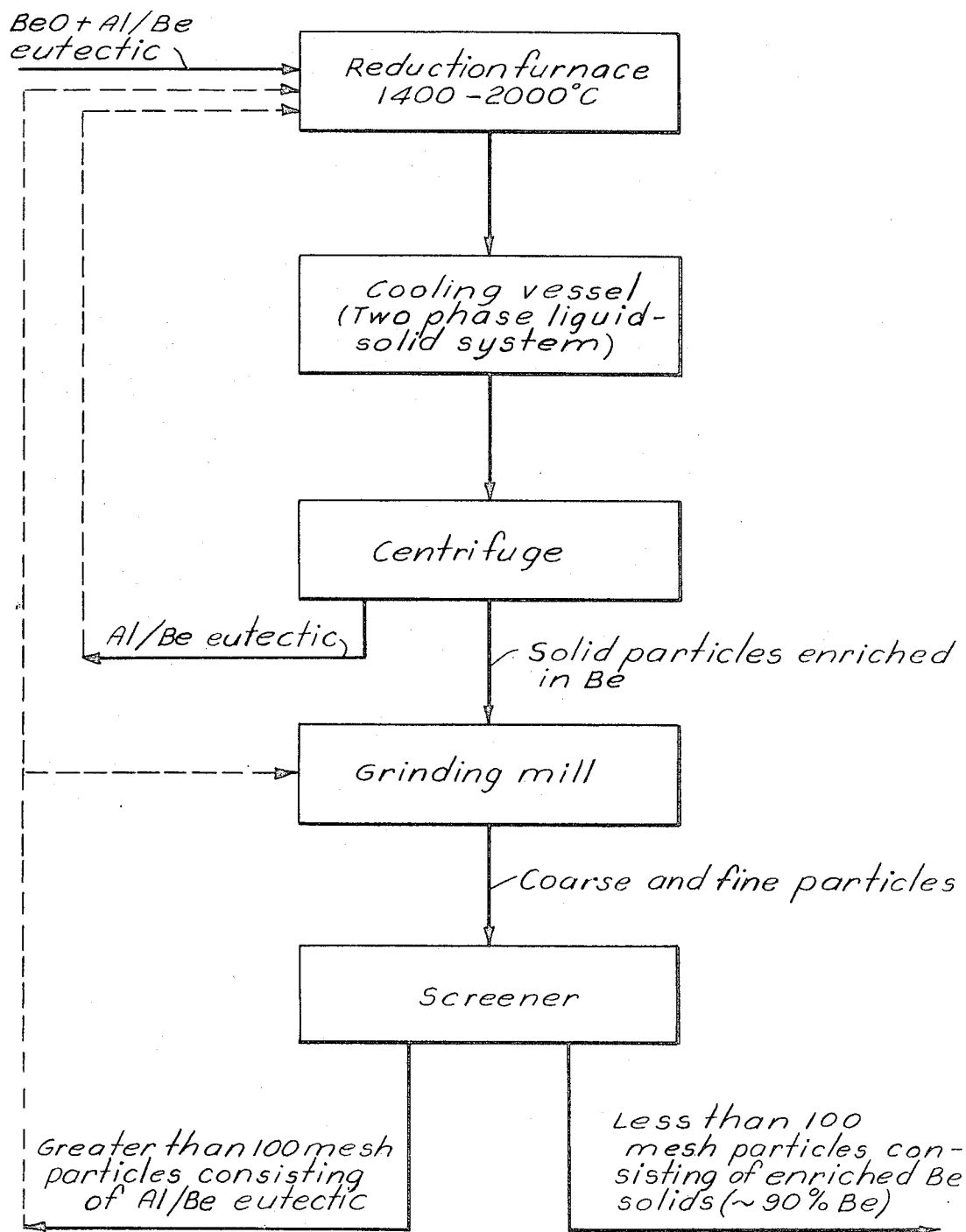


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PREPARATION OF BERYLLIUM

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PREPARATION OF BERYLLIUM

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10 Claims

ABSTRACT OF THE DISCLOSURE

A method for preparing a beryllium enriched material which comprises: comminuting an aluminum-beryllium alloy to obtain a coarse particulate fraction of an aluminum-beryllium eutectic composition and a fine particulate fraction comprising beryllium; and separating the coarse particulate fraction from the fine particulate fraction.

BACKGROUND

Beryllium is now produced by a number of well-known processes. For example, one method consists of the electrolysis of beryllium halides. Another process consists of the arc furnace reduction of BeO with carbon in the presence of a heavy metal, e.g. copper, nickel and the like, to form a beryllium-heavy metal alloy. A third method consists of reducing BeF₂ with magnesium or sodium metal. There are certain disadvantages associated with each of these processes. For example, when BeF₂ is reduced with magnesium metal it is difficult to separate the resulting beryllium metal from the magnesium fluoride salt produced. Usually the mixture must be heated to temperatures in excess of 1300° C. and the molten beryllium separated from the magnesium salt. When beryllium is formed by the arc furnace reduction of beryllium oxide with carbon to form an alloy, the formation of carbide impurities is a serious disadvantage. Furthermore, the beryllium is produced as an alloy with the heavy metal which is employed in the arc furnace.

The present invention affords an efficient and economical method for preparing beryllium metal. Furthermore, in the present invention the beryllium is produced in a desirable particulate form.

SUMMARY

The present invention takes advantage of the fact that the solid state solubility of beryllium in aluminum is very small, and therefore an aluminum-beryllium alloy exists substantially as a two phase composition. One phase consists of a ductile eutectic composition of aluminum with about 0.5 percent by weight of beryllium. The second phase consists of beryllium which is more brittle than the eutectic composition. Because the beryllium is more brittle than the eutectic composition it can be reduced by mechanical grinding to provide smaller size particles more readily than the eutectic composition.

Therefore, in accordance with the present invention, an aluminum-beryllium alloy is comminuted to produce a product mixture consisting of fine particles of beryllium and coarser particles of the eutectic composition. The finer beryllium particles are then separated from the eutectic particles by conventional separating methods, e.g. screening.

DRAWINGS

The figure schematically illustrates one embodiment of the method of the present invention.

PREFERRED EMBODIMENTS

In practice, an aluminum-beryllium alloy, usually containing at least about 1 percent by weight of beryllium,

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and preferably greater than 5% of beryllium, is comminuted e.g. by grinding in a ball mill, to produce a particulate product mixture comprising a coarse grade of particles comprising an aluminum-beryllium eutectic material ranging in size greater than about 100 mesh, preferably greater than about 200 mesh (U.S. Standard Sieve Series, 1940), and a finer grade of beryllium particles ranging in size smaller than about 100 mesh, preferably smaller than about 200 mesh, and greater than about 270 mesh. The finer beryllium particles are then separated from the coarser Al/Be eutectic particles such as by passing the mixture through a suitable size screen, or series of decreasing size screens, whereupon the smaller beryllium metal particles pass through the screen while the coarser eutectic particles are retained on the screen. The finer beryllium particles may also be separated from the coarser eutectic particles by other processes. For example fluid separation processes wherein the finer beryllium particles are separated from the coarser eutectic composition by the action of gas stream such as air, or liquid baths such as water can be employed.

The beryllium content of the mass consisting of the finer particles will increase with decreasing particle size. For example, when a comminuted aluminum-beryllium alloy is passed through a 100 mesh screen the fine particles passing through the screen are found to consist of about 90 percent by weight of beryllium metal; the greater portion of the remaining metal consisting of aluminum. Particles smaller than about 200 mesh are found to consist of greater than about 95 percent by weight of beryllium. The separated fine particles and/or coarse particles can be reprocessed in the manner heretofore described to further concentrate the beryllium metal. Although 100 and 200 mesh sizes are presented herein as the separation sizes between the coarser and smaller size particles it is understood that they are only exemplary and other size ranges may be employed for the separation process. The size range employed for separation is dependent on problems involved with fine beryllium powders and the like.

One embodiment of the present invention is schematically illustrated in the figure. In this embodiment an aluminum-beryllium alloy is provided in a molten one phase form, e.g. such as by reducing BeO in an arc reduction furnace with aluminum or an aluminum-beryllium alloy. The one phase molten mass is slowly cooled from the temperature of the initial molten mass, e.g. about 1100° C. or greater, to about 600° C. over a substantial period of time, e.g. 3 hours or more. As the mass cools a beryllium enriched solid phase, having a melting point which is higher than the aluminum-beryllium eutectic composition, crystallizes out forming a two phase system. One phase consists of beryllium enriched solids and the second consists of a molten aluminum-beryllium mixture. The solid beryllium enriched particles are separated from the molten phase, e.g. by filtration, decantation or preferably centrifugation. The beryllium enriched solids phase is then comminuted and processed by the method as defined hereinbefore. In a similar manner a solid material consisting of an aluminum-beryllium alloy may be first heated sufficiently to provide it in a molten one phase form and then slowly cooled to form a beryllium enriched solid phase. This solid phase is then comminuted and separated as defined hereinbefore.

As indicated, the separated coarse and fine particles may be further reprocessed to further concentrate beryllium metal. The coarse eutectic particles, prepared in the initial process, and usually containing less than about 1 percent beryllium, also find utility as an adjuvant in aluminum castings. The presence of a small percentage of beryllium promotes fluidity and tends to refine the grain in aluminum castings. Also the coarse eutectic particles

may be employed as a reducing agent in the aluminothermic reduction of BeO, to again form a beryllium containing starting material.

The aluminum-beryllium alloy employed in the present process can be prepared in a variety of ways. For example, BeO can be reduced, for example as described in U.S. 3,386,817, using aluminum or an aluminum-beryllium alloy at temperatures greater than about 1100° C. to produce an alloy usually containing from 3 to 10 percent by weight of beryllium. Thus, as indicated hereinbefore, the coarse particles from the comminuting and sizing steps of the present invention may be recycled, with or without further comminuting and sizing, to prepare an aluminum-beryllium alloy starting material.

A preferred method for preparing the alloy comprises the electrolysis of a fused Be-alkali metal halide salt mixture employing aluminum or an aluminum-beryllium eutectic mixture as a molten cathode.

As an example of the method of the present invention, beryllium metal powder of enhanced beryllium content is prepared in the following manner. A sample of an aluminum-beryllium alloy containing from 5 to about 7 percent beryllium, prepared by an electrolytic reduction process, is cooled slowly e.g. over a three hour period, from about 1000° C. to about 700° C. The molten portion, consisting essentially of an aluminum-beryllium eutectic alloy, is separated from the solids, consisting of a beryllium enriched material. Such a separation is made, for example, by a pressure filtration process. The beryllium enriched solids are comminuted, e.g. ground in a rod mill. Particles smaller than about 200 mesh are removed periodically by screening. These particles will consist of greater than about 95 percent by weight of beryllium with aluminum being the other constituent. The particles, which are greater than about 200 mesh in size, comprise greater than 98 percent by weight of aluminum with less than about 1 percent by weight of beryllium.

In a similar manner aluminum-beryllium alloys obtained from other processes can be comminuted and separated into coarse and fine fractions by separation methods such as screening, liquid or gas flow separation techniques and the like. The present process therefore affords an efficient economical method for producing beryllium enriched metals, especially in a highly useful powdered form.

What is claimed is:

1. A process which comprises:

(a) comminuting a solid aluminum-beryllium alloy starting material to provide a particulate product mixture comprising a coarse particulate fraction comprising an aluminum-beryllium eutectic composition and a fine particulate fraction comprising a beryllium enriched material and

(b) separating said fine fraction from said coarse fraction.

2. The process as defined in claim 1 wherein said aluminum-beryllium alloy starting material contains at least about 1 percent by weight of beryllium.

3. The process as defined in claim 1 wherein the aluminum-beryllium alloy starting material is comminuted to provide a particulate product mixture containing a coarse particulate fraction having a size greater than about 100 mesh and a fine fraction having a size less than about 100 mesh.

4. The process as defined in claim 1 wherein the aluminum-beryllium alloy starting material is comminuted to provide a particulate product mixture containing a coarse particulate fraction of a size greater than about 200 mesh and a fine particulate fraction of less than about 200 mesh.

5. The process as defined in claim 1 including in addition: initially providing said aluminum-beryllium alloy starting material in a molten form; slowly cooling said molten alloy starting material over a prolonged period of time to provide a beryllium enriched solid phase and a molten aluminum-beryllium eutectic phase, and separating said beryllium enriched solid phase from said liquid phase and processing the beryllium enriched phase through steps (a) and (b).

6. The process as defined in claim 5 wherein said molten aluminum-beryllium alloy starting material is cooled over a period of at least 3 hours from a temperature of about 1100° C. to about 600° C. to provide said molten and solid phases.

7. The process as defined in claim 6 wherein said aluminum-beryllium alloy starting material contains greater than about 5 percent by weight of beryllium.

8. The process as defined in claim 1 wherein said aluminum-beryllium alloy starting material contains greater than about 5 percent by weight of beryllium.

9. A process which comprises:

(a) contacting BeO at a temperature greater than about 1100° C. with a sufficient quantity of an aluminum source material selected from the group consisting of aluminum, an aluminum-beryllium alloy and mixtures thereof to produce an aluminum-beryllium alloy starting material containing greater than about 1 percent of beryllium;

(b) cooling said aluminum-beryllium alloy over a prolonged period of time to provide a beryllium enriched solid phase and a molten aluminum-beryllium alloy phase;

(c) separating said molten phase from said solid phase; (d) comminuting said beryllium enriched solid phase to provide a particulate product mixture containing a coarse particulate fraction greater than 100 mesh in size consisting of an aluminum-beryllium eutectic material and a fine particulate fraction less than 100 mesh in size consisting of an enriched beryllium material; and

(e) separating said coarse fraction from said fine fraction.

10. The process as defined in claim 9 wherein said molten aluminum-beryllium alloy starting material is cooled from about 1100° C. to about 600° C. over a period of at least about 3 hours to provide said molten aluminum-beryllium phase and said beryllium enriched solid phase, comminuting said beryllium enriched phase to provide a particulate product mixture consisting of a coarse particulate fraction greater than about 200 mesh in size and a fine particulate fraction less than about 200 mesh in size.

References Cited

UNITED STATES PATENTS

3,083,094	3/1963	Love	75—84
3,337,334	8/1967	Fenn	264—111
3,477,844	11/1969	Jackson	75—150

FOREIGN PATENTS

11,803	9/1960	Japan	75—62
270,740	6/1928	Great Britain	75—63

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