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DETONATING EXPLOSIVE IN POLYTETRAFLUOROETHYLENE MATRIX AND PREPARATION

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This invention relates to explosive compositions. More particularly, this invention relates to deformable, self-supporting, explosive compositions comprising a cap-sensitive, solid, detonating explosive admixed with a certain binding agent and to a method of fabricating said compositions.

Many applications of explosively generated pressures as, for example, in metal hardening preferably use or even require that the detonating explosive be in the form of a self-supporting unit rather than in loose crystalline or granular form. Although some detonating explosives can be compressed or cast into rigid self-supporting forms, conventional casting, and mechanical pressing and molding operations may be hazardous; the solid aggregates which are obtained are relatively massive, brittle, and lacking in flexibility and, further, if formulated with a binder, usually have too little cohesion and flexibility for satisfactory use, for example, in the form of a sheet. However, deformable, self-supporting, high explosive compositions which are convenient and safe to handle more recently have become available. These are made by combining a crystalline high explosive compound with suitable polymeric binders as described, for example, in U.S. Patents Nos. 2,992,087, 2,999,743, and 3,093,521.

Explosive compositions in the form of deformable self-supporting sheets and cords thus obtained by the procedures of said prior art may include as little as 7.5% but generally include 15% or more by weight of binder to provide practically usable physical properties. As the proportion of binder is increased, the concentration of detonating explosive is decreased, with resultant reductions in the available explosive energy by unit weight or volume of the composition and simultaneous decreases in sensitivity and ability to propagate a detonation. Since a maximum concentration of the explosive ingredient is desired for many applications of said compositions, there has been a need for compositions which contain an even higher proportion of explosive and which have physical properties equivalent to or better than those known in the art.

Problems are encountered particularly when the aforementioned compositions of the prior art must be exposed for appreciable periods of time to high temperatures as, for example, above 300° F. A number of detonating compounds are available for high temperature use, but when such compounds are formulated with conventional binders, the resulting products degrade or soften and lose their strength at elevated temperature. A goal in the art, therefore, has been to find explosive compositions which combine resistance to oxidative degradation, thermal stability, high strength and fabricability, and good explosive properties, particularly after prolonged exposure to elevated temperatures, e.g., above 300° F.

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This invention provides deformable, self-supporting explosive compositions which contain one or more high explosives dispersed in a polymer matrix and which retain to the necessary degree the properties of flexibility, physical strength, sensitivity to initiation, and ability to propagate a detonation even after exposure to elevated temperature.

The compositions of this invention comprise about from 50 to 95% by weight of finely-divided, cap-sensitive, solid detonating explosive in a matrix of tetrafluoroethylene resin, i.e., polytetrafluoroethylene. The compositions of this invention are prepared by:

(a) Forming a slurry of powdered tetrafluoroethylene resin, finely-divided, cap-sensitive, solid detonating explosive, wetting liquid and any optional additives, e.g., diluents, pigments, flexibilizers, etc.,

(b) Separating excess fluid from the solids in the slurry,

(c) Subjecting the resulting damp mass to compressive shear stress in more than one direction and shaping said stressed mass into sheet-like form, and

(d) Removing residual solvent retained within the shaped explosive composition.

Optionally, the dried, shaped composition can be sintered at temperatures of about from 621 to 650° F. for about 0.5 to 10 minutes.

The term "matrix" as used herein denotes that material which gives form to the shaped mass and retains the high explosive particles included or embedded therein. The preferred material for forming the tetrafluoroethylene matrix in the compositions of the present invention is fine powder polytetrafluoroethylene resin such as is commercially available under the name of "Teflon" [1]-6 TFE fluorocarbon resin. This preferred resin is a homopolymer of tetrafluoroethylene in the form of spheroidal porous aggregates, 5 to 700 microns in diameter, of generally spheroidal colloidal resin particles 0.05 to 0.5 micron in diameter.

As indicated above, fine powder tetrafluoroethylene resin is preferred for practice of the process of this invention, especially in combination with nonaqueous wetting liquids. However, tetrafluoroethylene resin aqueous dispersions can be used in the process of the invention, as exemplified hereinbelow, provided that none of the components to be incorporated into the tetrafluoroethylene resin matrix are soluble in or are adversely affected by water. Generally, however, the resin matrix is formed less readily when aqueous liquids are used as the wetting and mixing medium.

The solid high explosive is in finely divided form, substantially all passing through No. 00 (0.0296-in. opening) silk bolting cloth. Preferably, however, in order to achieve greater sensitivity to initiation in the finished sheet explosive composition, the cap-sensitive crystalline high explosive is in the superfine crystalline condition, i.e., the particles have a maximum dimension in the range of 0.1 to 100 microns and average less than 50 microns in the maximum dimension.

Examples of cap-sensitive, solid detonating explosives which can be used in the compositions of this invention are solid organic nitro compounds such as trinitrotoluene, picryl sulfone, and tetranitrodibenzotetraazapentalenes in-

[1] Registered trademark.

cluding both tetranitro-2,3;5,6-dibenzo - 1,3a,4,6a - tetraazapentalene and tetranitro - 2,3;4.5 - dibenzo-1,3a,6,6a-tetraazapentalene (see U.S. Patent 3,140,212); solid organic nitrates such as pentaerythritol tetranitrate (PETN) and nitromannite; organic nitramines such as tetryl, cyclotrimethylenetrinitramine (RDX), cyclotetramethylenetetranitramine (HMX); solid inorganic explosives such as lead azide; and mixtures of one or more of the foregoing explosives. Tetranitrodibenzotetraazapentalenes, especially tetranitro - 2,3;5,6 - dibenzo-1,3a,4,6a-tetraazapentalene, are particularly preferred because of the optimum thermal stability and constituent compatibility of compositions formed therefrom.

Other particulate solid ingredients can be used in the novel compositions of this invention. For example, pigments such as carbon can be incorporated to color the composition for easy identification. Also, finely-divided metallic fuels such as aluminum can be included as well as finely-divided oxidizing agents such as potassium perchlorate and barium peroxide.

As previously indicated, the first step in the process of this invention comprises forming a slurry of particles or aggregates of tetrafluoroethylene resin and explosive in a liquid which is capable of wetting the solid particles or aggregates and which does not dissolve or react with the solid ingredients to any significant extent. The wetting liquid performs two functions: (1) in the mixing step, it provides a fluid medium which permits the formation of a homogeneous mixture of the solid particles in an easy and safe manner, and (2) in the sheet-forming step, it acts as a pressure transmitting medium to effect distortion (i.e., elongation) and slippage of the resin particles. The wetting liquids are inert organic liquids, preferably hydrocarbons, which form a contact angle of less than 90° and preferably less than 45° with a smooth tetrafluoroethylene resin surface. Preferably, the liquids have a viscosity in the range of about 0.4 to 1000 centiposes at room temperature, although liquids of higher or lower viscosities can be used. Relatively volatile hydrocarbon liquids such as kerosene, naphtha, or Stoddard solvent are generally preferred wetting liquids for use in the present process. However, less volatile liquids such as white mineral oil can be used and later extracted with more volatile liquids. As shown in the examples, water-based liquids can be used as the fluid medium for the process of this invention, but they generally produce less satisfactory products than are obtained with nonaqueous liquids.

The amount of wetting liquid used to prepare the slurry is chosen primarily on the basis of the amount required to achieve ease and safety of mixing. With more sensitive explosives, a larger amount of liquid will be used to provide adequate dilution and good desensitization. The specific liquid-to-solids weight ratio used in the slurrying step to permit ease of mixing also will depend on the size and porosity of the solid particles being incorporated in the liquid and on the wetting power of the liquid toward the particles. In general, a practical working weight ratio of liquid-to-solids (i.e., explosive, pigments, etc., and tetrafluoroethylene resin) is about from 1:1 to 20:1. The maximum amount of solvent used is determined solely on the basis of economic factors, size of equipment available, and handling consideration, since increasingly large excesses of liquid have no deleterious effect. The wetting liquid is not a component of the finished explosive composition, but is essential for carrying out the process of the invention, as disclosed herein.

To achieve uniform distribution of the solids throughout the liquid, the slurry is mixed vigorously, preferably by subjecting it to the cutting action of a high-speed, bladed, rotary stirrer. The temperature at which the mixing is effected is determined chiefly on the basis of the thermal stability of the ingredients and the volatility of the wetting liquid. Room temperature is suitable, although generally any temperature from about 0 to about 100° C. will be adequate. Mixing time is dependent on the speed of mixing used, the temperature, and the degree of wettability of the solids by the liquid. In general, about 0.25 to about 15 minutes is a sufficient time to achieve adequate dispersion.

After the solids have been uniformly dispersed in the wetting liquid, the dispersion is subjected to a liquid-removing procedure, e.g., filtration, centrifugation or the like, whereby a solid mass is obtained which contains residual liquid in an amount such that, when the mass is confined, pressure is transmitted hydrostatically to the solids therein to effect deformation and slippage of the resin aggregates and thereby to coalesce the mass into a sheet-like structure. The liquid-containing solid mass thus is said to be in a hydrostatically coalescible condition and is maintained in such condition through the subsequent compressive deforming steps. The liquid-to-solids volume ratio in the mass is a critical factor in attaining this coalescible condition. If the liquid-to-solids volume ratio is inordinately large, the desired deformation and orientation of aggregates may not be achieved. On the other hand, if too little liquid is used, a fragile, crumbly structure may result. The liquid-to-solids volume ratio required to maintain a hydrostatic condition in the mass depends on the size, shape, density, porosity, and surface characteristics of the solid particles therein and, therefore, differs for different formulations. For a solid mass comprising tetrafluoroethylene resin fine powder in a wetting liquid, e.g., conventional fine powder, wherein the porous resin aggregates average about 300 microns in diameter, usually about 0.5 to 2 volumes of liquid per displacement volume of resin are required prior to compression to achieve the hydrostatic coalescible condition. The presence of the high explosive, and possibly other optional ingredients such as pigments, inert diluents and the like, in the wet mass may increase the amount of liquid needed if the solids are porous, of relatively large size and do not wet easily, etc. Assuming, however, that the ingredients do have a small particle size, are only moderately porous, and are readily wet by the liquid, the foregoing liquid-to-total solids volume ratio will be suitable in the mass prior to compression and working.

The hydrostatically coalescible solid mass obtained after separation of the free liquid from the dispersion is formed into a structure by the application of compressive shear stresses, that is, stress having components both normal and parallel to the surface of the composition. Preferably, the compositions are shaped into sheets by the sequential application of compressive shear stresses in which the shear stresses are alternately directed along lengthwise and crosswise axes to induce biaxial orientation. Simultaneously liquid is expressed from the mass but sufficient liquid is retained in the mass to keep the mass hydrostatically coalescible during the compression steps. The amount of liquid required to maintain the hydrostatically coalescible condition decreases as the mass is compressed, or as the aggregates come closer together. Therefore, the controlled expression of liquid from the mass by compression is achieved without destroying its coalescibility. In general, the liquid-to-solids volume ratio will not go below about 0.2 during the compressing steps.

The application of compressive shear stresses to the solid mass is preferably effected by rolling out the mass sequentially first in one direction and then in a direction which is approximately 90° to the direction of the first rolling and in the same horizontal plane. The alternation of rolling directions is continued until the desired sheet strength is achieved. Because the rolling causes an elongation of the mass in the direction of rolling and a reduction in thickness of the mass, it is preferably after the first rolling to fold the elongated, thinned mass back on itself in the direction of the longest dimension to obtain two approximately congruent layers, and to roll the two layered mass in a direction parallel to the fold until the layers are themselves coalesced. The steps of folding back

and rolling the folded layered mass in a direction perpendicular to the last previous fold can be repeated until the desired uniformity and knitting of layers are achieved. The strength which is required in the sheet is a prime factor in determining the conditions employed in the rolling operation, i.e., the number of rolling steps, the rate at which pressure is applied to the mass and at which liquid is expressed from the mass, the temperature, and the particular liquid-to-solids volume ratio maintained. In preparing the compositions of this invention, it is not always necessary that a sheet of maximum strength be formed since additional strength eventually may be imparted to the sheet by compressing or sintering the biaxially stressed mass. Therefore, in this process the minimum strength of the sheet will be that which will permit it to hold together during the subsequent operations or in use. To achieve this minimum strength it generally is necessary to perform the folding and rolling operations at least about four times. A greater number of rolling and folding operations can be used, but there generally is no advantage to having more than about 25 such steps.

Generally, it is best to introduce the deformation of the resin particles and aggregates gradually rather than abruptly and severely. Therefore, the pressure is applied to the mass incrementally in the several successive rolling steps, thereby reducing the thickness of the sheets in small increments and expressing the liquid from the mass at a low rate. If the mass is subjected to abrupt and severe application of pressure, flaws and fissures in the resulting sheet may be observed. These can be removed, if produced in the early stages of the process, by continued rolling, possibly with the addition of liquid to the mass. However, in the preferred procedure the thickness of the sheet is reduced gradually, e.g., by about 20 to 50% in each rolling step. Such a procedure results in gradual expression of liquid from the sheet.

The rolling operation conveniently is carried out at room temperature or slightly elevated temperatures. Temperatures below 19° C. are avoided because of the diminished coalescibility of the mass at such temperature. Since higher rolling temperatures result in higher sheet strength, and therefore fewer rolling operations are required to achieve a desired strength, it may be advantageous to operate at elevated temperatures provided such temperatures are below the decomposition temperature of the ingredients of the mass being rolled. A preferred temperature range is about from 25 to 70° C., but the temperature can be higher depending on the thermal characteristics of the components of the mixture.

After the sheet has been formed by the desired number of biaxially oriented rollings, the residual liquid is removed. The manner in which this liquid removal is accomplished depends on the properties of the wetting liquid. If the latter is relatively volatile, the sheet can be kept at a somewhat elevated temperature for the time required for all of the liquid to volatilize. If the wetting liquid is not sufficiently volatile for ready removal by evaporation, the liquid, such as white oil, used in formulation can be displaced by a miscible and more volatile liquid which subsequently can be removed by volatilization during a drying period.

Sheet from which the liquid has been removed is a porous matrix of intertwined fiber lamella surrounding the particles of high explosive. If desired, the porosity of the sheet can be reduced to a chosen level by densifying the sheet, e.g., by compacting it compressively. The total pressure applied to the sheet in this operation is dependent on the amount of densification desired. The ultimate thickness of the sheets can vary as desired, but generally, however, sheets thinner than 5 mils are not strong enough to withstand the subsequent operations. A preferred range of sheet thickness is about from 10 to 500 mils; thicknesses above 500 mils are not preferred because of possible density gradients. The flexible sheet can be used in this form or it can be modified further by sintering along, or by compacting followed by sintering at a temperature of 320° C. to about 380° C., preferably about 338° C., for ½ to 5 minutes.

Examination with the electron microscope of the sheeted composition hereinbefore described shows that a substantial degree of biaxial molecular orientation exists in the sheeting and is retained upon drying and heating the sheet even above the sintering point of polytetrafluoroethylene, i.e., above about 320 to 327° C. The mechanical and other properties of the sheet are quite remarkable. The unsintered structures are surprisingly strong and can be inelastically extended 50% or more, then dried and sintered with little tendency to return to the former dimensions. A piece of sheet of any length can be folded over on itself without cracking. The unsintered sheets can be laminated and the laminate healed by sintering and pressing to a coherent structure. After sintering, tensile strength exceeding 1,000 p.s.i. can be obtained, a value in excess of that usually achieved in flexible self-supporting explosive compositions.

A further remarkable feature of this process is that extremely large amounts of solid ingredients can be incorporated in the sheet explosive composition formed as described herein. Concentration of solid explosives, or explosives with other solid filler materials, up to about 95% by volume can be incorporated in the manner disclosed herein without inhibiting the formation of a sheet.

In order to operate the process of the invention, at least about 5% by volume of total solids should consist of tetrafluoroethylene resin. Preferably, the resin amounts to about 5 to 25% by weight of the total solids. Generally speaking, about 95% by volume of the total solids in the pressure coalescible compositions can consist of solid explosive or filler. Preferably, however, the volume of explosive and filler solids should not exceed 90% by volume of the total solids in the composition. Examples of fillers and additives which also can be incorporated in the porous structure, usually in amounts of 50% by weight or less based on the total composition, along with the explosive are metal powders, pigments, flexibilizers, and the like, which impart or enhance color, thermal conductivity, lubricity, flexibility and other properties as required.

The products of this invention have excellent strength, resistance to oxidative degradation, and thermal stability coupled with good explosive properties. In addition, the compositions of this invention can be worked into sheet that is cohesive even at a thickness as low as 10 mils. Furthermore, good composition cohesiveness is maintained even at low resin volume, e.g., as low as about 5%. As illustrated above, the compositions of this invention can be formed into sheets and used as such in known applications for sheet explosives, particularly those wherein the thermal stability and resistance to oxidative attack of conventional sheet explosives is deficient. Alternately, compositions of this invention can be fabricated into spheres, slabs, blocks, rods, tubes, cords, and other shapes, for example, by laminating or rolling sheets.

In the following examples, which illustrate this invention, parts and percentages are by weight unless otherwise indicated.

Example 1

A charge consisting of 22 parts by weight of finely divided tetranitro-2,3;5,6-dibenzo-1,3a,4,6a - tetraazapentalene, prepared as disclosed in British Patent No. 930,304, in the form of crystals 80% of which are less than 10 microns in diameter (herein designated as "Tacot" [2] high explosive), 3 parts of a commercial tetrafluoroethylene resin fine powder, comprising discrete spheroidal aggregates averaging about 300 microns in

[2] Registered trademark.

diameter, and about 187 parts of Stoddard solvent as the wetting liquid are mixed about for 15 to 90 seconds by the intensive mixing action of a high-speed, high-shearing blade rotating at about 4000 r.p.m. (Waring Blendor or Cowles dissolver). The suspension is filtered to remove the free liquid and the wet filter cake containing about from 20 to 35% liquid by weight is consolidated on a flat surface by four successive passes of a hand-operated roller applied alternately in directions at right angles to each other. This preliminary rolling imparts enough strength to permit handling the sheet for processing on power-driven rolls. The damp, sheeted composition is next passed through the power rolls, then is folded over one-half on the other half, and passed through the rolls in a direction parallel to the fold. This combined folding and rolling operation is carried out two more times with gradual reduction of sheet thickness by increments of 10 to 20% per pass to a final thickness of about 0.040 in. The sheet then is allowed to remain at room temperature until the residual wetting liquid has evaporated, after which the dried sheet is rerolled without folding to increase its density and tensile strength. The final density is about 1.05 g./cc. The sheeted composition now has a tensile strength of about 415 p.s.i. at ambient room temperature. The sheet, although consisting of about 88% "Tacot" high explosive is surprisingly insensitive to mechanical impact; no detonation of the explosive sheet is observed in the standard drop test (5-kg. weight) at 56 in. of fall. The "Tacot" high explosive itself in the same 5-kg. drop test likewise shows no detonations in the drop test. A 0.120-in.-thick laminate of three pieces of 0.040-in.-thick sheet is primed by a standard No. 6 electric blasting cap and detonates completely without confinement. The sheeted composition has remarkable stability to high temperatures. After being held at 500° F. for 12 hours, there is no perceptible change either in physical properties of the sheet explosive or in its ability to detonate upon being primed by a blasting cap. The outstanding stability in both physical and explosive properties upon exposure to elevated temperatures permits application of this material for severance of space-flight units under conditions where explosive means of separation heretofore could not be employed.

The composition, density, and velocity of detonation of the sheet explosive are controllable variables, as are the processing conditions so that, within limits, a reproducible range of properties is achievable in the finished sheet explosive. Thus, for example, a composition formulated as in Example 1 when rolled to a thickness of 250 mils at a density of 1.23 g./cc. detonates at a velocity of 5258 m./sec. when initiated by an electric blasting cap. After being further compressed by rolling, the density increases to 1.52 g./cc. and the detonation velocity is 6350 m./sec. A dry sheet explosive composition formulated as in Example 1, at a density of 1.3 g./cc. in a thickness of 160 mils detonates at 5860 m./sec. and has a tensile strength of about 600 p.s.i. After sintering at 645° F. for 10 minutes, the sheet is less flexible, has a tensile strength of about 1050 p.s.i., and detonates at a velocity of 6350 m./sec. Lower velocities of detonation are observed if thinner sheets are used. In order to insure detonation and propagation in "Tacot" high explosive/"Teflon" fluorocarbon resin sheets or laminates, at least 80 mils thick are used. As illustrated in succeeding examples, thinner sheets containing other high explosive ingredients will detonate completely.

Example 2

In a manner similar to that of Example 1, a charge consisting of 45 parts of commercial dextrinated lead azide, 5 parts of tetrafluoroethylene resin powder, and 200 parts of Stoddard solvent (wetting liquid) is mixed for about 60 seconds at 4000 r.p.m., and excess solvent is removed. The cake of wet material is placed on a stainless steel surface between two spacers each 1/16 in. in thickness. Using a wooden roller, the cake is consolidated until the thickness is reduced to 1/16 in., i.e., until the ends of the roller are supported by the 1/16-in.-thick spacers. The mass now can be transferred to power-driven rolls without disintegration during handling. The flattened cake, damp with wetting liquid, is now passed between power-driven 4-in.-diameter aluminum rollers set at a spacing of 0.025 in. between the rolls. The sheet is then folded back on itself and passed through the rolls in a direction parallel to the fold, this operation being repeated four times to develop additional strength and achieve the desired degree of consolidation of the sheet. After drying for about 16 hours at 160° F. to remove residual liquid, the sheet explosive composition still has a thickness of about 0.025 in., does not detonate in the 5-kg. drop test at 25 in., is primed in single thickness of sheet by a standard No. 6 strength electric blasting cap, and detonates completely at a velocity of 2580 m./sec.

Example 3

This example illustrates the process of the invention wherein the composition includes a flexibilizing agent and a pigment-fuel, in addition to high explosive and the tetrafluoroethylene resin binder.

A charge consisting of 15 parts of fine particle tetrafluoroethylene resin, 15 parts of fine aluminum powder (Aluminum Company of America, Grade No. 140), 15 parts of linear addition polymer of trifluorovinyl chloride (having a viscosity of 31 ± 7 centipoises at 100° F. and $d=1.895\pm0.015$; "Fluorolube," Grade MO–10, Hooker Chemical Company), 55 parts of superfine PETN, and 400 parts of Stoddard solvent is processed as described in Example 2 above, except that the series of biaxially oriented rolling operations is terminated when the sheet thickness is reduced to approximately 1/8 in. The sheeted composition has a density of about 1.5, and when primed with an electric blasting cap detonates at 6930 m./sec. A 6-in.-long strip 1 in. in width at −65° C. can be folded back on itself until the ends meet without fracturing. Analogous material made without the flexibilizing "Fluorolube" additive is quite brittle at −65° C.

Examples 4 to 7

These examples illustrate the preparation of a series of tetrafluoroethylene resin-bonded sheet explosive compositions, each containing a different high explosive, and all employing substantially the same preparative procedure wherein the ingredients shown in the table below, together with 350 ml. of Stoddard solvent, are mixed for about 90 seconds at high speed on a Waring Blendor and filtered with suction until the filter cake contains about 25 to 30% by weight of liquid. Next, the cake is compacted by hand rolling four times in alternate directions, then the resulting compacted mass is passed at room temperature through mechanical rolls set at a spacing of about 0.4 in. and rotated at a peripheral linear speed of about 2 ft./in., with successive 90° alternation of direction of rolling, until solvent content is reduced to about 10% by weight. The rough sheet is dried overnight at a temperature of about 125–130° F. and the dried rough sheet is passed through the same set of mechanical rolls (without rotation or folding of sheet) until the thickness of the sheet is reduced to the value shown in Table 1. After final rolling, each composition is sufficiently flexible that a strip of any length can be bent back on itself until the ends meet without producing cracks or fractures in the sheeted material. Even after being held for two hours at −65° F., sheets about 50 mils thick can be bent rapidly over a 0.5-in.-diameter dowel without cracking or fracturing. Other properties of the resulting tetrafluoroethylene resin-bonded sheet explosive compositions also are shown in Table 1.

TABLE 1

	Example 4		Example 5		Example 6		Example 7	
Ingredients, percent:								
"Teflon" 1 6 [a]	5		5		10		10	
HMX [b]	95							
RDX [c]			95					
PETN [d]					90			
Dipicryl sulfone [e]							90	
Sheet thickness, inch	0.105	0.100 0.050	0.100	0.050	0.100	0.102	0.100	0.150
Sheet densito, g./cc.	1.23	1.27 1.66	1.28	1.35	1.08	1.30	1.34	1.39
Sheet velocity of detonation, m./sec	5,900	6,250 6,350	6,680	6,680	6,150	6,180		6,150
Impact Sensitivity inch: [f]								
Sheet	15		>45		>16		>28	
High explo. only	13		15		17		22	
	Sheet	Explos. only	Sheet	Explos. only	Sheet	Explos. only	Sheet	Explos. only
Thermal stability: [g]								
Temperature, ° F	400	400	350	350	275	275	425	425
Time for ignition, hours	21	15	>21	>21	5	11	9	11

[a] A commercially available grade of tetrafluoroethylene resin fine powder.
[b] Cyclotetramethylenetetranitramine, average particle size about 14 microns.
[c] Cyclotrimethylenetrinitramine, average particle size about 8 microns.
[d] Pentaerythritol tetranitrate, average particle size about 7 microns.
[e] See U.S. Patent 2,952,708; predominately through 48 and on 100 mesh Tyler screen.
[f] Height of a drop of 5 kg. weight.
[g] High explosive (b, c, d, e) from the same lot used in making the sheet explosive composition.
[1] Registered trademark.

Example 8

This example illustrates the use of an aqueous dispersion of tetrafluoroethylene resin instead of dry fine powder tetrafluoroethylene resin in the process of the invention for the preparation of 90/10 "Tacot"/tetrafluoroethylene resin sheet explosive.

A mixture of 45 parts of finely divided "Tacot" high explosive and 160 parts of water is vigorously agitated while a mixture of 50 parts of water and 15 parts of a tetrafluoroethylene resin aqueous dispersion containing 33.5% by weight of resin solids (commercially available as "Teflon" 41BX) is added during about 20 to 30 seconds, and mixing is continued for about 1 minute at which time the curdy solid is separated by filtration. The moist cake is rolled by hand, repeatedly, during about 15 minutes, rotating the mass through about 90° between each successive rolling. The dry sheeted composition has a thickness of about 0.200 inch, a density of about 0.98 g./cc. and, when primed with an electric blasting cap, detonates with a velocity of 3760 m./sec. In general, the strength and flexibility of such a sheet are lower than for sheet of a similar composition prepared as in Example 1 above. Upon being consolidated under a pressure of 40,000 p.s.i., the sheet has a thickness of only about 40 to 50 mils, a density of 1.34 g., and at this thickness and density fails to propagate a detonation.

I claim:

1. An explosive composition comprising about from 50 to 95% by weight of finely-divided, cap-sensitive, solid detonating explosive in a matrix consisting essentially of tetrafluoroethylene resin.

2. A sheet explosive of the composition of claim **1**.

3. A product of claim **2** wherein the detonating explosive is tetranitro-2,3;5,6-dibenzo-1,3*a*,4,6*a*-tetraazapentalene.

4. A product of claim **2** wherein the detonating explosive is lead azide.

5. A product of claim **2** wherein the detonating explosive is pentaerythritol tetranitrate.

6. A product of claim **2** wherein the detonating explosive is cyclotrimethylenetrinitramine.

7. A product of claim **2** wherein the detonating explosive is cyclotetramethylenetetranitramine.

8. A product of claim **2** wherein the detonating explosive is dipicryl sulfone.

9. A composition of claim **1** comprising aluminum and about from 50 to 95% by weight, based on the total composition, of finely-divided, cap-senstive, solid detonating explosive in a matrix of about from 5 to 25% of tetrafluoroethylene resin, based on the total composition.

10. A process which comprises:

(a) forming a slurry comprising wetting liquid and, based on the total weight of solids, at least about 5% of powdered tetrafluoroethylene resin and 50 to 95% of finely-divided, cap-sensitive, solid detonating explosive;

(b) separating excess liquid from the solids in said slurry to form a damp mass;

(c) subjecting said mass to compressive shear stress in more than one direction and shaping said stressed mass into sheet-like form; and

(d) removing residual liquid retained within the shaped explosive composition.

11. A process which comprises:

(a) forming a slurry comprising a mixture of, based on the total weight of solids, at least about 5% of powdered tetrafluoroethylene resin and about from 50 to 95% of finely-divided, cap-sensitive, solid detonating explosive dispersed in about 1 to 20 times its volume of inert liquid hydrocarbon which wets said resin;

(b) separating excess liquid from the solids in said slurry to form a damp mass having a ratio of liquid-to-solids of about from 0.5:1 to 2:1;

(c) subjecting said damp mass to compressive shear stress in more than one direction until a coherent sheet-like structure is obtained; and

(d) removing residual liquid retained in the shaped explosive composition.

12. A process of claim **11** wherein said explosive is tetranitro-2,3;5,6-dibenzo-1,3*a*,4,6*a*-tetraazapentalene.

13. A process of claim **12** wherein the dried product is sintered at about from 621 to 650° F. for about from 0.5 to 10 minutes.

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BENJAMIN R. PADGETT, *Primary Examiner.*

Notice of Adverse Decision in Interference

In Interference No. 100,877, involving Patent No. 3,326,731, G. A. Noddin, DETONATING EXPLOSIVE IN POLYTETRAFLUOROETHYLENE MATRIX AND PREPARATION, final judgment adverse to the patentee was rendered Nov. 2, 1982, as to claims 1, 2, 6 and 7.

[*Official Gazette Feb. 1, 1983.*]