated. The plant is mechanized in its greater part. In the example, also, the copper having been part of the anode in the first vessel and being used for the liquid alloy, can be impure, without this becoming disadvantageous for the final aluminum. As far as the impurities go into the liquid aluminum-copper alloy, they can be taken out of it without disturbing the course of production. Impurities in the raw alumina, so far as they accumulate within the electrolyte of the first vessel (for example, in case the raw material has extraordinarily great quantities of calcium or of vanadium) are separated from the electrolyte in a separate apparatus before they reduce the solubility of the alumina in the electrolyte or its conductivity. This manner of working does not reduce the productiveness of the units as is the case with conventional processes.

The production units of the invention do not need collector bars in their bottoms, as are used in producing primary or refined aluminum by conventional processes. Therefore, the losses of voltage related with the bottoms are eliminated as well as the strain and wear resulting from the flow of current. Thus, less enegy, material, fluoride bath, and work are spent than are necessary in conventional processes for changing used-up bottoms for new bottoms.

The protecting casings around the anodes in the first vessel and the contact pins, supplying the current into these electrodes, consist for example of an aluminum-copper alloy, that has a higher melting temperature than temperature existing at the anodes within the fluoride bath or they consist of copper. Hence the anodes still have their complete original cross-section down in the fluoride bath. Further, one of the hot workings at the furnace is eliminated, in case of vertically as well as laterally inserted contact pins, because these parts of the electrical connections remain in the electrodes and melt and drop into the aluminum-copper alloy. Because conducting the current under the surface of the bath becomes more favorable, the losses of voltage in the anodes are reduced.

In the usual processes for the production of primary aluminum, the crusts on the surfaces of the fluoride baths are pushed into the baths by means of power operated tools in order to supply the baths with the necessary alumina. Contrary to this working process, the surfaces in the new process are kept closed, except a little opening necessary to feed liquid bath into the first vessel. The heat losses are thus reduced. By means of the channels, the second vessel is equally heated by the bipolar alloys. Because the alloy is circulating as explained hereinbefore, the voltage necessary for polarization is less. The new process eliminates several causes for losses of thermal and electric energy, so that the density of the current can be reduced in both vessels. The efficiency of the current becomes more favorable, because by means of the channels the surface of the anodic metal regularly, locally and continuously is supplied from the first vessel with fresh alloy having a high proportion of aluminum. This is not possible with the conventional three layer process because the aluminum from the depth of an aluminum-copper alloy has to be brought upwards to the electrolyte. Moreover, the layer of the alloy is relatively high and the least mobile, and that part of the alloy which is least hot and in the cell in its region, least warm. This is also not possible when a cell for the production of primary aluminum is directly connected together with a cell working the three layer process without the connecting channels.

By the new process, the conventional electrodes, consisting of baked carbon or of graphite, can partly or wholly be replaced by electrodes consisting of metal. In the first vessel, these metal anodes, for example, are designed with vertical shafts for the exhaust of the gases at the anode, for automatic charging of alumina, or to insert liquid fluoride bath with alumina in it. In the second vessel, from a metal cathode of appropriate dimensions and current load, practically no metal is transported by diffusion against the flow of current into the aluminum that is below this cathode. The consumption of baked carbon and of graphite electrodes becomes less when using metal electrodes, for example also when these are put in to keep the exact quantity and composition of the aluminum-copper alloy, because the current efficiency and the voltage of the production unit can be adjusted to their optimal values and constantly maintained there. The metal electrodes, for example, consist of aluminum, chromium, cobalt, copper, manganese, nickel or of titanium or of their alloys with one another or with other metals.

In the following description and in the attached drawing an example is given of the process and apparatus of the invention.

In the drawing:

FIGURE 1 is a diagrammatical vertical cross-section through an apparatus for effecting the process of the invention; and

FIGURE 2 is a top plan view of the apparatus shown in FIG. 1, empty and omitting the electrodes.

The illustrated production unit is mainly composed of three structural parts: a first vessel **1**, a second vessel **2**, and channels **4** connecting these vessels. The vessels and the channels each consist of a metal casing **15** with an insulating layer **16** and a lining **17** consisting of alumina and magnesium oxide refractories.

In the second vessel **2**, a wall **5**, which leaves free passage at least at one place at the bottom thereof, separates a draw-room **3** from the portion of the room opposite the channels **4**. The alloy can be taken from said draw-room.

To the first vessel **1**, electric current is lead via electrodes **6**. In the second vessel **2**, electrodes **7** are provided.

In the first and second vessels **1**, **2**, is the liquid aluminum alloy **8** that also fills the lower part of the channels **4**. The ways into and out of the channels are located beneath the surface of the alloy. The fluoride bath **9** of the first vessel **1** therefore has no bath-to-bath connection with the fluoride bath in the second vessel **2**. In the second vessel **2** the openings are also under the surface of the aluminum alloy in the lower part of the separating wall that leaves free passage at several places to the draw-room **3** for the alloys only. In the channels **4** and in the draw-room **3**, the alloy is under protective baths **11**, **12**. In the second vessel **2** upon the fluoride bath **10** above that portion of the alloy **8** that is not in the draw-room **3**, is the layer of refined aluminum **13**.

In the first vessel **1**, the electrodes **6** consist of carbon electrodes, copper electrodes, and aluminum-copper electrodes. The fluoride bath **9** belonging to the first vessel, is composed of fluorides of aluminum, barium, calcium and sodium, and further of alumina dissolved in this bath. The liquid aluminum-copper alloy **8** consists of one-third copper—the balance in this example being aluminum and small amounts of other metals. The aluminum is previously produced in the first vessel, whereas the copper comes from the copper electrodes and from the aluminum-copper electrodes.

The fluoride bath **10** in the second vessel **2** in this example is composed of the same fluoride as are in the first vessel. Converted by calculation to the composition of fluoride of aluminum, the bath in the second vessel has 50% of the aluminum fluoride, while the total content of fluorides in the bath is kept at 50%, and the bath is free of oxides. This process is working with "warm" aluminum-copper alloy. Contrary to conventional methods, it is avoided that parts are deposited on the bottom below the alloy in the second vessel, hampering the efficiency of the production.

From the first vessel **1**, and supported by controlled circulation by means of the channels **4**, the liquid alloy is always kept at such a temperature that it is possible to effect production without difficulties with a composition range, for example, of 62% copper, with a balance of aluminum, plus silicium, plus iron, and small amounts of other metals. With 32% copper, it is possible to work with good efficiency and without difficulties with an alloy having as much as 11% silicon plus iron, the rest being aluminum plus small amounts of other metals.

The large constant caloric capacity of the production unit eliminates also that in the first and in the second vessels **1** and **2** crusts are formed narrowing unfavorably the upper portion of the baths. After a short period of working, it is possible to produce aluminum-copper alloys and refined aluminum **13** with the desired quality, because the periods for heating up and baking the electrodes and the bottoms of the cells are shortened or eliminated. The reason for this is that the casing and the contact pins for the continuous anode take an essential part of the electric current at the most downward part of this anode. Therefore, at the beginning of the production process also, coking of the carbonaceous mass of the anode has to take place only in a relatively shorter part, compared with the conventional method of aluminum production. As the bottom and the sides of the production unit are lined with refractory material and, by this lining, practically no current is flowing through the bottom, because the liquid alloy is the conductor to the second vessel, the full load of the current can be charged much earlier than with the conventional method upon the bottom as well as upon the anode.

Impurities brought in with the supplied material are separated from the liquid alloy **8** within the draw-room **3**. The separated and segregated material is taken out. The purified aluminum-copper alloy **8** stays with the production unit or it is brought to further manufacturing units. The draw-room **3** alternatively can be arranged at another place in the production unit, or it can be connected with the unit by channels.

In order to avoid that the liquid alloy **8** takes in gas, or that it become superficially oxidized when in the channels **4** or in the draw-room **3**, the alloy is below a protective layer of, for example, molten salts **11**, **12**, with which the channels and/or the draw-room have been filled similar to the working process in German Patent 718,189. In the same manner the first and second vessels **1**, **2** can be charged, for starting the production, with molten fluoride baths **9**, **10** that have been previously purified, in order to keep low quantity of gas occlusion by the aluminum-copper alloy **8** and by the refined aluminum **13** from the start. Undesired side-reactions can occur, due to incoming oxygen, nitrogen or hydrogen, the latter for example, originating from humidity adhering to unmolten salts in the first as well as in the second vessel. To keep low from the beginning the portion of gas and oxide in the aluminum-copper alloy **8** and consequently in the refined aluminum **13**, the rooms to be filled with the alloy can wholly or partly be lined with copper or an aluminum-copper alloy, which are taken in by the liquid alloy after the remainder of the humidity has been driven out of the furnace brick lining, rammed paste, etc., at high temperature. By this working process, it is excluded that gases are brought into the fluoride bath by surface skin owing to oxidation of metal, as is the case with conventional methods. The refined aluminum **13** has therefore substantially less gas occlusion that usual. Consequently, the periods for de-gassing in the melting furnaces are shorter or can be eliminated and flawless rolling blocks can be cast.

In the channels **4**, the circulation of the liquid alloy can be checked and influenced (for example, by narrowing the cross-section of the free passage in one of the channels). They are covered when the production is running.

The temperature of the liquid aluminum-copper alloy can be adjusted from the first vessel **1**. From there, the temperature in the second vessel **2** is especially easily influenced, when a lower working temperature is used for the fluoride bath **10** in the second vessel **2** than in the first vessel **1**. Contrary to conventional working processes for the production of primary aluminum with a cathode consisting of aluminum, the new process with the cathode of the heavier aluminum-copper alloy **8** is independent of the density of the fluoride bath **9** in the first vessel **1**. The process allows adding substances to the fluoride bath **9**, in order to raise the value of its conductivity, or to bring metals into the alloy. Because the second vessel **2** is heated by the alloy **8** from the lower part, there are only small losses of fluoride bath by evaporation. For example, when using, in the second vessel **2**, a bath **10** with compounds of fluorides of natrium, aluminum, calcium and barium, and working at 720° C. in the lower part of the bath **10**, this temperature can be kept at the same magnitude and the distance between the liquid alloy **8** and the cathode **7** kept constant, by correspondingly adjusting in the first vessel **1** the distance between the electrodes **6** and the liquid alloy **8**.

A shaft **14**, arranged for example between the channels **4**, extends into the liquid aluminum alloy **8**, which from the channels **4** can flow into the shaft **14** via its lower opening. Out of the shaft **14**, aluminum can be distilled under reduced pressure. The shaft **14** itself consists, for example, of a graphite tube, or within the shaft into the liquid alloy a graphite tube is inserted. A metal tube, the melting temperature of which is at least 100° C. higher than the temperature of the liquid alloy can also be used. A graphite tube inserted into the shaft **14** can serve as an electrode for the outgoing current or for part of it in order to influence the production within the second vessel or to electrically cut it off from production. At the same time, when current is flowing through the graphite tube, and the latter is connected with the vacuum system, condensing of aluminum within the tube is eliminated by the heat effect of the current. By this method, the current for the second vessel **2** is reduced, or the second vessel is practically cut off. In case the tube or the shaft **14** becomes connected with the bar for the incoming electric current, the process can temporarily be effected without the first vessel **1**. In case the shaft **14** itself, for example, as a graphite tube, is connected with the vacuum system, its height has to correspond to the conditions of the vacuum. The shafts can also be arranged at another place or at other places in the production unit.

EXAMPLE

According to the above process, refined aluminum and aluminum alloys can be produced as follows:

Crushed bauxite is dried in a story-furnace and at the same time the iron oxides are partly reduced. The dried and hot bauxite next enters a rotary kiln, of which the outgoing gases, together with other gases, are used in the aforementioned story-furnace. The bauxite, mixed with carbonaceous material, is exposed to the reducing effect of a mixture of gases containing carbon monoxide and hydrogen. The hot material, the compounds of iron being for the most part reduced to metallic iron, enters through an opening, an otherwise closed large vessel that contains liquid fluoride bath which has been made of cryolite.

Carbon electrodes, connected with the secondary windings of a three-phase tranformer, extend deep into the fluoride bath which is covered with carbon in surplus. In the large vessel, the impure alumina is dissolved in the fluoride bath, whereas the spongy iron accumulates in the lower part of the vessel. Through a shaft, the specific heavier parts of the bath with the spongy iron in it flow upwards into a second vessel when liquid fluoride bath, taken out of the electrolysis plant, is poured into the large vessel. After the part of the bath containing the iron thus has been pushed away into the second vessel, direct current is switched onto the electrodes in the large vessel. The carbon bottom of this large vessel is the negative electrode. Electrolysis is continued as long as the iron has practically all been deposited. When adding once more fluoride bath out of the transport crucibles, then the part with the iron also flows through the shaft into the second vessel. The impure bath from the second vessel is electrically heated so that the spongy iron flows together forming liquid iron and can be separated form the bath, that is above the iron. If necessary, this part of the circulating bath can be purified of the calcium oxide accumulated in it by means of known processes and then added into the bath in the large vessel.

In the large vessel, containing bath that is practically free of iron, more bath is poured upon the fluoride bath and by this the raw fluoride bath, saturated with alumina, is pushed from down through the shaft into a crucible for the transport of raw-fluoride bath. This raw-fluoride bath is put into the first production unit's first vessel through a suitable pouring opening. Impurities still in the raw-fluoride bath, such as iron, titanium, silicon, chromium, manganese, vanadium or traces of zinc, are electrolytically brought into the liquid aluminum-copper alloy, and they are separated from the alloy by segregation.

The bath can by several steps be refined further, by passing it from the first production unit successively into the second and third production units by means of large transport crucibles. It is thus so purified that, since it contains still enough Al_2O_3, this bath can be put into conventional cells for the production of primary aluminum.

Before starting the electrolysis and before charging the fluoride baths into the vessels, these and the channels are filled to a sufficiently high level above the bottom of the walls **5** with aluminum-copper alloy, so that the fluoride baths cannot mix while being poured in. Though the process can be started with originally impure fluoride bath, during production only pure, high-grade and purest aluminum come out. That is only possible because the working process and the apparatus are designed to secure a highly equalized running of the production units. Due to the process, the resulting aluminum alloy also has uniform composition, because it is continuously adjusted to the desired condition, without the interrupted steps of conventional production. With the above process, the electrolysis plant is almost fully mechanized, skilled workman and staff may be concentrated at the necessary places, the input of electric current, electrodes, fluoride bath, size of buildings for the plant, and time required are reduced. For cooperation with the copper industry, the new working process is a marked step forward because raw materials for the copper industry can be worked to aluminum-copper alloys in the electrolysis plants of the aluminum industry and because the total input of material and energy is less.

What is claimed is:

1. A process comprising forming first and second spaced bodies of liquid aluminum alloy, connecting said bodies through liquid aluminum alloy in at least one channel, superposing molten fluoride baths with alumina therein on said bodies and maintaining the baths separate from each other, inserting into the baths electrodes which are spaced from said bodies and which are constituted of carbon compounds of metal, metal alloys and metals, and passing current between the electrodes via said bodies and the liquid alloy in the channel, the electrodes associated with said first body losing material into said first body, the electrodes associated with said second body being formed of a material of which the melting temperature is at least one hundred degrees centigrade higher than the working temperature at the cathode, refined aluminum being withdrawn from said second body.

2. A process as claimed in claim **1**, comprising distilling aluminum out of said second body under reduced pressure.

3. A process as claimed in claim **2**, comprising adding aluminum to said first body while current is being passed between said electrodes.

4. A process as claimed in claim **3**, comprising adding supplemental fluoride bath with alumina therein to the bath associated with said first body while current is being passed between the electrodes.

5. A process as claimed in claim **4**, wherein the bath superposed on said first body includes fluorides of aluminum, barium, calcium and sodium.

6. A process as claimed in claim **4**, wherein the alloy includes from one-fifth to two-thirds of copper.

7. A process as claimed in claim **4**, comprising supporting the bodies in vessels which are dehumidified before being loaded.

8. A process as claimed in claim **4**, comprising electrically heating said first body and controlling the heat of said second body from said first body.

9. A process as claimed in claim **4**, comprising spacing the electrodes from said second body at a greater distance than the electrodes are spaced from said first body.

10. Apparatus for processing aluminum, comprising a first vessel adapted for containing liquid aluminum alloy, a second vessel adapted for containing liquid alloy, said vessels being spaced from each other, means defining at least one channel connecting the vessels at lower portions thereof and through which said liquid alloy can flow from one vessel to the other, and electrodes extending downwardly into said vessels to electrolysis positions, said vessels extending above said at least one channel to encompass said electrodes at least in part and to accommodate molten fluoride baths superposed on the liquid alloy, said vessels and means cooperatively isolating the baths from each other, whereby current can flow from one vessel to the other through alloy in said channel.

11. Apparatus as claimed in claim **10**, wherein said means defines an opening above the said channel adapted to accommodate a bath of molten salt superposed on the alloy in said channel.

12. Apparatus as claimed in claim 11, comprising means defining a draw-room in the production unit consisting of said first vessel, said at least one channel and said second vessel and adapted to receive alloy at the lower part thereof and to accommodate a bath of molten salt superposed on the alloy in the draw-room.

13. Apparatus as claimed in claim 12, wherein the material for the electrodes in the first vessel is carbon, a compound of metal, a metal alloy or a metal.

14. Apparatus as claimed in claim 13, comprising means defining in said first vessel a vertical shaft of which the lower opening is above said liquid alloy and of which the upper opening serves as a spillway for the liquid bath.

15. Apparatus as claimed in claim 13, comprising means defining a vertical shaft extending down into said alloy and enabling the distillation of aluminum under reduced pressure.

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