June 21, 1966 W. SCHMIDT 3,257,199

THERMAL REDUCTION

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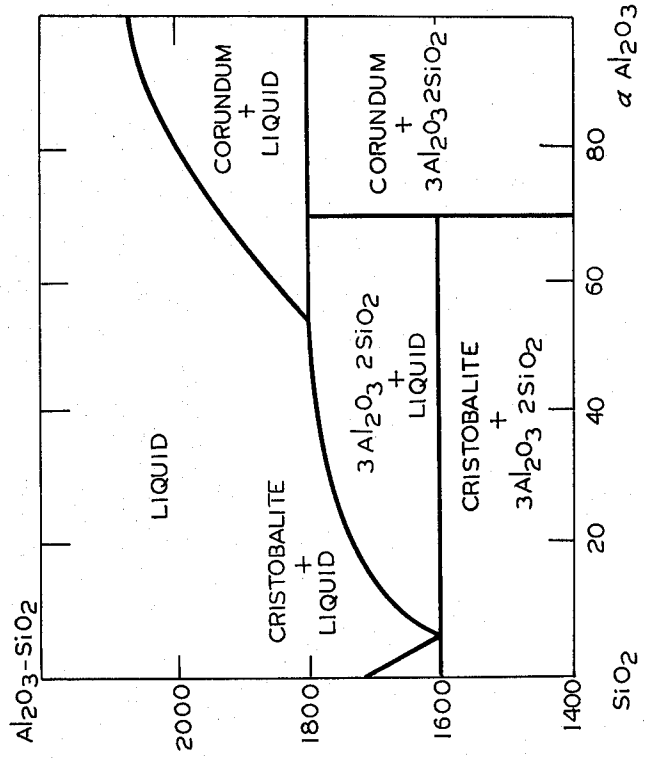



FIG. 1

INVENTOR

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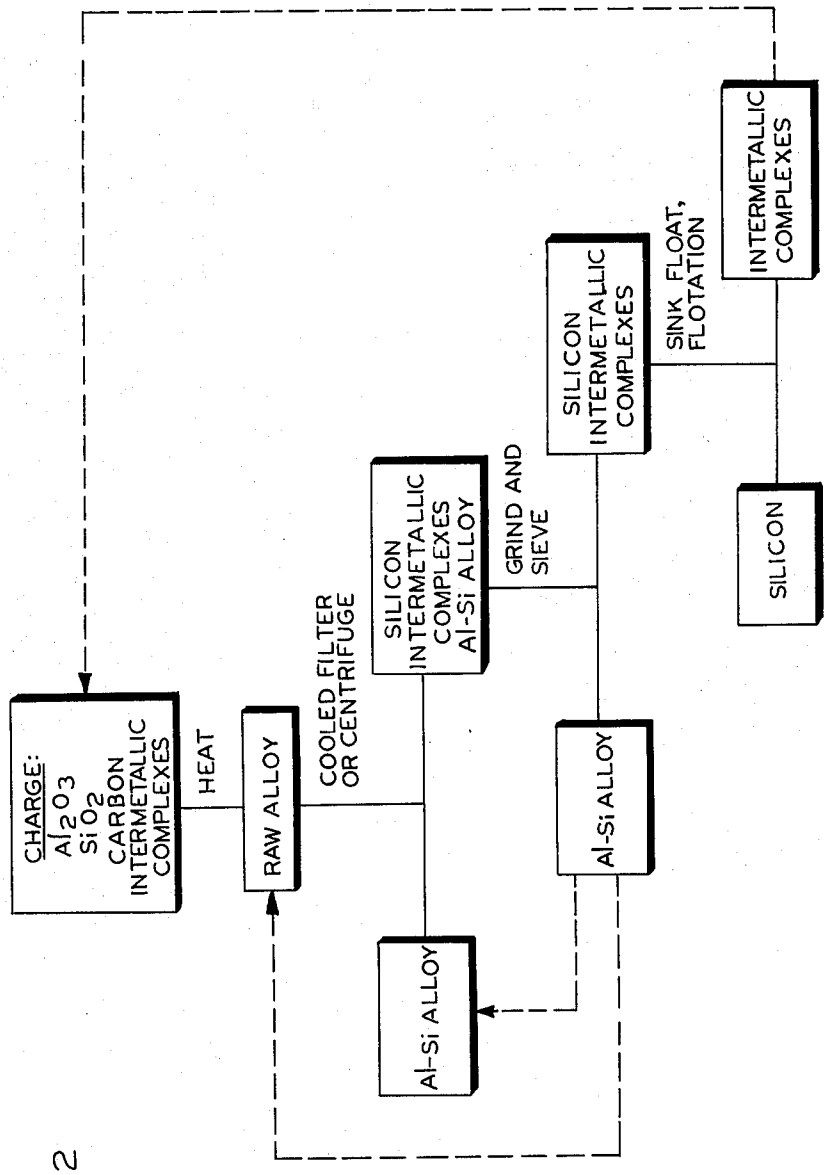



FIG. 2

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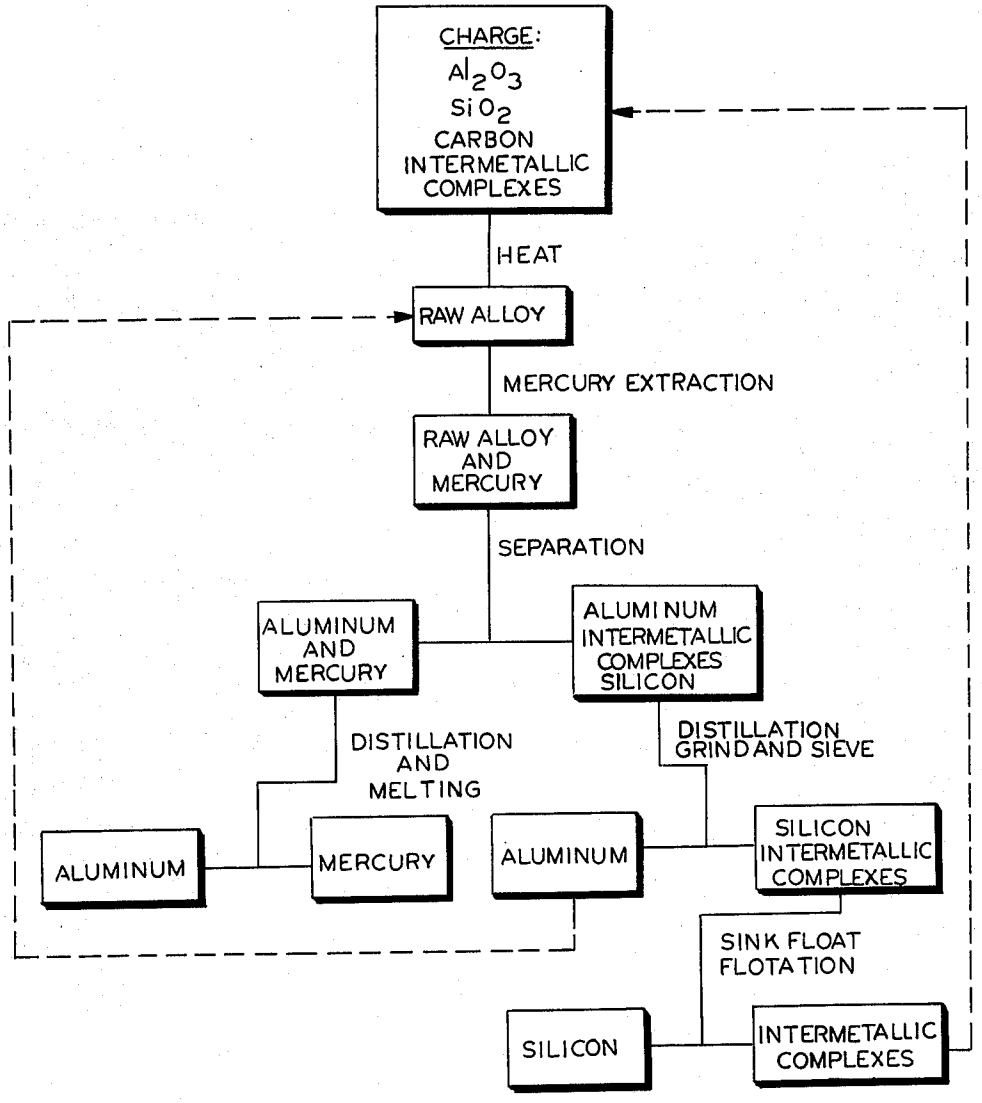



FIG. 3

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3,257,199

# THERMAL REDUCTION

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14 Claims. (Cl. 75—68)

This invention relates to the direct thermal reduction of alumina-bearing materials to form aluminum alloys and more particularly, to the thermal smelting of raw aluminum-silicon alloys for the specific purpose of further purifying them so as to obtain either partially purified alloys or aluminum of commercial purity.

The thermal reduction of alumina-bearing materials to form aluminum alloys is well known in the art and usually involves treatment of oxidic ores such as bauxite, clay, kaolin, kyanite, mixtures of alumina and silica, etc. with sufficient carbon, at elevated temperatures, in order to reduce the charge to the metallic state. However, the many problems attending such a reduction reaction are also well known in the art and have, in fact, seriously hindered the heretofore practiced thermal reduction processes from becoming economically optimized. As is well known, it takes a certain amount of energy, usually in the form of electrical energy, to reduce alumina-bearing materials to the corresponding liquid metal. If, in fact, the liquid metal cannot be fully recovered, a significant economic defect in the reduction process is developed in that expended energy, which has a direct relationship to cost, has served no useful purpose. In this regard, it is known, for example, that the aluminum metal resulting from the reduction of the alumina-bearing ores has a tendency to be vaporized out of the reaction zone and into the atmosphere, thereby detracting from the overall yield of aluminum. Additionally, and perhaps more significantly, the reduced aluminum metal easily reacts with the carbon present as the reducing agent or the carbon monoxide inherently formed in the reaction system to yield aluminum carbide. The presence of aluminum carbide is extremely undesirable mainly due to the fact that aluminum carbide can form a fused ternary system with aluminum and aluminum oxide. The fused ternary system is extremely viscous and creates many practical operating problems since it accumulates at the cooler parts of the furnace building up gummy masses which ultimately must be removed by shutting down the furnace and cleaning it.

There have been many proposals made in the prior art in an attempt to solve the problems hereinabove referred to but, for the most part, the prior art solutions have not been completely satisfactory. For example, it has been proposed to add heavy metals such as iron, copper and lead to the feed in order to dissolve the aluminum produced, thereby lowering its vapor pressure, so that the loss of aluminum by vaporization will be minimized. In addition, these heavy metals contribute gravity and bulk to aluminum, thereby enhancing the tendency of the liquid metals to rapidly settle down from the hot zone of the furnace to minimize the loss of aluminum due to vaporization. However, most heavy metals react with considerably more than their own weight of aluminum and silicon upon solidification to form intermetallic compounds which have little or no commercial significance. In this regard, the gains made by decreasing the amount of aluminum lost by evaporation were more than offset by the losses of aluminum and silicon due to their combination with the heavy metals added so that the overall effect of the process was to make it even more inefficient.

In addition, most of the heavy metals cannot counteract the formation of aluminum carbide because the latter is more stable than the carbides of the metals added.



Another proposal heretofore suggested involved counteracting the formation of aluminum carbide by the simultaneous reduction of silica to form silicon. It has long been recognized that 2 moles of aluminum carbide will react with 3 moles of silica to give 8 moles of aluminum plus 6 moles of silicon. The addition of silica to the feed was for the purpose of destroying the aluminum carbide formed by the reaction of the aluminum and the carbon present in the system. However, a very practical problem encountered in the use of silica is the fact that severe losses of aluminum from vaporization will result unless an excess of silica is employed in order to lower the vapor pressure of the aluminum. Thus, for commercial operations, a ratio of about 60% aluminum to about 40% silicon is a well established compromise between counteracting the formation of aluminum carbide and minimizing the loss of aluminum and silicon through vaporization.

However, when a relatively large amount of silica is employed as a furnace feed, the feed tends to form fused masses before silica will be substantially reduced. Reference to the phase diagram of alumina and silica which appears in FIGURE 1 will show that a ratio of 57% $Al_2O_3$ and 43% $SiO_2$ has a melting point of about 1810° C. It can be seen that operating with a relatively high portion of silica can lead to the formation of gummy viscous products containing oxides, carbides and reduced metals which tend to accumulate to the cooler parts of the furnace, thereby causing shutdown of the furnace and expensive cleaning operations. Moreover, it is conventional practice to charge the furnace feed in the form of briquets in which the carbonaceous reducing agent is finely divided throughout the briquet, and if fusion of the oxides occurs, the carbonaceous material has a tendency to segregate from the liquid, thereby leaving the oxide unreduced. Another problem encountered with the use of high amounts of silica is the fact that the fused masses which can form, crust over and hinder the escape of carbon monoxide, which is inherently formed in the reaction system. The rapid removal of carbon monoxide is necessary in order to prevent the formation of aluminum carbide and aluminum oxide by its reaction with the reduced aluminum. It is therefore essential to provide a furnace feed which does not readily fuse but tends to keep in shape until it is reduced to metal. Therefore, again with reference to FIGURE 1, it would be more preferred to operate to the right of the 1810° point, but at this point in the diagram insufficient silica would be present to counteract the formation of aluminum carbide as well as to sufficiently lower the vapor pressure of aluminum to minimize losses due to vaporization from the hot zone.

Therefore, it is the primary object of this invention to provide a novel process for the direct thermal reduction of alumina-bearing materials to form aluminum alloys.

It is another object of this invention to provide a novel process for the production of aluminum-silicon alloys wherein losses of aluminum due to vaporization from the reaction zone will be minimized.

It is a further object of this invention to provide a novel process for the production of aluminum-silicon alloys wherein considerably less silica is employed in the feed material thereby minimizing the danger of forming fused masses in the reduction furnace.

It is still another object of this invention to provide a novel process for the direct thermal reduction of alumina-bearing materials wherein materials of relatively high specific gravity are added to the furnace zone in order to aid in the rapid withdrawal of the formed aluminum from the reaction system.

It is still another object of this invention to provide a novel process for the direct thermal reduction of alumina-bearing materials wherein the formation of aluminum carbide is minimized but not at the expense of losing more aluminum due to the formation of intermetallic compounds by the reaction of the formed aluminum and the heavy metals added.

It has now been found that the above objects can be attained by carrying out the direct thermal reduction of alumina-bearing materials in the presence of intermetallic complexes of iron and/or titanium which have aluminum and silicon chemically bonded thereto. The intermetallic complexes, which are added to the feed in accordance with the novel teachings of this invention, form a liquid solution within the smelting furnace with the aluminum and silicon formed by reduction of the corresponding oxides. They are substantially saturated with both aluminum and silicon so that no further reaction with these elements will be possible when the raw alloy solidifies.

While not wishing to be bound by any specific theory of operation, it nevertheless appears that the use of the novel intermetallics of this invention will accomplish all of the objectives attempted to be solved by the heretofore proposed processes without the disadvantages which inherently accompanied them. Thus, the intermetallics which are added will provide the necessary bulk and gravity so that the liquid metal can be discharged from the hot zone at a very rapid rate thereby minimizing aluminum losses due to vaporization without the inherent disadvantages of having the added compounds react with both aluminum and silicon.

In addition, the intermetallics of this process will counteract the formation of aluminum carbide without the inherent danger of forming fused oxidic masses. Another advantage of the instant invention is the fact that the materials added will substantially lower the vaporization losses of aluminum since it will be dissolved by the intermetallics. However, of primary importance is the fact that the novel process of this invention will permit decreasing the silica to alumina ratio of the furnace feed thereby minimizing the danger of forming fused oxidic masses within the smelting furnace. As has heretofore been pointed out, the approximate ratio of alumina to silica in the heretofore practiced commercial smelting processes was usually 60:40 for the reasons hereinabove stated. The process of this invention will permit the use of furnace feeds having alumina to silica ratios of 75–87:25–13, and preferably 82–85:18–15.

The intermetallic complexes of this invention consist substantially of complexes of iron and/or titanium chemically combined with aluminum and silicon. The term "intermetallic complex" as used herein is intended to include both compositions having exact formulas as well as those which cannot be defined by a precise stoichiometrical formula. As is well known, ternary compounds, i.e., compounds of either iron or titanium with both aluminum and silicon, can be defined by known phase diagrams provided equilibrium conditions are assumed. However, in compositions containing both iron and titanium, simultaneously chemically bonded with aluminum and silicon, a wide variety of complexes are possible whether it be by solid solution of one compound in another or by isomorphic substitution of atoms of one metal in the intermetallic compound formed primarily with the other metal. Nevertheless, the intermetallic complexes which are added to the furnace feed in accordance with the instant invention are characterized by the fact that they have a relatively high density and densities between 3.4 and 4.3 grams per cubic centimeters are particularly preferred in order to achieve fast removal of the liquid metal from the hot zone of the furnace. Another characteristic of the intermetallic compounds of this invention is the fact that the iron and/or titanium complex is substantially fully satisfied with aluminum and silicon so that when the complexes are added to the



furnace feed they will not react, upon solidification of the raw alloy, with additional aluminum and silicon, thereby detracting from the overall economy of the operation. Thus, these materials provide heavier bulk, have the property of lowering the vapor pressure of aluminum and counteract aluminum carbide formation without the inherent defects of the prior art compounds in that they are so inert to aluminum and silicon that they will no longer react to remove these desired products to any substantial degree.

As has heretofore been stated, the intermetallic complexes which can be employed in the novel process of this invention are complexes of iron and/or titanium with aluminum and silicon. If the intermetallic complex is based on iron, its content of iron can range from 25–30% by weight of the complex. If it is based on titanium, its content if titanium can range from 25–45% by weight of the complex. If the complex contains both iron and titanium, then the combined amounts of iron and titanium should not exceed 55 weight percent of the total complex. The balance of the complex is composed of aluminum and silicon and the relative proportions of these two materials must be chosen to effect chemical saturation, which commonly will be achieved by selecting from a weight ratio of 0.05–2 aluminum:1–4 silicon. The particularly preferred intermetallic complexes are those wherein the aluminum is present in the higher end of the range in order to insure that the complex will be substantially saturated by chemical combination with the aluminum in order to minimize any possible reaction between the complex added and the aluminum inherently reduced in the furnace during solidification of the raw alloy.

The intermetallic complexes which are employed in accordance with the instant process can be prepared simply by melting an alloy containing the desired components, i.e., an alloy containing aluminum and silicon and either or both of titanium or iron. The relative amounts of the individual components in the alloy are generally determined by the weight ratios desired in the intermetallic complex. However, the weight ratio of the individual components in the alloy need not be exactly the same as the weight ratios desired in the intermetallic complex since the formation of the intermetallic complex is a rather complicated reaction and allowances can be made for additional side reactions which must inherently take place. However, it has been found that the novel intermetallic compounds of this invention can be prepared by melting an alloy which contains the following weight ratios:

For 1 part of iron, 0.5–1 part silicon, 1.5–2 parts of aluminum

For 1 part of titanium, 1 part of silicon, 0.05–0.5 part of aluminum

Another embodiment of this invention resides in replacing a portion of the iron and/or titanium with manganese and/or chromium, especially in those cases where it is desired to obtain a casting alloy as a product of the reduction furnace. The intermetallic complexes containing manganese and/or chromium are prepared by including manganese and/or chromium in the alloy which is to be melted. The amount of manganese and/or chromium which can be present in the intermetallic complexes is not narrowly critical and advisably should not exceed 10% by weight of the total composition.

As has heretofore been pointed out, the intermetallic complexes which are used in the process of this invention are substantially saturated with both aluminum and silicon so that reaction with these elements during the solidification of the raw alloy will be minimized. It is an absolute requirement in the instant process that the intermetallic complexes employed be at least 75% chemically saturated with aluminum and/or silicon in order to minimize reaction with these compounds. It is

to be understood, however, that the preferred embodiment of this invention would reside in those complexes wherein the chemical saturation with aluminum and silicon approaches 100%.

Specific examples of intermetallic complexes which can be employed in the novel process of this invention are shown in Table I below. In each case, the percentages are expressed as weight percents.

TABLE I

| Example | Percent Iron | Percent Manganese | Percent Chromium | Percent Titanium | Percent Aluminum | Percent Silicon | Density in Grams per Cubic Centimeter |
|---|---|---|---|---|---|---|---|
| 1 | 28 | | | | 44 | 28 | 3.46 |
| 2 | 28 | | | | 38 | 34 | |
| 3 | 25 | | | 3 | 40 | 32 | 4.08 |
| 4 | 10 | | | 25 | 29 | 36 | 4.03 |
| 5 | 16 | | | 19 | 30 | 35 | 4.05 |
| 6 | | | | 44 | 12 | 44 | 4.1 |
| 7 | 5 | | | 30 | 17 | 48 | |
| 8 | 28 | | | 26 | 24 | 22 | 4.3 |
| 9 | 18 | 5 | | 4 | 49 | 24 | 4.0 |
| 10 | 14 | 3 | 3 | 16 | 31 | 33 | 4.2 |

The novel process of this invention is carried out simply by adding the intermetallic complexes to a furnace feed in a conventional reduction furnace. The furnace feed is composed of three components, one being a carbonaceous reductant, preferably in the form of coke or charcoal; the second being a mixture of alumina and silica or alumina and silica-bearing ores, such as bauxite, clay, koalin, kyanite, or mixtures thereof; and the third component being the intermetallic compounds hereinabove referred to. The amount of intermetallics which are added to the furnace feed should represent between 15 to 50 percent by weight of the total amount of silica and alumina present in the feed. The variation in the weight ratio of intermetallics added is obviously dependent on the specific nature of the furnace feed. For example, if relatively pure components are employed, such as Bayer-alumina and quartz, it is then preferred to operate in the higher end of the range; whereas if impure feed materials are employed, it is then preferred to operate in the lower end of the range because these materials will inherently form some intermetallic compounds due to the impurities contained therein.

As has heretofore been pointed out, the ratio of alumina to silica in the feed is one which is considerably high in alumina, so as to minimize the danger of forming fused masses of unreduced oxides. Therefore, the charge in the novel process of this invention will comprise alumina and silica having a weight ratio of 75–87 parts of alumina and 25–13 parts of silica; 15–50 weight percent of the total weight of both silica and alumina of intermetallic complexes; and sufficient carbon as a reductant, in order to reduce the metal-bearing ores to the liquid metal. The feed materials are usually heated in an electric furnace at a temperature of about 2000° C. until the oxides are reduced to the metallic state and the liquid alloys are tapped. The liquid alloys are then allowed to cool and the intermetallics plus elementary silicon, if present in amounts above the eutectic aluminum-silicon composition, begin to crystallize out of the charge at about 1000° C. thereby enriching the liquid in aluminum. When cooling approaches the eutectic point which occurs at about 578° C., an aluminum-silicon alloy exists in the liquid phase which contains about 12 percent silicon and this liquid phase is then separated from the solid phases in accordance with conventional techniques.

It is to be understood that there are many variations of the basic reduction process hereinabove described which fall within the scope of the instant invention and they are intended to be included herein. The variations in the basic process are governed by the desire to subject the raw alloys to steps of purification which produce either an aluminum-silicon alloy or commercially pure aluminum. It is to be understood that the novel process of this invention is applicable in either of the two instances.

FIGURE 2 represents a typical flow sheet including process variations for the manufacture of a partially purified aluminum-silicon alloy in accordance with the process of this invention. As can be seen by reference to said figure, intermetallic complexes are added to the charge in a reduction furnace and heat is applied until the ores are reduced to the liquid metal and then the liquid metal is tapped and cooled to a preselected temperature ranging from about 580° to 680° C. It is to be understood that the composition of the liquid aluminum-silicon alloy varies with the temperature to which the raw alloy is cooled and the specific compositions obtained at a particular temperature are extremely well known in the art and are a matter of preference, depending upon the intended use for the finished alloy. However, in the vast majority of commercial operations, it is customary to cool the liquid raw alloy to a temperature of approximately 600° C. wherein a eutectic composition of aluminum, silicon, iron and titanium exist containing approximately 12% by weight of silicon, 1% iron and 0.1% titanium. At this temperature the aluminum-silicon alloy exists in the liquid phase and the solid phases contain the intermetallic complexes, elementary silicon, if an excess is present, and various intermetallic compounds resulting from impurities contained in the feed material. The liquid alloy is then separated from the solid phases by conventional techniques including filtration, decantation or, more preferably, centrifuging. The liquid phase thus contains the desired product whereas the solid phase contains free silicon, various intermetallic complexes and a portion of the aluminum-silicon alloy due to the fact that the separation of liquid and solid phases can never be carried out at 100% efficiency and the portion of the alloy usually trapped in the solid phases is about 10–15%. At this point, the solid phases can be discarded if it is desired.

A more preferred embodiment of this invention would reside in treatment of the solid phases in order to recover the aluminum-silicon alloy remaining therein. This separation can be accomplished merely by comminuting the solid phases in a ball mill and then passing the reduced particle sizes through a screen ranging from 30 to 300 mesh Tyler and more preferably 140 to 200 mesh. It has been found that the fraction which does not pass through the screen will contain an extremely high percentage of the eutectic or quasi-eutectic aluminum-silicon alloy. This aluminum-silicon alloy can either be added to the aluminum-silicon alloy previously recovered in the liquid phase or, even more desirably, can be recycled back to the molten raw alloy and again reprocessed in the manner above-described.

A more particularly preferred embodiment of the invention would reside in further treating the solid phase remaining after removal of the partially purified aluminum-silicon alloy, that is, the solid phase containing silicon and intermetallic complexes, in order to recover the silicon especially if there is a substantial excess of elementary silicon present. The recovery of silicon from the intermetallic complexes can be accomplished by a

wide variety of ways including gravity separation or sink float techniques with a high density liquid such as carbon tetrachloride mixed with bromoform wherein a silicon-rich fraction can be recovered. The density of the liquid is adjusted to allow the silicon to float while the heavier intermetallics sink. Alternatively, the silicon can be separated from the intermetallic complexes by treatment with a flotation medium consisting of a fluorine-containing acid and a fluoride salt. The recovered silicon can then either be sold or used to blend with aluminum to produce other aluminum-silicon alloys. The intermetallic complexes obtained are then recycled back to the charge in the reduction furnace so as to make the process continuous. It is to be understood that if an impure feed material is employed which, as has heretofore been set forth, will inherently produce a certain amount of intermetallic complexes, then a like quantity of intermetallic complexes should be discarded from the intermetallic complexes recovered in order to prevent an undesirable build-up of these complexes in the furnace.

FIGURE 3 represents a variation of the reduction process wherein it is desired to produce commercially pure aluminum rather than an aluminum-silicon alloy. In this process, the steps are identical as those in FIGURE 2 until the raw alloy is produced whereupon the raw alloy is treated with a metallic solvent such as zinc, lead, mercury, etc., as is well known in the art, in order to leach out the aluminum followed by separating the liquid and solid phases. The aluminum is then recovered from the liquid solvent by a number of conventional techniques including distillation and melting in order to obtain commercially pure aluminum. At this point, the solid phases can be discarded if such is desired or the solid phases can be reprocessed as hereinabove described and that part of the aluminum which had been trapped when the solids were separated can be reclaimed and recycled back to the raw alloy and the intermetallic complexes recycled back to the furnace feed. In like manner, the intermetallic complexes which have been introduced into the raw alloy as a result of the reduction of oxides of heavy metals contained in the feed as impurities are eliminated by rejecting a corresponding quantity from the remaining intermetallic compounds. The rest of the material is then recycled to provide the intermetallic complexes for the next furnace charge.

As can be seen, the novel process of this invention is extremely beneficial in the thermal reduction of alumina-bearing ores whether it is desired to produce either aluminum-silicon alloys or commercially pure aluminum. Additionally, the process of this invention provides a method of recycling various intermediate products, thereby enhancing the overall economy of the operation.

Another preferred embodiment of this invention resides in having the feed materials charged to the reduction furnace in intimate contact with one another so as to be better able to dissolve the aluminum and silicon as soon as they are reduced from their corresponding ores. This can be accomplished by grinding all feed components to a size passing a screen below at least about 140 and preferably 200 U.S. standard mesh, mixing the finely divided particles thoroughly and forming them into briquets. Additionally, it is advisable to provide a certain amount of the feed in the form of a clay of sufficient plasticity in order to act as a binder for the briquets. The clay, which is also a portion of the feed, can be present in amounts ranging from 5 to 20 percent. It has been found that by grinding and blending the feed materials in this manner the otherwise prevailing difficulties in continuous furnace operations are very much alleviated.

The following examples will now illustrate the best mode contemplated for carrying out this invention but it is to be understood that it is not intended to be limited thereto.



*Example 1*

This example will illustrate the novel process of this invention for the preparation of an aluminum silicon alloy, having an approximate weight ratio of 88 percent aluminum and 12 percent silicon. In this example, relatively pure oxidic feed materials are employed.

A conventional electrical submerged arc furnace is charged with briquets in which the following feed is blended:

| | Kilograms |
|---|---|
| Bayer alumina | 359 |
| Ball clay | 100 |
| Quartz | 10 |
| Charcoal | 235 |
| Intermetallic complex | 200 |

The composition of the charged oxides is as follows:

TABLE II

| Oxides | Bayer Alumina, kg. | Clay, kg. | Ashes of Charcoal, kg. | Quartz, kg. | Total, kg. |
|---|---|---|---|---|---|
| $Al_2O_3$ | 359 | 34 | 0.5 | | 393.5 |
| $SiO_2$ | | 63 | 0.8 | 10 | 73.8 |
| $TiO_2$ | | 1.8 | | | 1.8 |
| $Fe_2O_3$ | | 1.2 | 0.1 | | 1.3 |
| Total | 359 | 100 | 1.4 | 10 | 470.4 |

The 200 kilograms of intermetallic complexes which are charged in the furnace correspond mainly to the formula $FeAl_3Si_2$ having a weight ratio of approximately 1 Fe:1.5 Al:1 Silicon. The intermetallic complex contains about 7 kilograms of aluminum and 5 kilograms of silicon in an uncombined form, and the total composition is as follows:

TABLE III

| | Kg. |
|---|---|
| Aluminum | 88 |
| Silicon | 58 |
| Titanium | — |
| Iron | 54 |
| Total | 200 |

The charge is then reduced in a conventional manner and tapped to yield a raw alloy of the following composition:

TABLE IV

| | Kg. |
|---|---|
| Aluminum | 275 |
| Silicon | 87.3 |
| Titanium | 1.1 |
| Iron | 57.0 |
| Total | 420.4 |

The raw allow is cooled to approximately 610° C. and is then subjected to separation by a centrifuge to yield a liquid phase and solid phases having the following composition:

TABLE V

| | Liquid Phase, kg. | Solid Phase, kg. |
|---|---|---|
| Aluminum | 169.8 | 105.2 |
| Silicon | 27.1 | 60.2 |
| Titanium | 0.2 | 0.9 |
| Iron | 2.5 | 54.5 |
| Total | 199.6 | 220.8 |

The 199.6 kilograms of the liquid phase is an aluminum-silicon alloy having an approximate ratio of 88 aluminum: 12 silicon. The remaining solid phases are then

comminuted in a ball mill and sieved through a 100 mesh screen with the following results:

TABLE VI

| Metal | +100 Mesh (kg.) | −100 Mesh (kg.) |
|---|---|---|
| Aluminum | 21.30 | 83.90 |
| Silicon | 5.50 | 54.70 |
| Titanium | 0.25 | 0.65 |
| Iron | 1.55 | 52.95 |
| Total | 28.60 | 192.20 |

The plus 100 mesh fraction can then either be added to the aluminum-silicon alloy previously obtained or can be recycled back to the raw alloy and reprocessed. The minus 100 fraction can either be further processed in order to remove the free silicon or can be recycled directly back to the furnace feed. From this example, it can be seen that a makeup 7.8 kilograms of intermetallics would have to be added.

*Example 2*

This example will also illustrate the preparation of aluminum silicon alloy but it differs from Example 1 in that an impure feed material is employed rather than the pure Bayer alumina.

A conventional electrical submerged arc furnace is charged with the following feed:

| | Kilograms |
|---|---|
| Guiana bauxite | 400 |
| Bauxitic clay | 100 |
| Ball clay | 100 |
| Quartz | 10 |
| Charcoal | 250 |
| Intermetallic complex | 100 |

The composition of the charge oxides is as follows:

| Oxides | Quartz kg. | Guiana Bauxite kg. | Bauxitic Clay kg. | Ball Clay kg. | Total kg. |
|---|---|---|---|---|---|
| $Al_2O_3$ | | 340 | 58 | 34.0 | 432.0 |
| $SiO_2$ | 10 | 34.8 | 34.5 | 63.0 | 147.3 |
| $TiO_2$ | | 18.0 | 3.0 | 1.8 | 22.8 |
| $Fe_2O_3$ | | 7.2 | 4.5 | 1.2 | 12.9 |
| Total | 10 | 400 | 100 | 100 | 615.0 |

The 100 kilograms of intermetallic complexes which are charged in the furnace correspond to that of Example 5 in Table I of the specification. The intermetallic complex has the following composition:

| | Kg. |
|---|---|
| Aluminum | 30.0 |
| Silicon | 35.0 |
| Titanium | 19.0 |
| Iron | 16.0 |
| Total | 100.0 |

The charge is then reduced in a conventional manner and tapped to yield a raw alloy of the following composition:

| | |
|---|---|
| Aluminum | 236.0 |
| Silicon | 93.3 |
| Titanium | 32.7 |
| Iron | 27.1 |
| Total | 389.1 |

The raw alloy is cooled to approximately 630° C. and is then subjected to separation by centrifuging to yield



a liquid phase and solid phases having the following composition:

| Metal | Liquid Phase, kg. | Solid Phase, kg. |
|---|---|---|
| Aluminum | 165.0 | 71.0 |
| Silicon | 29.5 | 63.8 |
| Titanium | 0.2 | 32.5 |
| Iron | 3.0 | 24.1 |
| Total | 197.7 | 191.4 |

The 197.7 kilograms of the liquid phase is an aluminum-silicon alloy having an approximate ratio of 88 aluminum:12 silicon. The remaining solid phases are then comminuted in a ball mill and sieved through a 100 mesh screen with the following results:

| Metal | +100 mesh (kg.) | −100 mesh (kg.) |
|---|---|---|
| Aluminum | 26.4 | 44.6 |
| Silicon | 7.8 | 56.0 |
| Titanium | 2.8 | 29.7 |
| Iron | 1.8 | 22.3 |
| Total | 38.8 | 152.6 |

The plus 100 mesh fraction can either be added to the aluminum-silicon alloy previously obtained or can be recycled back to the raw alloy and reprocessed.

As can be seen, the minus 100 mesh fraction contains 152.6 kilograms of intermetallic complex and it can either be further processed in order to recover the free silicon or can be recycled directly back to the furnace feed. If this fraction is to be recycled directly back to the furnace feed without removal of silicon, then it can be seen that 52.6 kilograms must be discarded since this is approximately the amount of intermetallics which are produced by the impure feed materials of this example.

*Example 3*

This example will illustrate the novel process of this invention when it is desired to produce commercially pure aluminum rather than aluminum silicon alloy.

In this example, the process of Example 1 is repeated up until the 420.4 kilograms of the raw alloy is tapped. At this point, the raw alloy is subjected to conventional mercury extraction and the liquid amalgamated aluminum and the solid phases are separated. The aluminum amalgam is then purified by techniques involving distillation and melting to yield purified aluminum of the following composition:

| Metal: | Aluminum (kg.) |
|---|---|
| Aluminum | 177.00 |
| Silicon | 0.20 |
| Titanium | 0.05 |
| Iron | 0.30 |
| Total | 177.55 |

The solid phases have the following composition:

| Metal: | Solids (kg.) |
|---|---|
| Aluminum | 98.00 |
| Silicon | 87.10 |
| Titanium | 1.05 |
| Iron | 56.70 |
| Total | 242.85 |

The solid phases are then comminuted in a ball mill and sieved through a 100 mesh screen with the following results:

| Metal | +100 mesh (kg.) | −100 mesh (kg.) |
|---|---|---|
| Aluminum | 15.0 | 83.0 |
| Silicon | 3.75 | 83.35 |
| Titanium | 0.38 | 0.67 |
| Iron | 1.60 | 55.1 |
| Total | 20.73 | 222.12 |

The plus 100 mesh fraction is advisably recycled back to the raw alloy to be re-extracted with mercury when the next batch is processed. The minus 100 mesh fraction can either be discarded or is preferably subjected to a sink float or gravity separation with a high density liquid such as a mixture of bromoform and carbon tetrachloride, having a specific gravity of about 2.42. If this treatment is carried out, a float and a sink fraction will occur having the following compositions:

| Metal | Float, kg. | Sink, kg. |
|---|---|---|
| Aluminum | 1.5 | 81.5 |
| Silicon | 29.5 | 53.85 |
| Titanium | .1 | .57 |
| Iron | .15 | 54.95 |
| Total | 31.25 | 190.87 |

As can be seen, the float fraction is silicon having a relatively high degree of purity and this material can either be sold or used in the formation of other types of aluminum-silicon alloys. The sink portion can be recycled back to the furnace feed in order to make the process continuous. It can be seen that on the next charge of the furnace a makeup of 9.13 kilograms of intermetallic complexes will be necessary.

Another modification of this invention involves further processing the partially purified aluminum-silicon alloy, obtained as the liquid phase in Examples 1 and 2, by leaching out aluminum by means of a metallic solvent such as zinc, mercury and lead.

Many other modifications and variations of the above process will be obvious to those skilled in the art and it is not intended that this process be limited except as necessitated by the appended claims.

What is claimed is:

1. In the process for the production of a raw aluminum-silicon alloy wherein oxidic ores comprising substantially alumina and silica are charged into a reduction furnace with sufficient carbon as a reductant, heated until the ores are reduced to the metallic state, followed by tapping the raw alloy, the improvement which comprises adding to the furnace feed a substantially fully satisfied intermetallic complex of aluminum and silicon with at least one metal selected from the group consisting of iron and titanium in an amount ranging from 15–15 percent by weight of the total amount of alumina and silica charged.

2. The process of claim 1 wherein the intermetallic added is a complex of both titanium and iron with aluminum and silicon wherein the total amount of both iron and titanium ranges from 25–55 percent by weight of the total composition.

3. The process of claim 1 wherein the intermetallic added is a complex of iron, aluminum and silicon wherein the iron is present in an amount ranging from 25–30 percent by weight of the total composition.

4. A process for the production of a partially purified aluminum-silicon alloy from oxidic ores which comprises subjecting a furnace feed comprising alumina, silica, and an intermetallic complex to reduction in the presence of a sufficient amount of a carbonaceous reducing agent until the oxides are reduced to the metallic state and a raw alloy is formed, tapping the resulting raw liquid alloy, cooling the raw alloy to a temperature sufficient to obtain a liquid phase and solid phases and separating the resulting liquid phase consisting of partially purified aluminum-silicon alloy from the solid phases, said intermetallic complex consisting of substantially fully satisfied complexes of aluminum, silicon and at least one metal selected from the class consisting of iron and titanium, wherein the amount of fully satisfied intermetallic complexes in the furnace feed ranges from 15 to 50 percent by weight of the total amount of alumina and silica charged and the weight ratios of said alumina and silica are as follows:

$$Al_2O_3:SiO_2=75–87:25–13$$

5. The process of claim 4 wherein the complex is one of iron, aluminum and silicon, wherein the iron represents 25–30 percent by weight of the total complex.

6. The process of claim 4 wherein the complex is one of titanium, iron, aluminum and silicon, wherein the total amount of iron and titanium ranges from 25–55 percent by weight of the total complex.

7. The process of claim 4 wherein the solid phases are substantially recycled back to the furnace feed.

8. The process of claim 4 wherein the solid phases are subjected to a further separation by comminution and sieving through a 30–300 mesh screen and the finer fraction is recycled back to the furnace feed.

9. The process of claim 4 wherein the solid phases are further separated by comminution and sieving through a 30–300 mesh screen, the coarser fraction being recycled to the raw alloy, and the finer fraction being recycled to the furnace feed.

10. The process of claim 4 wherein the partially purified aluminum-silicon alloy is further purified by treating it with a metallic solvent selected from the group consisting of zinc, mercury and lead, separating the aluminum-metallic solvent liquid phase from the resulting solid phases and recovering aluminum.

11. The process for the production of aluminum which comprises subjecting a furnace feed comprising alumina, silica and intermetallic complexes to reduction in the presence of a sufficient amount of a carbonaceous reducing agent until the oxides are reduced to the metallic state and a raw liquid alloy is formed, tapping the resulting raw liquid alloy, treating the raw alloy with a metallic solvent for aluminum, separating the aluminum-metallic solvent liquid phase from the resulting solid phases and recovering the aluminum, said intermetallic complex consisting of substantially fully satisfied complexes of aluminum, silicon and at least one metal selected from the class consisting of iron, and titanium, wherein the amount of said fully satisfied intermetallic complexes added to the furnace feed ranges from 15–50 percent by weight of the total amount of alumina and silica charged and the weight ratios of said alumina and silica are as follows:

$$Al_2O_3:SiO_2=75-87:25-13$$

12. The process of claim 11 wherein the metallic solvent for aluminum is selected from the the class consisting of zinc, mercury and lead.

13. The process of claim 11 wherein the metallic solvent is mercury.

14. A process for the production of aluminum from oxidic ores which comprises subjecting a furnace feed comprising alumina, silica, and an intermetallic complex to reduction in the presence of a sufficient amount of a carbonaceous reducing agent until the oxides are reduced to the metallic state and a raw alloy is formed, tapping the resulting raw liquid alloy, cooling the raw alloy to a temperature sufficient to obtain a liquid phase and solid phases, separating the resulting liquid phase consisting of partially purified aluminum-silicon alloy from the solid phases, treating the partially purified alloy with a metallic solvent for aluminum, separating the aluminum-metallic solvent liquid phase from the resulting solid phases and

recovering the aluminum, said intermetallic complex consisting of substantially fully satisfied complexes of aluminum, silicon and at least one metal selected from the class consisting of iron, and titanium, wherein the amount of said fully satisfied intermetallic complexes added to the furnace feed ranges from 15–50 percent by weight of the total amount of alumina and silica charged and the weight ratios of said alumina and silica are as follows:

$Al_2O_3:SiO_2$=75–87:25–13


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