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3,420,137 CONTAINED COMPACTED AMMUNITION PRIMER COMPOSITION AND METHOD OF PREPARATION

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No Drawing. Filed Aug. 18, 1967, Ser. No. 661,514
U.S. Cl. 86—1 13 Claims
Int. Cl. F42b 5/00

ABSTRACT OF THE DISCLOSURE

Improved process and compositions for ammunition priming are provided by suspending finely divided priming ingredients in water or other volatile inert liquid to form a stable concentrated fluid suspension, containing about 20% to 65% solids, preferably 45% to 55% solids. The charging of containers with priming is then effected easily and uniformly, by automatic means if desired, by dispensing one or more drops, or other predetermined volume, of such suspension into each container. On subsequent removal of the water or other liquid, the priming composition is obtained, without a pressing step, as a substantially pore-free coherent pellet which is firmly bonded to the container, particularly when the surface thereof is initially permeable. The priming ingredients having a specific gravity over 2.5 are preferably used in the form of particles having the largest dimension less than 15 microns, and those having a specific gravity less than 2.5 are used in the form of particles the largest dimension of which is less than about 60 microns.

Background of the invention

This invention relates to an improved process and compositions for ammunition priming, for example, for the priming of the propellant charge in rim-fire or center-fire cartridges for use in rifles, shotguns or explosive-actuated tools, and especially effective for the priming of caseless ammunition adapted for such uses.

In both the dry and wet primer charging methods utilized heretofore, many sequential steps had to be performed with great care, several of which involved unavoidable difficulties. The initial mixing step had to be carried out with the observance of necessary precautions in order to guard as much as possible against explosions. Generally, the mixing period was prolonged in order to give assurance that the ingredients, which usually differ quite widely in specific gravity and other properties, were distributed with sufficient uniformity throughout the mixture. In the mixing of dry ingredients, gentle and prolonged handling was especially advisable. Generally, the charging operation involved the complete filling of rows of openings of the proper volume in the charge plate with the priming mixture, then placing a tray of properly placed primer cups or shells under the plate, and later dropping into each cup or shell a primer charge liberated by a knock-out pin from the charge plate opening above it. Completion of the operation required in the case of rim-fire shells, the transfer of the charge into the rim by means of spinning punches, and with primer cups, the assembly with other elements such as the anvil and cover as well as the implantation and consolidation of the charge by the application of punches under suitable pressure. Typically, the applied pressure with dry primers was in the range of about 500 to 5000 pounds per square inch of punch area, and with wet primers was in the range of about 50 to 500 pounds per square inch of punch area. Each of these steps required close supervision by trained

personnel who could make the necessary modifications and adjustments as needed.

In attempting to apply the foregoing procedure to the priming of caseless ammunition or consumable rounds, some of the disadvantageous features of the charging or loading operations are considerably magnified. For example, a pressing operation appears indispensable for the use of prior art primer compositions in order to obtain a well-bonded primer pellet displaying adequate sensitivity to assure certainty of firing under the action of the firing pin. However, the application of the necessary high punch pressures increases the chances for the occurrence of accidental explosions, of greatly magnified force, in addition to the possibility of damaging or weakening some of the briquetted propellant charges, generally consisting of propellant granules bonded together by means of an intergranular explosive binder. Furthermore, due to the larger dimensions of the container as compared to conventional metallic primer cups or shells, the use of the known charge plate method for loading the priming charges would involve a considerable decrease in the number of units which could be charged in each operation.

Summary of the invention

In accordance with this invention, generally stated, a process and compositions are provided for ammunition priming wherein the primer ingredients are uniformly mixed and maintained in fluid suspension and the charging operation is effected by dispensing into each unit container a predetermined volume of the said suspension. On removal of the liquid suspending medium, a consolidated effective priming pellet is obtained which is substantially non-porous and which is firmly bonded to the container. Primers characterized by the desired excellent functioning are thereby produced with the elimination of troublesome operations or disadvantages which were unavoidable in accordance with prior art practices.

The concentrated liquid suspension priming or slurry priming process and compositions in accordance with the present invention essentially involves the use of solid ingredients in finely divided form and of a liquid having the desired fluidity and volatility, in which the said ingredients are substantially insoluble and readily dispersed as a stable suspension, particularly in the presence of a binder or binders dissolved in the liquid, which is preferably such as to impart increased viscosity and surface-active effectiveness thereto.

The provision of each solid primer component in finely-divided form enables the ready dispersion thereof in the suspending liquid to yield a suspension or slurry of excellent stability. Generally, a suitable state of subdivision is attained when the largest particle dimension is in the micron range, of the order of one-tenth or less of the particle size customarily used in primer compositions. Preferably, those ingredients having a specific gravity of at least 2.5 are used in the form of particles whose largest dimension is 15 microns or less and averaging 1 to 15 microns, while those having a specific gravity of less than 2.5 may be used in the form of particles whose largest dimension is 60 microns or less and averaging 5 to 50 microns. Thus, the term "finely divided" as used herein refers to particle sizes comparable to those of pigments as generally utilized in paints. In fact, the priming suspensions of this invention resemble many commercial paint compositions with respect to mixing and handling properties.

The solid components of the priming composition, particularly the explosives, are most conveniently handled in liquid-wet condition during the formulation, mixing and dispensing operations.

The preferred liquid suspending medium is water, which for best and consistent results should be distilled or de-

ionized, and which should contain sufficient dissolved natural gum or synthetic water-soluble polymer or high molecular weight compound to provide a suitable level of viscosity and protective colloidal or similar surface or capillary activity and to exert a binder effect on the primer composition when it has been dried. Typical natural gums which are suitable for use, alone or as a mixture of two or more thereof, are gum arabic, karaya gum, partly deacetylated karaya gum, gum tragacanth, guar gum, locust bean gum, and arabinogalactan, as derived from giant western larch. Suitable water-soluble polymeric or high molecular weight compounds, which may be used alone or in combinations of two or more, are methyl cellulose, hydroxyethyl cellulose, carboxymethyl cellulose, dextrin, polyvinyl-pyrrolidone, linear high molecular weight poly(ethylene oxide), polyvinyl alcohol, and urea-formaldehyde or melamine-formaldehyde polymers.

It may likewise be advantageous at times to use an organic liquid or mixture of such liquids as the suspending medium, which is not so volatile at ordinary temperatures as to permit undue losses by vaporization during the handling and charging steps, but is sufficiently volatile so that thorough drying can readily be effected at about 100° to 150° C. Such liquid or mixture, having a suitable desensitizing action on the explosive ingredient or ingredients and compatible with the primer components, may consist of a hydrocarbon, halogenated hydrocarbon, alcohol, ether, or ether having a normal boiling point between about 65° C. and 200° C. Suitable liquid compounds are hexane, cyclohexane, carbon tetrachloride, ethylene dichloride, ethylene dibromide, trichloroethane, tetrachloroethane, pentachloroethane, dichloroethyl ether, dichloromethyl ether, ethanol, methoxyethanol, butyl alcohol, and ethyl acetate. A suitable gum or other binder compound, or mixture thereof, also imparting a desired viscosity, is present in dissolved or colloiddally dispersed form in the liquid medium. Examples of such materials are shellac, rosin, calcium resinate and ester gum.

The primary explosive component of the priming suspension consists of any suitable percussion-sensitive compound, or mixture of such compounds, in finely-divided form. The requisite fine particle size may be obtained through control of the precipitation conditions, as by mixing concentrated reagent solutions at temperatures of about 0° to 50° C., or by mixing a concentrated solution of the explosive with an excess of a non-solvent at about 0° to 25° C., or by mechanically subdividing coarser particles in the presence of a non-solvent liquid. Most, if not all, of the known initiating explosives can readily be obtained in the form of needle-shaped crystals, in which the length is many times greater than the transverse dimensions. Such elongated particles are readily subdivided to the required extent during high speed agitation, as by means of rapidly rotating propeller blades, in preparing the priming suspension. The particle size may likewise be reduced to the requisite extent by ball milling in liquid suspension or other suitable means for subdivision.

It is generally preferable to utilize a primary explosive precipitated in the form of needle crystals, which after the application of high speed mixing during the preparation of the priming suspension are present as very finely divided particles of the desired size. An advantageous example thereof is the use of needle crystals of a double salt of lead styphnate and lead nitroaminotetrazole, "Stabanate," prepared as disclosed in U.S. 3,310,569 issued Mar. 21, 1967. Likewise, other normal or basic metal salts of aromatic nitro compounds, particularly lead salts thereof, and their double salts may be used advantageously. Such salts are exemplified by lead salts of dinitrophenol, dinitrocresol, picric acid, and styphnic acid, and the double salts thereof with another lead salt, such as lead acetate or lead propionate.

In instances requiring a highly brisant primary explosive, lead azide may be utilized with advantage. For use in suspension priming in accordance with this invention,

the material may be precipitated in the desired very fine particle size by reacting cold concentrated solutions of lead nitrate and sodium azide, particularly in the presence of a dissolved high molecular weight substance such as dextrin, carboxymethyl cellulose, or polyvinyl alcohol.

Certain of the primary explosives useful in percussion priming mixtures are readily obtainable in the desired finely divided state by any of the above-described procedures. For example, diazodinitrophenol is obtained as yellow microscopic needle-like crystals by rapidly mixing picramic acid with an excess of nitrous acid at 15° C. (Ind. & Eng. Chem. 25, pp. 663-669, 1933). Coarse crystals or particles can readily be converted to fine particle size by the rapid agitation of an acetone solution with water. Coarse needle crystals can be subdivided to the desired extent by the rapid stirring applied during the formation of the priming suspension. The wet material can be suitably subdivided by ball milling in the presence of water or other liquid, or by other convenient method of comminution. Although diazodinitrophenol displays less favorable thermal stability than most of the metal salt initiating explosives, its sensitivity and igniting power render it excellent for use in priming compositions which are not to be subjected to prolonged storage at elevated temperatures, and it is particularly advantageous in that on explosive decomposition, only gaseous reaction products are formed without the deposition of any solid ignition residue.

For certain percussion primer applications, suitable compositions may be comprised entirely or preponderantly of a primary explosive ingredient of fine particle size, as described above, as the sensitivity, brisance, igniting power, and other ballistic properties can be balanced to provide the requisite functioning. This is readily accomplished by the use of a mixture of two or more primary explosives, and particularly by the use of a double salt as described above.

Frequently, however, the presence in the priming mixture of other ingredients is preferred as facilitating the obtaining of compositions which provide excellent functional characteristics over a range of operating conditions. Such additional ingredients of known types include sensitizers for improving the percussion sensitivity, fuels, and oxidizing agents, for providing the proper power of igniting the propellant charge. Such added ingredients should likewise be in finely divided form for use in suspension priming compositions, and as indicated above are either precipitated as fine particles or subdivided mechanically to proper size.

When a sensitizer is used, it is generally present in the range of about 0.5% to 5% by weight of the solid ingredients. Advantageously, it may consist of tetrazene precipitated as fine particles by the reaction of aminoguanidine with nitrous acid in a cold aqueous solution.

The primer composition may include about 5% to 25% by weight of one or more relatively insensitive organic explosive compound, such as pentaterythritol tetranitrate (PETN), nitroaminoguanidine (NAG), nitrosoguanidine, cyclotrimethylenetrinitramine, dinitrotoluene, trinitrotoluene, nitrocellulose, nitrostarch, erythritol tetranitrate, or mannitol hexanitrate.

The conversion of commercially available coarse crystals of PETN to the desired particle size range is readily accomplished by dissolving 175 grams in about 1 liter of acetone at 30° to 35° C., filtering the solution, and adding it gradually with thorough agitation to 7 to 8 liters of water at about room temperature. After filtration on a Buchner funnel and washing several times with deionized water, the moist fine PETN is stored ready for use in suspension priming.

Finely-divided NAG is similarly prepared from initially coarse material by dissolving in water at 95° to 100° C. to form a nearly saturated solution, then mixing the solution rapidly with an excess of flaked ice, and filtering and washing as above.

A further component may be one or more inorganic oxidizing agents, generally in the range of about 5% to 50% by weight, such as metal oxide or peroxide or oxidizing metal salts. When water is used as the suspension medium, substantially water-insoluble compounds such as lead oxide, lead peroxide, ferric oxide, magnetic iron oxide, barium chromate or lead chromate, must be used, as otherwise the fine particles would undergo crystal growth during handling and storage and thus tend to settle rapidly from suspension. However, water-soluble inorganic oxidizing salts such as metal nitrates, permanganates, chlorates, and perchlorates, and ammonium and substituted ammonium salts of oxidizing acids, may be utilized advantageously with the use of organic liquids as suspension medium.

A fuel component may be added, generally to the extent of about 5% to 25% by weight, which may consist of one or more metals, metal alloy, or other combustible element or compound, such as aluminum, magnesium, zirconium, titanium, zinc, iron, antimony, boron, silicon, ferrosilicon, calcium silicide, carbon, sulfur, red phosphorus, antimony sulfide, lead hypophosphite, or lead thiocyanate. Aluminum powder is particularly advantageous, as the commercially available pigment grade has the desired finely-divided particle size. Any adhering oil or grease can be removed readily by extraction with petroleum ether.

Additional ingredients may likewise be present in the primer composition, in order to provide any desired functional improvement. For example, should greater abrasive action be desired, as for a rim-fire primer, a suitable addition of finely-divided coal displaying conchoidal fracture or of other abrasive material may be made.

The binder component of the suspension priming composition, such as gum arabic and karaya gum for use in aqueous suspensions and ester gum for use in non-aqueous suspensions, also enable the control of viscosity and surface-active properties so as to provide suspensions of desired fluidity and stability. Such colloidal ingredient or mixture thereof is generally used in the range of about 0.1% to 10%, most often in the range of 0.5% to 5%, by weight of the total primer solids.

The preparation of the priming suspension is carried out by combining measured portions of the ingredients and mixing. The combining and mixing may advantageously be effected in the same vessel used to store the completed suspension, the vessel being provided with a variable speed agitator so as to permit initial mixing at low speed and completing the blending at high speed to form a substantially homogeneous suspension.

For safety and convenience in handling, the explosive material of the primer composition is preferably added moist with liquid, which is generally the form in which it has been prepared or stored. The inert material, such as inorganic oxidizing agents or fuels, abrasive, or the gum or other binder, may be added in a dry or moist state as is most convenient. In the case of wet material, the content of solids and of liquid must be known so that the proper weights or volumes of the ingredients can be added to form the desired composition.

The blending of the finely-divided solid components and the liquid suspension medium occurs quickly, particularly at high stirring rates sufficient to cause an appreciable vortex around the mixing propeller shaft. Such rapid stirring is discontinued when a substantially homogeneous suspension has been formed and any needle-shaped particles have been sufficiently subdivided, which can readily be ascertained by microscopic examination of a number of drops of the suspension. The completed suspension flows readily, at the preferred solids' content of about 50% by weight or even up to about 65% by weight, and is quite similar to aqueous latex paints with respect to fluidity and consistency.

After stationary storage, a layer of fairly clear liquid generally appears at the upper surface and some pasty

sediment collects at the bottom of the container. However, the dispersion is easily and quickly restored to substantial homogeneity and made ready for use by brief mixing or stirring, even after storage for months. In use, the homogeneity of the priming suspension is maintained and stratification is completely avoided by gentle stirring, for example by means of a rotating magnetic stirrer.

Thus, the suspension or slurry priming compositions in accordance with this invention are advantageous with respect to the ease, rapidity and safety features of mixing and handling operations.

The charging of primer compositions into containers, such as the usual metal cups or shells as used in standard types of ammunition, or briquettes of propellant as used in caseless ammunition, is readily carried out by operations, readily adapted to mechanization, which are significantly simplified over prior art procedures and provide many significant advantages. The priming suspension has the proper fluidity so that a closely reproducible volume thereof may be dispensed or metered by any of a variety of techniques.

An excellent procedure which is of great versatility is the dip-pin method. In this method, a rod of predetermined diameter is dipped into the priming suspension to a suitable depth and is then withdrawn vertically so that a pendant drop clings to the end of the rod. Then, on contacting the base of the cavity of the container being charged, the drop transfers thereto. With the use of a container whose cavity walls are porous or permeable, the water or other liquid medium of the priming suspension migrates into the walls through capillary action. Because of this phenomenon and through the progressive evaporation of the liquid on subsequent drying, the primer charge shrinks to a well-implanted, substantially non-porous consolidated mass without any additional pressing step. Because of the penetration of the liquid into the container walls and the deposit of the binder ingredient within the crevices thereof during subsequent drying, the primer pellet is firmly bonded in place.

The weight of the primer pellet thus implanted is influenced by a number of factors, the principal ones being the dimensions, profile and surface of the dipping portion of the rod, the depth to which it is immersed, the rate of withdrawal from the suspension, and the characteristics of the suspension, such as the concentration of solids therein and its viscosity. When all such factors are maintained constant, successive charges resulting from the transfer of a single drop are substantially identical. In general, the volume of the drop and thus of the charge weight increases with an increase of the rod diameter or of the depth of immersion.

Test series were run with given dip-pins of differing diameters, all conditions being otherwise essentially constant except with respect to the rod withdrawal rate. The following data on charge weight per drop of suspension priming were obtained for ten successive charges:

Rod diameter (inch)	Depth of immersion (inch)	Charge weight (in grains)		
		Average	Maximum	Minimum
0.080.....	5/8	0.260	0.290	0.240
0.106.....	1/2	0.297	0.345	0.250
0.124.....	3/8	0.346	0.405	0.29

It is apparent that a sufficient uniformity of charge, that is adequate for most uses, is obtainable even without any provision for an accurately reproducible rod withdrawal rate.

The mechanized charging of a number of primers at each cycle is readily effected by mounting a row of dip-pins on a holder, which is cam-actuated so that it is raised and lowered cyclically at controlled rates. A row of primer cups is moved into position under the pins by synchronized mechanism, the pendant drops are transferred simultaneously from each pin to its cup and the

charged cups are withdrawn for subsequent passage through a drying station.

A workable modification of the dip-pin method involves the use of tubes having a central bore instead of the solid rods.

Likewise, the dispensing of a predetermined volume can be effected by the use of a suitable metering pump. For example, a reciprocating piston can be operated to displace a predetermined small volume of suspension at each stroke through a discharge orifice into a primer container. The latter may be fed into proper position on a conveyor operated synchronously with the pump, so as to charge successive containers in turn on continued operation.

While the above and other similar mixing and charging procedures may be applied to any effective primer composition, it is essential that each solid ingredient be present in the form of finely-divided particles, preferably such that the largest dimension does not exceed about 60 microns, and with respect to ingredients having a specific gravity above 2.5, does not exceed about 15 microns. With the observance of this feature, the principles of this invention are broadly applicable and not to be limited to the components and formulations specifically described herein for illustrative purposes.

Further examples of primer compositions which may be used are disclosed in my U.S. Patent 3,310,569 issued Mar. 21, 1967 and U.S. Patent 3,321,343 issued May 23, 1967, including the cited references.

In applications in which the container that is charged with the priming suspension is a metal cup or shell, the interior wall thereof which is contacted by the suspension is preferably provided with a porous surface layer. For example, this may consist of a porous metal oxide layer, which may be formed anodically, as in the case of aluminum or aluminum alloy, or through the formation of a surface conversion coating by chemical treatment, as by treatment with a phosphate-containing solution. Likewise, other porous interior surface layers may be used in order to promote the formation of a well-implanted, strongly bonded, and compact primer pellet.

When the container is a cup, as in primers such as commonly used in centerfire ammunition, the dried primer pellet entirely fills the bottom of the cup cavity and presents a dish surface somewhat depressed at the center. The initial capillary penetration of the liquid suspending medium into the porous surface layer of the container wall provides a suction compacting effect on the suspended solid particles of the composition and the subsequent progressive removal of liquid by evaporation is accompanied by further compacting of the pellet due to shrinkage effects.

The charged primers may be dried by conventional methods, as by circulating warm dry air thereover or with the use of infra-red heating or by means of conventional drying ovens. At times, vacuum drying may be utilized, particularly when it is desirable to accomplish rapid and thorough drying at close to room temperature, for example, at 40° to 80° C.

In charging suspension priming compositions in accordance with this invention into rim-fire shells, the initial dispensing is effected by the dip-pin or other volumetric procedure as described above, thus securing the indicated advantages. The charged shells may then be subjected to a partial drying carried out so that the charges retain about 15% to 30% of water or other liquid suspension medium. Transfer of the charges to the hollow rims may then be effected by the conventional spinning process and after a further drying step, the priming process is completed.

However, the use of the present suspension priming process enables the securing of further advantages, such as the elimination of the need for the use of spinning punches to effect the movement of the primer composition into the rim. For example, this is accomplished by the rapid rotation of the charged shells so as to cause the transfer of the particles of primer composition into the

rim by centrifugal force, followed by drying. The centrifugation may be carried out after exhaustion of the air in the shell by means of a vertically mounted arbor, rotatable at high-speeds and provided with an effective vacuum pick-up device.

Another procedure involves the progressive drying of each charged shell by means of a jet of warm air or other gas directed centrally at the base of the shell with sufficient velocity and under conditions such that the composition fills the rim portion as well-implanted coherent annular primer pellets. In a further variation, a row or rows of charged shells may be advanced through a drying enclosure, which is provided with air convection or infra-red drying means or both, while being tilted at an angle of about 5° to 15° to the vertical, first in one direction and then in the opposite direction so as to effect the implantation of the primer pellet in the rim.

The priming of rim-fire shells without need for any spinning step or for any departure from conventional drying procedure may be accomplished by the use of shells having a centrally indented base, so that the hollow rim of the shell in upright position is below, or mostly below, the top surface of the indentation. When a drop or other metered volume of the priming suspension is dispensed into such shells, and is then subjected to suitable drying conditions, the rim is charged with a well-implanted coherent primer pellet.

The priming process and compositions in accordance with the present invention are particularly advantageous for application to preformed propellant charges adapted for use in caseless ammunition. As stated above, the advantages of increased ease and safety in mixing, handling, and loading are generally available regardless of the specific type of ammunition primer being fabricated. However, further benefits are available in the manufacture of primers adapted for use in consumable rounds of ammunition, that is, in the provision of primers contained in or attached to preformed propellant charges.

The preformed propellant charges for such use are generally prepared by bonding relatively small grains of nitrocellulose (single base), nitrocellulose-nitroglycerine (double base), or plasticized nitrocellulose-nitroglycerine (multiple base) smokeless powder under pressure in a suitable mold, at times while moistened with a volatile solvent, to result in a coherent, but somewhat friable, briquette or agglomerate. Microscopic crevices and cracks persist in the completed preformed charge between the starting grains, and also within such grains when they have a porous or fibrous structure. Frequently, the provision of proper friability in the preformed charge is intentional so that shattering thereof occurs when the primer is actuated and proper ignition and rapid combustion of the charge occurs so as to result in the desired ballistic performance.

When fluid priming suspension is dispensed on a surface of such preformed propellant, as within a cavity therein provided for the purpose, the aqueous or other suspension liquid readily penetrates the capillary passages, with several beneficial results.

On passage of the liquid into the container walls, the solid particles of the suspension are carried along up to any opening of finer dimension, resulting in the formation of tightly packed layers of the fine particles. The action and result are much like those involved in the suction filtration of a fluid suspension of solid particles. On subsequent evaporation during the drying period, shrinkage effects occur which likewise favor the consolidation of the pellet to a substantially non-porous condition.

At the same time, the liquid which migrates into the capillary passages of the wall contains dissolved binder. After the removal of the liquid medium by evaporation, the solid binder residue effects bonding between the fine particles within the pellet, at the boundary between the pellet and the adjacent wall as well, and also into the capillary passages within the wall. Thus, the result is the

ready obtainment of firmly implanted and well consolidated primer pellets.

Such primer pellets may be implanted in accordance with the present process in shallow cylindrical containers, open at the top, consisting of preformed propellant. Subsequently, such primed containers may each be solvent bonded in an appropriate cavity in a larger preformed propellant charge.

Another advantage which is provided is that the process and compositions of this invention are particularly adapted for primers which function without depositing any bothersome solid residue. Conventional priming mixtures, containing particles much larger in dimension than those of the present compositions, tend to deposit adherent solids on the firing pin and on the walls of the firing chamber which interfere with proper continued functioning, at times producing undesired erosive and corrosive effects. Compositions are readily formulated in accordance with this invention which function either with the formation of little or no solid products or with the formation of very fine particles of extremely high melting point, such as very fine metal oxide particles, all but a harmless trace of which are carried off with the gaseous decomposition products of the propellant charge.

As stated above, the fine particle size of the primer ingredients is an essential feature enabling the easy and rapid preparation and maintenance of stable and uniform priming suspensions. In addition, the fine particle size is significantly advantageous in providing functionally superior primer compositions displaying excellent sensitivity to percussive initiation, ignition power such as to promote the desired ballistic efficiency, and general reliability, even in low temperature environments. The improvement in functional characteristics appear to be effected by the finely divided state because of the very greatly increased reactive surface area of the primer ingredients as compared to that characterizing prior art components. Such increased surface area is particularly important with respect to the functioning of the explosive ingredients and to the reactions between fuel and oxidizer components.

While generally, best results are attained when all the particles are finely divided to the extent that the largest dimension does not exceed 60 microns and, with respect to ingredients of specific gravity over 2.5, does not exceed 15 microns, it will be understood that some advantages may be available at times through the use of somewhat larger particle sizes. Thus, the primer ingredients may be effectively used when the average particle size is within the range of about 10 to 30 microns, particularly when the priming suspension is used soon after preparation and without any intermediate extensive storage before charging. Also, in the case of particles in the form of flakes, wherein the thickness is far smaller than the length and breadth, the largest dimension of usable particles may somewhat exceed that specified in the preferred range. For example, finely divided aluminum flakes may be utilized in preferred compositions, wherein the largest dimension may exceed 15 microns by 5 or 10 microns, without difficulty in the process or detriment in the resulting primer pellet.

It will also be apparent that a suitable protective surface layer or cover may be provided over the primer pellet. Conveniently, such layer may be obtained by dispensing a drop or several drops of an effective lacquer of nitrocellulose or shellac or both to the exposed surface of the pellet and drying.

Preferred embodiments

Suspension priming compositions, exemplary of this invention, which are particularly effective in combination with preformed propellant for use in consumable am-

munition rounds, include compositions containing the following solid finely-divided ingredients:

	Percentage by weight	Maximum particle dimension (microns)
Stabanate ¹	5 to balance	1-15
Tetrazene	0-10	1-60
PETN ²	0-15	1-60
Aluminum	0-15	1-15
NAG ³	0-50	1-60
Karaya ⁴ gum	0.1-5	
Gum arabic	0.1-10	

¹ Double salt of lead styphnate and lead nitroaminotetrazole (U.S. Patent 3,310,569).

² Pentaerythritoltetranitrate.

³ Nitroaminoguanidine.

⁴ Preferably partly deacetylated Karaya gum, as disclosed in my co-pending application, Ser. No. 639-289, filed May 18, 1967.

The following compositions constitute preferred specific examples:

	Percent by weight	
	Example 1	Example 2
Stabanate	48.5	73.5
Tetrazene	3.0	
PETN	14.0	15.0
Aluminum	10.0	10.0
NAG	23.0	
Karaya gum	1.0	1.0
Gum arabic	0.5	0.5

The process of preparing a priming suspension in accordance with this invention is exemplified by the following procedure, applied specifically to the composition of Example 1. However, such procedures are generally applicable to the preparation of effective suspension priming compositions.

The primary explosive, Stabanate, was precipitated as finely divided needle crystals in accordance with Example 1 of my U.S. Patent 3,310,569 issued Mar. 21, 1967, washed by decantation, allowed to settle, and separated from the supernatant liquid. After the solids' content of a representative sample had been determined, the proper amount of moist solid was weighed and transferred to a cylindrical container.

The other explosive ingredients were filtered on a Buchner funnel, and the solids' content of a representative sample of each was determined, typical values being 71.7% for tetrazene, 55.5% for PETN, and 53.5% for NAG. Proper weights of the moist PETN and NAG were added to the container and the contents were briefly mixed. Next, the weighed dry aluminum pigment and gum component were added and mixed at low speed, for example, by means of a three-bladed propeller operated for several minutes at about 200 r.p.m. After addition of the moist tetrazene during continued low speed stirring, the mixing speed was increased to about 2000 r.p.m. Stirring was continued for 30 to 60 minutes to result in a uniform fluid suspension wherein needle crystals had been suitably subdivided and with all ingredients homogeneously dispersed.

The solids' content of the resulting slurry or suspension is generally within the range of about 50% to 55%, depending on the solids' content of the moist explosives added. If a less concentrated suspension is desired, a measured amount of liquid is added before the mixing is completed.

At 50% solids' content, 140 ml. of suspension contain 100 grams of composition, and at 55.6%, the corresponding volume is 120 ml.

Suspensions of higher solids' content are readily obtainable by starting with moist explosives of increased solids' content, obtainable for example by suction filtration or by centrifuging. Thus, the primary explosive may be prepared at about 65% solids' content by suction filtration. In that case, the resulting priming suspension contains 63.4% by weight of solids.

As each of several variable features can be accurately controlled independently, for example, the concentration of solids in the suspension and the volume of the suspen-

sion that is dispensed, the values may be selected for a given operation in accordance with the greatest reproducibility and convenience.

Thus, suitable fluid uniform suspensions may readily be prepared at a desired concentration, generally within the range of about 20% to 65% by weight, and preferably of about 45% to 55% by weight. An optimum concentration for a given primer-propellant combination may generally be set so that a predetermined volume of suspension will deposit a compact primer pellet of the selected standard weight. When the charging is effected by the dip-pin method, the volume of drop, and thus the weight of primer deposited, is controlled as stated above by controlling the pin dimensions, its depth of immersion in the priming suspension and by its rate of withdrawal therefrom. Other volumetric charging methods, as described above, are likewise subject to close control so that the primer deposited from suspension yields reproducible results in each particular combination.

Primer compositions, deposited by the dip-pin or other above-described procedures and comprised of the ingredients in accordance with the specific examples or the above-indicated modifications, have been found to exhibit excellent sensitivity to percussive initiation over a wide range of temperatures, from about -40° to 120° F. Also, the ignition of adjacent propellant charges on actuation of these primers is effected over the above wide temperature range reliably and with the attainment of excellent ballistic uniformity.

When the coherent primer pellet has been implanted within a cavity in the preformed propellant charge, being intimately bonded to the cavity walls, as described above, the combination is such as to provide excellent ballistic efficiency. This is believed to illustrate an enhancement of the igniting power of the priming composition which is effected by the fine state of subdivision and uniformity of mixing of the priming ingredients and also by the intimate proximity of the propellant and primer. For example, a 20 grain preformed propellant was found to be reliably and uniformly ignited by means of a 0.3 grain implanted primer pellet, deposited from suspension, while in contrast the same propellant charge required 0.8 grain of ordinary priming to achieve comparable ignition.

Accordingly, it will be understood that the suspension priming process and compositions are widely applicable for the provision of excellent percussive ignition of ammunition propellants with the use of finely divided components of varied nature and proportions, as indicated above. Modifications may therefore be made in the specific illustrative details within the spirit and scope of the appended claims.

What is claimed is:

1. In the manufacture of ammunition primers, the process comprising forming a stable fluid concentrated suspension of solid primer ingredients in finely divided state in a volatilizable liquid,

said liquid being a non-solvent for said solids and containing an organic binder dissolved therein,

said ingredients consisting essentially of a substantial proportion of at least one primary explosive selected from the group consisting of explosive metal salts, diazodinitrophenol and tetrazene, and substantially any balance thereof of at least one of auxiliary primer ingredients selected from the group consisting of combustible fuels, nitrated organic compounds, abrasives, and inorganic oxidizing agents, dispensing a predetermined volume of said suspension into a primer container, and removing said liquid to form

a coherent compact primer pellet within and adhered to said container.

2. A process according to claim 1, wherein the said solid ingredients are predominantly of said primary explosive.

3. A process according to claim 1, wherein the said solid ingredients are predominantly of an explosive lead salt.

4. A process according to claim 1, wherein the said binder comprises a high molecular weight compound or polymer which increases the viscosity of said liquid when dissolved therein.

5. A process according to claim 1, wherein the said liquid is water.

6. A process according to claim 1, wherein the said liquid is an organic liquid having a desensitizing action on explosive ingredients and having a normal boiling point between about 65° and 200° C.

7. A process according to claim 1, wherein the said solids consist of particles having the largest dimension less than about 60 microns, with particles, of specific gravity greater than 2.5, having the largest dimension less than about 15 microns.

8. A process according to claim 1, wherein the said solid ingredients include about 5% to 25% of a nitrated organic compound.

9. A process according to claim 1, wherein the said binder is present in an amount of about 0.1% to 10% by weight of the said solid ingredients.

10. A process according to claim 1, wherein the said primer container has a porous interior surface.

11. A process according to claim 1, wherein said dispensing is effected dropwise by means of a dip-pin.

12. An ammunition primer comprising a container and a compact pellet of priming composition implanted within and adhered to said container, said pellet consisting entirely of finely divided solid particles of ingredients consisting essentially of a substantial proportion of at least one primary explosive selected from the group consisting of explosive metal salts, diazodinitrophenol and tetrazene and substantially any balance thereof of at least one of auxiliary primer ingredients selected from the group consisting of combustible fuels, nitrated organic compounds, abrasives, and inorganic oxidizing agents, said particles having the largest dimension less than about 60 microns, being bonded together by a colloidal organic binder and consolidated without mechanical pressure by having been deposited in said container as a concentrated fluid suspension in a liquid, said liquid having been subsequently removed from the suspension to effect shrinkage of the said pellet.

13. An ammunition primer according to claim 12, wherein the said container has a porous interior surface, said surface being penetrated by capillary portions of the said composition.

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U.S. Cl. X.R.

102—86.5; 149—105, 23, 24, 25, 26, 27, 28; 264—3

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,420,137

January 7, 1969

Edward A. Staba

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 3, line 28, "ether", second occurrence, should read -- ester --.
Column 6, line 62, in the table, second column, line 2 thereof, "1/16" should read -- 9/16 --.

Signed and sealed this 10th day of March 1970.

(SEAL)

Attest:

Edward M. Fletcher, Jr.

Attesting Officer

WILLIAM E. SCHUYLER, JR.

Commissioner of Patents