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REFINING AND PRODUCING ALUMINUM

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Aluminum, as produced by the ordinary commercial processes now in use, or as recovered as secondary metal or scrap, normally contains impurities, the amount and nature of which depend upon the origin of the metal. It is frequently desirable to remove from aluminum these impurities, which usually take the form of other metallic elements such as iron, silicon, copper, titanium, zinc, magnesium, cadmium, lead, and the like. Such methods as have been proposed for this purpose are quite expensive.

The present invention relates to a distillation process of refining aluminum. Such processes have been previously proposed, but have heretofore not been considered commercial because of the high temperatures at which they must be operated. It is therefore the object of this invention to provide a distillation process of refining aluminum which is operable at temperatures consistent with inexpensive and commercial apparatus and at a cost less than that of previously proposed processes of this general type.

I have discovered that when halides are mixed with impure aluminum and the mixture heated to elevated temperatures in a heating chamber, relatively pure aluminum will vaporize at commercially operable temperatures and rates and may thereafter be recovered by condensing the vapors in a suitable condensing chamber. Of the halides the fluorides are preferred for this purpose, even over other somewhat cheaper halides, because it has been found that the general efficiency of the process is better when fluorides are used. Of the fluorides the alkaline earth fluorides and the double fluorides containing aluminum are preferred, and aluminum fluoride is a compound which has been found satisfactory. Of the alkaline earth fluorides, magnesium fluoride is preferred, although in high temperature operation the higher vaporizing temperature of calcium fluoride may make the use of this compound desirable. Of the double fluorides containing aluminum, the alkali metal double fluorides are regarded as most satisfactory, and of these the sodium aluminum fluorides, such as cryolite or chiolite, have proven the more efficient.

The selection of the halide will be governed by considerations of the thermal characteristics of the halide in question and the temperature of operation. Halides which are unstable at the temperature of operation, or which vaporize readily at that or lower temperatures, thus leaving the reaction zone impoverished in halide prior to effective reaction, are usually not desirable in practice.

It would appear, although this surmise is theoretical and my invention is not limited thereto, that the halide and the available aluminum form some low boiling mixture, compound, or analogous body vaporizing at relatively low temperature, which leads to the useful result that the aluminum may be distilled over at temperatures far below those at which aluminum can be practically distilled at any appreciable rate under the action of temperature alone and in the absence of halides.

I have further discovered that whenever available aluminum is present, as, for instance, the aluminum which would result from the dissociation of aluminum carbide under heat, that this aluminum, or a substantial proportion thereof, is also, in the presence of halide, distilled over at temperatures below which any aluminum would be normally distilled. My invention therefore contemplates not only the refining of metallic aluminum, but also the recovery of aluminum from those aluminum compounds, or mixtures of aluminum compounds and other substances, which produce, in part at least, under the action of heat, available elementary aluminum. I have mentioned aluminum carbide, but mere mixtures of alumina and carbon have likewise given results, as would be expected whenever the mixture of substances is such that aluminum carbide or other easily dissociated aluminum compounds are formed.

In accordance with my refining process, impure aluminum is first put into a finely divided or comminuted state by a ball-milling operation, atomizing, or by other well known methods for producing small particles. The size of the particles depends, to some extent, on the other conditions hereinafter described, but should be such as to permit an intimate mixture of the particles with the halide used. For most purposes, particles having a size less than 14 mesh are preferred. The particles are then mixed with a suitable halide and placed in a heating chamber or furnace. The mixture may be loose when placed in the furnace, but it is preferred to briquette the mixture before introducing it into the furnace. This may be done by subjecting it to pressure in a mold, or by employing a binder. Likewise, it is advantageous to sinter the briquettes before placing them in the furnace.

After the mixture has been placed in the heating chamber, the process is carried out in an atmosphere inert to aluminum, under conditions which permit the gases evolved from the mixture of impure aluminum and the halide to pass away from the mixture and toward a condensing zone or chamber. This condition may be readily obtained by reducing the pressure in the chamber by means of a vacuum pump and maintaining the action of the vacuum pump during the heating step hereinafter described. It may also be secured by passing a current of a gas which is substantially effectively inert to aluminum across the briquettes during the heating step. For example, a current of hydrogen may be used.

The mixture in the heating chamber is then heated at an elevated temperature. The temperature used varies, depending upon the particular halide employed in the mixture. For example, when aluminum fluoride is used in the mixture, a temperature of between 900° and 1000° C. is satisfactory. When sodium cryolite (Na_3AlF_6) is used, a temperature of between 900° and 1000° C. may be employed. If the halide is magnesium fluoride, the mixture is heated to a temperature of between 900° and 1300° C. in the heating step.

The heating of the mixture may be accomplished by applying heat externally to the portion of the furnace in which the mixture is located, or internal heating means, such as an electric resistor, may be employed for the purpose. When the mixture is heated as above described, vapors are evolved which pass to a cooling zone and condense. The condensing zone may constitute a part of the chamber in which the mixture is heated, but remote from the heating zone, or it may be a separate chamber from which the vapors evolved from the mixture are led from the furnace. The material found in the condensing zone or chamber at the end of the heating step consists principally of a mixture of the halide and pellets of aluminum. The aluminum may be separated from the balance of the condensate mechanically, as by a screening process, to remove the pellets of aluminum, but it is preferred to accomplish the separation by melting the condensate, in which case the aluminum forms a liquid layer separate from the salt and can be tapped off readily. The salt, which is likewise purified by the process, may be recovered and used again in the purification of further aluminum or otherwise.

As exemplary of the specific operation of my process, I have chosen the following halides which have been found particularly suitable to a high efficiency operation, viz., aluminum fluoride, magnesium fluoride, and the double fluorides of aluminum and the alkali metals, of which latter cryolite is preferred. When aluminum fluoride is used, it is preferably mixed with particles of impure aluminum in the proportions of about 3 parts of aluminum fluoride to 1 part of the aluminum by weight. The temperature at which the mixture is heated when aluminum fluoride is employed is between 900° and 1000° C., the exact temperature depending, to some extent, on such factors as the size of the aluminum particles and the speed and economy with which it is desired to have the process proceed. Ordinarily, a heating period of from 1 to 4 hours is used.

When magnesium fluoride is used as the halide, it is preferably present in the original mixture in the proportions of about 1½ parts of magnesium fluoride to 1 part of impure aluminum by weight, and the mixture is heated to a temperature of between 900° and 1200° C. When the halide employed is cryolite, the most efficient proportions are about 2 parts of cryolite to 1 part of aluminum by weight, and the temperature to which the mixture is heated between 900° and 1000° C.

As an example of the operation of my process, aluminum containing 0.16 per cent silicon, 0.31 per cent iron, 0.01 per cent copper, and 0.008 per cent titanium was comminuted to 28 mesh size. This material was mixed with cryolite in the proportions of 2 parts of cryolite to 1 part of aluminum by weight, and the mixture was briquetted in a mold by applying pressure to it. Briquettes of the mixture were placed in one end of a closed horizontal retort having a vacuum pump attached to the other end thereof. The end of the retort containing the briquettes was then heated to a temperature of 800° C. for a period of 3½ hours. During that time, the retort was maintained by the vacuum pump at a residual gas pressure of .7 millimeter of mercury. At the end of the heating period, the retort was opened, and the deposit found at the cooler end of the retort was removed. The deposit consisted principally of a mixture of cryolite and aluminum, the aluminum being present in the form of pellets. Upon separation of the aluminum from the cryolite, the aluminum was found to contain only 0.01 per cent silicon, 0.05 per cent iron, 0.002 per cent titanium, and no copper. Likewise, briquettes composed of a mixture of magnesium fluoride and aluminum containing 0.15 per cent silicon, 0.36 per cent iron, 0.01 per cent copper, and 0.007 per cent titanium in the proportions of slightly less than 1½ parts of magnesium fluoride to 1 part of the impure aluminum by weight were heated in a horizontal retort for 2 hours at 1120° C. and a pressure of 4 millimeters of mercury. Upon separation of the pellets of aluminum from the deposit found in the cooler end of the retort, the aluminum was found to contain 0.01 per cent silicon, 0.01 per cent iron, 0.001 per cent titanium, and only a trace of copper.

Although the process has been described above as involving the use in conjunction with impure aluminum of only one of the halides or double halides mentioned, it is to be understood that the mixture may contain two or more of the halides.

I claim:

1. The process comprising forming a mixture comprising aluminum and a halide, heating said mixture in an atmosphere effectively inert to aluminum to vaporize therefrom metallic aluminum and halide, condensing the resulting vapors and separating the aluminum from the condensate.

2. The process comprising forming a mixture comprising aluminum and a fluoride, heating said mixture in an atmosphere effectively inert to aluminum to vaporize therefrom metallic aluminum and fluoride, condensing the resulting vapors and separating the aluminum from the condensate.

3. The process comprising forming a mixture comprising aluminum and aluminum fluoride, heating said mixture in an atmosphere effectively inert to aluminum to vaporize therefrom metallic aluminum and fluoride, condensing the resulting vapors and separating the aluminum from the condensate.

4. The process comprising forming a mixture comprising aluminum and magnesium fluoride, heating said mixture in an atmosphere effective-

ly inert to aluminum to vaporize therefrom metallic aluminum and fluoride, condensing the resulting vapors and separating the aluminum from the condensate.

5. The process comprising forming a mixture comprising aluminum and double fluorides of an alkali metal and aluminum, heating said mixture in an atmosphere effectively inert to aluminum to vaporize therefrom metallic aluminum and fluoride, condensing the resulting vapors and separating the aluminum from the condensate.

6. The process comprising forming a mixture comprising aluminum and sodium cryolite, heating said mixture in an atmosphere effectively inert to aluminum to vaporize therefrom metallic aluminum and fluoride, condensing the resulting vapors and separating the aluminum from the condensate.

7. The process comprising forming a mixture comprising aluminum and two or more of the fluorides from the class composed of aluminum fluoride, magnesium fluoride, alkali metal double fluorides, and the alkaline earth fluorides, heating said mixture in an atmosphere effectively inert to aluminum to vaporize therefrom metallic aluminum and fluoride, condensing the resulting vapors and separating the aluminum from the condensate.

8. The process of forming a mixture comprising a halide and substance which becomes dissociated by heat at the temperature of the process to produce available elementary aluminum, heating said mixture in an atmosphere effectively inert to aluminum and at a temperature below the temperature at which aluminum would appreciably distill in the absence of a halide, thereby to vaporize therefrom metallic aluminum and halide, condensing the resulting vapors and separating the aluminum.

9. The process of forming a mixture comprising a fluoride and substance which becomes dissociated by heat at the temperature of the process to produce available elementary aluminum, heating said mixture in an atmosphere effectively inert to aluminum and at a temperature below the temperature at which aluminum would appreciably distill in the absence of a fluoride, thereby to vaporize therefrom metallic aluminum and fluoride, condensing the resulting vapors and separating the aluminum.

10. The process of heating elementary aluminum in the presence of a halide and in an atmosphere relatively inert to aluminum to vaporize aluminum and halide, condensing the resulting vapors and separating the aluminum from the condensate.

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