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PROCESS FOR PREPARING ROCKET FUEL CONTAINING POLYMERIZED OLEFINS AND BORON

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7 Claims. (Cl. 52—13)

The present invention relates to improvements in the surface moderation of double base smokeless powders applicable only to double base propellent compositions containing nitroguanidine as an ingredient and to the resulting improved surface moderated double base smokeless powders of progressively burning composition characteristics. The invention is applicable to double base smokeless propellent powder compositions of forms and sizes suitable for use as propellent powders in small arms, ordnance and other projectors and for use as gas generating charges for such purposes as pneumatic actuation of mechanical devices or propulsion of rockets and generally for any purpose for which it is desired the rate of combustion for the same area of the propellent charge should increase from the surface inwards at least for the outer portion of thickness of the grain. The double base powders whose surface burning characteristics are moderated in an improved fashion according to the invention must contain nitroguanidine distributed through them as an ingredient, and as the presence of this compound tends to reduce the muzzle flash from the propellent powder, as is well known, the present invention is applicable to the production of the so-called flashless double base propellent powders. By the term "double base propellent composition" as used herein, we mean one comprising nitrocellulose colloided with a medium comprising a liquid explosive nitric ester such as nitroglycerine, diethylene glycol dinitrate or the like, with or without non-explosive ingredients such as stabilisers and cooling agents, whether or not forming part of the liquid medium with which the nitrocellulose is colloided, antifouling agents and inorganic salts.

The object of the invention is to produce double base surface moderated propellent charges of progressive burning characteristics of improved stability in their physical characteristics.

In general, surface moderation of propellent powder compositions is carried out by exposing the surface of the propellent powder to a liquid comprising or containing a compound that is a non-volatile solvent for the nitrocellulose and is of substantially lower calorific value, the object being that for at least a certain thickness of the powder grain the non-volatile solvent liquid should be able to exert a cooling effect diminishing gradually from the surface inwards. It is well known that it is much more difficult to obtain a satisfactory surface moderated double base powder than to obtain a satisfactory surface moderated single base powder, especially when the proportion of nitroglycerine or other explosive liquid nitric ester to the nitrocellulose is relatively high. The reason for this difficulty is that the surface moderating liquid is usually a solvent for the liquid nitric ester, and the latter is liable to diffuse into the surface coating and render it ineffective. Solid compounds such as diethyl diphenyl urea are soluble in nitrocellulose solvents so that although they can be used satisfactorily as surface moderants for single base propellent powders they are thus normally unsuitable for use for the surface moderation of double base propellent powders. When the surface moderants chosen are not such that the liquid nitric ester diffuses into them the coating is liable to lack adequate adhesion or to lead to difficulties in the ignition of the powder.

In British specification No. 621,699 it has been proposed to form propellent explosive compositions of suppressed flash characteristics comprising substantially 50 to 65% of nitroguanidine, the particles of which have a specific surface of at least 9000 square centimetres per cubic centimetre dispersed in nitrocellulose which is gelatinised by 0.8 to 1.5 times its weight of a liquid explosive nitric ester and a small proportion of a moderant, which may be for instance diethyl diphenyl urea, the composition having a calorimetric value of substantially from 650 to 825 calories/gram, water gaseous. Symmetrical diethyl diphenyl urea and nitroguanidine form a double compound insoluble in liquid explosive nitric esters, and according to the above mentioned proposal unless the nitroguanidine is first intimately mixed with the nitrocellulose and nitroglycerine the double compound forms aggregates which are not well dispersed in the colloidal matrix so that higher rates of burning result.

According to the present invention the method of effecting the surface moderation of a double base smokeless propellent powder containing nitroguanidine comprises treating the powder surface with symmetrical diethyl diphenyl urea in a quantity sufficient to form in situ by interaction with the nitroguanidine present in the neighbourhood of the surface a zone of diminishing concentration in said neighbourhood and inwards from the surface of said powder of the chemical complex formed by said interaction.

In accordance with the method of the present invention the double compound of symmetrical diethyl diphenyl urea and the nitroguanidine is formed mainly at the surface but also to a decreasing extent for some distance into the interior of the powder grain whereby the adherence of the surface moderating coating is rendered very satisfactory and the insolubility of the aforesaid double compound prevents the inter diffusion of the liquid explosive nitric ester from within the grain and the symmetrical diethyl diphenyl urea from the outer zone. The resulting surface moderated double base smokeless propellent powders have progressive burning composition characteristics and their ballistic properties are substantially unaltered by climatic storage.

The nitroguanidine is preferably used in a very finely divided form of average crystal diameter not exceeding 5μ and average length not exceeding 80μ. If necessary any amount of nitroguanidine up to the maximum imposed by the art of practical powder making may be included in the composition. For the purposes of the invention the symmetrical diethyl diphenyl urea cannot be replaced by any of the chemically nearly allied derivatives of urea that have found utilisation as stabilisers or surface moderating agents for nitrocellulose propellent powders, for instance symmetrical dimethyl diphenyl urea, since these do not form with nitroguanidine double compounds insoluble in liquid explosive nitric esters.

It will be understood that a stabiliser or a moderant may be distributed homogeneously through the composition from which the propellent powder is formed, and it is even possible to include in this way, in addition to the symmetrical diethyl diphenyl urea to be used for the surface moderation, a proportion of symmetrical diethyl diphenyl urea although this will react with some of the nitroguanidine distributed through the composition of the grain. The symmetrical diethyl diphenyl urea used for the surface moderation may be applied in the undiluted state, or as an emulsion or in solution in some volatile solvent.

The invention is not limited to the surface moderation of propellent powders intended to be used in guns or other firearms, although it is very suitable for this and

finds ready application in the surface moderation of those flashless smokeless powders in which flash suppressing properties are associated with the inclusion of a relatively high proportion of nitroguanidine in the composition. Thus the invention may be applied for moderating the initial rate of burning of gas generating smokeless powder charges for use in cartridges for the operation of diesel engine starters, or aeroplane engine starters and the like or the actuation of other kinds of mechanical devices.

The invention is illustrated by the following examples.

Example 1

A mixture of 53 parts of nitrocellulose (nitrogen content 13.1%) 36 parts of nitroglycerine, 10 parts of nitroguanidine and 1 part of symmetrical dimethyl diphenyl urea is incorporated with acetone solvent and is pressed through a 0.040 inch die with a 0.014 inch pin. The extruded tubular cord is cut into 0.05 inch lengths, and stoved to remove residual volatile solvent. It is then surface moderated in a rotating pan by spraying with a solution containing 1 part of symmetrical diethyl diphenyl urea in 10 parts of alcohol, and is finally graphited. A charge weight of 12 grains of this powder when fired in the .22 Hornet rifle gives a velocity (measured over sixty yards) of 2370 feet per second, and a mean maximum pressure of 14.9 tons per square inch.

After storage of this propellent at a temperature of 65° C. for six months, no significant change in ballistics is recorded.

Example 2

A mixture of 30 parts of nitrocellulose (nitrogen content 13.4%), 19 parts of nitroglycerine, 50 parts of nitroguanidine and 1 part of symmetrical dimethyl diphenyl urea is incorporated with acetone solvent and is pressed through a 0.035 inch die with a 0.014 inch pin. The extruded tubular cord is cut into 0.05 inch lengths and stoved to remove residual volatile solvent. It is then surface moderated in a rotating pan by spraying with a solution containing two parts of symmetrical diethyl diphenyl urea in 10 parts of alcohol, and is finally graphited. A charge weight of 42 grains of this powder when fired in the .303" Lee Enfield rifle gives a velocity (measured over 60 yards) of 2360 feet per second, and a mean maximum pressure of 18.5 tons per square inch.

After storage of this propellant at a temperature of 65° C. for six months, no significant change in ballistics is recorded.

A charge weight of 36.5 grains gives the same velocity (2391 ft./sec.) with the powder produced in accordance with this example before it is treated with said alcoholic solution of symmetrical diethyl diphenyl urea. 36.5 grains of the untreated powder generates a mean maximum pressure of 20.2 tons per square inch.

What I claim is:

1. A method of effecting the surface moderation of a pre-formed double base smokeless propellent powder containing chemically uncombined nitroguanidine which comprises treating the pre-formed powder surface with symmetrical diethyl diphenyl urea in a quantity sufficient to form in situ by interaction with the said nitroguanidine a zone of inwardly diminishing concentraton in the neighborhood of the surface only of the said pre-formed powder, of the chemical complex formed by said interaction.

2. A method as claimed in claim 1 wherein the nitroguanidine is in a finely divided form of average crystal diameter not exceeding 5μ and average length not exceeding 80μ.

3. A method as claimed in claim 1 wherein the symmetrical diethyl diphenyl urea used for the surface moderation is applied in the undiluted state.

4. A method as claimed in claim 1 wherein the symmetrical diethyl diphenyl urea used for the surface moderation is applied as an emulsion.

5. A method as claimed in claim 1 wherein the symmetrical diethyl diphenyl urea used for the surface moderation is applied in solution in solvent.

6. A surface moderated double base smokeless propellent powder containing chemically uncombined nitroguanidine characterised in that it has in the neighborhood of its surface only a zone of inwardly diminishing concentration of the chemical complex formed in situ by interaction of said nitroguanidine and symmetrical diethyl diphenyl urea.

7. A method as claimed in claim 1 wherein a stabilizer is distributed homogeneously throughout the composition from which the propellent powder is formed, said stabilizer being symmetrical diethyl diphenyl urea present in quantity insufficient to react with all of the nitroguanidine.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,992,911 July 18, 1961

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It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

In the heading to the printed specification, lines 2 and 3, strike out "PROCESS FOR PREPARING ROCKET FUEL CONTAINING POLYMERIZED OLEFINS AND BORON" and insert instead -- PROPELLENT POWDERS --.

Signed and sealed this 12th day of December 1961.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents

USCOMM-DC

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