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PROCESS FOR CONTROLLED SURFACE OXIDATION OF BERYLLIUM POWDERS

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

More particularly, the invention is directed to a process of controlling surface oxidation of beryllium metal powders.

Beryllium metal powders, particularly those of subsieve particle size have many applications wherein the amount or extent of surface oxidation of the particles has an important influence or effect upon the product resulting from the use of such powder. Accordingly the use of such beryllium subsieve powders having controlled amounts of surface oxide is highly desirable.

It is a particular object of the present invention to provide a process for treating beryllium subsieve powders whereby controlled amounts of surface oxide upon the powder particles are obtained.

Among the many areas of use of beryllium metal subsieve powders in which the amount of surface oxide upon the particles is critical, are the production of beryllium metal parts by powder metallurgy where the properties of such parts vary with the amount of surface oxide on the starting powders, and in the production of certain rocket and missile propellants wherein the degree of surface oxidation of the beryllium powders affects ballistics performance.

A further object of the invention is to provide a process wherein the desired surface oxidation and the reduction of the beryllium metal to subsieve particle size are obtained concurrently.

A still further object of the invention is to provide a process for effecting controlled surface oxidation of beryllium metal subsieve powder by incorporating controlled amounts of water in an otherwise anhydrous liquid vehicle in which the milling operation of the metal is carried out.

In my prior application Ser. No. 302,828, now abandoned, there is set forth a process of wet milling beryllium metal by which a powder of subsieve particle size having a beryllium oxide free surface is obtained.

In accordance with the present invention, it has been discovered unexpectedly that in carrying out the process of comminuting beryllium metal for producing powders of subsieve particle size range by means of the wet milling operation, the powdered particles may be produced with a predetermined or controlled amount of surface oxidation which is directly proportional to the amount of water added during or prior to the milling operation.

In comminuting of the beryllium metal, as the average particle size of the powder goes below 25 microns, the freshly fractured powder surfaces become chemically reactive. In the wet milling of the powder, the freshly fractured surfaces are, therefore, continually exposed to contact with the liquid media. When water is present, or is added to the liquid milling vehicle, the freshly fractured powder surfaces react practically stoichiometrically with the water in accordance with the following chemical equations.

(A) $Be+2H_2O=Be(OH)_2+H_2$

(B) $Be+H_2O=BeO+H_2$

In each of the foregoing equations it will be seen that hydrogen gas is generated, this being a fact that has been verified by experience. It has also been demonstrated as a result of tests that the over-all reaction occurs according to Equation B. Beryllium hydroxide, if it does form as indicated by Equation A, must decompose to BeO and release additional water which reacts with more beryllium to form BeO according to the following chemical equations.

(C) $Be(OH)_2=BeO+H_2O$

(D) $Be+H_2O=BeO+H_2$

The controlled surface oxidation according to the process of the present invention, has been found applicable for the production of beryllium subsieve powder using as a starting feed metal nuclear grade chips, technical grade chips, nuclear grade crushed beads, and technical grade crushed beads. The term "chips" as here used represents lathe turnings from vacuum cast billets and "beads" represents the as-reduced metal in the form of irregular spheroids.

Following are four examples of the working of the process of the present invention with a tabulation of the results obtained according to the examples.

Example I

80 pounds of cobalt bonded, one-half inch diameter tungsten carbide spherical balls were introduced into a 2.5 gallon total volume beryllium metal jar mill. A charge of 1500 grams of nuclear grade beryllium metal chips, 1500 ml. anhydrous naphtha, and 45 ml. of distilled water were introduced into the mill. The mill was closed and rotated at 45 r.p.m. for 12 hours. The mill was vented to release hydrogen pressure that had generated, resealed, and rotated an additional 8 hours. The slurry was removed from the mill, filtered, and vacuum dried. The resulting dry powder had a weighted avearge particle size of 12 microns.

Example II

The procedure was the same as for Example I, except that 1500 grams of minus ⅛ inch crushed nuclear grade beads were charged instead of chips, 35 ml. of distilled water was added instead of 45 ml., and the total time for rotation was 28 hours instead of 20 hours. The resulting dry powder had a weighted average particle size of 13 microns.

Example III

300 pounds of cobalt bonded, one-half inch diameter tungsten carbide spherical balls were introduced into a 10 gallon total volume beryllium metal mill. A charge of 12 pounds of technical grade chips, 1.5 gallons of anhydrous naphtha, and 90 ml. of distilled water were introduced into the mill. The mill was closed and rotated at 45 r.p.m. for 15 hours. The mill was vented to release hydrogen pressure that had generated, resealed, and rotated an additional 9 hours. The slurry was removed from the mill, filtered, and vacuum dried. The resulting dry powder had a weighted average particle size of 10 microns.

Example IV

The procedure was the same as for Example III, except that the metal charge was technical grade minus ⅛ inch

crushed beads, the water added was 180 ml., the mill was vented after 20 hours, and the total time for rotation was 40 hours. The resulting dry powder had a weighted average particle size of 8 microns.

The following tabulation based on the foregoing examples illustrates the results obtained.

Charge Weight	Charge Type *	Water Added, gms.	Percent BeO Pickup A	Percent BeO Pickup B	Percent BeO Start	Percent BeO Product
1,500 gms	N.C.	45	2.05	4.00	.32	4.31
1,500 gms	N.B.	35	1.59	3.12	.18	3.24
12 lbs	T.C.	90	1.14	2.26	.35	2.62
12 lbs	T.B.	180	2.26	4.39	.29	4.65

*N.=nuclear grade, T.=technical grade, C.=chips, B.=beads.

As shown, this tabulation sets forth the beryllium metal batch size; the type of beryllium feed; the amount of water added per batch; theoretical percent surface oxidation pickup for Equation A and Equation B calculated from the weight of water added to the respective batch size; the percentage of beryllium oxide (BeO) present in the starting metal charge, and the total BeO found in the respective beryllium powders.

It will be apparent from the above tabulation that the over-all reaction occurs according to Equation B, hereinabove referred to. The columns marked "Pickup A" and "Pickup B" represent the theoretical oxide pickup to be expected according to the chemical Equations A and B and that the actual pickup of BeO substantially conforms to that to be expected or indicated by Formula B. For instance, considering Example I, the percent of BeO in the starting charge is given as .32 percent and the final product shows a BeO percent of 4.31. Deducting the .32 percent of BeO in the starting charge from the 4.31 percent of BeO in the product shows a total increase of the beryllium oxide of 3.99 percent as against 4.00 percent theoretically expected.

In referring hereinabove to "subsieve" powder, this defines a powder whose weighted average particle size is below 25 microns.

In the examples of the process as hereinabove set forth, reference has been made to the use of anhydrous naphtha as the hydrocarbon vehicle in which the metal is ground or milled and to which the measured amount of distilled water is added either before starting the milling operation or during the same. It is to be understood, however, that use may be made of any one of a number of other anhydrous, non-halogenated hydrocarbons, such as benzene, n-hexane, n-heptane, and the like.

I claim:

1. A process for controlled surface oxidation of beryllium metal powders of a weighted average particle size below 25 microns which comprises oxidizing the said metal particles with a stochiometric amount of chemically pure water.

2. A process for controlled surface oxidation of beryllium metal powder particles, which comprises milling beryllium metal to a weighted average particle size below 25 microns in an anhydrous non-halogenated hydrocarbon to which has been added a predetermined amount of distilled water, based upon the quantity of metal being milled.

3. A process for controlled surface oxidation of beryllium metal powders, which comprises milling beryllium metal to a weighted average particle size of below 25 microns in an anhydrous non-halogenated hydrocarbon vehicle having incorporated therein controlled amounts of chemically pure water and thereby obtaining pickup of an amount of surface oxidation on the powder directly proportional to the amount of water added.

4. A process for controlled surface oxidation of beryllium metal powder particles, which comprises milling the metal to a weighted average particle size below 25 microns in an anhydrous non-halogenated hydrocarbon vehicle selected from the group consisting of naphtha, benzene, n-heptane, and n-hexane and having therein a calculated amount of chemically pure water based upon the quantity of metal taken such as to effect a stoichiometric reaction thereof with the metal particles to obtain a pickup of a desired amount of surface oxidation on the powder.

5. A process according to claim 4 wherein the said calculated amount of water is combined with the hydrocarbon vehicle prior to milling.

6. A process according to claim 4 wherein the said calculated quantity of water is introduced into the hydrocarbon vehicle during the milling operation.

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