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METHOD FOR REDUCING THE PYROPHORICITY OF METALLIC POWDERS

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The present invention relates generally to pyrophoric metal powders and more specifically to a novel method for reducing the pyrophoricity of such powders.

It has long been known that certain metals, when reduced to a suitably fine state of division are pyrophoric, i.e. said metals undergo spontaneous combustion when exposed to an oxidizing atmosphere. Thus, depending somewhat upon the particular metal involved and the average particle size, powders comprising metals and metal alloys such as iron, titanium, zirconium, cobalt, lead, nickel, uranium, manganese, zinc, cadmium, aluminum, silver/thorium or iron/cobalt alloys etc., can be pyrophoric. Obviously, handling of such powdered metals is difficult, and moreover, can present a serious fire or explosion hazard. In accordance with the present invention, the pyrophoric character of such finely-divided metals and metal alloys is vastly reduced or eliminated.

It is a principal object of the present invention to provide a novel method for reducing the pyrophoric character of powdered metals.

It is still another object of the invention to provide a method for reducing the pyrophoric character of powdered metals which method does not alter the physical structure of the powder.

Other objects will in part be obvious and will in part appear hereinafter.

In accordance with the present invention it was discovered that the pyrophoric character of pyrophoric powdered metals is vastly reduced when said metals are contacted with certain gases at low temperatures.

Generally speaking, any pyrophoric powdered metal or metal alloy can be treated by the process of the present invention. As is well recognized in the art, metal and metal alloy powders, depending to some extent upon the particular metals involved, can be pyrophoric when the average particle size thereof is less than about 5 microns and particularly when said average particle size is less than about 1 micron.

Gases suitable for use in the present invention are those chosen from the group consisting of carbon dioxide, carbon monoxide, sulfur dioxide, sulfur trioxide, hydrogen sulfide, ammonia and the halogens. However, it should be borne in mind that it is also important that the gas not react chemically with the metal powder under the environmental conditions encountered during treatment of the powder. Obviously then, of the above-mentioned gases only carbon dioxide and carbon monoxide are considered generally chemically inert with respect to most of various types of pyrophoric metal and metal alloy powders which can be so treated. For instance, hydrogen sulfide would normally be unsuitable for use with pyrophoric silver powder because said gas tends to react chemically with silver and to form sulfides therewith. For this reason as well as because the treatment can be effected conveniently, carbon dioxide is much preferred as the treatment gas.

It is important that the metal powder be contacted with the gas at low temperatures, i.e. at less than about —20° C. and preferably below about —30° C. If temperatures above about —20° C. are utilized little or no reduction in pyrophoricity will normally be achieved. Said contact is normally achieved most conveniently when

the gas is cooled to the solid or liquid state. A particularly simple method of effecting the treatment when carbon dioxide is the gas utilized comprises contacting the metal powder with Dry Ice, i.e. carbon dioxide cooled to the solid phase.

Having reduced the pyrophoric character of the metal powder said powder can be handled more easily and with less danger than has been formerly encountered in the art. For instance, the metal powder product of the present invention having reduced pyrophoric character can often be pressed into desired shapes in oxidizing atmospheres such as air, thereby greatly simplifying forming apparatus and procedures. Heretofore, it was often necessary to carry out pyrophoric metal powder forming operations in an inert atmosphere. Another important aspect of the invention resides in the further discovery that, once having reduced the pyrophoric character of the powder in accordance with the process, said character remains depressed even though the powder is subsequently subjected to elevated temperatures which might have been expected to result in complete recovery of its pyrophoric character.

For instance, as will be demonstrated in the examples the treated powders of the present invention can be heated in an inert atmosphere, cooled to room temperature and remain non-pyrophoric. Conversely, when an inert gas such as nitrogen or a member of Group VIII*a* of the Mendeleev Periodic System is contacted with a pyrophoric metal powder at low temperature, pyrophoricity is normally regained even at room temperature (i.e. about 20° C.). It should be noted, however, that if the treated metal powders of the present invention are exposed to an oxidizing atmosphere at elevated temperatures, i.e. above about 200° F., pyrophoric activity will usually be elicited. Apparently there exists an "activation" temperature for each powder/gas combination in an oxidizing atmosphere above which pyrophoricity results and below which little or no pyrophoric character is expressed.

EXAMPLE 1

About 50 grams of a pyrophoric iron powder having an average particle diameter of about 45 millimicrons and which had been stored at 20° C. in a nitrogen atmosphere was charged, without exposure to the atmosphere into a 200 ml. beaker charged with about 80 grams of Dry Ice. The Dry Ice was placed in the beaker about 5 minutes prior to the charging of the iron powder thereinto so as to insure that air would be substantially completely displaced by CO_2. After the Dry Ice passed entirely into the gas phase, the iron powder was exposed to air at room temperature and it was found that no pyrophoric reaction occurred. However, when an untreated sample of the powder was exposed to air, said powder burned violently.

Next, about 40 grams of the treated powder was placed in a tablet mold and a tablet having a radius of about 1 inch and a thickness of about ¼ inch was prepared. The resulting tablet was also non-pyrophoric.

The tablet and the remaining 10 grams of treated powder were then placed in a nitrogen swept oven and heated to about 400° F. for about 30 minutes. The oven and contents thereof were cooled to room temperature with continuous nitrogen sweep. When exposed to air neither the powder nor the tablet were pyrophoric.

When a similar tablet and powder sample are heated as mentioned above with the exception that no cooling is effected prior to exposure to the atmosphere both the powder and tablet are pyrophoric.

When the carbon dioxide treatment is carried out as described above with the exception that the Dry Ice is allowed to vaporize completely and the interior of the beaker attains a temperature of about —10° C. prior to

the introduction thereinto of the metal powder little or no reduction in pyrophoricity of said powder is achieved.

EXAMPLE 2

This example is essentially a duplicate of Example 1 with the exception that liquid nitrogen (at 760 mm. Hg) is utilized as the treating gas. When the treated powder was exposed to the atmosphere at about 70° F., pyrophoricity thereof was exhibited by violent burning.

It is believed, although there is no intent to be bound by this explanation, that treatment of the pyrophoric metal powder with certain inert gaseous compounds under conditions of low temperatures results in a peculiarly occluded metal surface in which active sites for oxidation are stringently deactivated. This explanation would appear to be additionally supported by the fact that even when a metal powder which has been rendered non-pyrophoric by the process of the present invention is thereafter subjected to temperatures normally sufficient to desorb the gas from the powder surface, the prior pyrophoric character nevertheless does not return.

Obviously, many changes can be made in the above examples and description without departing from the scope of the invention.

For instance, pyrophoric metal powders, other than the iron powder specifically mentioned in the example, such as lead, silver, thorium, zirconium, zinc, etc., are also suitable for treatment by the process of the invention.

Moreover, gaseous compounds other than carbon dioxide are also suitable, provided of course that adherence to the temperature and chemical inertness limitations set forth hereinbefore is maintained. Thus, gases such as carbon monoxide, hydrogen sulfide, and the like are often suitable.

Accordingly, it is intended that the above disclosure be regarded as illustrative and as in no way limiting the scope of the invention.

What is claimed is:

1. A process for reducing the pyrophoric character of pyrophoric metallic powders which comprises contacting said powder at temperatures below about −20° C. with a gas chemically inert with respect to said metal and chosen from the group consisting of carbon dioxide, carbon monoxide, sulfur dioxide, sulfur trioxide, hydrogen sulfide, ammonia and the halogens.

2. The process of claim **1** wherein contact between said gas and said powder is accomplished at below about −30° C.

3. The process of claim **1** wherein said gas is carbon dioxide.

4. The process of claim **3** wherein said gas is carbon dioxide cooled to the solid phase.

5. A process for handling pyrophoric metallic powders which comprises:

(1) contacting a pyrophoric metallic powder at temperatures below about −20° C. with a gas chemically inert with respect to said metal and chosen from the group consisting of carbon dioxide, carbon monoxide, sulfur dioxide, sulfur trioxide, hydrogen sulfide, ammonia and the halogens, and

(2) thereafter exposing the resulting treated powder to an oxidizing atmosphere at temperatures below about 200° F.

6. The process of claim **5** wherein contact between said gas and said powder is accomplished at temperatures below about −30° C.

7. The process of claim **5** wherein said metallic powder comprises iron.

8. The process of claim **5** wherein the gas utilized is carbon dioxide.

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