Patented July 24, 1934 1,967,913

UNITED STATES PATENT OFFICE

1,967,913

PROCESS OF MAKING PROPELLANT POWDERS

Gordon C. Tibbitts and Frederick R. Seavey, Alton, Ill., assignors to Western Cartridge Company, East Alton, Ill., a corporation of Delaware

No Drawing. Application November 7, 1930, Serial No. 494,183. Renewed June 24, 1933

20 Claims. (Cl. 52—20)

This invention relates to propellant powders, and more particularly powders of the type which are surface-treated with a deterrent to retard the initial combustion rates.

One of the objects of this invention is to provide a process of surface treating propellant powders, and more particularly double base propellant powders, adapted to improve the ballistic characteristics of such powders.

Another object is to provide a process of the character described, whereby the application of the deterrent to the powder grain can be controlled to secure the desired penetration.

Further objects will appear from the detail description, in which will be described an illustrative embodiment of this invention; it is to be understood, however, that this invention is susceptible of various other embodiments.

In the surface treatment of propellant powders with a deterrent, it has been found advantageous to apply the deterrent to be employed with a vehicle which is a solvent for the deterrent. Such a vehicle may or may not be a solvent for the powder grains. In applying a given surface coating to powder grains in this manner, i. e., by merely adding the solvent solution of the deterrent to the powder, difficulty is experienced in controlling the depth of penetration upon which variable the ballistic properties of the finished powder are dependent. Also the grains are liable to stick together due to the softening of their surfaces by the solvent or because of the adhesive properties of the coating of the deterrent. Even where a highly volatile solvent is used, it is difficut to evaporate this solvent quickly enough to prevent sticking, and to secure the desired degree of penetration.

Generally stated in accordance with this invention, the deterrent is carried in a vehicle and applied to the powder grains by effecting deposit of the deterrent on the grains. There are several procedures which have been found suitable to effect the deposit of the deterrent upon the grains. In accordance with an embodiment of this invention the deterrent is dissolved in the vehicle. The vehicle may or may not be a solvent for the powder grains. In order to effect deposition of the deterrent, there is added an agent, preferably a non-solvent for both the deterrent and the powder grains which is adapted to effect precipitation of the deterrent from the vehicle. Where the vehicle is volatile, it is separated from the surface-treated powder grains by evaporation. For this purpose, however, the heating of the mass is carried out at a temperature and for a period of time sufficient not only to effect evaporation of the vehicle, but also to secure the desired degree of penetration of the deterrent thus controlling the ballistic properties of the finished powder. In the application of the deterrent, carried by the vehicle, to the powder grains, the vehicle is applied in an amount only sufficient to wet the grains and the precipitation of the deterrent and the evaporation of the vehicle is then proceeded with at the desired rate. In general, the process is so carried out that the contact of the deterrent-carrying vehicle, the precipitation of the deterrent and the evaporation of the vehicle are all controlled so as to effect the desired degree of penetration of the deterrent into the grain thus imparting the desired ballistic properties to the finished powder.

In accordance with another embodiment of this invention, the deterrent carried in solution in a vehicle, is dropped slowly onto the powder grains which are in contact with an excess of a non-solvent for both the deterrent and the powder. In this manner, the deterrent is precipitated from its solvent solution immediately upon coming in contact with the non-solvent and the deterrent being in very finely divided form is readily picked up by the powder grains. For a given coating, the rate of addition of the deterrent solution and the temperature and time of treatment may be so controlled as to effect the desired degree of penetration of the deterrent.

In order to generally accomplish the objects of this invention, the deterrent may be one of the usual ones employed for that purpose. A deterrent which has been found particularly useful is diamyl phthalate; a related deterrent, dibutyl phthalate has also been found satisfactory. Moreover, deterrents, such as tricresyl phosphate, ethyl lactate, and mono, di and trinitro toluene may be employed. Any coating material may be applied by means of the principles outlined herein. The materials used for this purpose may or may not be plasticizers for the nitrocellulose of the powder grains. When the deterrent employed is not a plasticizer for nitrocellulose, the temperature and time of treatment is so regulated that the desired degree of spreading rather than penetration is obtained.

It will be understood, of course, that suitable solvents may be employed which are adapted for the particular deterrents chosen and the particular powder under treatment. The word "modifier" is employed in the appended claims

in its usual sense in this art as contemplating the various substances employed for modifying or varying the burning rate or ballistic properties of the powder, of which substances the so-called "deterrents" constitute a sub-class.

Many of the advantageous features of this invention may be secured by the surface treatment of smokeless powder grains generally, when employing the principles embodying this invention. This invention is applicable, therefore, to both powders of the straight nitrocellulose and double base, or nitrocellulose-nitroglycerin types. This invention has been found particularly applicable to the treatment of double-base powders, for example, a surface treated nitrocellulose-nitroglycerin which is of particular utility is one containing 15% nitroglycerin and 6% diamyl phthalate.

As an illustrative embodiment of this invention, a nitrocellulose-nitroglycerin powder grain containing 15% nitroglycerin is surface treated with diamyl phthalate in the following manner: The deterrent is carried in the vehicle such as methyl or ethyl alcohol, which is a mild solvent for the powder grain; however only sufficient of the vehicle is employed to wet the surface of the mass of powder grains being treated. After the solution of the deterrent has been thoroughly agitated with the powder being treated to the extent desired to secure the desired ballistic properties, the whole mass is drowned in water; the alcohol and water being completely miscible, the precipitation of the deterrent is accomplished. The water slurry of the surface treated powder, the alcohol and the water are then heated to a temperature of 75° C. and held there for thirty minutes to accomplish the evaporation of the alcohol from the solution. The grains can then be drained and dried in any suitable manner, and the grains, if desired, can be graphited. It is found that by varying the period of heating, different ballistic results are secured; accordingly, the time of treatment must have reference to the desired ballistic properties.

There is thus produced a powder grain, in this particular instance, a double base powder grain, having the desired ballistic properties. The powder is thus rendered progressive burning to the desired extent by the control of time and temperature which factors govern the degree of penetration when employing a given amount of a particular deterrent. There is also secured an efficient and uniform distribution of the deterrent over the grains and on account of the presence of the non-solvent such as water, sticking together of the grains is obviated. It will be understood, of course, that suitable means is provided to recover the solvent in a manner well known to those skilled in the art.

As a further illustrative embodiment of this invention a powder grain of straight nitrocellulose composition is surface treated with dinitrotoluene in the following manner: The deterrent is carried in solution in the vehicle such as benzol or alcohol and this solution is dropped into an agitated mixture of the grains with an excess of water. When the solvent solution comes in contact with the water, the DNT is precipitated in a very finely divided condition and is applied to the grains in a uniform layer. The degree of penetration of the deterrent is controlled to the desired extent by the time of treatment and the temperature to which the mass is heated. The solvent is evaporated and recovered. The grains may be drained and dried in any suitable manner and then may be graphited, if desired.

It will be understood, of course, that either straight nitrocellulose or nitrocellulose-nitroglycerin grains may be surface coated by either one of the illustrative procedures cited above. It will further be understood that various features and subcombinations are of utility and may be employed without reference to other features and subcombinations; that is, contemplated by and is within the scope of the appended claims. It will be further understood that various changes may be made in detail without departing from the spirit of this invention; it is therefore understood that this invention is not to be limited to the specific details described.

Having thus described the invention what is claimed is:

1. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent dissolved in a vehicle and precipitating the deterrent from the vehicle in the presence of the vehicle.

2. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent carried in a vehicle and adding an agent adapted to effect deposit of the deterrent on the grains.

3. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent dissolved in a vehicle and adding an agent adapted to effect precipitation of the deterrent from the vehicle.

4. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent dissolved in a vehicle and adding an agent which is a non-solvent for the deterrent.

5. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent carried in a vehicle which is a solvent for the powder grains and for the deterrent and adding an agent which is a non-solvent for the powder grains and the deterrent.

6. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent carried in a volatile vehicle, effecting deposit of the deterrent and thereafter evaporating the vehicle.

7. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent dissolved in a volatile vehicle, precipitating the deterrent from the vehicle and thereafter evaporating the vehicle.

8. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent carried in a volatile vehicle, adding an agent adapted to effect deposit of the deterrent and thereafter evaporating the vehicle.

9. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent dissolved in a volatile vehicle, adding an agent adapted to effect precipitation of the deterrent and subsequently evaporating the vehicle.

10. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent carried in a volatile vehicle which is a solvent for the powder grains and for the vehicle, adding an agent which is a non-solvent for the powder grains and for the deterrent and subsequently evaporating the vehicle.

11. In the art of making propellant powders, the process comprising, treating powder grains with a deterrent carried in a volatile vehicle, effecting deposit of the deterrent and subsequent-

ly heating the mass at a temperature and for a period sufficient to evaporate the vehicle and to secure the desired ballistics.

12. In the art of making propellant powders, the process comprising, mixing a deterrent in a vehicle treating powder grains with only sufficient of the mixture to wet the powder grains, and effecting deposit of the deterrent on the grains in the presence of the vehicle.

13. In the art of making propellant powders, the process comprising, suspending a deterrent in a volatile vehicle treating powder grains with only sufficient of the deterrent suspension to wet the powder grains, effecting deposit of the deterrent and thereafter evaporating the vehicle.

14. In the art of making propellant powders, the process comprising, immersing powder grains in a non-solvent bath, and slowly adding to the bath a deterrent carried in a vehicle, the non-solvent and vehicle being so selected as to effect precipitation of the deterrent upon contact.

15. In the art of making propellant powders, the process comprising, immersing powder grains in a non-solvent bath, and slowly adding to the bath a deterrent solution the solvent of which is completely miscible with the non-solvent so as to precipitate the solute upon contact of the solution with the bath.

16. In the art of making propellant powders, the process comprising, immersing powder grains in a non-solvent bath and slowly adding to the bath a modifier solution the solvent of which is so selected as to effect precipitation of the modifier upon contact with the bath.

17. In the art of making propellant powders, the process comprising, agitating powder grains in an excess of non-solvent, and slowly dropping a solution of a modifier into the mixture.

18. In the art of making propellant powders, the process comprising, agitating powder grains in an excess of non-solvent, slowly dropping a solution of a modifier into the mixture to precipitate the modifier from the solution and thereafter evaporating the solvent.

19. In the art of making propellant powders, the process comprising, agitating powder grains in an excess of non-solvent, and precipitating a modifier from a carrying vehicle in the non-solvent and in the presence of the vehicle.

20. In the art of making propellant powders, the process comprising, agitating powder grains in an excess of non-solvent, and continuously precipitating a modifier from a carrying vehicle in the non-solvent during agitation of the mixture, and thereafter removing the vehicle from the non-solvent.

GORDON C. TIBBITTS.
FREDERICK R. SEAVEY.