METHOD OF EXTRACTING ALUMINUM FROM ALUMINUM-SILICON ALLOYS BY LOW PRESSURE

Max J. Spendlove, Takoma Park, Md., and Herbert S. Caldwell, Washington, D. C., assignors to the United States of America as represented by the Secretary of the Interior

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The invention described herein may be manufactured and used by or for the Government of the United States for governmental purposes without the payment to us of any royalty thereon in accordance with the provisions of the Act of March 3, 1946 (60 Stat. 523). This invention relates to the production of metallic aluminum and more particularly to the production of aluminum from alloys thereof with silicon. Broadly stated, this invention relates to methods and means for the extraction of high-grade metallic aluminum from low-grade alloys thereof comprising silicon as a major component. Heretofore, aluminum has largely been produced from imported bauxite, or the like, which necessarily made domestic aluminum largely dependent upon foreign sources of supply. More recently, there has been reported a process for the reduction of clays with coke to yield aluminum alloys containing from about 45 to about 35 percent by weight of silicon, the balance being largely aluminum contaminated with minor proportions of iron, titanium and the like. In an effort to produce high-grade aluminum from the foregoing alloys thereof with silicon, zinc and other metals have been employed in a distillation process as set forth in United States Patent Nos. 2,196,673 and 2,513,339 in the name of Hirsch Loewenstein. However, the prior processes are subject to the disadvantage that very large quantities of zinc or the like are required per unit of aluminum extracted. It has now been found, by the invention to be described below, that high-grade aluminum can be produced employing as little as 2½ parts of zinc per part of aluminum, and that substantially the entire quantity of zinc can be recovered for re-use.

Accordingly, this invention has for its principal object the provision of a method and means for the systematic extraction of high-grade metallic aluminum from alloys thereof with silicon employing zinc as the extraction agent in cyclic fashion. Another object is to provide suitable apparatus for efficiently conducting the extraction in cyclic fashion with minimum losses of heat and materials. Other and further objects will be apparent as the ensuing description proceeds.

The foregoing and related objects hereinabove apparent are accomplished by this invention wherein there is provided a method for the production of metallic aluminum from alloys thereof with silicon which comprises extracting such alloys at a temperature between 382° C. and 600° C. at an absolute pressure not higher than 100 microns Hg with liquid zinc, whereby aluminum is dissolved and silicon remains undissolved, separating the zinc-aluminum alloy from the undissolved silicon, then evaporating the aluminum alloy and returning the zinc for further extraction of silicon alloy as before until extraction is completed, and lastly, condensing the zinc vapors apart from the aluminum and silicon.

In the preferred embodiment the invention provides apparatus for the production of metallic aluminum from alloys thereof with silicon wherein a closed receptacle is adapted to be evacuated to subatmospheric pressure, and contains circumferentially disposed means for heating a zinc-evaporation zone in the lower portion of said receptacle, having combined therewith a condensing zone including the cooling mass positioned in the upper portion of said receptacle, and means defining an extraction zone below said condensing zone adapted to receive condensed liquid zinc from said condensing zone, said extraction means comprising a vessel open at its upper end and adapted to hold coarsely granulated aluminum-silicon alloy and provided with an outlet near its lower end for discharging liquid zinc-aluminum alloy into said zinc-evaporation zone. The invention will be made clear by reference to the ensuing description and accompanying drawings in which:

Fig. 1 is a sectional elevation of one form of apparatus for carrying out the invention.
Fig. 2 is a sectional elevation of a modified form of apparatus for carrying out the extraction wherein the upper portion of the extraction zone is below the lower portion of the extraction zone. Suitable alloys of aluminum and silicon for extraction in accordance with this invention including the alloys containing from about 45 to 35 percent by weight of aluminum and from 35 to 20 percent by weight of silicon, the balance being aluminum contaminated with minor proportions of iron, titanium and the like. Obviously, the higher grade alloy is more easily extracted but this invention will cleanly separate aluminum from its alloys with silicon in substantially all proportions.

A suitable proportion of zinc for employment as an extractant in accordance with this invention comprises about 2.5 parts by weight of zinc per part by weight of aluminum to be extracted up to 11 or more parts by weight of zinc per part by weight of aluminum to be extracted. One of the significant advantages of this invention resides in the ability of the apparatus to separate and purify the aluminum alloy on a continuous basis, thereby substantially the entire of the aluminum content is extracted from the alloy thereof with silicon. In general, it is preferred to employ between about three to five parts by weight of zinc per part by weight of aluminum to be extracted from its alloy with silicon.

The suitable temperature for extracting the aluminum content of the silicon alloy thereof with zinc in the liquid phase lies between the melting point of the zinc-aluminum eutectic mixture (about 382° C.) up to the boiling point of the zinc under the low absolute pressure employed. In general, it is preferred to extract the aluminum with molten zinc at a temperature between about 419.4° C. and 450° C. Inasmuch as considerable heat is saved and the extraction is most efficiently performed at a temperature just sufficient to insure the maintenance of all zinc employed in liquid phase.

The extraction of the alloy is completed by repeatedly passing the molten zinc over the alloy to be extracted, then separating the dissolved zinc from the dissolved aluminum, and repassing the molten zinc over the alloy to be extracted. The separation of the zinc from the aluminum is accomplished by evaporating the zinc from the extracted aluminum. A suitable temperature at which to evaporate the zinc from the aluminum under the preferred low-pressure conditions of this invention, lies between about 540° C. and 850° C. but in general it is preferred to employ a more restricted temperature range for evaporation between about 540° C. and 600° C. A higher evaporation temperature will, of course, result in a faster circulation rate for the zinc extractant but on the other hand requires for best results.
that the zinc vapors be condensed and cooled to the preferred extraction temperature as set forth above.

The normal boiling point of zinc under atmospheric pressure is about 907°C. It has been found that by carrying out the entire operation on an absolute pressure of not higher than 100 microns Hg, that the extraction efficiency is very markedly enhanced over any procedures heretofore known while at the same time the separation of the zinc from the produced alloy thereof with aluminum can be accomplished at a temperature which is but very little higher than that required for the most efficient extraction. Thus, it is preferred to carry out both the extraction and the evaporation at an absolute pressure lying between about 25 and 75 microns of mercury. By virtue of this total pressure, the extraction of the aluminum-silicon alloy can be carried out as low as 420°C to 425°C whereby as high as 80 percent extraction is quickly accomplished. Quantitative extraction of all aluminum in the alloy thereof with silicon can be accomplished under the preferred pressure conditions at this temperature range with but very few passes of zinc over the alloy to be extracted.

Under the low total pressure preferred of about 25 to 75 microns Hg, the effluent zinc-aluminum alloy, low in silicon, and the extraction zone is led into the evaporation zone and there purged of its zinc content by evaporation at a temperature but slightly higher than that prevailing in the extraction zone. With the preferred apparatus to be described below, it is possible to control the zinc circulation rate with ease by controlling the heat input to the zinc evaporation zone. In general, it has been found desirable to heat the evaporation zone to a temperature between about 450°C to 650°C depending upon the rate at which it is desired to recirculate the zinc to the condensing and extracting zones.

When the extracted aluminum-zinc alloy is heated, the zinc immediately begins to evaporate and the vapor is led to a condensing zone positioned to return liquid condensed zinc free of aluminum back to the extraction zone. In general, it is desired to condense and cool the zinc to a temperature at which extraction of the aluminum-silicon alloy can be most efficiently accomplished. Accordingly, the condensing zone is maintained at a temperature sufficient to yield condensed zinc at about 420°C to 425°C.

For a practical embodiment of suitable apparatus in which to carry out the process of this invention, and referring now to Fig. 1, there is shown a flexible gasket 23 serves to maintain a fluid-tight engagement between the cylinder 22 and the ring 19. Surrounding the cylinder 22 at its lower portion adjacent the gasket 23 is a cooling pipe 24 adapted to prevent the disintegration of the gasket 23 during operation at high temperatures. Surrounding the cylinder 22 is a second ring 25 sealed to the cylinder 22 by a gasket 26. Rigidly affixed to the ring 25 and extending downward therefrom is a cooling cylinder 27 provided with a depending carbon cylinder 28. The carbon cylinder 28 is supported in vertically moveable relationship to the cooling cylinder 27 by a shaft 29 passing through a cap 30. The shaft 29 is threaded at its upper end and provided with a hand wheel 31 in threaded engagement with the shaft 29. A yoke 32 is mounted on the top of the cooling cylinder 27. The shaft 29 is rotationally connected with the handle wheel 31 as its dependent screw cylinder 28 whereby a chamber 34 is defined. The chamber 34 is provided with an inlet 35 near the lower portion thereof and an outlet 36 near the upper portion thereof. During operation cooling fluid, for example, water, is admitted through the inlet 35 passed upwardly around the member 28 and outlet 36. The shaft 29 when threaded and outward from the jacket 27 through the outlet 36.

In the cylinder 22, radially extending therefrom, is an exhaust pipe 37 adapted to be connected to a vacuum pump, not shown.

In operation, the vessel 13 is filled with a charge of aluminum-silicon alloy to be extracted. For initial operation, a charge of zinc is placed in the pot 11, the ring 12 and the member 17 are placed in the apparatus and the ring 25 with the cooling structure 27 is placed in position as shown. The entire structure is evacuated through the exhaust line 37 and the induction heating coil 8 is suitably energized from a source of high frequency electricity not shown. Upon reaching the boiling temperature, the zinc evaporates from the pot 11 proceeds upwardly through the spacing ring 12 through the orifice 36 in the inner portion of the vessel 13 and is condensed to the liquid form in the inverted member 17. The liquid zinc condensate is then dispensed through the neck of the member 17 into the vessel 13 and extracts metal aluminum from the contained charge of aluminum-silicon alloy. The resulting liquid alloy of aluminum and zinc passes downwardly through the outlets 16 into the pot 11 where the aluminum content remains and the zinc is re-evaporated for the extraction of further quantities of aluminum as before. The leaching vessel 13 until the termination of the process.
cooling member 28 is lowered by means of the hand wheel 31 into contact with the inverted member 17. Cooling fluid passes into the upper housing 27 through the inlet 35 and out through the outlet 36 whereby the member 28 extracts heat from the member 17 which in turn results in the accumulation of solidified vaporized zinc in the upper surface of the member 17. Thus, at the completion of the extraction and heating the zinc employed is evaporated from the purified aluminum remaining in the pot 11 and from the residual silicon remaining in the vessel 13 and collected on the upper inside surface of the member 17. The electric heating is then discontinued, the vacuum on the line 37 is slowly released and the apparatus can be opened by lifting the cooling assembly. Thereupon the member 17 is moved with its attached burden of solidified zinc and the vessel 13 is removed for disposal of its load of extracted silicon. The spacing ring 12 is removed and the pot 11 is removed for discharge of its volume of purified metallic aluminum. In repeating the cycle, it is expedient to replace the pot 11 by the member 17 containing the solidified zinc for use in extracting an additional batch of aluminum-silicon alloy.

A modified form of apparatus suitable for employment in the high-vacuum extraction of aluminum-silicon alloys with metallic zinc is shown in Figure 4. The supporting base structure 38 holds rigidly mounted thereon a housing 39. In the housing 39 is mounted an extraction pot 40 provided with a moveable bottom 41. The bottom 41 is made preferably of a dense form of carbon. Similarly, the extraction pot 40 is made of carbon in order to resist the corrosive action of molten alloys of aluminum and zinc. Horizontally disposed beside the extraction pot 40 is an evaporating pan 42 which is adapted to provide a large surface area and a thin depth for evaporating zinc from molten aluminum. The pan 42 is similarly constructed of dense carbon as are all the portions of the apparatus in contact with molten or vaporized metals. Connecting the extraction pot 40 and the evaporating pan 42 is a carbon tube 43 provided with an offset portion 44 whereby the liquid contents of the pot 40 are transferred to the pan 42 by gravity while the solid contents of the pot 40 cannot pass over into the pan 42. A tap hole 45 closed by a plug 46 is provided in the evaporating pan 42 for the ultimate withdrawal of molten aluminum from the apparatus.

Covering the pan 42 and the pot 40 is a dense carbon cover 47 which is in turn spaced apart from a metallic cover 48. The metallic cover 48 is joined to the supporting base structure 38 and the housing 39 to form a gas-tight housing for the entire assemblage. Suitable resistance heating elements 49 are disposed about the pan 42 and the body 40 in order to regulate the temperature at which evaporation and extraction take place. The housing 48 is provided with an exhaust outlet 50 for evacuating the entire assembly to an absolute pressure not higher than 100 microns of mercury. Mounted in the housing 48 over the pot 40 is a carbon hollow cooling block 51 adapted to condense the zinc vapors hinging on the lower side thereof and transfer the condensed liquid zinc into the pot 40.

The temperature of the cooling block 51 is regulated by cooling fluid admitted through the inlet pipe 52 and withdrawn through the outlet pipe 53. As shown, the outlet cooling pipe is co-axially mounted around the inlet cooling pipe 52 and the block 51 is suspended from and mounted upon the vertical portion of the outlet cooling pipe 53. The housing 48 is surrounded by a vacuum seal 54 by gasket 55. The offset portion 56 of the housing 39 is similarly capped by a plate 57 and sealed by a gasket 58.

In the operation of the device shown in Figure 2, the bottom 41 of the pot 40 is closed and a charge of metallic aluminum-silicon alloy is placed in the pot 40. The zinc vapors pass upwardly through the throat 59 and are condensed on the lower surface of the block 51 to the liquid form. Liquid zinc falls by gravity then back into the pot 40 for further extraction of metallic aluminum. Upon conclusion of the extraction cycle, cooling fluid is admitted through the pipe 52 to the inner portion of the block 51 and thence upwardly and outwardly through the outlet pipe 53. This strongly cools the block 51 and metallic zinc collects in solid form on the lower face of the block 51. When all of the zinc in the system has condensed in solid form upon the lower face of the block 51 the heat in the pot 40 is discontinued, the vacuum in the line 50 is slowly released to atmospheric pressure, and metallic aluminum is tapped off from the tap hole 45 by removing the plug 46 and associated vacuum-retaining seal 59.

After removal of metallic aluminum, the entire structure is cooled by discontinuing further heating and the bottom 41 of the pot 40 is dropped by moving the associated supporting plate 60 which serves the function of not only supporting the bottom of the plate 41 but maintains a vacuum in the housing 39. The structure is then ready for recharging with additional aluminum-silicon alloy and any small amounts of make-up zinc that may be required due to mechanical losses.

Since many apparently differing embodiments of this invention will appear to one skilled in the art, various changes can be made in the particular details shown and described without departing from the spirit and scope of this invention.

What is claimed is:

1. A method for the production of metallic aluminum from alloys thereof with silicon which comprises extracting aluminum from such alloys at a temperature between 382° C. and 600° C. at an absolute pressure not higher than 100 microns Hg., with liquid zinc, whereby the aluminum is dissolved and silicon remains undissolved, separating the zinc-aluminum alloy from the undissolved silicon, then evaporating the zinc from the alloy and returning the zinc for further extraction of silicon alloy as before until extraction is completed, and lastly, condensing the zinc from the aluminum and making up the aluminum for further use.

2. The process of extracting aluminum from an aluminum-silicon alloy, which comprises washing the alloy with molten zinc at an absolute pressure of between about 25 to 75 microns of mercury at temperatures of about 420° C. to 450° C., whereby rapid solution of the aluminum from the alloy is obtained.

3. The process of continuously extracting aluminum from an aluminum-silicon alloy, which comprises repeatedly washing the alloy in a closed circuit with a volume of molten zinc equal to about two and one-half times the amount of aluminum contained in the alloy, at an absolute pressure of between about 25 to 75 microns of mercury at a temperature of about 420° C. to 450° C., whereby rapid solution of the aluminum from the alloy is obtained.

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