

UNITED STATES PATENT OFFICE.

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PROGRESSIVELY-BURNING POWDER AND PROCESS OF MAKING THE SAME.

1,354,442.

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No Drawing.

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To all whom it may concern:

Be it known that we, RICHARD G. WOODBRIDGE, JR., KELLOGG K. V. CASEY, and CLARENCE I. B. HENNING, citizens of the United States, and residents of Wilmington, county of New Castle, and State of Delaware, have invented a certain new and useful Progressively-Burning Powder and Process of Making the Same, of which the following is a specification.

This invention relates to propellant powders which have been provided with a deterrent material to retard their rate of combustion; and it comprises a powder of this type coated with an easily ignitable propellant, and a process of making such powder.

While all smokeless propellant powders are progressive burning, the term "Progressive burning powders" has in recent years been applied to powders of the surface treated type which are extensively used in military rifles. In powder of this type the burning of the powder grain is regulated by treating the surface of the powder grain with a deterrent material such as an explosive, or nonexplosive, deterrent, or a mixture of these, thereby causing the outer layer of the powder to burn more slowly, which results in reduced pressure at the breech of the gun and does not unduly increase the pressure along the bore of the rifle. As might be expected, surface treated powders are more difficult to ignite than non-surface treated powders but this difficulty in ignition has been largely overcome by the use of decidedly stronger primers. Thus, by the use of decidedly stronger primers for the ignition of surface treated powders, ballistic results have been obtained very much in advance over ballistic results obtainable from non-surface treated powders. In view of this fact that surface treated powders have been used quite successfully in this manner, it had not been realized that greater possibilities existed in connection with the use of powder of this type.

In seeking to overcome this difficulty in ignition, it has been proposed by one of us to incorporate in the smokeless powder charge a supplementary priming charge composed preferably of black powder, either by mixing it with the smokeless powder or by placing it in the base of the shell. This

procedure, however, has been found in practice to entail certain disadvantages. Thus, if the black powder is mixed with the smokeless powder, there is a tendency for the black powder to segregate upon handling or shipment, especially when the larger size nitrocellulose grains are used. If the black powder is placed at the base of the shell, two loading operations are required for filling the shell, and there is liability of a variation in the amount of black powder added to different shells.

A line of attack on this problem which appeared promising lay in coating the difficultly ignitable progressive burning powder with an easily ignitable propellant. Previous experiments in this connection had indicated that difficulty would probably be encountered in causing an easily ignitable propellant, such as black powder, to adhere so strongly that dusting of the black powder would not occur during handling of the coated grains, this dusting (that is, formation of black powder dust) being liable to lead to premature explosions. We have now discovered that a progressive burning powder having excellent ballistic properties, and free from any tendency to form dust, may be prepared by coating a deterrent-carrying nitrocellulose powder with a composition containing both black powder, or some other easily ignitable propellant, and a substantially non-volatile binder which is preferably both a solvent for the nitrocellulose and an explosive.

Our invention may be described in greater detail as follows:

The smokeless powder grains to which a coating of black powder is applied according to our invention may be made according to the usual methods of manufacture. For example, sufficient nitrocellulose to give 100 lbs. by weight of nitrocellulose, which may be a mixture of nitrocellulose soluble in ether-alcohol, with nitrocellulose relatively insoluble in ether-alcohol, or may be soluble nitrocellulose alone, is dehydrated with ethyl alcohol of approximately 95% strength by volume by means of a dehydrating press in order to displace the water. The excess alcohol is removed from the nitrocellulose, leaving about 33 $\frac{1}{3}$ parts, by weight, of alcohol in the dehydrated nitro-

cellulose. To this is added approximately 66 $\frac{2}{3}$ parts by weight of ethyl ether in a suitable mixing machine, preferably provided with brine refrigeration. To the nitrocellulose and solvent in the mixer there may be added one-half or more parts of diphenylamin or other stabilizer, together with graphite, powdered metals, nitrocompounds, etc., in varying amounts. The composition is thoroughly mixed and colloided, which operation in the mixer requires about one-half hour or more, depending on the efficiency of the mixer. After thoroughly mixing, the colloided composition is formed into preliminary blocks by means of a press. These blocks are then pressed in the so-called finishing press and the composition pressed through dies into strings, which strings are granulated by means of a cutting machine. The granulated powder is then placed for one or more days in solvent recovery apparatus to recover the alcohol and ether, and then dried in water or air to reduce the solvent remaining in the powder to a low amount.

The powder may, if desired, then be surface coated with a deterrent, such as dimethyldiphenylurea, or an explosive deterrent, such as dinitrotoluene, or a mixture of these or other explosive or non-explosive deterrents, according to the process described in Patent 1,310,848 to O. J. Teeple, Jr., 1,313,459 to A. L. Broadbent and R. G. Woodbridge, Jr., or 1,312,463 to R. G. Woodbridge, Jr.

Smokeless powder grains which have been manufactured as above and whose surfaces have been treated with a deterrent material or a deterrent explosive or a mixture of these or other deterrent materials or deterrent explosives, are then, in accordance with our invention, surface coated with a black powder composition by a process such as that illustrated in the following example:—

100 parts of the above described smokeless powder grains are mixed in a revolving drum with 1 to 10 parts of a black powder composition and about 2 parts of a non-volatile solvent for nitrocellulose, such as dinitrotoluene. The dinitrotoluene may be any one of several isomers, or a mixture of the same obtained by the direct nitration of toluene, but we prefer to use pure dinitrotoluene *i. e.*, 1-2-4 dinitrotoluene. After the materials in the drum have been thoroughly mixed, *i. e.*, in from 5 to 10 minutes, heat is applied in any suitable manner to the outside of the drum or to the jacket of the drum if so provided, until the mixture of powder grains, black powder and dinitrotoluene is heated to a temperature slightly above the melting point of the dinitrotoluene. In the case of the 1-2-4 dinitrotoluene derivative a temperature of 70° to 75° C. is usually sufficient, depending upon

the purity of the dinitrotoluene. At a temperature of about 70° C. the dinitrotoluene melts and being a solvent for nitrocellulose it colloids with the nitrocellulose of the surface of the powder grains and impregnates the surface to a substantial distance while the black powder composition becomes firmly attached to the surface of the powder grains partly as an exterior coating but also at the same time and to a considerable extent is carried into the pores of the nitrocellulose colloid. In this manner the black powder composition is so firmly attached to the smokeless powder grains that it is essentially an integral part of the powder and cannot be removed without destroying the powder grains.

We do not limit ourselves to the use for every 100 parts of powder grains of 1 to 10 parts of black powder composition and 2 parts of dinitrotoluene or other non-volatile solvent for nitrocellulose, as it is clear that we can vary the amount of black powder mixture and percentage of dinitrotoluene contained therein according to the ballistics desired.

In place of dinitrotoluene we may use other non-volatile nitrobenzenoid hydrocarbons such as dinitrobenzene, mono- or tri-nitrotoluene, and mono- or poly-nitroxylenes; or other nitroaromatic hydrocarbons, as for example mono-nitronaphthalene; or aromatic amids such as phenylacetanilide and dimethyldiphenylurea, provided the substance to be used is a solvent to some extent for nitrocellulose. In the case of non-volatile solvents with a relatively high melting point, such as dimethyldiphenylurea, which has a melting point of 120° to 121° C., when pure, we prefer to add to this 25% or more of dinitrotoluene in order to obtain a mixture with a melting point considerably below 100° C.

While we prefer to use a non-volatile solvent for nitrocellulose as a binding agent for the easily ignitable explosive, we may also use other binding agents such as diphenylamin, rosin, etc. which, though not solvents from nitrocellulose, can be melted at a temperature which has no injurious effect on the stability of the powder.

The black powder which we prefer to use is of the composition generally used for black sporting powder and of approximately the following composition: 74.0% saltpeter, 10.4% sulphur, 15.6% charcoal.

However, we do not limit ourselves to any particular composition of black powder as it is evident that we would use that composition which by tests gives the best results and likewise that granulation which by tests is found most suitable for the particular purpose desired. In general we prefer to use black powder meal.

The ballistic characteristics of the smoke-

less powder grains prepared according to our process are illustrated as follows:—

5 Nitrocellulose powder grains were prepared from a mixture of soluble and insoluble nitrocellulose and surface treated with 6 parts of dimethyldiphenylurea per 100 parts of powder. This powder before being treated with black powder composition according to our invention above described 10 gave in the .30/06 Springfield rifle with 180-grain bullet using a weight of charge of 55.0 grains, a mean instrumental velocity at 78 ft. of 2,572 feet per second, with a mean pressure of 37,350 pounds. This powder 15 was difficult of ignition, due to the considerable amount of dimethyldiphenylurea with which the surface of the powder grains had been treated. A portion of the same powder grains was treated with 6 parts of a 20 mixture composed of 5 parts of black powder composition and 1 part of dinitrotoluene, according to our invention as above described. The powder grains so treated were found to be more readily ignited and with a 25 57.5 grains weight of charge gave in the .30/06 Springfield rifle, with 180-grain bullet, a mean instrumental velocity at 78 ft. of 2,458 feet per second, with a mean pressure of only 28,490 lbs.

30 We claim:—

1. The process of coating deterrent-carrying nitrocellulose powder grains with an easily ignitable propellant, which comprises mixing said grains and said propellant in 35 the presence of a nonvolatile binding agent meltable at a temperature that has no injurious effect on the stability of the powder, while heating the mass to a temperature slightly above the melting point of the binding agent. 40

2. The process of coating deterrent-carrying nitrocellulose powder grains with an easily ignitable propellant comprising potassium nitrate, which comprises mixing said 45 grains and said propellant in the presence of a non-volatile binding agent meltable at a temperature that has no injurious effect on the stability of the powder, while heating the mass to a temperature slightly above 50 the melting point of the binding agent.

3. The process of coating deterrent-carrying nitrocellulose powder grains with an easily ignitable propellant comprising potassium nitrate, which comprises mixing said 55 grains and said propellant in the presence of a non-volatile binding agent meltable at a temperature that has no injurious effect on the stability of the powder and capable, when melted, of dissolving nitrocellulose, 60 while heating the mass to a temperature slightly above the melting point of the binding agent.

4. The process of coating deterrent-carrying nitrocellulose powder grains with an 65 easily ignitable propellant, which comprises

mixing said grains and said propellant in the presence of dinitrotoluene, while heating the mass to a temperature slightly above the melting point of dinitrotoluene.

5. The process of coating deterrent-carrying nitrocellulose powder grains with black 70 powder which comprises mixing 100 parts of said grains with from 1 to 10 parts of a black powder composition containing about 20% of dinitrotoluene, while heating the 75 mixture to a temperature slightly above the melting point of the dinitrotoluene.

6. A propellant powder comprising deterrent-carrying nitrocellulose powder grains having a firmly adherent coating of an 80 easily ignitable propellant.

7. A propellant powder comprising deterrent-carrying nitrocellulose powder grains having a firmly adherent coating of an easily ignitable propellant comprising potassium 85 nitrate.

8. A propellant powder comprising deterrent-carrying nitrocellulose powder grains and an easily ignitable propellant held to said grains by a non-volatile binding agent 90 meltable at a temperature that has no injurious effect on the stability of the powder.

9. A propellant powder comprising deterrent-carrying nitrocellulose powder grains and an easily ignitable propellant held to 95 said grains by a non-volatile binding agent meltable at a temperature that has no injurious effect on the stability of the powder, and capable, when melted, of dissolving nitrocellulose. 100

10. A propellant powder comprising deterrent-carrying nitrocellulose powder grains and an easily ignitable propellant held to said grains by a non-volatile nitrocellulose- 105 solvent which is itself an explosive.

11. A propellant powder comprising deterrent-carrying nitrocellulose powder grains and an easily ignitable propellant comprising potassium nitrate held to said grains by 110 a non-volatile binding agent meltable at a temperature that has no injurious effect on the stability of the powder, and capable, when melted, of dissolving nitrocellulose.

12. A propellant powder comprising deterrent-carrying nitrocellulose powder grains 115 having a coating of black powder held to the surface of the grains by an aromatic nitro-hydrocarbon capable of forming a colloidal solution with nitrocellulose.

13. A propellant powder comprising deterrent-carrying nitrocellulose powder grains 120 having a coating of black powder held to the surface of the grains by a nitro-benzenoid hydrocarbon.

14. A propellant powder comprising deterrent-carrying nitrocellulose powder grains 125 having a coating of black powder held to the surface of the grains by a dinitro-benzenoid hydrocarbon.

15. A propellant powder comprising de- 130

- rent-carrying nitrocellulose powder grains having a coating of black powder held to the surface of the grains by dinitrotoluene.
- 5 16. A propellant powder comprising deterrent-carrying nitrocellulose powder grains having a coating of black powder held to the surface of the grains by substantially pure 1, 2, 4-dinitrotoluene.
- 10 17. A propellant powder comprising deterrent-carrying nitrocellulose powder grains having a coating of black powder consisting of 75 parts of potassium nitrate, about 10 parts of sulfur, and about 15 parts of charcoal held to the surface of the grains by an
- 15 aromatic nitro-hydrocarbon capable of forming a colloidal solution with nitrocellulose.
18. A propellant powder comprising 100 parts of nitrocellulose powder grains carrying sufficient deterrent material to render
- 20 them difficult to ignite, and from about 1 to 10 parts of black powder held to the surface of said grains by a non-volatile solvent for nitrocellulose.
19. A propellant powder comprising 100 25 parts of nitrocellulose powder grains carrying sufficient deterrent material to render them difficult to ignite, and from about 1 to 10 parts of black powder held to the surface of said grains by a binding agent comprising 30 an aromatic nitro-hydrocarbon capable of forming a colloidal solution with nitrocellulose.
20. A propellant powder comprising 100 parts of nitrocellulose powder grains carry- 35 ing sufficient deterrent material to render them difficult to ignite, and from about 1 to 10 parts of black powder held to the surface of said grains by dinitrotoluene.
- In testimony whereof we affix our signa- 40
tures.
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