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Julian et al.

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[54] **IGNITER COMPOSITION**

[76] Inventors: **Elmo C. Julian; Frank G. Crescenzo,** both of China Lake, Calif.; **Robert C. Meyers, deceased,** late of China Lake, Calif. by Bette C. Meyers, executrix

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[52] **U.S. Cl.** **149/19,** 149/20, 149/22, 149/44, 149/87

[51] **Int. Cl.** **C06c 1/00**

[58] **Field of Search** ..... 260/92.1; 52/2, 2.1, 52/23, 23 X, 24, 15; 149/19, 87, 44, 20, 22

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Primary Examiner—Carl D. Quarforth
Assistant Examiner—E. A. Miller
Attorney—K. F. Ross and Q. B. Warner

EXEMPLARY CLAIM

1. A composition consisting essentially of about 30 to about 85 weight percent of a material selected from the class consisting of polytetrafluoroethylene and polytrifluorochloroethylene; about 15 to about 70 weight percent of a material selected from the class consisting of magnesium, aluminum, boron, titanium, ziroconium, thorium, mixtures thereof and lithium nitride; about 1 to about 10 weight percent of a material selected from the class consisting of potassium dichromate, manganese dioxide, ammonium nitrate and ammonium perchlorate; and from about 1 to about 10 weight percent of a material selected from the class consisting of lead fluoride and sodium fluoride.

2 Claims, 3 Drawing Figures

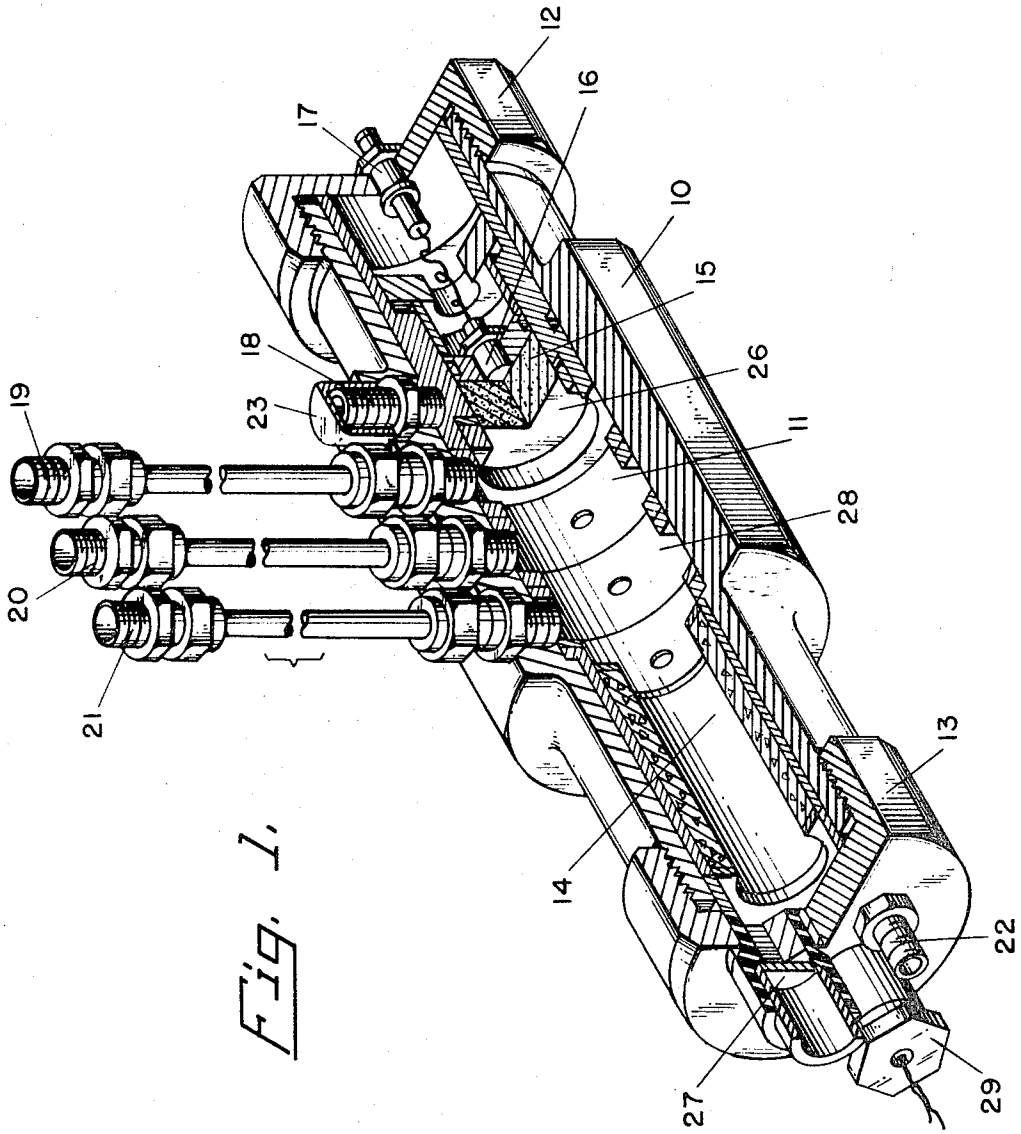


Fig. 1.

INVENTORS
ELMO C. JULIAN
FRANK G. CRESCENZO
ROBERT C. MEYERS, DECEASED,
BY BETTE C. MEYERS, EXECUTRIX,
BY
ATTORNEYS

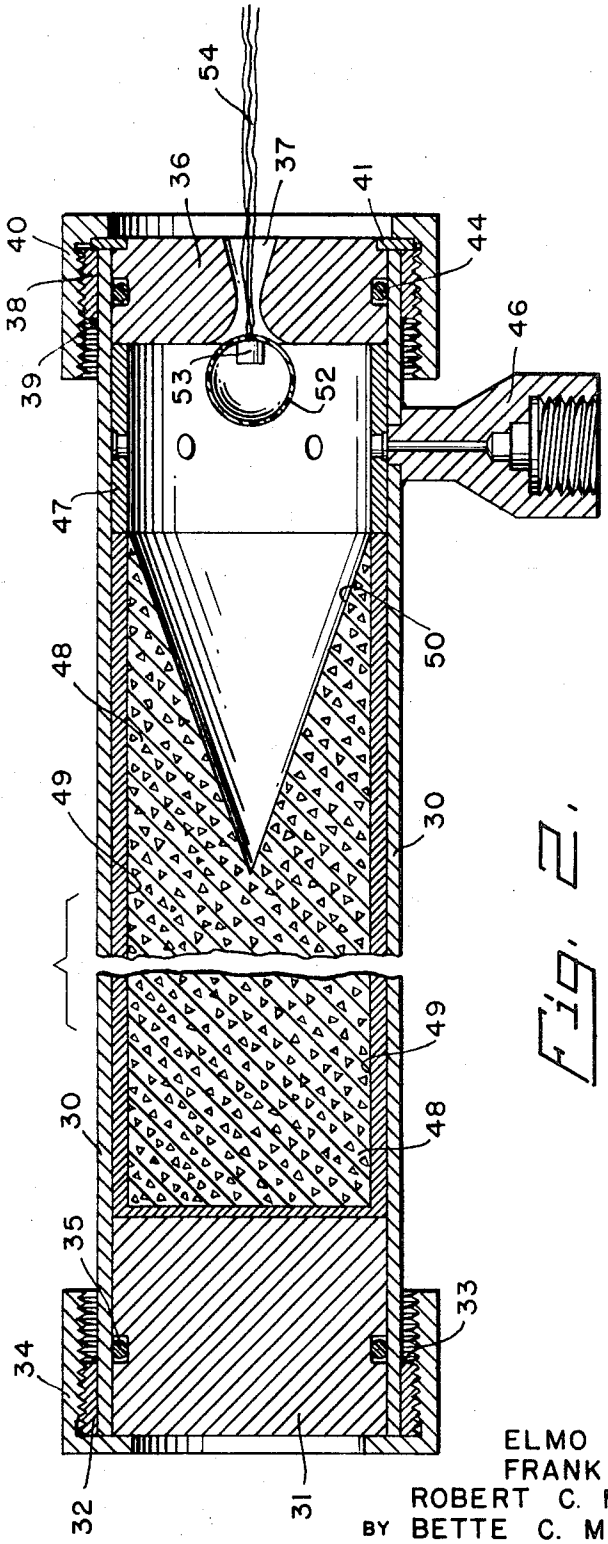


Fig. 2.

INVENTORS
ELMO C. JULIAN
FRANK G. CRESCENZO
ROBERT C. MEYERS, DECEASED,
BY BETTE C. MEYERS, EXECUTRIX,
BY
ATTORNEYS

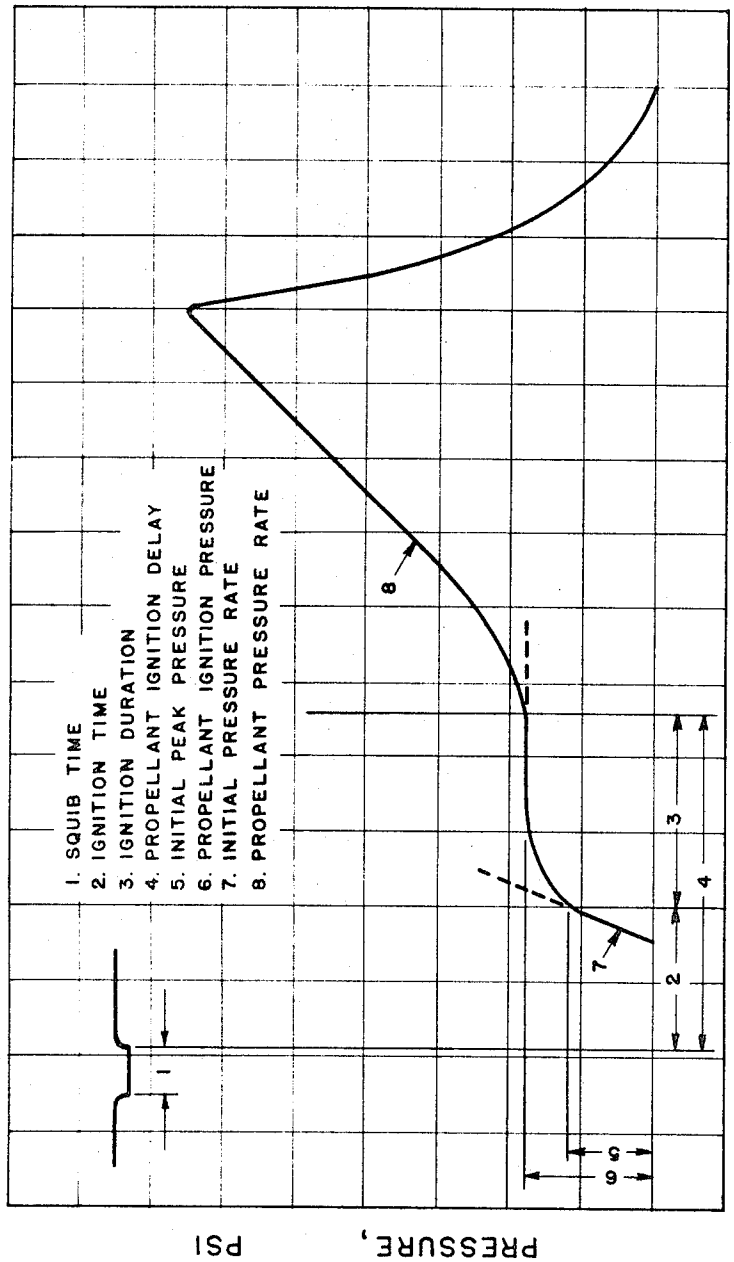


Fig. 3.

INVENTORS
ELMO C. JULIAN
FRANK G. CRESCENZO
ROBERT C. MEYERS, DECEASED,
BY BETTE C. MEYERS, EXECUTRIX,
BY
ATTORNEYS

IGNITER COMPOSITION

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

This invention relates to a new igniter, more particularly, it relates to an igniter mixture for gas producing charges such as propellants, fuels and explosives.

The ideal igniter should produce short ignition delays with reproducible output characteristics independent of environmental temperature and pressure conditions without causing damage to any component parts. In addition, igniter materials should be sufficiently inert to avoid hazard when handling. They should have good surveillance characteristics. The principal igniter materials used in the past have consisted of black powder used alone, or a mixture of 80 percent black powder and 20 percent magnesium. The above materials are subject to the disadvantages that they are highly sensitive to static electricity and friction, thus making their handling extremely dangerous. Also, they absorb moisture from humid air, this factor adversely affecting their ignition characteristics in that it leads to delayed ignition and complete ignition failures. Additionally, the above materials are highly sensitive in their operation to changes in temperature and pressure. Further, mixtures of black powder and magnesium have a tendency to segregate, thus vitiating the advantages of the combination.

It is therefore an object of this invention to provide an igniter which has an acceptable range of ignition time delay.

It is another object of this invention to provide igniter mixtures which are inert to static electricity, impact and friction. It is another object of this invention to provide an igniter mixture which is inert to temperatures below 400°C.

It is another object of this invention to provide an igniter mixture which has good surveillance characteristics and which is unaffected in its operation by moisture in the atmosphere.

It is a further object of this invention to provide an igniter which gives reproducible output characteristics which are independent of environmental temperature and pressure conditions.

It has been found that the above objectives are accomplished by an igniter incorporating a composition comprising a mixture of a material from the class consisting of magnesium, aluminum, thorium, zirconium, titanium, molybdenum, boron and lithium nitride, with a fluorinated alkene. To this composition may be added oxidizing agents such as lithium perchlorate, potassium dichromate, manganese dioxide and ammonium nitrate. Catalytic agents, such as, inorganic fluorides may also be added. Examples of these inorganic fluorides are fluorides of the alkali metals and lead.

The invention is best understood by reference to the following description taken in connection with the accompanying drawings hereby made a part of this specification and in which:

FIG. 1 is a cut-away showing of an ignition testing semi-vented bomb, used to test the compositions of the invention;

FIG. 2 is a horizontal cross-section of a two inch steel static firing motor for testing the ignition of end burning propellent charges; and

FIG. 3 is a typical pressure-time graph showing the igniter performance characteristics of interest obtained from test firing the two inch semi-vented bomb of FIG. 1.

The bomb of FIG. 1 comprises generally an internal pressure chamber in which the propellent grain is ignited by means of a conventional squib and the igniter composition of this invention. The pressure chamber is provided with means for measuring internal pressures at various times and intervals of space along the bomb. Referring to FIG. 1, the outer case of the bomb is represented by numeral **10** and the internal pressure chamber by the numeral **11**. The case **10** is provided with threaded end plugs **12** and **13** for engagement with the ends of case **10**. The end plug **12** contains the igniter assembly and the end plug **13** contains the pressure relief assembly. A three inch section of an internal burning, externally inhibited, star perforated propellent grain is shown at **14**. The grain has an outside diameter of 1.9 inches. The igniter material is shown at **15**. For igniting the igniter material **15** a conventional squib is provided at **16** which in turn is provided with a pressure seal electrical lead **17**. For measuring pressure in the chamber at various intervals of time and space, pressure taps **18, 19, 20, 21,** and **22** are provided. These taps are connected by hydraulic lines to Wiancko conventional type pressure gages. A cap **23** is provided for pressure tap **18** to prevent pressure leak when the device is inoperative. Rupture discs **26** and **27** designed to rupture at predetermined pressures are provided as shown. For adjusting the distance between propellent grain and igniter, removable collars **28** are provided in the pressure chamber. A thermocouple assembly **29** is supported by end plug **13**.

In operation of the test bomb, a propellent charge **14** is placed in the inner cavity **11** of the bomb as shown, and the igniter composition **15** of the invention is inserted in the case 4 inches from the propellant **14** and adjacent the squib **16**. After the pressure gages have been connected to suitable recorders, the igniter is ignited by introduction of current to the squib **16** through electrical lead **17**. During the process of ignition of the squib, igniter and propellant, pressure-time measurements are recorded.

Referring to FIG. 2, there is shown a static testing rocket motor having an outer casing **30** and provided with head end closure **31**. Head end closure **31** is sealed to the rocket body by means of threaded ring **32** attached to the case **31** by fillet **33**, and internally threaded cap **34** as shown. O ring **35** is provided to furnish a gas tight seal. The rocket nozzle is shown at **36** having an exhaust port **37**. The nozzle **36** is attached to the rocket body by means of threaded ring **38** secured to the outer casing or rocket tube **30** by means of fillet **39**, and internally threaded cap **40**. Nozzle shear ring **41** is provided as shown. The end assembly is made gas tight by means of O ring **44**. Pressure plugs **46** are provided for measuring pressure in the cavity aft of the rocket grain. A series of these plugs located circumferentially in the outer casing may be used. Spacing ring **47** is provided for holding the grain secure. For testing the igniter, a double base end burning propellent grain **48** having an outer inhibiting coating **49** is located in the rocket motor chamber as shown. The grain is provided with a conical shaped burning surface **50**. The igniter material of the invention in a plastic bag **52** with a conventional squib **53** in proximity thereto is posi-

tioned as shown. Electrical leads **54** are provided as shown for conducting current from a source not shown to squib **53.** In operation of the motor, current is introduced to ignite the squib, igniter material and propellant in turn. Internal pressure is measured by means of the pressure plugs **46** which are hydraulically connected to pressure gages.

Referring to FIG. **3,** there is shown a typical pressure-time graph used to present important igniter test data obtained by testing compositions of the invention in the test device of FIG. **1.** The compositions are operative as igniter materials under actual operating conditions. In the graph, pressure is plotted on the ordinate against time delay in milliseconds on the abscissa. The legend for the numerals is shown on the drawing. Squib time is the interval of time between the application of current to fusion of the bridge wire in the squib. Igniter time is the time elapsing from fusion of the bridge wire to ignition of the igniter or until the first indication of initial pressure, this later indicating the ignition of the igniter. The ignition duration is the time between ignition of the igniter and ignition of the propellant and is indicated on the graph by the inflection of the pressure time curve as indicated by the numeral **3.** The propellant ignition delay is the sum of the igniter time and the ignition duration as represented by the numerals **2** and **3** on the graph. This is the most significant factor in the evaluation of igniter mixtures. Numerals **5, 6, 7** and **8** represent supplementary pressure information shown on the graph.

The invention is illustrated by the examples included in Tables 1–4, inclusive, which are illustrative only of the invention but not limiting thereof. Tables 1 and 2 present comparative results of ignition tests performed on black powder, black powder-magnesium mixture, and a polytetrafluoroethylene-magnesium mixture of the invention. Tables 3 and 4 present examples of ignition mixtures of the invention and test data therefor. The data for the examples was obtained with the ignition testing, semi-vented bomb of FIG. **1.**

The compounding of the igniter mixtures is illustrated by the following: Metal powder 100 mesh or finer was added to powdered Teflon polymer of 50 mesh or finer in ratios of 50 to 800 percent of the stoichiometric quantities. These materials were then thoroughly mixed. They may be blended, rolled or treated in a signal blade mixer. Alternate methods of making the mixtures is as follows: Teflon is reduced to less than 5 micron particle size by techniques well known in the art and introduced to a Waring blendor. An inert solvent such as acetone or a hydrocarbon is added and the mixture blended for 10 seconds in the blendor. This material is transferred to a suitable vessel where the metal is added and the mixture is stirred for 5 minutes. The material is then filtered to remove the bulk of the solvent and transferred to a mixer where the remainder of the components are added. Complete grinding followed by further mixing completes the compounding.

The fluorocarbon compound is a polymeric halogenated alkene, preferably, polytetrafluoroethylene or polytrifluorochloroethylene or mixtures thereof. Other fluorinated alkenes may be used, such as, the fluorinated higher alkenes. By the term "fluorinated" as used in this specification and claims is meant either totally or partially fluorinated. The polytetrafluoroethylene may have a molecular weight range from 100,000 to 9,000,000. This compound is known commercially as Teflon and its chemical composition is published in the literature. The polychlorotrifluoroethylene may have a molecular weight range from 303 to 10,000. This compound is known commercially as Kel-F. It may be used in the composition in liquid, solid or waxy form depending upon the amount of plasticization which is to be effected by its use in non-solid form. Plasticization to a degree, may be effected through the use of liquid polymers such as KEL-F polymer oil of various molecular weights. A portion of the solid polymer may thus be replaced by a chemically equivalent weight of the liquid polymer. When used in solid form the fluorocarbon compound is a powder of less than 20 mesh particle size.

The following is an example of compounding the composition when plasticization is required. The metal powder, 100 mesh or finer, is added to somewhat less than the intended quantities of the powdered solid polymer, 50 mesh or finer. Enough liquid polymer to realize the intended ratio of total polymer to powdered metal, 50 – 200 percent of the stoichiometric quantities, is added. The liquid polymer may be added in the form of a solution in a solvent such as benzene or methylene chloride. In this case the solvent must be removed by evaporation. The material may be mixed in a sigma blade mixer.

As respects the metals used, the heat of formation of the metal-fluorine bond must exceed 67 kilocalories per gram molecule of the condensed halide. As the examples show, the nitrides of these metals may be used. The carbides and hydrides, having similar chemical properties and similar heats of formation as the nitrides may be used also. The metals and their compounds may be in divided form of less than twenty mesh, or part of the finely divided metal may be replaced by wire of 0.0020 inches in diameter or granules of 20 – 25 mesh particle size.

Oxidation of one of the reaction products, carbon, may be effected by the addition of an oxidant, such as, ammonium nitrate, or lithium perchlorate in amounts of not more than 10 percent by weight. A small increase in gas production is thus effected. The oxidant may be added to the mixture without affecting the polymer-metal ratio. The oxidant may be in powdered or pellet form.

The propellants used to test the igniter composition are the double base propellants, N–5 and X–11. A representative N–5 composition is as follows:

Component	Wt. Percent
Nitrocellulose (12.6%N)	50.00
Nitroglycerin	34.9
Diethyl Phthalate 10.50	
2-Nitrodiphenylamine	2.00
Lead 2-Ethyl Hexoate	1.20
Lead Salicylate	1.20
Candelilla Wax	0.20

A representative X-11 composition is the following:

Component	Wt. Percent
Nitrocellulose (12.6%N)	50.00
Nitroglycerin	33.1
Diethyl Phthalate	12.4
2-Nitrodiphenylamine	2.0
Lead Salicylate	1.2
Lead β-resorcylate	1.2
Candellia Wax	0.1
Carbon Black	+0.05

As stated previously, the percentage of metal to fluorocarbon which may be used is preferably from about

50 to about 800 percent of the stoichiometric. The preferred percentage range is from about 15 to about 70 percent by weight of metal and about 30 to about 85 percent by weight of fluorocarbon.

same composition and ignition delay values obtained show a definite relationship between the composition of the mixture and its heat of explosion. Variation in ignition time delay results from the extreme conditions

TABLE I

Summary of Performance Characteristics of Igniter Materials Test Fired at −65° F. in 2.0-Inch Diameter Experimental Test Vehicles with N-5 Propellant

Igniter material	Igniter charge wt.	Squib time, msec.	Igniter time, msec.	Ignition duration, msec.	Propellant ignition delay, msec.	Initial peak pressure, p.s.i.	Initial rate of pressure rise, p.s.i./msec.	Propellant ignition pressure, p.s.i.	Propellant rate of pressure rise, p.s.i./msec.	Average heat of explosion
Black powder	3.0	14.0	2.0	12	14	326	137	510	50.6	700
Black powder, 80%-Mg, 20%	3.0	9.0	20.0	3	23	460	213	505	43.7	850
Polytetrafluoroethylene, 67%-Mg, 33%	3.0	20.0	14.7	9	24	474	301	637	56.6	1800

TABLE II

Test Made at −65° F. in 2.0 Diameter Inch Experimental Test Vehicle with X-11 Propellant

Igniter material	Igniter charge wt.	Squib time, msec.	Igniter time, msec.	Ignition duration, msec.	Propellant ignition delay, msec.	Initial peak pressure, p.s.i.	Initial rate of pressure rise, p.s.i./msec.	Propellant ignition pressure, p.s.i.	Propellant rate of pressure rise, p.s.i./msec.	Average heat of explosion
Black powder	3.0	18.6	8.7	13.4	22.1	368	119	543	87	700
Black powder, 80%-Mg, 20%	3.0	18.7	8.2	10.2	18.4	325	94	581	111	850
Polytetrafluoroethylene, 67%-Mg, 33%	3.0	20.2	8.3	8.3	16.6	443	188	600	92	1800

TABLE III

Three gram charge weights were used. Tests were run at −65° F. The propellent charge was spaced 4" from the igniter. The examples are given in percentage composition by weight

Example No.	Component (wt. percent)						H.E.	P.I.D
	Magnesium	Boron	PTFE	PCTFE	NaF	$K_2Cr_2O_7$		
1	50.0		25.0	10.0	7.5	7.5	1,281	32.0
2	40.0		35.0	10.0	7.5	7.5	1,597	17.5
3	30.0		45.0	10.0	7.5	7.5	1,846	14.5
4	20.0		55.0	10.0	7.5	7.5	1,378	24.5
5	60.0		15.0	10.0	7.5	7.5	1,059	59.9
6	25.0	25.0	25.0	10.0	7.5	7.5	1,347	26.9
7	31.08		63.92		2.5	2.5	1,964	14.5

LEDGEND.—PTFE=Polytetrafluoroethylene; PCTFE=Polychlorotrifluoroethylene; H. E.=Heat of explosion in calories per gram; P. I. D.=Propellant ignition delay in milliseconds.

TABLE IV

A three gram charge weight was used. Mixtures were tested at −65° F. with the propellant being spaced 4" from the igniter. The examples are given in percentage compositions by weight

Example No.	Component (wt. percent)											H.E.	P.I.D.
	Th	Zr	Ti	Mo	Al	Li_3N	PTFE	$K_2Cr_2O_7$	NaF	PbF_2	$LiClO_4$		
8	66.40						28.60	2.50	2.50			647	70.7
9		33.39			9.00		36.61	3.00		9.00	9.00	1320	31.4
10			21.53		9.00		46.82	3.00		9.00	9.00	1200	42.5
11				27.31	9.00		42.69	3.00		9.00	9.00	430	28.8
12					25.13		69.87	2.5	2.5			1623	
13						30.12	64.88	2.50	2.50			1715	

LEGEND. PTFE=Polytetrafluoroethylene; H.E.=Heat of explosion in calories per gram; P.I.D.=Propellant ignition delay in milliseconds

It will be noted that all data, with the exception of the heat of explosion tests, was taken at temperatures of −65°F for three gram charge weights. The igniter was spaced a distance of four inches from the propellent charge. Test firings were conducted at low temperatures with small igniter charge weights, as these conditions indicate successful functioning at extreme conditions as well as extend the difference in the ignition time delays of similar compositions. Examples 1 through 5 in Table III show varying proportions of the under which the tests were made. It will be noted from the results of Tables I and II that the propellant ignition delay obtained with magnesium-Teflon mixtures is as good as that obtained with the ignition compositions of the prior art, that is, black powder used alone and black powder-magnesium mixture. For propellant ignition in general, it is to be noted that differences in propellant ignition delay when the total time is less than 35 milliseconds are insignificant as any ignition delay under 35 milliseconds is highly acceptable. The advantages of

the ignition mixtures of the present invention over prior art ignition mixtures stem from other properties. It will be noted from all the examples that the heat of explosion of the igniter compositions of this invention can be adjusted to fit the circumstances by choice of the metal used. In example 8 of Table IV, a composition is shown in which the heat of explosion is only 647 calories per gram. This composition could be used in applications wherein heat damage to surrounding metal parts is to be avoided. It is to be noted that in all examples, with the possible exception of examples 5 and 8 of Tables III and IV, respectively, an acceptable ignition time delay is obtained. The examples show that the igniter mixtures of the invention produce extremely high energy output, and have reproducible output characteristics under severe conditions. The mixtures were tested and found to be inert to shock, friction, or static electricity, and insensitive to temperatures of 400°C or less. The mixtures are simple to manufacture from readily obtainable materials; they are not subject to segregation, they are non-toxic and their operation is not affected by moisture and high humidity. The mixtures are compatible with propellent and explosive compositions in general, and can be assembled in contact with the propellant. This is in contrast to black powder which is incompatible with double base propellants, for example, and must be assembled in a cannister to avoid contact with the propellant, as well as to prevent absorbtion of moisture from the atmosphere by the black powder. To test the inertness of the mixtures to static electricity a spark from a spark coil was discharged through the ignition mixtures placed in the spark gap without ignition of the powder. Representative samples of the ignition mixtures were subjected to temperatures at 400°C for four hours in a muffle furnace with no signs of ignition. Drop tests performed on representative mixtures indicate that they are highly insensitive to shock. For example, only 50 percent ignition was obtained in drops of 85 centimeters with a two kilogram weight. As will be noted from the compositions of the examples, oxidizing agents such as potassium dichromate may be added. Catalytic agents, such as, fluorides of the alkali metals and lead fluoride may also be incorporated in the mixtures. It was noticed that a slight improvement was obtained by the combinations of oxidizing agent and catalytic agent. The effect of the catalytic agent is believed to be due to probable activation of the surface of the metal, thus enhancing its reaction with the fluorinated compound.

While the effectiveness of the ignition compositions of the invention have been illustrated by the ignition of double base propellants having a nitroglycerin-nitrocellulose base, they are by no means limited to this application as they are equally applicable to the ignition of other propellants and explosives in general.

Obviously, many modifications and variations of the present invention are possible in the light of the above teachings. It is therefore to be understood that within the scope of the appended claims the invention may be practiced otherwise than as specifically described.

What is claimed is:

1. A composition consisting essentially of about 30 to about 85 weight percent of a material selected from the class consisting of polytetrafluoroethylene and polytrifluorochloroethylene; about 15 to about 70 weight percent of a material selected from the class consisting of magnesium, aluminum, boron, titanium, zirconium, thorium, mixtures thereof and lithium nitride; about 1 to about 10 weight percent of a material selected from the class consisting of potassium dichromate, manganese dioxide, ammonium nitrate and ammonium perchlorate; and from about 1 to about 10 weight percent of a material selected from the class consisting of lead fluoride and sodium fluoride.

2. The composition according to claim **1** wherein the material selected from the class consisting of magnesium, aluminum, boron, titanium, zirconium, thorium, mixtures thereof and lithium nitride has a particle size which does not exceed 20 mesh.

* * * * *