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2,771,035

PROPELLANT

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This invention relates generally to propellants and more particularly to an improved propellant powder and to a process for making the same.

Ammonium nitrate, black powder, potassium nitrate, potassium sulfate, barium nitrate and the like have been utilized, heretofore, to modify the burning characteristics of propellant powder grains. Such modifiers have been either incorporated in the propellant grain or, probably more frequently, have been applied to the surface of the grain as a coating to improve the ignition characteristics of the grain as well as to reduce muzzle flash. The coating has usually been applied by tumbling the propellant grains in particles of the modifier and in a molten deterrent by a process similar to that disclosed in U. S. Patent 1,354,640. Modifiers of these types are hygroscopic, however, and the particles thereof rapidly cluster or agglomerate when exposed to the atmosphere. Because of this characteristic of the modifier, it has been impossible to apply a uniform coating of discrete particles of modifier to the surface of propellant powder grains. Consequently, the burning characteristics of some powders have not been sufficiently improved by the modifier to entirely eliminate muzzle flash or to improve the ignition of some propellants sufficiently to permit their utilization in some types of cartridges.

It is therefore an object of this invention to provide an improved propellant powder. It is another object of the invention to provide propellant powder grains having improved igniting and burning characteristics. A further object is to provide a novel process for coating propellant powder grains with a modifier. Another object is to provide a method for improving the ignition and burning characteristics of propellant powder grains. A more specific object is to provide powder grains having a modifier on their surface which improves the ignition characteristics thereof.

The foregoing objects as well as others are accomplished in accordance with this invention, generally speaking, by providing a process for coating propellant grains with particulate crystals of solid modifier. More specifically, the invention contemplates a method in which a modifier is ground into particulate form while it is dispersed in an anhydrous liquid and thereafter coating propellant powder grains with the resulting suspension of discrete particles of the modifier. It is preferred to grind the modifier in a liquid plasticizer for nitrocellulose and to coat the powder grains with the resulting suspension of particulate crystals of modifier although the modifier can be ground to particulate form in an anhydrous liquid other than a nitrocellulose plasticizer provided this liquid is replaced with, or has added thereto, a plasticizer which will soften the surface of the nitrocellulose base powder grains during the coating process. Plasticizers for nitrocellulose which are a solid at room temperature can be utilized when they are dissolved in a volatile non-solvent for the modifier. The greater the volume of liquid in the suspension the more rapidly the grain will become uniformly coated

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with the modifier. If a solid plasticizer is used it is dissolved in the liquid carrier of the modifier either before or during the step of coating the powder grains.

Particulate crystals of potassium nitrate substantially uniformly distributed over the surface of smokeless propellant powder grains have been found especially advantageous in improving the ignition characteristics of propellants having a heavy surface coating of a deterrent. In fact, none of the other modifiers enumerated hereinafter have been found equally effective for this purpose, although they do offer other worthwhile advantages when applied to the surface of powder grains in accordance with this invention.

"Particulate crystals" is used herein and in the appended claims to designate particles of the modifier which are not only of fine granulation as described more fully hereinafter but which are discrete particles as distinguished from clustered or agglomerated particles. The particles of modifier are bound to the surface of the grain or are embedded therein in such a way that at least one surface thereof protrudes from the surface of the powder grain.

Examples of suitable non-solvents which can be used as a grinding medium or as a diluent for the plasticizer are substantially anhydrous isopropyl alcohol, methyl alcohol, benzene, toluene, ethyl ether, and the like but other liquid non-solvents for the modifier which are compatible with the plasticizer and other components of the grinding medium and which can be removed from the coated powder grains at temperatures to which powder grains can be heated safely can be utilized. For most practical purposes, non-solvents having a boiling point of less than about 100° C. are preferred. Any plasticizer for nitrocellulose which is a liquid at ordinary room temperatures or which is soluble in the non-solvent diluent utilized in conjunction therewith can be utilized. Examples thereof includes the diaryl phthalates like diphenylphthalate, the dialkylphthalates such as dibutylphthalate and the like, the diaryl dialkylureas, dinitrotoluene, dimethylsebacate, triphenyl phosphate, triethylene glyco di-2 ethylbutyrate and the like. Both the plasticizer and the diluent must be substantially anhydrous.

The modifier must be ground to a granulation such that substantially all of the particles are less than 30 microns across their major axis but it is preferred that the particles of the modifier be ground to a particle size within the range of from about 3 microns to about 10 microns. Any solid modifier for powder grains such as ammonium nitrate, potassium sulfate, potassium bichromate, barium nitrate, potassium nitrate, and the like can be ground and subsequently applied to powder grains in accordance with the process of this invention provided the grinding medium or liquid carrier is not a solvent for the particular modifier selected. Such modifiers when they are precipitated from their solution by ordinary methods have a particle size substantially greater than 30 microns due to normal crystal size and clustering. Furthermore, the invention has been found admirably suitable for applying other powdered modifiers such as powder metals and the like.

A minor amount of rosin or a synthetic resin such as ethyl cellulose, or nitrocellulose is dissolved in the grinding medium for best results. Judgment must, of course, be exercised in selecting the type and amount of resin in order to avoid a viscosity which will hinder or prevent proper grinding or prevent obtaining uniform distribution of the suspension of modifier over the propellant grains. Actually the purpose of the resin is not to form a film over the grains after solvent removal but is to aid in wetting and dispersing the particles of modifier during the grinding step in the process. For most practical purposes the amount of resin can vary from about 0.1 to about 1.0 part dissolved in a liquid carrier composed of from about

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20 parts to about 60 parts volatile non-solvent for the modifier or diluent and about 10 parts to about 20 parts plasticizer. The ratio of diluent to plasticizer must be varied with the solubility of nitrocellulose in the plasticizer because sufficient plasticizer must be used in the coating process to soften the surface of the powder grains. The particular ratio of amount of modifier to amount of carrier is not critical but best results are obtained if not more than about 1 part modifier to about 1 part liquid phase are utilized, although up to 4 parts liquid to one part modifier or as little liquid as one part to 3 parts modifier can be utilized.

As pointed out hereinbefore, water in quantities sufficient to dissolve an appreciable quantity of the modifier must be avoided in the liquid carrier. Small amounts of calcium carbonate or similar salts useful for neutralizing acids are advantageously included in the carrier to neutralize any acidic compounds therein and small amounts of a stabilizer such as diphenylamine or ethyl centralite can be conveniently added to the propellant grains by means of this process.

The invention is applicable to propellant powder grains of various sizes and shapes which have been formed by any of the conventional methods for making propellant grains. Globular propellant grains formed by a process similar to that disclosed by Olsen, Tibbetts, and Kerone in U. S. Patent 2,027,114, for instance, as well as powder grains formed in this way but subsequently rolled, may be treated in accordance with this invention with particular advantage including improved ignition properties. Grinding the modifier in the substantially anhydrous suspending medium or carrier which may be subsequently used for coating the powder grains substantially eliminates any possibility of the finely ground particles becoming agglomerated or clustered and insures that the resulting grains will have a substantially uniformly distributed coating of modified in particulate form over the surface. It has been found that the ignition properties of powder grains is further improved if a coating of about 0.1 to 1.0 part dinitrotoluene based on the weight of the powder grain is placed on the surface of the grains prior to the coating of solid modifier.

In order to more fully describe and to further clarify the invention, the following is a detail description of one embodiment thereof:

A grinding medium or liquid carrier for the modifier is prepared in a ball mill by dissolving about 3 parts ethyl cellulose in about 150 parts dibutyl phthalate, about 3 parts diphenylamine and about 360 parts substantially anhydrous isopropyl alcohol. About 4 parts calcium carbonate are then suspended therein.

About 600 parts of commercial grade potassium nitrate are ground in a hammer mill to break up the lumps and are then placed in the ball mill containing the above described liquid carrier. Porcelain balls having a diameter of about 1½ inches occupy about one-half the volume of the mill and the mill is run at about 32 R. P. M. for about 20 hours or until the average particle size of the potassium nitrate in the resulting suspension will be about 7 microns with substantially none of the particles being greater than about 30 microns along their greatest axis.

A sweetie barrel is charged with about 1000 lbs. globular nitrocellulose base powder grains which were formed by the process disclosed in the aforementioned U. S. Patent 2,027,114 and were later coated with dibutyl phthalate by the process disclosed by McBride in U. S. Patent 1,955,927. About one and one-half gallons of the potassium nitrate suspension prepared as described in the foregoing are diluted to about four gallons total volume with substantially anhydrous isopropyl alcohol and the resulting solution is added to the sweetie barrel. The sweetie barrel is closed and the contents thereof are tumbled for about five minutes or until the suspension is substantially uniformly distributed over the grains. The lid is then removed and air is introduced into the

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barrel for about 40 minutes or until the isopropyl alcohol has been substantially removed. The temperature of the powder grains is elevated to about 55° C. when removal of the alcohol is begun and is held at this temperature throughout the treatment with air at ambient temperature. The powder grains are next treated with graphite by a conventional sweetie barrel coating method and the powder is ready for charging into suitable cartridges. The foregoing salt treating process will leave a coating of about one percent of discrete crystals of potassium nitrate on the propellant powder grains.

In another embodiment of the invention, the modifier is ground and suspended as described in the foregoing embodiment. The powder grains are coated with about 2.5 parts powdered dinitrotoluene per 1000 parts powder grains, however, before the liquid suspension of the modifier is added thereto. The powder grains are preferably coated with the dinitrotoluene by tumbling them in a sweetie barrel. It is unnecessary to heat the powder grains as the dinitrotoluene particles will adhere to the surface of the grain and are subsequently softened and bound to the surface of the grains by the liquid carrier.

In still another embodiment the modifier is ground as before but before the particles thereof are applied to the powder grains, the modifier is separated from most of the grinding medium by filtering. A sweetie barrel is charged with about 1000 lbs. globular powder grains which were formed by the process disclosed in the aforementioned U. S. Patent 2,027,114 and were later coated with dibutyl phthalate by the process disclosed by McBride in U. S. Patent 1,955,927. About 0.7 part of powdered dinitrotoluene and about 2.5 parts of powdered diphenylphthalate and about 10 parts of the filter cake (about 90 percent modifier) obtained above are added to the sweetie barrel. The contents are tumbled for about 15 minutes when the temperature in the barrel is elevated to about 55° C. and tumbling continued about 45 minutes or until the grains have a substantially uniform coating of modifier. The grains are coated with graphite as before.

Even greater quantities than one percent solid modifier can be applied to the surface of propellant grains by the process of this invention, if desired. It is extremely difficult, if not impossible, to apply a coating of more than about one-half percent solid modifier to propellant grains in accordance with prior art processes and consequently, an occasional muzzle flash has been obtained with such propellant grains, particularly if the powder charge was composed of globular propellant grains. With most propellant powder grains, however, about one percent of unclustered or discrete particles of potassium nitrate is sufficient to completely prevent muzzle flashes although the process can be utilized to apply as much as about 5.0 percent modifier, if desired.

It was found that propellant grains coated in accordance with this invention are more easily ignited than similar propellants coated with larger particles or coated with clustered particles of potassium nitrate or similar modifier in accordance with the prior art. In fact, primers heretofore only suitable for igniting the more easily ignited propellants, such as perforated cylindrical powder grains coated with an explosive deterrent, can now be used as a result of this invention for igniting the most difficult to ignite propellants including propellant grains having a heavy deterrent coating. Hence, the hazard of an occasional hangfire due to poor ignition of the propellant charge has been substantially eliminated and a much more uniform velocity has been obtained in cartridges where the ignitability of the propellant is poor but not to such a degree as to cause a hangfire. These improvements are obtained without any substantial deleterious action on the propellant grains and without an appreciable increase in production costs. Moreover, it has been found that various other characteristics of the cartridges such as the profile of the bullet, for instance, do not have to be controlled so closely as was formerly

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required to prevent the ballistic performance of the cartridge from becoming unsatisfactory. The improvement in ignition is due not only to the improved distribution of the particles of modifier over the surface of the grains but also to the fact that the particles are in particulate form.

This invention has been described in detail in the foregoing embodiment but it is not limited to such details and variations can be made within the skill of the art without departing from the spirit of the invention. For example, other suitable grinding apparatus can be used and in some instances, depending upon the physical condition of the modifier and upon the interval of time available for suspending it in the carrier, grinding of the modifier in a hammer mill or similar equipment prior to suspension thereof in the carrier can be eliminated. Heat alone or other suitable solvent removal steps can be used to remove the diluent from the propellant grains after the coating process.

Having thus described the invention, what is claimed and is desired to be covered by Letters Patent is:

1. In the manufacture of propellants, a process adapted for coating nitrocellulose base propellant grains with discrete, particulate crystals of potassium nitrate, which process involves grinding in a ball mill from about 1 to about 3 parts potassium nitrate in from about 1 to about 4 parts liquid carrier comprising the following ingredients in the ratio of about 0.1 to about 1 part ethyl cellulose, about 10 to about 20 parts dibutyl phthalate, about 20 to about 60 parts substantially anhydrous isopropyl alcohol until the potassium nitrate is suspended in the liquid carrier in particulate form, and thereafter wetting propellant powder grains with the suspension and removing said isopropyl alcohol from said grains.

2. In the manufacture of propellants, a process adapted for coating nitrocellulose base propellant grains with discrete, particulate crystals of potassium nitrate, which process comprises grinding in a ball mill from about 1 to about 3 parts potassium nitrate and from about 1 to about 4 parts carrier comprising the following ingredients in the ratio of about 0.1 to about 1 part ethyl cellulose, about 10 to about 20 parts dibutyl phthalate, about 20 to about 60 parts substantially anhydrous isopropyl alcohol until the potassium nitrate is suspended in the liquid carrier in particulate form, and thereafter mixing in a sweetie barrel nitrocellulose base propellant powder grains with the suspension, heating and tumbling the mixture until the suspension is substantially uniformly distributed over the grains and thereafter removing the isopropyl alcohol from the grains.

3. In the manufacture of propellants, a process adapted for coating nitrocellulose base propellant grains with discrete, particulate crystals of a modifier of the ballistic properties of the powder grains, which process comprises grinding in a ball mill a mixture of from about 1 to about 3 parts potassium nitrate and from about 1 to about 4 parts carrier comprising the following ingredients in the

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ratio of about 0.1 to about 1 part ethyl cellulose, about 10 to about 20 parts dibutyl phthalate, about 20 to about 60 parts substantially anhydrous isopropyl alcohol, until the potassium nitrate is suspended in the liquid carrier in particulate form, and thereafter mixing in a sweetie barrel nitrocellulose base propellant powder grains, dinitrotoluene and said suspension, tumbling the resulting mixture until the grains are substantially uniformly coated with the suspension, and thereafter removing the isopropyl alcohol from the grains.

4. In the manufacture of propellants, a process adapted for coating nitrocellulose base propellant powder grains with discrete, particulate crystals of a solid modifier of the ballistic properties of a nitrocellulose base powder grain selected from the group consisting of solid inorganic oxidizing salts comprising, comminuting the modifier while it is submerged in a substantially anhydrous liquid carrier comprising from about 10 to about 20 parts plasticizer for nitrocellulose, from about 20 to about 60 parts of a non-solvent for nitrocellulose and for the solid modifier that is compatible with the plasticizer and has a boiling point of less than about 100° C. until the said modifier is suspended in the liquid carrier in particulate form, and thereafter wetting propellant powder grains with the suspension and removing said non-solvent from the propellant powder grains.

5. In the manufacture of propellants, a process adapted for coating nitrocellulose base propellant powder grains with discrete, particulate crystals of a solid modifier of the ballistic properties of a nitrocellulose base powder grain selected from the group consisting of solid inorganic oxidizing salts comprising, comminuting the modifier while it is submerged in a substantially anhydrous liquid carrier comprising from about 0.1 to about 1 part ethyl cellulose, from about 10 to about 20 parts plasticizer for nitrocellulose, from about 20 to about 60 parts of a non-solvent for nitrocellulose and for the solid modifier that is compatible with the plasticizer and has a boiling point of less than about 100° C. until the said modifier is suspended in the liquid carrier in particulate form, and thereafter wetting propellant powder grains with the suspension and removing said non-solvent from the propellant powder grains.

6. A nitrocellulose base propellant powder grain having up to as much as about 5 percent potassium nitrate in discrete, particulate crystals of less than 30 microns along their major axis and bound to the surface of the propellant grain.

References Cited in the file of this patent

UNITED STATES PATENTS

1,354,640	Woodbridge	Oct. 5, 1920
1,627,692	Du Pont et al.	May 10, 1927
1,955,927	McBride	Apr. 24, 1934
2,027,114	Olsen et al.	Jan. 7, 1936