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DRY PROCESS FOR CONTROLLED SURFACE OXIDATION OF BERYLLIUM POWDERS

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The invention relates to a process of treating beryllium metal powders.

More specifically, the invention is directed to a novel process for forming oxide upon the surfaces of beryllium metal powder particles and more particularly for controlling the amount or percent of such oxide formation upon powder of subsieve particle size.

Beryllium metal powders, particularly those of subsieve particle size have many applications or uses wherein the amount of surface oxidation on the powder particles has an important influence or effect upon products in which the powder is used, or products made entirely of the oxidized subsieve powder.

Accordingly, it is highly desirable to be able to control the amount of surface oxidation of the powder particles.

It is, accordingly, a particular object of the present invention to provide a novel process whereby surface oxidation of beryllium metal particles, more particularly particles of subsieve size, can be controlled whereby to obtain a predetermined percentage of such oxidation.

It is a further object of the invention to provide a novel process which is applicable to subsieve powders of beryllium metal, which carry an initial percentage of beryllium oxide coating, whereby such coating can be increased or added to up to a desired percentage.

A still further object of the invention is to provide a novel dry process for controlled oxidation of beryllium metal subsieve powders of both the high purity and technical grade purity.

Broadly the objects of the present invention are attained by the addition of controlled volumes of atmospheric air to beryllium metal powder which has been preheated to a specified temperature. By controlling the volume of air to which the beryllium metal powder particles are exposed, it is possible, according to the process of the present invention, to effectively control the degree of surface oxidation at any given temperature.

Subjecting preheated powders to uncontrolled volumes of air in a treatment area such, for example, as an open end tube furnace, many factors are involved which influence the amount of surface oxidation resulting on the powder particles which prevent the production of particles having a known or predetermined percentage of surface oxidation.

The process of the present invention involves the application of Avogadro's Law according to which 22.4 liters of any gas at standard conditions will contain one gram molecular weight of the substance comprising the gas.

Atmospheric air consists principally of 20% oxygen and 80% nitrogen. Therefore, according to the stated Law, since air contains 20% oxygen, 22.4 liters of air will contain .2 gram molecular weight of oxygen or 6.4 grams. Thus, one liter of air will contain .285 gram of oxygen which is equivalent to .446 gram maximum of oxide to be picked up by the beryllium metal, per liter of air. However, for any particular temperature the beryllium oxide pickup is not necessarily quantitative.

It has been discovered that beryllium metal subsieve powder when heated below certain temperatures and contacted with controlled volumes of atmospheric air will

show a beryllium oxide pickup on the surface of the particles which is not quantitative or in direct proportion to the volume of air to which the powder is exposed whereas, when heated to and above certain temperatures the degree of air oxidation is quantitative or substantially directly proportional or equivalent to the volume of air to which the powder is exposed or with which it is contacted. Accordingly, by controlling the temperature of the powder and the proportion of atmospheric air with which the heated powder is brought into contact, the amount of surface oxidation on the subsieve powder particles can be predetermined and controlled.

In explanation of the foregoing, it has been found that a surface beryllium oxide pickup of only .5% is obtained at a powder temperature of 190° C. even when sufficient air is introduced into the chamber containing the powder to theoretically produce a pickup of 4.5% BeO.

At a powder temperature of 230° C. there is a surface BeO pickup of .7% with an input of air into the powder chamber equivalent to 1.0% BeO and this only increases to 1.0% by adding a volume of air equivalent to 4.2% BeO.

In order to achieve a surface beryllium oxide pickup of 2.2%, a volume of air equivalent to 4.2% BeO is introduced into the powder chamber in which the beryllium powder is preheated to a temperature of 300° C.

At powder temperatures above 350° C., the degree of air oxidation is quantitative or substantially directly proportional to the volume of air with which the powder is contacted. Thus, a BeO increase of 4% is achieved by heating the beryllium metal powder to a minimum temperature of 350° C. and exposing it to, or contacting it with, a volume of air calculated to be equivalent to 4% BeO.

It has been determined that oxidizing beryllium powders by as much as 5% BeO at temperatures as high as 400° C., with controlled air volumes, results in a nitrogen pickup of less than .01% indicating a selective attack by oxygen.

In carrying out the process of the present invention, the air volumes do not necessarily have to be introduced into the powder chamber at one time but can be introduced in any number of separate or successive volumes. For example, if the powder is contained in a vessel such as a conical vacuum drier whose volume is 10 cubic feet and the calculated volume of air required to produce the desired oxidation, is 30 cubic feet at the operating temperature of the drier, the air can be added by evacuating the vessel and introducing the 10 cubic feet capacity of air and then repeating the evacuation and refilling the vessel with air until the total volume is added.

Above or more than 99% of the air is evacuated from the powder chamber for introducing a succeeding volume.

The new volume of air is introduced slowly into the chamber, that is, it is allowed to bleed in at a slow rate so as to take a predetermined period of time for it to contact and react with the heated powder and the desired temperature is maintained for a predetermined time as set forth in the following examples.

During the period of introducing the air into the chamber, the heated powder is continuously agitated in order to continually expose new metal particle surfaces for oxidation, and thereby produce a uniform surface oxidation around each particle.

By the stated process or procedure, the powder will react with the oxygen content of the air and the oxygen depleted air can then be withdrawn by means of a suitable vacuum system and replenished in the manner stated.

While in the preceding paragraph it has been set forth that the process is carried out in a conical vacuum drier,

this is only one type of treatment receptacle which may be employed. It is possible to use an externally heated rotary kiln for carrying out the process provided that precautions are taken to regulate the volume of air swept through the tube relative to the beryllium powder charge therein.

Following are examples of the working of the process of the present invention with a tabulation of the results obtained in connection with each of the examples.

The procedure of the present invention is applicable to subsieve powders of varying degrees of particle size.

By subsieve powders is meant powders of 25 micron average particle size and less. The examples herein set forth illustrate the successful operation of the process upon beryllium metal powders with 7 microns, 10 microns, and 14 microns average particle size, and of both the high purity and technical grade purity powder.

EXAMPLE I

Nuclear grade beryllium powder containing .07% Fe and .9% Mg and having a weighted average particle size of 10 microns was used for these experiments. Each experiment involved placing 180 grams of beryllium powder into a 4 liter suction flask whose total volume was 4.2 liters. The contents were evacuated with a vacuum pump and were heated to the respective temperature. Air was bled in slowly over a period of two minutes to atmospheric pressure, manually agitating the flask at the same time. The desired temperature was maintained for 30 minutes, the flask was evacuated with the vacuum pump, and the procedure was repeated for the desired number of air changes. Following are the results:

Temp.	Air Changes	Percent BeO Found	BeO Pickup, percent	
			Theoretical	Found
Blank		.48		
230° C.	1	1.22	1.04	.74
230° C.	4	1.50	4.16	1.02
300° C.	1	1.57	1.04	1.09
300° C.	4	2.70	4.16	2.22

EXAMPLE II

Technical grade beryllium powder containing .45% Fe and 1.0% Mg and having a weighted average particle size of 7 microns was used for these experiments. This series of tests included placing a 30 gram sample of beryllium powder in a flask whose over-all volume was 550 ml., heating the contents to the desired temperature under vacuum, then bleeding in air slowly (with manual agitation of flask contents) to atmospheric pressure. This procedure was repeated until a total of six air changes were completed at each respective temperature. The test results were as follows:

Temp.	Percent BeO Found	BeO Pickup, percent	
		Theoretical	Found
Blank	.30		
180° C.	.80	4.68	.50
220° C.	1.09	4.68	.79
300° C.	2.48	4.68	2.18
350° C.	4.20	4.68	3.90
400° C.	5.05	4.68	4.75

EXAMPLE III

Nuclear grade beryllium powder containing .09% Fe and 0.06% Mg and having a weighted average of 14 microns was used for these experiments. A total of 125 grams of beryllium powder was charged into a flask whose total volume was 4.2 liters. The flask contents were put under vacuum with a vacuum pump and were heated to 140° C. For one experiment air was bled in slowly to atmospheric pressure over a period of two minutes while maintaining manual agitation of the contents, the contents were reheated to 140° C. and maintained at that temperature for 20 minutes, the system was again evacuated and air bled in as before. This was repeated until the powder was subjected to three air changes for a total input of 12.6 liters of air.

A second experiment was conducted to determine the effect of adding oxygen gas in place of atmospheric air. An amount of 125 grams of beryllium powder was added to a flask whose total volume was 2.2 liters. The flask contents were put under vacuum, heated to 140° C., oxygen was bled in slowly over a period of three minutes while agitating the contents manually, maintained at a temperature of 140° C. an additional 20 minutes, and the procedure was repeated. Results of the above tests were as follows:

Temp.	Oxidation Media	Percent BeO Found	BeO Pickup, percent	
			Theoretical	Found
Blank		.72		
140° C.	Air	1.26	4.50	.54
140° C.	Oxygen	1.33	11.80	.61

With reference to the tabulations following the examples set forth, it will be noted that at the lower temperatures employed, the beryllium oxide pickup is considerably less than would be expected from the theoretical calculations but at higher elevations and particularly after the temperature employed rises above 300° C., the actual pickup of beryllium oxide more closely approaches what is to be expected from the theoretical calculations. For example, considering the figures set forth after Example I. The beryllium metal powder at the start was found to carry .48% of beryllium oxide. At a temperature of 230° C., the pickup to be expected according to the theoretical calculations would be 1.04%. With the powder at the temperature indicated and after being subjected to one air change, the beryllium oxide found was 1.22% which, considering the .48% initially on the powder gave an actual beryllium oxide pickup of .74%.

At 300° C. with four air changes,, the total beryllium oxide found on the particles was 2.70% which, considering the initial .48% gave an indicated beryllium oxide pickup of 2.22%.

Now, upon reference to Example II, it will be seen from the results tabulated that with the beryllium metal subsieve powder raised to a temperature of 400° C. and subjected to a total of six air changes, the beryllium oxide found on the powder amounted to 5.05% which, considering the initial oxide present of .30%, gave an indicated beryllium oxide pickup of 4.75% which is actually greater than was to be expected from the theoretical calculations which indicated an expected pickup of 4.68%.

The examples set forth indicate the presence of a certain amount of beryllium oxide on the particles started with. However, it would be obvious that the process may be successfully carried out upon a powder which may have been previously formed under conditions of control which would result in the production of oxide free particles.

From the foregoing, it will be apparent that there is provided by the present invention a novel process by which controlled oxidation of beryllium subsieve metal powders can be carried out. By following the teachings of this process, powder particles may be obtained with an accurately detailed percentage of oxide coating.

We claim:

1. A process for producing sub-sieve beryllium metal powders containing a predetermined amount, up to about 5 percent beryllium oxide, comprising heating said powders in the presence of an oxygen containing gas at temperatures of from 140° to 400° C., said amount of beryllium oxide being directly proportional to the increase in temperature.

2. The process of claim **1**, wherein the beryllium metal powders are of a weighted average particle size of from 7 to 25 microns.

3. A process for producing sub-sieve beryllium metal powders containing a predetermined amount, up to about 5% beryllium oxide, comprising heating said powders in

a closed chamber to a temperature above 350° C. and contacting said powders with a gas containing oxygen in an amount up to about 5% by evacuating the chamber and refilling the same with a fresh charge of gas for as many times as may be necessary to furnish the required quantity of oxygen corresponding to the beryllium oxide desired.

4. The process of claim 3, wherein the beryllium metal powders are of a weighted average particle size of from 7 to 25 microns.

5. The process of claim 3, wherein the reaction takes place in a closed chamber and the oxygen containing gas is air.

6. The process of claim 3, wherein the beryllium is exposed to the gas for a period of from about 20 to 30 minutes.

7. The process of claim 3, wherein the powders are agitated.

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