Aug. 17, 1937. O. KRUH 2,090,451

MANUFACTURE OF ALUMINIUM

Filed May 29, 1935 2 Sheets-Sheet 1

Fig.1

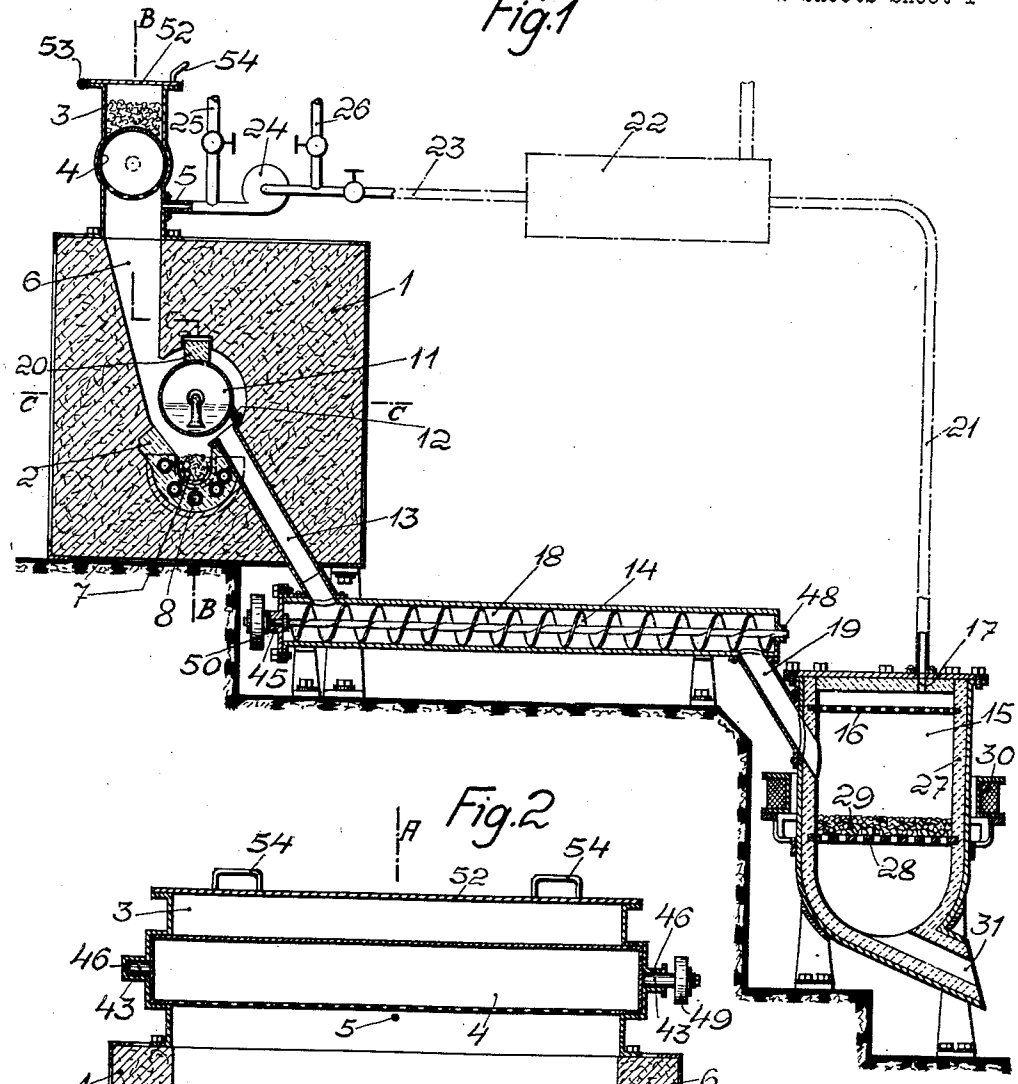
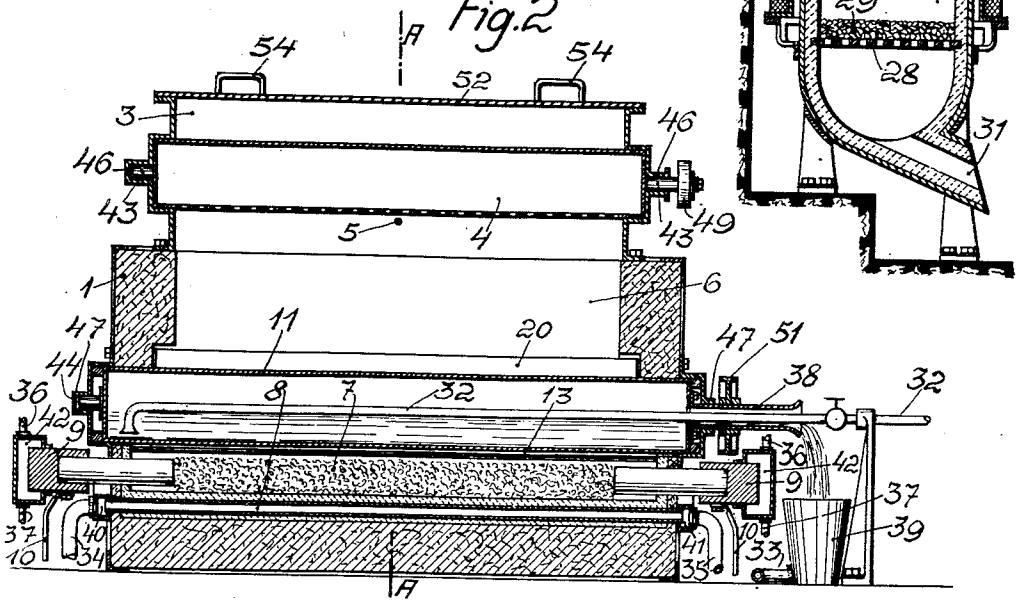



Fig.2



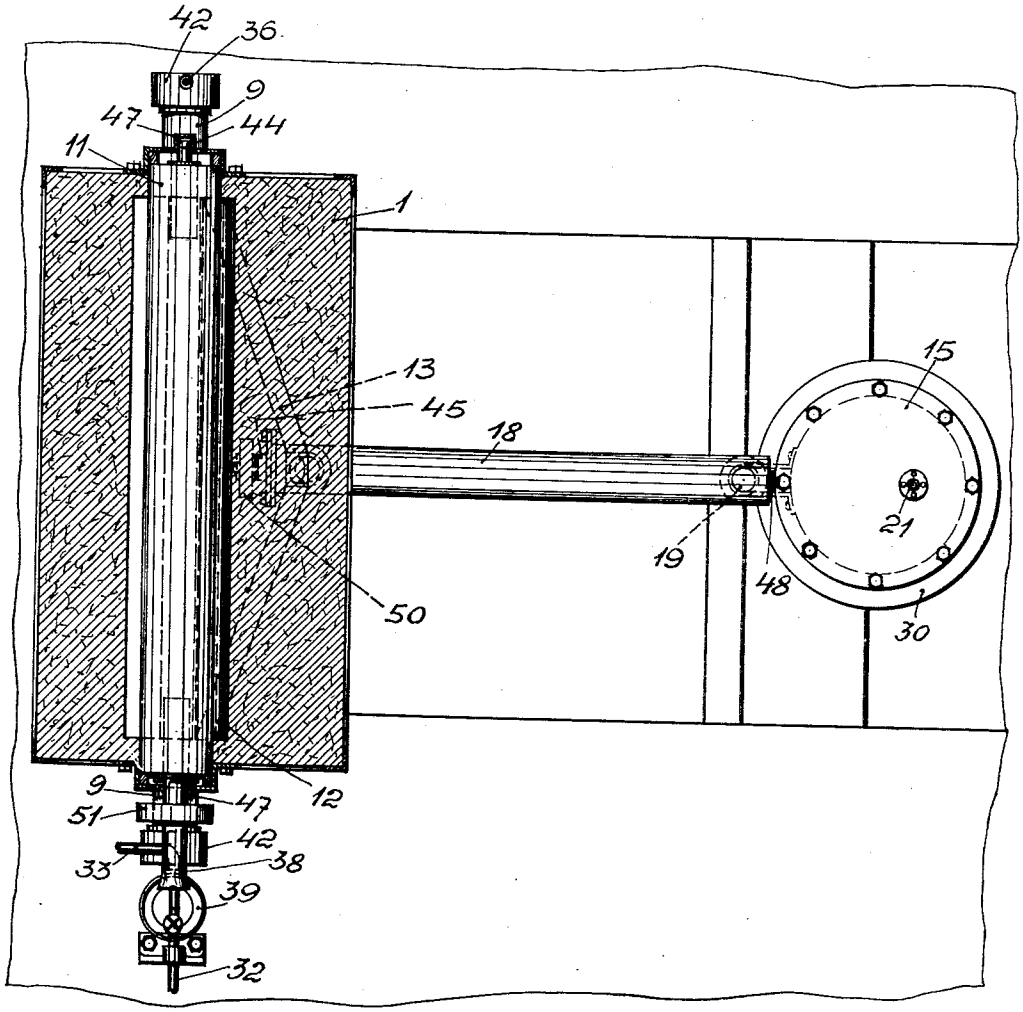

Inventor
Otias Kruh
By Bonnie Davis Marvin & Edmonds
Attorneys

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MANUFACTURE OF ALUMINIUM

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Fig.3




Inventor
Oscar Kruh
By Pennie Davis Marvin & Edmonds
Attorneys

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# UNITED STATES PATENT OFFICE

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## MANUFACTURE OF ALUMINIUM

Osias Kruh, Vienna, Austria

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In Austria June 5, 1934

10 Claims. (Cl. 75—68)

My invention relates to the production of pure aluminium by the process of reducing aluminium oxide by means of carbon or carbonaceous reducing agents at a high temperature under certain new conditions. Experiments have already been made to reduce aluminium oxide by carbon at a high temperature; however, without success. It was, for instance, proposed to form a compressed bar of a mixture of aluminium oxide with carbon and to use this bar as an electrode in an arc-discharge furnace in the presence of an inert or reducing gas like nitrogen, hydrogen or carbon hydride. Trials based on this proposal failed for the reason that the gas having no access to the inner particles of the mixture, the reaction stops at once after reduction of the surface layer of the bar. It has further been proposed to reduce a mixture of aluminium oxide with different hydrocarbons such as turpentine together with soot, this mixture forming a paste, and heating this mixture in a furnace in an atmosphere of acetylene at a high temperature. The acetylene was expected to prevent the reoxidation by the air of the aluminium formed. This proposal could not succeed, as acetylene decomposes at a high temperature, and forms soot which soon fills the furnace. This soot combines with the aluminium produced to form aluminium carbide. Another proposal was made to reduce aluminium oxide by carbon in vacuo at a high temperature with the idea that carbon monoxide, formed on account of the reaction, could be removed quickly from the reaction chamber, and that aluminium could be so distilled without being oxidized by the carbon monoxide. This third proposal could not succeed either, because the carbon monoxide and the aluminium have the same weight and, therefore, the same velocity. On account of those difficulties aluminium has hitherto been manufactured usually by an electrolytic process. Pure aluminium oxide is dissolved in fused cryolite and mixture of cryolite and electrolized. Aluminium is deposited at the cathode, and forms there a liquid which is scooped off from time to time. The production of one kilogramme of aluminium requires theoretically 23.5 kwh.; in practice, however, 28–30 kwh. are consumed. This large consumption of electrical energy in the electrolytic process has made the cost of aluminium high. The cost of the high current generators, the regulation of the voltage of the bath and the necessity of using only very pure aluminium oxide increase the cost of the aluminium further.

My invention has for its object to obviate those disadvantages, and to make the production of aluminium simpler, and cheaper. This is possible since the production of one kilogramme of aluminium by the new process requires only about 10 kwh. The cost of the electrical energy is considerably lower as alternating current of higher voltage may be used.

According to my invention aluminium is made by the following process: Aluminium oxide is loosely mixed with a carbon containing material (preferably in proportions corresponding to the stoichiometrical relation between aluminium oxide and carbon) and this mixture is heated in a reaction chamber to a high temperature, while a continuous protective gas flows through the reaction chamber, and carries away from the reaction chamber the gases formed by the reaction, mainly carbon monoxide, and the aluminium vapour. The aluminium particles are surrounded by the protective gas, and so protected from the attack by other gases present. The mixture of gas and vapour when leaving the reaction chamber is cooled at once to a temperature at which the aluminium particles remain intact. The aluminium vapour is precipitated to a greater part on a cooling surface, while the gases pass on. The precipitated aluminium is removed by mechanical means to a chamber where the aluminium is collected. That part of the aluminium that is not precipitated and is carried further by the gases is partly removed from them by a conveyer screw, and the last traces are retained by a filter.

I have found that it is advantageous to give the reaction chamber the shape of a hollow channel, and to set immediately above the reaction chamber a cooling device extending along the full length of the chamber. The cooling device consists of a rotating cylinder cooled from the inside so that the ascending aluminium vapour is precipitated on its surface from which the aluminium is scraped off by means of a scraper and removed from the furnace.

The contents of the reaction chamber may be heated in any known manner, the best method being to utilize electrical power.

The use of the hydrogen as a protective gas has given very good results, and the reaction involves an intermediate formation of acetylene. At the high temperature of the furnace the hydrogen reacts with the carbon to which aluminium oxide is admixed, and considerable quantities of acetylene are formed. The energy required for the formation of acetylene is provided for by the heat of the furnace, since this re-

action is endothermic. This energy is by no means lost, since it is recovered at the reduction of aluminium oxide by acetylene, the latter being a very effective reducing agent. The hydrogen of the consumed acetylene is set free and instantaneously forms with the carbon present fresh acetylene on account of the temperature equilibrium between the hydrogen, carbon, and acetylene being affected by the disappearance of the acetylene. The velocity of the reaction is further increased by the circumstance that acetylene reacts more readily with aluminium oxide than does carbon. The reduction of aluminium oxide may be attained by acetylene alone without coal; hydrogen, however, must be mixed with the acetylene in a proportion corresponding to the temperature equilibrium between hydrogen, carbon, and acetylene.

The amount of protective gas required for removal of the gases produced by the reaction, especially carbon monoxide, and for the transportation of the aluminium vapour is not great, since the molecular weight of hydrogen is small, and the average molecular velocity of hydrogen is much higher than that of carbon monoxide and that of the aluminium vapor, provided the calculation of the velocities be based on the same temperature. For instance, the average velocity of the hydrogen molecules is 3.74 times higher than that of the molecules of the carbon monoxide, and, accordingly, the hydrogen fills the space 3.74 times more than carbon monoxide. Therefore, by using an amount of hydrogen about twice that of the carbon monoxide, the space will be filled 7.48 times more by hydrogen than by carbon monoxide, and this will be sufficient to protect the aluminium particles from the attack by carbon monoxide, as there are always hydrogen molecules interfering between the molecules of aluminium and carbon monoxide.

Hydrides of carbon, or mixtures thereof may be used for the reduction of aluminium oxide, but hydrogen must be added in the correct proportion, as otherwise soot is formed. The correct proportion may be determined in advance by well-known simple preliminary, quantitative tests to that end.

The accompanying drawings illustrate diagrammatically a device for carrying out the process according to my invention and referring thereto:

Fig. 1 is a partly sectional side elevation, taken on lines A—A of Fig. 2; Fig. 2 is a partly sectional front elevation, taken on lines B—B of Fig. 1; and Fig. 3 is a partly sectional plan view, taken on lines C—C of Fig. 1.

In the furnace 1 there is provided an elongated shallow channel 7 which serves as a reaction chamber for the production of aluminium. The walls of this chamber consist of a lining 2 of fireproof material, protected from the action of excessive temperatures by the cooling tubes 8.

The reaction chamber 7 communicates through a channel 6 with a top 3 of the furnace, this top being provided with a rotary sieve 4 and, at a lower point, with a gas inlet 5. In order to carry out the process of reducing aluminium oxide the reaction chamber 7 is to be filled with this oxide, with the reducing agent and with the protective gas. For instance a mixture of aluminium oxide and coal, in the correct proportion, is supplied to the chamber 7 through the top 3 and the channel 6, the sieve 4 being rotated with proper speed to maintain the balance between the material fed to the reaction chamber and the products leaving the same, and at the same time hydrogen enters at 5 and is introduced into the chamber through the channel 6. Electric current is supplied to the reaction chamber by the leads 9 and the electrodes 10, provided on both sides of the chamber. By the passage of the electric current through the mixture of aluminium oxide and carbon contained in the reaction chamber the mixture is heated to a high temperature, and in the presence of hydrogen the reduction of aluminium oxide to aluminium takes place.

The protective gas flows through the reaction chamber and carries reaction gases, mainly carbon monoxide, and the aluminium vapour towards an internally cooled rotary cylinder 11, situated above and along the reaction chamber. This gaseous mixture is cooled by the cylinder to a temperature at which carbon monoxide is no longer able to oxidize aluminium. The aluminium vapour is precipitated on the surface of the cylinder to a kind of metallic snow, and is scraped off the cooling cylinder by a movable scraper 12, and conveyed through the funnel-shaped chute 13 to a cylindrical channel 18 wherefrom it is transported by a conveyor screw 14 through the discharge pipe 19 to the chamber 15 where the aluminium collects. The part of the aluminium not precipitated on the cooling surface but carried further by the flowing gas mixture settles on the conveyor screw 14, and the last traces of aluminium are retained by the filter 16 in the chamber 15. A block-shaped packing 20 interposed between the top of the cylinder 11 and the inner wall of the furnace 1 prevents the flow of hydrogen from shunting the reaction chamber 7.

The mixture of hydrogen and carbon monoxide leaves the chamber 15 at 17 and may be used, then, for any purpose. I prefer, however, to recover the pure hydrogen from this mixture and to reconduct it into the furnace 1 in order to obtain in the device a closed circulation of the protective gas. To this end, as shown in Fig. 1 by the dot and dash lines, the mixture of gases is passed from the chamber 15 through the conduit 21 to a device 22 wherein the hydrogen is separated from the carbon monoxide and from other impurities, and returns to the furnace through the conduit 23, a pump or exhauster 24 of known construction being inserted in this conduit for maintaining the required circulation of the hydrogen. Fresh hydrogen may be supplied to the furnace or may be entered into circulation by the pipe 25 or by the pipe 26. Devices for separating pure hydrogen from a gaseous mixture containing hydrogen are well-known in the art and their special construction does not constitute any part of my invention. Thus by the rectangle 22 I only have indicated where such a device is to be disposed without showing the known parts of the device.

The finely divided aluminium collected in the chamber 15 may be used in this state for paints, pyrotechnics, for chemical or other purposes. It may be cast into bars and used for any purpose for which aluminium is manufactured.

Preferably, the casting process may be carried out at once in the collecting chamber 15 so that it takes place in the presence and under the protective action of the protective gas (f. i. hydrogen). For this purpose the chamber 15 may be constructed in any known manner, but preferably in the manner shown in Fig. 1 by way of example. In this figure the inner walls of the

chamber 15 are covered with a lining 27 consisting of a refractory material which does not react with molten aluminium, for instance cryolite, magnesite or any other suitable stone or brick, but I prefer to use sintered aluminium oxide, as this lining material gives excellent results. The aluminium collected in the chamber 15 is melted upon a casting bed which is constructed like a filter and consists of a perforated bottom 28 carrying a heaped layer 29 of a refractory material incapable of reacting with the aluminium at elevated temperatures. This material may be the same as that of the lining 27, for instance cryolite, magnesite or the like, but preferably sintered aluminium oxide. To melt the aluminium any suitable heating means may be provided for, preferably electrical means, for example a high-frequency coil 30 arranged outside the chamber 15, as shown in Fig. 1. The construction of electrical heaters for melting and casting purposes is well-known in the art and, therefore, does not constitute a part of my invention. The melted aluminium passes into the bottom part of the chamber 15 through the filter 28, 29 which retains any impurities, and may be discharged from the chamber 15 through the outlet.

The device is to be fitted with the required number of cooling and driving implements, it being understood that I do not claim the special constructions of such implements, since they are frequently used in metallurgical and electrometallurgical processes. In the device illustrated by the drawings the cylinder 11, the tubes 8 and the electrodes 10 are to be cooled by water during operation. For this purpose water inlet pipes 32, 34, 36 and water outlet pipes 33, 35, 37 are provided for. The pipe 32 projects into the interior of the cylinder 11 so as to discharge cooling water thereinto. The water leaving the cylinder 11 through the spout 38 flows into the vessel 39 and from there into the pipe 33. The ends of the cooling tubes 8 are fitted into water distributing chambers 40 and 41 which are connected to the pipes 34 and 35, respectively, so as to permit the cooling water to flow from 34 through 40, 8 and 41 into 35. Each electrode 10 is partly surrounded by a water-filled casing 42 which is connected on one side to the inlet pipe 36 and on the other side to the outlet pipe 37.

The axles 43, 44 and 45 of the sieve 4, of the cylinder 11 and of the conveyor 18, respectively, are rotatably mounted in bearings 46, 47 and 48, respectively. The axles 43 and 45 are provided with pulleys 49 and 50, respectively, for being rotated by the latter with proper speed. The cylinder 11 is driven by a pulley 51 which is secured to the cylindrical portion of the tube 38. Of course, the pulleys 49, 50 and 51 may be replaced by any other driving means, for instance by electric motor drives.

The top 3 of the furnace 1 may be closed by a cover plate 52 pivoted about 53 and provided with handles 54.

What I claim is:

1. The process of manufacturing aluminium, which comprises preparing a loose mixture of aluminium oxide and carbonaceous reducing material so that gas brought into contact with said mixture has access to the inner particles thereof; exposing said mixture to a flowing protective gas inert to aluminium while heating the mixture to a temperature sufficient to reduce the aluminium oxide by the action of the carbonaceous material and to vaporize into said protective gas the aluminium formed by reduction; carrying away the composite of aluminium vapour, protective gas, and gaseous reaction products formed during the said reduction process; and cooling said composite to a temperature sufficient to transform the aluminium vaporized into the solid state and to prevent the aluminium particles from reacting with the said gaseous reaction products.

2. The process of manufacturing aluminium, which comprises reducing aluminium oxide by carbonaceous material in the presence of a gas mixture containing hydrogen and acetylene, said reducing process being carried out at a temperature sufficient to vaporize into said gas mixture the aluminium formed by reduction.

3. The process of manufacturing aluminium, which comprises preparing a loose mixture of aluminium oxide and carbonaceous reducing material so that gas brought into contact with said mixture has access to the inner particles thereof; exposing said mixture to flowing hydrogen while heating the mixture to a temperature sufficient to reduce the aluminium oxide by the action of the carbonaceous material and to vaporize into said hydrogen the aluminium formed by reduction; carrying away the composite of aluminium vapour, hydrogen and gaseous reaction products formed during the said reduction process; and cooling said composite to a temperature sufficiently low to prevent the aluminium particles from reacting with the said gaseous reaction products and to condense the aluminium vapours so as to obtain the aluminium in the solid state.

4. The process of manufacturing aluminium, which comprises loosely mixing aluminium oxide and carbon; heating said mixture to a temperature sufficient to reduce said oxide by the action of the carbon and to vaporize the aluminium formed thereby; surrounding the vaporized aluminium particles with a protective gas inert to aluminium so as to protect said particles from the chemical attack by other gases present; carrying away the gaseous mixture of aluminium vapour, protective gas, and reaction gases formed during the reduction process; cooling said gaseous mixture to a temperature sufficient for preventing the aluminium particles from being oxidized by the said reaction gases; and removing from the said gaseous mixture the aluminium in the solid state.

5. The process of manufacturing aluminium, as claimed in claim 1, wherein the aluminium oxide and the carbonaceous material are mixed in proportions corresponding to the stoichiometrical relation between aluminium oxide and carbon.

6. The process of manufacturing aluminium, which comprises reducing aluminium oxide by acetylene in the presence of hydrogen at a temperature sufficient for vaporizing into said hydrogen the aluminium formed by reduction.

7. The process of manufacturing aluminium as claimed in claim 1 wherein the said protective gas contains hydrogen maintained in a proportion which is sufficient to protect the aluminium particles from the attack by other gases present and to prevent the formation of aluminium carbide.

8. The process of manufacturing aluminium as claimed in claim 3, wherein the amount of hydrogen supplied to the process is at least twice that of the carbon monoxide formed by the reduction of the aluminium oxide.

9. The process of manufacturing aluminium, which comprises reducing aluminium oxide by carbonaceous material in the presence of a gas

mixture containing hydrogen and acetylene in proportions corresponding substantially to the temperature equilibrium between hydrogen, carbon and acetylene, said reducing process being carried out at a temperature which is sufficiently elevated for vaporizing into the said gas mixture the aluminium formed by reduction.

10. The process of manufacturing aluminium, which comprises heating a loose mixture of aluminium oxide and carbonaceous material in a reaction chamber to a temperature which affords the reduction of the aluminium oxide by carbon and the vaporization of the aluminium formed thereby; supplying to said chamber a protective gas inert to aluminium; withdrawing from the reaction chamber the mixture of aluminium vapour, protective gas, and gaseous reaction products formed during the said reduction process; cooling said mixture immediately after leaving the reaction chamber, to a temperature sufficient to prevent oxidation of the aluminium and formation of aluminium carbide and to condense the aluminium vapour; carrying away the condensate of aluminium from the reaction chamber together with the said gaseous mixture; and separating from this latter the aluminium in the solid state.

OSIAS KRUH.