Patented Apr. 27, 1948 2,440,327

UNITED STATES PATENT OFFICE

2,440,327

"HOT" DOUBLE-BASE POWDER

Bryce L. Crawford, Jr., Minneapolis, Minn., assignor to Regents of the University of Minnesota, Minneapolis, Minn., a corporation of Minnesota

No Drawing. Application December 29, 1943, Serial No. 516,081

4 Claims. (Cl. 52—13)

1

This invention relates to explosives and more particularly to propellants of the so-called "hot," double-base powder types, wherein difficulty has heretofore been encountered due to ignition by radiation and fissuring during the use of thick powder sections. The unstable burning characteristics of such double-base powders has been found to be induced by the formation of minute holes, described in the trade as "fissures" or "wormholes," in the interior of the powder grain during burning, and such characteristics have a serious deterrent to the use of this type of powders in jet-propulsion motors, projectiles and the like.

When intense radiant energy radiations impinge upon powder, ignition occurs, and this is effected by any fissure or crack in the powder section, or bit of dust, or other irregularity. Where there is an irregularity or bit of dust, the first effect is a local accumulation of radiant energy, which then causes a minute cavity having the appearance of a "fissure" or "wormhole." If the radiant energy is continued, the energy accumulation at the irregularity, "fissure" or "wormhole" continues until ignition takes place. Burning may thus be initiated at many places nearly simultaneously in a single large section of powder. This is most unsatisfactory and frequently very dangerous where the powder is used as the propellant in a jet propulsion device such as a rocket-type projectile.

In order to overcome this difficulty, it has heretofore been proposed to render the powder opaque by inclusion of a small amount of opaque substance such as carbon black or nigrosine or by inclusion of diphenylamine, subsequently darkened by exposure to heat or sunlight. Such powders have satisfactory burning characteristics and appear to be less susceptible to fissuring, possibly because of lower flame temperatures, but they are seriously deficient in that examination of the interior of the otherwise clear powder grain by visual inspection, using transmitted light, is rendered impossible in such opaque powders.

It is an object of the present invention to provide an improved propellant, specifically an improved double-base powder capable of being visually inspected and yet having satisfactory

2

burning characteristics and free from the difficulties of "worming."

It is also an object of the invention to provide an improved explosive capable of such clarity as to allow visual inspection through large sections by utilizing transmitted light and containing a substance capable of substantially diminishing the "worming" and ignition by radiation.

Other and further objects of the invention are those inherent in the following description and claims.

In carrying out the invention there is preferably used any suitable explosive, preferably a double-base powder such as

A

Trench mortar type:	Per cent
Nitrocellulose (13.25% nitration)	51.5
Nitroglycerin	43.0
Ethyl centralite	4.0
Potassium nitrate	1.5

B

"Modified bullseye" type:	Per cent
Nitrocellulose (13.25% nitration)	59.0
Nitroglycerine	40.0
Ethyl centralite	1.0

These explosives exhibit the "worming" effect, ignition by radiation and fissuring effects, and have unsatisfactory characteristics when used in large solid sections for jet propulsion devices such as rocket projectiles. To the selected explosive there is added a small percentage of pigment or dye, or substance capable of being developed into a dye, this addition being preferably made at the time the explosive is being made, and while it is in a freely plastic or fluid stage, such that the pigment or dye may be evenly distributed throughout the powder by milling or working. The admixture of the coloring material may be effected at any time during manufacturing, for example after the nitrocellulose and nitroglycerin of the powder are mixed. Thus, admixture of the coloring matter may be made during the "colloiding" step of the usual powder-making procedure. The pigment or dye is applied either dry or in a suitable solvent or carrier compatible with the powder while mixing.

For the pigment or dye there is used any coloring compound having a high radiant energy ab-

sorption coefficient in the wave length range of .8 to 5.0 μ, capable of passing visible light and which is stable in the powder and does not adversely affect the stability of the powder. The pigment or dye (herein sometimes collectively referred to as "coloring material") may either be a soluble coloring material or of colloidal character such that it is capable of being evenly dispersed or dissolved in the powder. The coloring material must not be of such a type as will preclude passage of sufficient light through the powder section for visual inspection of the section and must be stable in the powder, and must not adversely affect the stability of the powder.

As examples of suitable pigments or dyes there may be mentioned copper chloride, Prussian blue, methylene blue, ferric chloride, Luxol green, nitro-sodium methylaniline, chromium chloride, Cyanine XXXIX (1,1'-diethyl-4,4'-tricarbocyanine iodide), p-nitroisodiphenylamine. All of these materials exhibit some improvement, though certain of the pigments, notably Prussian blue, copper chloride and methylene blue exhibit the most pronounced effect and are preferred. When these materials are added to a powder, the amount of coloring matter added depends upon its inherent effectiveness in absorbing radiant energy radiations, and the amount added to a given basic powder is therefore determined by the type of coloring agent and the characteristics desired. Without limiting the invention reference is made to the following table:

Table A.—Activity of representative coloring agents in HES 4021 powder

Coloring Agent	Percent	Activity (0.104 cm.^{2} per calorie)
$CuCl_2 \cdot 2H_2O$	1.00	0.70
Do	0.40	.61
Prussian blue	0.05	.52
Methylene blue	0.10	.41
$FeCl_3$	1.00	.34
Luxol green	0.10	.28
Nitro-sodium methylaniline	0.10	.25
$CrCl_3 \cdot 6H_2O$	1.00	.24
Cyanine XXXIX (1,1'-diethyl-4,4'-tricarbocyanine iodide) p-Nitroisodiphenylamine	0.05	.23
p-Nitroisodiphenylamine	0.10	.21
Methylene blue	0.01	.21
Plain cast powder section	----------	.15
Plain sheet powder section	----------	.15

The foregoing examples illustrate the range of concentration of coloring matter, so as to yield a powder in which radiant energy between the wave lengths of .8 to 5.0 μ is largely absorbed, but in which sufficient visible light is transmitted to allow visual inspection of slabs up to several inches in thickness for cracks, fissures, bubbles, specks, etc.

When ignition time for a given sample of powder is plotted as a function of the intensity of incident radiation, the plotted points fall on a hyperbolic curve. While a comparison of the activity of different basic powder compositions, or containing for example different coloring materials, can be made from such curves, a more convenient method of comparison is to plot the reciprocal of the ignition time as a function of light intensity. When this is done a straight line results. This is supported by the following approximate analysis:

Powder will ignite when it has accumulated a quantity of energy Q_i sufficient to reach the ignition temperature. The increment of energy dQ accumulated per unit area of incident surface in time dt will be

$$(1) \qquad dQ = k_1 I dt - k_2 dt$$

where I is the intensity of incident radiation, k_1 is proportional to the fraction of light absorbed by the sample and k_2 is the rate of energy loss from a unit area at the powder surface and so will be dependent on t. However, as a first approximation one may consider the average value k_2 between the initial time t_0 and the time of ignition t_i to be a constant for any given powder. Then, when ignition occurs

$$(2) \qquad Q_i = [k_1 I - k_2(\text{average})] t_i$$

and

$$(3) \qquad 1/t_i = [k_1 I/Q_i] - [k_2(\text{average}) Q_i]$$

The quantity Q_i is nearly constant for a given basic powder and varies only slightly for small changes in powder composition, such as the addition of dyes and pigments; k_1 is an effective overall absorption coefficient which will be strongly influenced by any coloring agent placed in the powder.

The slope k_1/Q_i of the straight line obtained from the plotting of the reciprocal time versus radiation intensity, according to (3) may be taken as an indication of the activity of a given powder toward light of a given wave length range. The relative effectiveness of the several powder compositions listed in Table A are thus able to be compared conveniently.

The foregoing analysis is presented as an aid in understanding the invention but is not intended as a limitation thereon.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that I do not limit myself to the specific embodiments herein except as defined by the appended claims.

What I claim is:

1. A thick section of double-base powder of the "hot" type that is normally susceptible to "worming" in the presence of intense radiations developed during burning, said powder being colored with a coloring matter capable of absorbing radiant energy in the wave length range of .8 to 5.0 μ, said coloring matter being of a type and present in an amount such that visible light is capable of being transmitted through the powder, thus permitting visual inspection thereof, and present in an amount such that radiations between the wave lengths of .8 to 5.0 μ are largely absorbed.

2. A thick section of double-base powder of the "hot" type that is normally susceptible to "worming" in the presence of intense radiations developed during burning, said powder being colored with copper chloride, said copper chloride being present in the powder in an amount such that visible light is capable of being transmitted through the powder, thus permitting visual inspection but present in an amount such that radiations in the wave length range of .8 to 5.0 μ are largely absorbed.

3. A thick section of double-base powder of the "hot" type that is normally susceptible to "worming" in the presence of intense radiations developed during burning, said powder being colored with Prussian blue, said Prussian blue being present in the powder in an amount such that visible light is capable of being transmitted through the powder, thus permitting visual inspection but present in an amount such that

radiations in the wave length range of .8 to 5.0 μ are largely absorbed.

4. A thick section of double-base powder of the "hot" type that is normally susceptible to "worming" in the present of intense radiations developed during burning, said powder being colored with methylene blue, said methylene blue being present in the powder in an amount such that visible light is capable of being transmitted through the powder, thus permitting visual inspection but present in an amount such that radiations in the wave length range of .8 to 5.0 μ are largely absorbed.

BRYCE L. CRAWFORD, JR.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
437,499	Mindeleff	Sept. 30, 1890
979,431	Claessen	Dec. 27, 1910
1,202,712	Hawkins	Oct. 24, 1916

FOREIGN PATENTS

Number	Country	Date
12,325	Great Britain	1895
387,612	France	July 18, 1908

OTHER REFERENCES

Marshall-Explosives, vol. I, Phila., 1917, pages 323, 326 and 329. (Copy in Div. 43.)

Certificate of Correction

Patent No. 2,440,327. April 27, 1948.

BRYCE L. CRAWFORD, JR.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows:

Column 3, Table A, first column thereof, in the ninth item under the heading "Coloring Agent", after "iodide)" strike out "p-Nitroisodiphenylamine";

and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 22nd day of March, A. D. 1949.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.

Certificate of Correction

Patent No. 2,440,327. April 27, 1948.

BRYCE L. CRAWFORD, JR.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows:

Column 3, Table A, first column thereof, in the ninth item under the heading "Coloring Agent", after "iodide)" strike out "p-Nitroisodiphenylamine";

and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 22nd day of March, A. D. 1949.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.