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(54) **EPDM ROCKET MOTOR INSULATION**

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(51) **Int. Cl.**[7] **C08L 9/00**; C08F 210/16; C08K 3/36; F02G 1/00; B63H 11/00

(52) **U.S. Cl.** **523/138**; 524/492; 524/526; 525/331.7; 525/332.3; 60/200.1; 60/204

(58) **Field of Search** 524/492, 526; 525/331.7, 332.3; 523/138; 60/200.1

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(57) **ABSTRACT**

A novel and improved EPDM formulation for a solid propellant rocket motor is described wherein hexadiene EPDM monomer components are replaced by alkylidene norbornene components and with appropriate adjustment of curing and other additives functionally-required rheological and physical characteristics are achieved with the desired compatibility with any one of a plurality of solid filler materials, e.g. powder silica, carbon fibers or aramid fibers, and with appropriate adhesion and extended storage or shelf life characteristics.

18 Claims, 3 Drawing Sheets

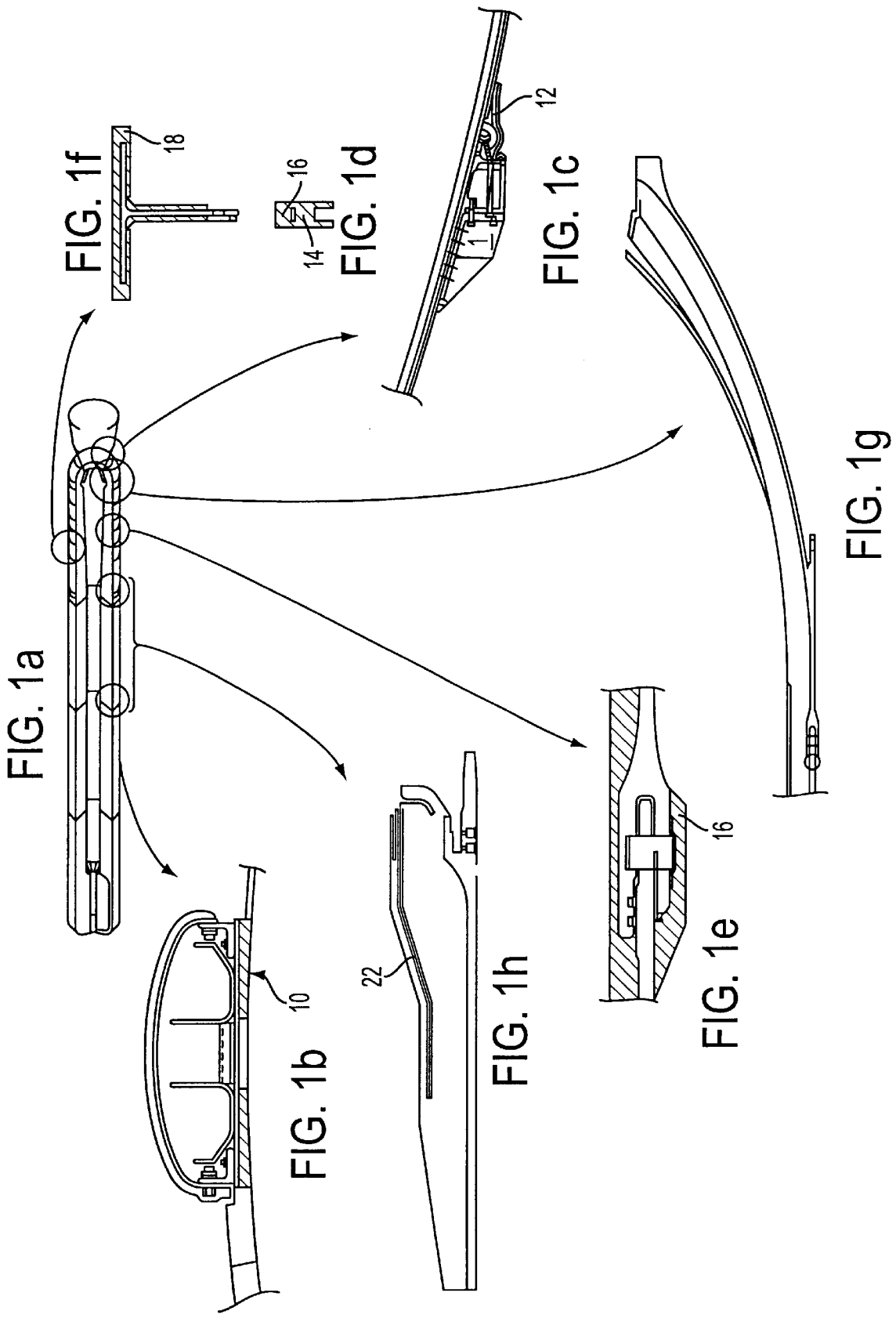
FIG. 1a
FIG. 1b
10
FIG. 1c
12
FIG. 1d
16
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FIG. 1e
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FIG. 1f
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FIG. 1g
FIG. 1h
22

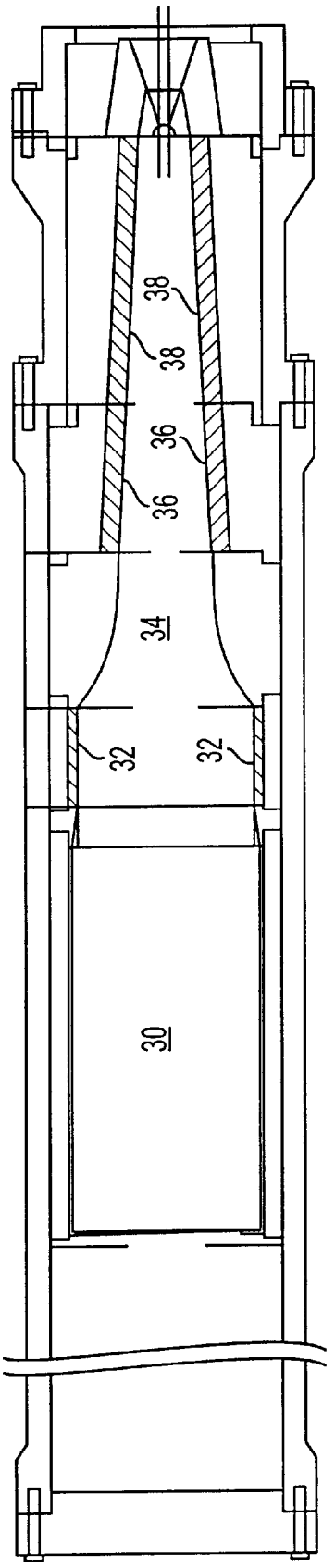

FIG. 2

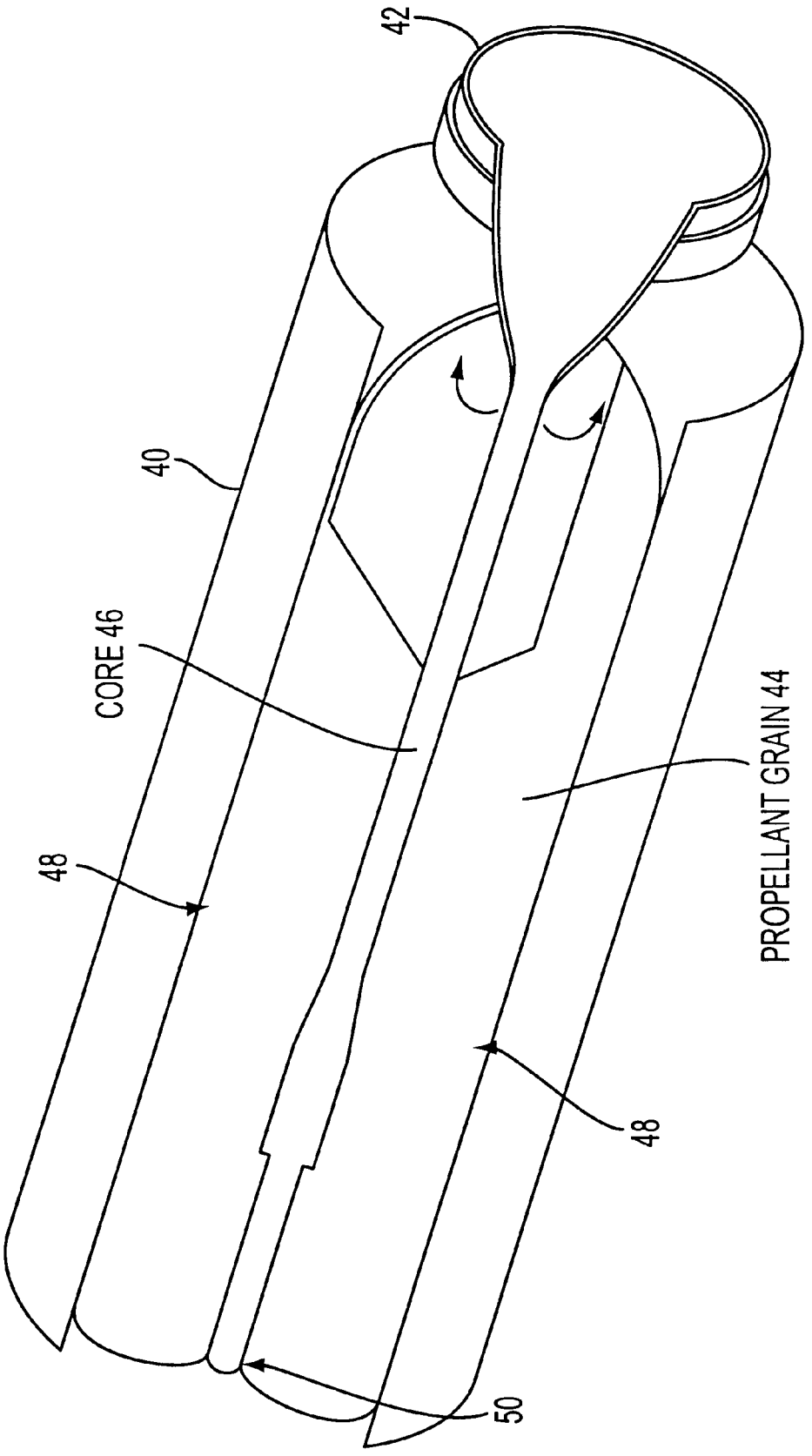

FIG. 3

EPDM ROCKET MOTOR INSULATION

This application claims the benefit of the priority from Provisional Application Nos. 60/115,855, 60/115,856, 60/115,859 and 60/115,857 filed on Jan. 13, 1989.

ORIGIN OF THE INVENTION

The U.S. Government has a paid-up license in this invention and the right in limited circumstances to require the patent owner to license others on reasonable terms as provided for by the terms of contract NAS 8-38100 awarded by the National Aeronautics and Space Administration (NASA) and contract N00030-97-C-0100 awarded by the U.S. Department of the Navy.

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a novel composition and method for providing insulation for solid propellant rocket motors, and more particularly to EPDM compositions having fibrous components such as carbon fibers or powder fillers such as silica, or also containing Kevlar reinforcing fibers and suitable for internal and external insulation applications on such rocket motors.

2. Background and Description of the Related Art

It is generally accepted current industry practice to prepare insulations for solid propellant rocket motors from a polymeric base importantly composed of an EPDM (ethylene-propylene-diene monomer) terpolymer blend and containing as one of the diene monomer components of the EPDM blend a 1,4-hexadiene (HD).

This EPDM terpolymer, which is commonly designated as the primary EPDM terpolymer since it is present in a higher concentration than the secondary EPDM terpolymer, has been established as a standard for solid propellant rocket motor insulations due to its superior ablation characteristics, excellent physical properties and processability.

For instance, an exemplary carbon fiber-filled rocket motor insulation composed of NORDEL 1040 as the primary terpolymer is commonly known in the industry as the STW4-2868 thermal insulation and has the following composition as shown in Table 1:

TABLE 1

STW4-2868 THERMAL INSULATION FORMULATION (carbon fiber; parts by weight)

Ingredient	Function	Parts by Weight
NORDEL 1040	Primary EPDM terpolymer base	80
Neoprene FB	Secondary polymer base	20
Zinc oxide	Activator	5
Sulfur	Curative	1
HAF carbon black	Pigment	1
MBT	Accelerator	1
AGERITE Resin D	Antioxidant	2
AGERITE HPS	Antioxidant	1
Tellurac	Accelerator	0.50
Sulfads	Accelerator	0.75
VCM carbon fibers	Filler	41
Total Parts by Weight		153.25

Alternatively, solid rocket motor insulations are also composed of compositions employing finely divided powder silica as a filler, with or without the added presence of a fibrous reinforcing agent.

Exemplary silica-filled rocket motor insulations have also included NORDEL 1040 and NORDEL 2522 as the primary terpolymer in their formulations and the resulting compositions are respectively commonly known in the industry as the 053A and DL1375 thermal insulations. They have the following compositions shown in Table 2:

TABLE 2

THERMAL INSULATION FORMULATION (silica filled; parts by weight)

Ingredient	Function	DL1375 (parts by weight)	053A (parts by weight)
NORDEL 1040	Primary EPDM terpolymer base		80
NORDEL 2522	Primary EPDM terpolymer base	80	
Neoprene FB	Secondary polymer base	20	20
Zinc oxide	Activator	5	5
Sulfur	Curative	1	
AGERITE Resin D	Antioxidant	2	2
AGERITE HPS	Antioxidant	1	1
Captax	Accelerator	1	1
Tellurac	Accelerator	0.5	0.5
Sulfads	Accelerator	0.75	0.75
HiSil 233	Filler	35.5	35.5
Total Parts by Weight		146.75	146.75

In addition, an EPDM terpolymer comprising the HD monomer is sold under the tradename NORDEL 2722E. An exemplary silica-filled rocket motor insulation comprising NORDEL 2722E as the secondary terpolymer is commonly known in the industry as the DL1552A thermal insulation and has the following composition as shown in Table 3:

TABLE 3

DL1552A THERMAL INSULATION FORMULATION WITH SILICA

Ingredient	Function	Parts by Weight
Buna EP T 3950 (Bayer Corp., Fiber, Additives and Rubber Division of Orange, Texas)	Primary EPDM terpolymer base	75
NORDEL 2722E (DuPont Dow Elastomers)	Secondary EPDM terpolymer base with high ethylene content	20
WINGTACK 95 (hydrocarbon resin) (Goodyear Tire and Rubber Co., Chemical Division of Beaumont, Texas)	Tackifier	7
IRGANOX 1010 (tetrakis[methylene-3-(3'-5'-di-tert-butyl-4'-hydroxyphenyl) proprionate]methane) (Ciba Specialty Chemicals, Additives Division, Tarrytown, N.Y.)	Antioxidant	1
TRYCOL DA-6 (decyl polyoxyethylene alcohol)(Chemical Associates, Inc. of Copley, Ohio)	Wetting agent	0.5
Stearic acid (including palmitic acid) (Harwick Standard Distribution Corp. of Akron, Ohio)	Cure activator	1
HiSil 233 (silica hydrate)(PPG Industries, Inc. of Lake Charles, Louisiana)	Reinforcing filler	45
Aluminum oxide C (Al_2O_3)(Degussa Corporation of Ridgefield Park, N.J.)	Reinforcing filler	0.3
N330 carbon black (Columbian Chemicals Co. of Marietta, Ga.)	Pigment and reinforcing filler	
KALENE 1300 (butyl gum elastomer)	Co-vulcanizing	20

TABLE 3-continued

DL1552A THERMAL INSULATION FORMULATION WITH SILICA

Ingredient	Function	Parts by Weight
(Hardman Division of Harcros Chemicals, Inc. of Belleville, N.J.)	plasticizer	
HYPALON 20 (chlorosulfonated polyethylene)(DuPont Dow Elastomers)	Cure activator	5
AGERITE Resin D (polymerized trimethyl dihydroquinone)(R.T. Vanderbilt Co., Inc. of Buena Park, Ca.)	Antioxidant	0.25
TZFD-88p (zinc oxide dispersed in an EPDM binder)(Rhein Chemie Corp. of Trenton, N.J.)	Cure activator	2
SP 1056 (bromomethyl alkylated phenolic resin)(Schenectady Int'l, Inc. of Schenectady, N.Y.)	Curing agent	15
Total Parts by Weight		193.05

An exemplary aramid fiber filled rocket motor insulation comprising NORDEL 1040 is commonly known in the industry as R196 thermal insulation and has the following composition as shown in Table 4:

TABLE 4

R196 THERMAL INSULATION FORMULATION WITH KEVLAR

Ingredient	Function	Parts by Weight
NORDEL 1040 (EPDM terpolymer)	Polymer base	80
NATSYN 2200 (polyisoprene) (Goodyear Tire and Rubber Co., Chemical Division of Akron, Ohio)	Polymer base	20
WINSTAY S (styrenated phenols) (Goodyear Tire and Rubber Co., Chemical Division of Akron, Ohio)	Antioxidant	1.0
Dechlorane Plus 515 (1,2,3,4,7,8,9,10,13,13,14,14-dodecachloro-1,4,4a,5,6,6q,7,10,10a,11,12,12a-dodecahydro-1,4,7,10-dimethanodibenzo (a,e) cyclooctene) (Occidental Chemical Corporation of Dallas, Texas)	Flame retardant	40
Antimony oxide (Sb_2O_3)(Harcros Chemicals, Inc. of Kansas City, Kansas)	Flame retardant/-filler	20
¼" KEVLAR fiber (aramid staple fiber) (E.I. duPont de Nemours and Co., of Wilmington, Delaware)	Fiber	20
VAROX DBPH-50 (2,5-dimethyl-2,5-di(t-butylperoxy)hexane on a carrier) (R.T. Vanderbilt Co., Inc. of Buena Park, Ca.)	Curing agent	2.5
Total Parts by Weight		183.5

Numerous past efforts to develop effective replacements for these standard solid rocket motor insulation formulations have not been successful.

The only manufacturer currently producing the foregoing primary EPDM terpolymer in adequate quantities to meet the demands of the rocket motor insulation industry is DuPont Dow Elastomers of Beaumont, Tex., which markets and sells an EPDM terpolymer comprising the HD monomer under the tradename NORDEL 1040 and Nordel 2522.

However, the ability of the industry to produce STW4-2968, DL1375, 053A, DL1552A, R196 and other thermal insulations containing NORDEL 1040 and NORDEL 2522, and NORDEL 2722E as a primary or secondary EPDM terpolymer has recently been placed in jeopardy due to the announcement by DuPont of its intention to cease production of NORDEL 1040, 2522, 2722E and, generally, other EPDM polymers formed from 1,4-hexadiene. There is therefore a need in this industry, previously not satisfied, to find an effective alternate or a replacement for the above-described standard STW4-2868, DL1375, 053A DL1552A and R196 thermal insulations. Development and formulation of a suitable primary EPDM terpolymer replacement is especially critical for these discontinued NORDEL insulation formulations.

The requirements for an acceptable, functionally effective, insulation for solid propellant rocket motors are well known to be quite severe due to the extreme conditions to which the insulation is exposed. These conditions not only include exceedingly high temperatures but also severe ablative effects from the hot particles (as well as gases) that traverse and exit the rocket motor interior. Unless the insulation will withstand such conditions, catastrophic failure may (and has) occur.

U.S. Pat. No. 3,347,047, an early patent describing asbestos fiber filled insulations, states that flame temperatures encountered in the combustion of propellants, particularly when used as source of propulsion, necessitating the confinement of the gases of combustion and ultimate release thereof through orifices, are usually accompanied by extremely turbulent flow conditions. All of these features place considerable stress and strain upon the member defining the escape passageway. While the combustion of the propellant in the case of rockets and the like will usually be of short duration, the temperatures and turbulence encountered have been found to very easily destroy even the strongest and most exotic alloys formed of iron, steel, titanium, magnesium, silicon, chromium, beryllium and the like. As a consequence, the projectile structure fails leading to total destruction thereof through explosion or in the event that only the exit passageway is destroyed, the projectile proceeds in an erratic uncontrollable path since its trajectory or path is at least in part dependent upon the contour of the passageway through which pass the gaseous products of combustion. That statement still remains fully applicable today.

Therefore any replacement insulation should exhibit at least comparable temperature resistant and ablation characteristics and rheological and physical properties (e.g., Mooney viscosity) at least equivalent to that of STW4-2868, DL1375, 053A, DL1552A and R196, yet should not otherwise significantly alter the formulation techniques employed for the production of the such rocket motor thermal insulation. Additionally, due to the large and growing quantities of solid propellant rocket motor insulation required by the industry, any such replacement EPDM terpolymer candidate should be abundantly available now and into the foreseeable future.

In addition, any replacement EPDM or like terpolymer should satisfy a number of other requirements including wettability of and bond strength with such diverse filler additives as a carbon fiber, aramid fiber, and a silica powder. It is also necessary that such additives be substantially homogeneously dispersed throughout the insulation composition as it is being produced. While standard mixing devices can be employed in the practice of this invention, such as a Banbury mixer, it is a common experience that substantially homogeneous distribution of fibrous additives is not achieved, or achieved only with difficulty, with many elas-

tomeric compositions. Difficulties have been described as in, for instance, during mixing of the components, it can be observed that premature vulcanization may occur as well as other problems that may impede, or entirely frustrate, effective distribution of the various additives which are essential to the ultimate production of the insulation.

Further, once formulated, the elastomeric composition must also possess acceptable shelf life characteristics such that it remains sufficiently pliable, without becoming fully cured, until used in application to the rocket motor casing. This requirement is essential because the production of a given lot of insulation may have to wait in storage for a number of months prior to use. Typically, the insulation may be stored in large rolls in an uncured, or at most a partially cured, state until ready for use. A number of curing agents are well known and are conventionally employed but still must be compatible with the overall EPDM formulation to permit satisfactory shelf life. This in turn requires a balancing of curing agent activity.

In addition, the formulated insulation should be substantially odorless for obvious reasons and this can require special adjustment of the curing agent components.

After application to the interior (or if desired the exterior) of the rocket motor casing, and subsequent curing thereof, an acceptable insulation must also exhibit satisfactory bonding characteristics to a variety of adjacent surfaces. Such surfaces include the internal surface of the rocket motor casing itself and the insulation must also exhibit adequate bonding characteristics between itself and the propellant grain, typically with an intermediate liner surface. In turn, the propellant grain in a solid propellant rocket motor is composed of a variety of materials notably including still another elastomer, various combustible materials, and such additional components as aluminum particles.

A functionally acceptable solid propellant rocket motor insulation must meet those requirements and must also survive aging tests. Such rocket motors may be fully fabricated even many months before actual firing, and for tactical weapons especially sometimes even more than a year or even a plurality of years. For instance, strategic missiles may be stored in silos or submarine launch tubes for decades. Over that period of time, the insulation must continue to remain fully functional without unacceptable migration of its components to or from adjacent interfacial surfaces and adequately retain its elastomeric characteristics to prevent brittleness. This requirement also needs to be satisfied under wide temperature variations. The vibration and physical stress placed on a rocket motor at the time of launch, whether a ground launch or an air firing, is exceedingly high, and brittleness and cracking in the insulation is effectively intolerable, whether from premature or gradual overcure or whatever cause. Even at the end of the burn of the propellant grain within the rocket motor casing the insulation must remain substantially and functionally intact to avoid potentially catastrophic failures of the entire launch vehicle.

In turn, this means that the insulation composition must meet the ablation limits for protection of the casing throughout the propellant burn without adding undue weight to the motor.

A number of past patents have been granted proposing various solutions to the insulation formulation problem. These include U.S. Pat. No. 3,421,970 (generically describing elastomeric formulations with asbestos); U.S. Pat. No. 3,562,304 (generically describing an elastomeric formulation with asbestos fibers); U.S. Pat. No. 3,637,576 (describing an EPDM formulation with a norbornene component with asbestos fibers); U.S. Pat. No. 4,492,779 (generically describing elastomeric formulations with Kevlar fibers); U.S. Pat. No. 4,514,541 (generically a du Pont "master batch" formulation with Kevlar fibers, but not an insulation); U.S. Pat. No. 4,550,130 (generically describing a moldable carboxylic acid modified EPDM to enhance affinity to various fillers); U.S. Pat. No. 4,878,431 (generically describing an elastomeric formulation using the EPDM Nordel 1040, with Kevlar fibers); U.S. Pat. No. 5,364,905 (describing a technique for the in situ polycondensation formation of aramid fibers, but not referring to rocket motor insulations); U.S. Pat. No. 5,498,649 (describing a polyamide/maleic anhydride modified EPDM with Kevlar fibers for a rocket motor insulation); U.S. Pat. No. 5,821,284 (a Kevlar fiber filled insulation containing an EPDM illustrated by Nordel 2522 in combination with ammonium salts); and U.S. Pat. No. 5,830,384 (generically referring to EPDM's with a "dry water" silica additive for cooling purposes). None of these patents address nor effectively solve the problem faced by the present invention. In fact the frequent reference to Nordel 1040 or Nordel 2522 serves to confirm the observation that these particular elastomers are well-nigh the standard in the rocket motor insulation industry.

Accordingly, the search for a functionally satisfactory elastomeric insulation composition requires discovery and implementation of an extraordinarily complex combination of characteristics. The criticality of the material selection is further demonstrated by the severity and magnitude of the risk of failure. Most insulations are of necessity "man-rated" in the sense that a catastrophic failure can result in the loss of human life—whether the rocket motor is used as a booster for launch of the space shuttle or is carried tactically underneath the wing of an attack aircraft. The monetary cost of failure in satellite launches is well-publicized and can run into the hundreds of millions of dollars.

One well known potential point of failure is the appearance of voids or cracks in the insulation which could lead to the penetration of the rocket motor casing itself. The resultant dispersion of hot gases may not only lead to destruction of the motor generally or can at least lead to its being thrown of its intended course or trajectory with several unhappy results. In such events, either the vehicle itself will self-destruct, or will be intentionally destroyed, or the satellite will be launched into a useless orbit.

Therefore, one of the most difficult tasks in the solid propellant rocket motor industry is the development of a suitable, acceptable insulation composition that will meet and pass a large number of test criteria to lead to its acceptability.

Furthermore, any replacement EPDM terpolymers should not be susceptible to obsolescence issues nor discontinuance in future supply thereof.

SUMMARY OF THE INVENTION

It is, therefore, an object of this invention to address a crucial long-standing need in the industry for an acceptable substitute for the STW4-2868, DL1375, 053A, DL1552A and R1961 insulations by providing a reformulated rocket motor thermal insulation notably comprising a suitable primary or secondary terpolymer replacement for the 1,4-hexadiene-based EPDM and one that minimizes the degree of modification to the existing formulation methods and also as to the ultimate functional properties of the STW4-2868, DL1375, DL 1552A, 053A and R196 thermal insulations.

In accordance with the principles of this invention, these and other objects of the invention are attained by the

discovery and provision of a rocket motor insulation formulation comprising, as a primary or secondary terpolymeric base, an EPDM terpolymer formed from at least one alkylidene norbornene, especially ethylidene norbornene (ENB) as the diene component.

Exemplary EPDM terpolymers that may be used according to this invention comprise those having an alkylidene diene, particularly an ENB diene, component include KELTAN 4506, KELTAN 1446A, KELTAN 2308, NORDEL IP NDR-4520, and NORDEL IP NDR-4640, each of which may be substituted into the STW4-2868, DL1375, R196 and 053A insulation for the NORDEL 1040 without requiring significant modifications to the standard STW4-2868, DL1375, 053A, DL1552A and R196 thermal insulation formulation methods nor as to the resulting multitude of functionally acceptable properties. Other exemplary terpolymers include high-ethylene-content EPDM terpolymers formed from an ENB diene component are NORDEL IP NDR-3722p and BUNA EP T 2370, which may be substituted into the DL1552A for the NORDEL IP NDR-2722E without requiring significant modifications to the DL1552A formulation. Nordell IP NDR-3725 has also been used but the supplier (du Pont) has indicated that due to low demand it now prefers a different formulation, Nordel IP NDR-3722, with a lower diene content of about 0.5% versus about 2.5% for Nordel IP NDR-3725p.

It has now been found that only a small proportion of ENB diene component is sufficient for incorporation in such elastomers, say from about 2 to about 10 wt. %, preferably from about 2 to about 7 wt. %, and with the balance of the olefin content of the composition composed of ethylene and propylene, with the ethylene forming from about 40 to about 80 wt. %, preferably from about 50 to about 75 wt. %, and with the remainder being propylene. Trace amounts of other dienes may also be present to induce branching in the elastomer. Generally, the only significant modification that is required involves the selection of a less reactive curing agent to offset the higher reactivity (relative to HD) of ethylidene norbornene (ENB). Furthermore, NORDEL IP NDR-3722 and BUNA EP T 2370 are not presently foreseen as being susceptible to obsolescence issues.

Other objects, aspects and advantages of the invention will be apparent to those skilled in the art upon reading the specification and appended claims which, when read in conjunction with the accompanying drawings, explain the principles of this invention.

BRIEF DESCRIPTION OF THE DRAWINGS

The accompanying drawings serve to elucidate the principles of this invention. In such drawings:

FIG. **1** is a schematic cross-sectional view of a rocket motor depicting various regions in which the insulation of this invention may be applied;

FIG. **2** is a schematic of a char motor suitable for conducting the ablation tests reported herein; and

FIG. **3** is a cut-away view of a solid propellant rocket motor illustrating the outer case, the internal insulation layer, the propellant grain, and the insulation of the ignitor closure structure.

DETAILED DESCRIPTION OF THE DRAWINGS

As shown in schematic FIG. **1**, and its components in FIGS. **1***a*–**3***b*, illustrate the manner in which the inventive insulation may be applied to various parts of a rocket assembly. These include, but are not limited to, the system tunnel floor plate shear ply **10**, the LSC blast shield **12**, the stiffener stub hole plugs **14**, stiffener stub insulation **16**, the external joint weatherseal **18**, T-ring insulation **20**, aft dome internal insulation **22**, and center segment aft end internal insulation (underneath the flap) **24**.

In FIG. **2**, the schematic cross-section of a char motor test assembly is illustrated wherein the propellant is contained in the beaker **30**, and low velocity insulation test specimens located at **32** upstream of the throat **34**, with medium velocity test specimens located in the section at **36** and with high velocity insulation test specimens located in the region **38**. Generally, such a char test motor assembly permits the location of a plurality of different insulation formulation test specimens about the circumference at any of locations **32**, **36** or **38**, in the conventional manner.

FIG. **3** is a cut-away schematic view of a typical rocket motor illustrating the case **40**, the nozzle **42**, the propellant grain **44** with its center bore **46** and the internal insulation layer shown at **48**. The insulation of the ignitor closure structure is indicated at **50**.

DETAILED DESCRIPTION OF THE INVENTION

In accordance with one embodiment of this invention, the inventive rocket motor insulation formulation comprises, as a primary polymeric base, an EPDM terpolymer in which the diene component of the EPDM is composed of at least one alkylidene norbornene, and in particular ethylidene norbornene (ENB).

The selected EPDM terpolymer should be substitutable into the STW4-2868, DL1375, 053A, DL1552A and R196, thermal insulation formulation (Tables 1 and 2) without requiring significant modification of the present techniques employed for the formulation thereof. It is a further highly desirable feature of the present invention that the insulation formulation may be composed of readily available commercial materials, provided that such compositions are properly assembled and blended together for the final insulation material.

Suitable EPDM terpolymers having an ENB diene component for use in this invention include, without limitation, and as stated above, KELTAN 4506, KELTAN 1446A, KELTAN 2308, each of which is available from DSM of the Netherlands, and NORDEL IP 4520 and NORDEL IP 4640, both of which are and continue to be available from DuPont Dow Elastomers.

These materials have the following respective contents as derived from the manufacturer's data literature:

	ENB Content Wt %	Ethylene Content Wt %	Mooney Viscosity
Keltan 4506	4.5	54	40
Keltan 1446A	7	59	10
Keltan 2308	2	74	24
Nordel IP 4520	5	51	20
Nordel IP 4640	5	55	40

The remaining content is propylene with traces of certain dienes used to produce branching in the molecular structure.

In accordance with another preferred embodiment, the secondary EPDM terpolymer is/are NORDEL IP NDR-3722 and/or Buna EP T 2370, which include ENB as their diene monomers and are respectively available from DuPont Dow and Bayer Corporation and manufactured in relatively large capacities.

An exemplary formulation is set forth in TABLE 5:

TABLE 5

Ingredient	Parts by Weight
Primary EPDM terpolymer	70–80
Secondary EPDM terpolymer with ENB diene monomer	15–25
Tackifier	5–10
Antioxidant	1–3
Wetting agent	0–1
Curing activator	5–10
Silica filler	40–50
Pigment	0–3
Plasticizer	15–25
Curing agent	10–20

The primary EPDM preferably has a sufficiently high diene content to provide a more reactive polymer to decrease cure time. Preferably, the alkylidene diene content of the primary EPDM is in a range of about 2–12 wt %, more preferably about 10–12 wt %. Additionally, the primary EPDM terpolymer preferably has a medium ethylene content of from about 56 wt % to about 65 wt %.

In a more preferred embodiment, the primary and secondary terpolymer components, tackifier, antioxidant, wetting agent, curing activator, filler, pigment, plasticizer, and silica are the same as those set forth in Table 3 above and are present in the concentrations specified in Table 3. However, when following that formulation composition on simply a "drop in" basis, it was observed that an unsatisfactory scorch characteristic developed (e.g. a scorch time of about 5.9 minutes as against and 8 minute minimum time allowed per specification. Similarly, substituting Buna EP T 2370 in place of Nordel 2722E led to similar results of an unsatisfactory scorch time of 6.6 minutes and a high Mooney viscosity of 119. Consequently, according to this invention, a less reactive brominated phenolic resin curing agent, for instance, having a lower reactivity than that used in the DL1552A formulation (i.e., SP 1056 containing about 6 wt % bromine) was required and was selected in order to compensate for the observed higher reactivity of ENB. This then led to acceptable scorch characteristics and the use of cure temperatures of about 320° F. provided similar cure rates as were used for DL 1552A. The resulting physical properties were also acceptable. Preferably, the resin curing agent of the reformulated insulation formulation is SP 1055 (manufactured by Schenectady International), which contains about 3% bromine by weight.

It will be observed that these materials may have significant variations in specific norbornene content, and also in the ethylene/propylene content ratio as well as in the Mooney viscosity, yet each has been found to be effective if selected for use in production of a rocket motor insulation.

Adhesion-promoting secondary polymers that may also be used in the formulation include elastomer modifiers, especially polar polymers. Among suitable such secondary polymers are chlorosulfonated polyethylene, such as Hypalon 20 from DuPont Dow, and polychloroprene. Polychloroprene polymers are available from DuPont Dow under the tradenames Neoprene FB, Neoprene TW, and Neoprene GRT.

An exemplary plasticizer for the inventive formulation is the EPDM-based Trilene 67A (Uniroyal).

Tackifiers may also optionally be used. An example of a suitable tackifier is Akrochem P-133.

One or more antioxidants are also preferably included within the inventive insulation formulation. Preferred antioxidants include polymerized 1,2-dihydro-2,2,4-trimethylquinoline (Agerite Resin D) and mixed octylated diphenylamines (Agerite Stalite S), each of which is available from R. T. Vanderbilt Co.

Various powder silica fillers are well known in other elastomeric combinations and may be used, including HiSil 233.

An exemplary carbon fiber is VMC carbon fiber.

Suitable cure activators may include metal oxides, such as zinc oxide and magnesium oxide (e.g., Elastomag 170, from Morton Chemical Co.).

The curing package preferably includes at least one phosphate cure accelerator, including by way of example, Rhenocure AP-5, Rhenocure AP-7, Rhenocure AP-3, Rhenocure ZADT/G, and Rhenocure S/G, which are available from Rhein Chemie and Accelerator VS, available from Akro Chem. Additional cure accelerators that may be used in combination with the phosphate cure accelerator include butyl zimate, Altax, Akroform Delta P.M., Sulfads. While the use of Accelerator VS was initially unacceptable because of the foul odor problem it generated, it has also been now found that such formulations can be prepared with no significant odor when about 1.0 phr magnesium oxide is added thereto.

Sulfur curing agents are preferred for the formulation. A suitable sulfur-curing agent is Akrosperse IS-70 from Akro Chem. Elemental sulfur can also be used.

Batches of insulation containing silica powder generally may be formulated in an internal mixer with the following two-pass mix procedure. In the first pass, the mixer speed may be set to approximately 40 rpm and the all of the components other than the curing agents and accelerators are added to the internal mixer. (The silica was added last in the first pass.) The mixing may be performed at a temperature of about 300° F. Suitable mixing times depend on the temperature and mixing speed, and are ascertainable to the skilled artisan without an undue amount of experimentation.

In the second pass, the mixer speed was set to about 40 rpm. In a sequential manner, half of the master batch was added to the mixer, then the curatives were added, then the remaining half of the master batch was added to the mixer. The second mixing step was performed at a temperature of approximately 180° F. to 190° F. The material was sheeted out onto the laboratory mill and allowed to cool to room temperature prior to evaluating its rheological and physical properties.

The shaping and curing of the inventive insulation may be performed in accordance with techniques known in the art.

Exemplary new formulations containing a silica powder filler are set forth in TABLE 6 below with concentrations shown by weight.

TABLE 6

EXAMPLES OF SILICA FILLED EPDM INSULATION FORMULATIONS

Ingredient	RDL5338	RDL5342	RDL5343	RDL5347	RDL5363	RDL5255	RDL5335	RDL5201	RDL5220	RDL5370
Keltan 4506	70.0	73.0	73.0	55.0	55.0					
Keltan 1446A	20.0	17.0	17.0							
Keltan 2308				35.0	35.0					
Nordel IP 4520								80.0	80.0	80.0
Nordel IP 4640						80.0	80.0			
Hypalon 20	10.0	10.0	10.0	10.0	10.0					
Neoprene FB						20.0	20.0			
Neoprene TW								20.0	20.0	
Neoprene GRT										20.0
Trilene 67A										
Kraton L-2203										
Akrochem P-133	5.0	5.0	5.0	5.0	5.0			5.0	5.0	5.0
Agerite Resin D						2.0	2.0	2.0	2.0	2.0
Agerite Stalite S	2.0	2.0	2.0	2.0	2.0					
HiSil 233	35.5	35.5	35.5	35.5	35.5	35.5	35.5	37.0	37.0	37.0
Elastomag 170								0.5	0.5	0.50
Zinc Oxide	3.0	3.0	3.0	3.0	3.0	4.0	4.0	4.0	4.0	4.00
Butyl Zimate	0.50	0.50							0.50	
Rhenocure AP-5	3.50	3.50							3.50	
Rhenocure AP-7										
Rhenocure AP-3						2.90		3.85		3.85
Rhenocure ZADT/G						0.80		1.00		1.00
Rhenocure S/G						0.60		0.80		0.80
Altax			1.10	1.10	1.20		1.10			
Akroform Delta P.M.					0.45		0.20			
Sulfads					0.50					
Accelerator VS			2.70	2.70						
Akrosperse IS-70	1.10	1.10	1.20	1.20	1.40				1.00	
Sulfur						0.35	0.45	0.40		0.40

Ingredient	RDL5380	RDL5381	RDL5194	RDL5222	RDL5273A	RDL5298	RDL5277	RDL5279	RDL5319	RDL5320A
Keltan 4506										
Keltan 1446A										
Keltan 2308										
Nordel IP 4520	80.0	80.0	90.0	90.0	90.0	80.0			40.0	40.0
Nordel IP 4640							90.0	90.0	50.0	50.0
Hypalon 20			10.0	10.0	10.0	10.0			10.0	10.0
Neoprene FB										
Neoprene TW										
Neoprene GRT	20.0	20.0								
Trilene 67A						10.0				
Kraton L-2203							10.0	10.0		
Akrochem P-133	5.0	5.0	5.0	5.0	5.0	5.0	9.0	9.0	5.0	5.0
Agerite Resin D	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0		
Agerite Stalite S									2.0	2.0
HiSil 233	37.0	37.0	35.5	35.5	35.5	36.5	38.0	38.0	35.5	35.5
Elastomag 170	0.50	0.50								
Zinc Oxide	4.00	4.00	3.00	3.00	4.00	3.00	3.00	3.00	3.0	4.0
Butyl Zimate	0.50			0.40		0.15			0.40	
Rhenocure AP-5	3.40			3.15		4.45			3.15	
Rhenocure AP-7							3.00			
Rhenocure AP-3			3.85							
Rhenocure ZADT/G			1.00				0.90			
Rhenocure S/G			0.80							
Altax		1.00			1.00			1.20		1.00
Akroform Delta P.M.								0.25		
Sulfads								0.82		
Accelerator VS		2.70			2.70					2.70
Akrosperse IS-70				0.95	1.10	1.34	1.25	1.40	1.00	1.00
Sulfur	0.50	0.50	0.40							

Ingredient	RDL5350	RDL5351A	RDL5367	RDL5185	RDL5186	RDL5205	RDL5276
Keltan 4506							
Keltan 1446A							
Keltan 2308							
Nordel IP 4520	45.0	45.0	45.0				
Nordel IP 4640	45.0	45.0	45.0	80.0	80.0	80.0	80.0
Hypalon 20	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Neoprene FB							
Neoprene TW							
Neoprene GRT							
Trilene 67A				10.0	10.0	10.0	10.0
Kraton L-2203							

TABLE 6-continued

EXAMPLES OF SILICA FILLED EPDM INSULATION FORMULATIONS

Akrochem P-133	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Agerite Resin D				2.0	2.0	2.0	2.0
Agerite Stalite S	2.0	2.0	2.0				
HiSil 233	35.5	35.5	35.5	35.5	35.5	35.5	35.5
Elastomag 170							
Zinc Oxide	3.0	4.0	3.0	3.0	3.0	3.0	3.0
Butyl Zimate	0.40						0.40
Rhenocure AP-5	3.15						3.50
Rhenocure AP-7					3.00		
Rhenocure AP-3				3.85		3.85	
Rhenocure ZADT/G				0.80	0.80	1.00	
Rhenocure S/G				0.80		0.80	
Altax		1.00	1.10				
Akroform Delta P.M.			0.35				
Sulfads			0.50				
Accelerator VS		2.70					
Akrosperse IS-70	1.00	1.00	1.00				1.05
Sulfur				0.40	0.80	0.40	

In addition, individual batches of insulation formulations containing silica filler were prepared with NORDEL IP NDR-3725 (Example 1, Table 8 below), BUNA EP T 2370 (Example 2), and NORDEL 2722E (Comparative Example) as the secondary EPDM terpolymers having high ethylene contents. However, in these formulations it has been observed that there is an increased reactivity of the ethylidene norbornene monomers as used in Examples 1 and 2 (compared to the 1,4-hexadiene monomer of the Comparative Example A), and accordingly, SP 1055 (3 wt % bromine) was used in Examples 1 and 2 and SP 1056 (6 wt % bromine) was used in the Comparative Example A (Table 8) in equal quantities for comparison purposes. In addition, another formulation, RDL 5654 was also prepared in much the same way, but substituting NORDEL IP NDR-3722p for NORDEL IP NDR-2722E, and SP 1055 was used in place of SP 1056.

TABLE 7

Polymer	Ethylene content %	Propylene content %	Diene content % type	Viscosity
NORDEL IP NDR-3722	71	26.5	2.5/ENB	25
BUNA EP T 2370	71	26	3/ENB	16
NORDEL 2722E	72	22	6.4/HD	26

The batches were each formulated in a laboratory scale Reliable Rubber & Plastics Machinery Company Model R-260 internal mixer having a net chamber volume of 4260 cubic centimeters. A 3000 gram batch was prepared by a two-pass mix procedure.

In the first pass, the mixer speed was set at 40 rpm, and the primary and secondary polymer components, tackifier, and antidegradant were added to the mixing chamber in the concentrations set forth in Table 3 and masticated for one minute. Subsequently, the remaining ingredients were added (same concentrations as in Table 3), and the mixer speed was increased to 60 rpm. The batches were removed from the internal mixer after mixing for a total of approximately 7 minutes. The temperature at the time of removing the batch was 300° F. to 320° F. The master batch was sheeted out on a 6"×13" Farrel Corporation laboratory two-roll mill and allowed to cool to room temperature.

In the second pass, the mixer speed was set to 40 rpm. In a sequential manner, half of the master batch was added to the mixer, then the curatives were added (same concentrations as set forth in Table 3), then the remaining half of the master batch was added to the mixer. After a total mix time of approximately 40 seconds, the final mix was removed from the mixer. The actual temperature at the time of removal was approximately 200° F. The material was sheeted out onto the laboratory mill and allowed to cool to room temperature prior to evaluating its rheological and physical properties.

The rheological properties of these several formulations are reported in Table 8.

TABLE 8

RHEOLOGICAL PROPERTIES (silica filler)

Property	Example 1 [RDL5294]	Example 2 [RDL5331]	RDL5654	Comparative Example A
(1) Mooney viscosity (ML 1 + 4 at 212° F.)(ASTM D 1646)	63.4	61.0	65.4	68.8
(2) Mooney scorch (MS + 1 at 250° F., min.)(ASTM D 1646)	21.5	18.1	27.1	11.8
(3) Oscillating disk rheometer (ODR at 320° F., 5° arc)(ASTM D 2084) properties:			—	
(a) ML (minimum torque, in.-lb.) (ASTM D 2084)	17.8	16.4	17.1	21.4

TABLE 8-continued

RHEOLOGICAL PROPERTIES (silica filler)

Property		Example 1 [RDL5294]	Example 2 [RDL5331]	RDL5654	Comparative Example A
(b)	MH (maximum torque, at 2 hrs, in.-lb.)(ASTM D 2084)	91.0	95.0	86.8	97.7
(c)	Ts2, (time to 2 point rise above minimum viscosity, in min.) (ASTM D 2084)	2.3	1.8	2.4	1.5
(d)	MC(90)(in.-lb.)(ASTM D 2084)	83.7	87.1	79.8	90.1
(e)	Tc (90)(min)(ASTM E 2084)	74.0	73.5	77.1	75.5

The physical characteristics of these formulations are reported as follows in Table 9.

TABLE 9

PHYSICAL PROPERTIES

Properties	Specification Requirement	Example 1 [RDL5294]	Example 2 [RDL5331]	RDL 5654	Comparative Example A
Shore A hardness (ASTM D 2240)	69–79	73.8	74.2	72.4	71.8
Ash content (%) (ASTM D 297)	20–26	22.6	22.4	22.2	22.7
Specific gravity (ASTM D 792)	1.04–1.07	1.051	1.058	1.056	1.054
Tensile strength, perpendicular (psi; ASTM D 412)	1450 min.	2420	2480	2210	2280
Elongation parallel (%; ASTM D 412)	450 min	703	693	643	636
100% modulus (psi)	—	347	370	377	371
Tear resistance (pli) (ASTM D 624)	170 min.	233	238	230	221
Specific heat (BTU $(lb.)^{-1}(° F.)^{-1}$ (ASTM E 1269)	—	0.445	0.442	—	0.443
Thermal conductivity (BTU $(ft)^{-1}(in.)^{-1}(° F.)^{-1}$ (ASTM E 1225)	—	0.134	0.133	—	0.129

Measurements were also made of the resulting ablation rates for these formulations.

TABLE 10

MATERIAL ABLATION RATE (MILS/SECOND)

	Example 1	Example 2	Comparative Example A
Low Mach Region (0.0028)	3.00	3.13	3.40
Medium Mach Region (0.0318–0.0386)	7.34	7.94	7.60
High Mach Region (0.0397–0.0634)	19.60	17.37	20.11

The ablation tests for Table 10 were performed as follows. The tests were performed in a char motor, schematically illustrated in FIG. **2**. The samples for the three velocity regions of the motor (low, medium, and high Mach) were net molded using conventional compression molding techniques. The low Mach region had space for 15 specimens, while the medium and high Mach regions had space for 4 specimens. The thickness of each specimen was measured at several axial locations before and after firing. The char motor itself featured a propellant beaker filled with Thiokol Propulsion propellant TP-H1148 to a sufficient depth to provide for a 12-second burn with a tungsten nozzle radius of 0.24 inches. The actual action time and average operating pressure for the test motor were 11.7 seconds and 936 psi, respectively.

The cure characteristics and processability of the inventive Examples closely matched those of the Comparative Example A. The cure rates, as indicated by the 90 percent cure time tc(90), of Examples 1 and 2 were equivalent to that of the Comparative Example A. Similarly, the physical properties and ablative performance of the materials prepared in accordance with Examples 1 and 2 were substantially equivalent to those of the Comparative Example and were well within the specification requirements. Thus these results indicate that the new formulations have the capability of being substituted for the old standard to-be-discontinued insulations.

In addition, a comparison was made between DL1552A and RDL 5654 in a low mach test char motor using TP-H1148 propellant and a 30.8 second firing time at an average pressure of 881 psi. The following results indicate that RDL 5654 is a viable replacement for DLI 552A.

TABLE 10A

COMPARISON OF THE MATERIAL AFFECTED RATE (MAR) OF DL1552A AND RDL5654 IN THE LOW MACH CHAR MOTOR

	Mach No. Range	D-5 DL1552A Average MAR (mils/sec.)	RDL5654 Average MAR (mils/sec.)
Test motor		HPCAT-07	MIR-05
Low velocity test section	0.0027	3.83	3.86
Medium velocity test section	0.0027–0.0081	3.79	3.80
High velocity test section	0.0094–0.044	5.36	5.49

In the following TABLE 11 there are set forth examples of the new EPDM insulation formulations but now containing carbon fibers.

Batches of carbon fiber containing insulations generally may be formulated in a mixer via a two-pass mix procedure. In the first pass, the mixer speed may be set to approximately 40 rpm and the all of the components other than the curing agents, accelerators, and carbon fiber are added to the internal mixer. As the mixing proceeds, the temperature will increase up to about 300° F., or even higher. Suitable mixing times depend on the temperature and mixing speed, and are ascertainable to the skilled artisan without an undue amount of experimentation.

In the second pass, the mixer speed may be set to about 40 rpm., but this speed is dependent on the size of the mixer. In a sequential manner, half of the master batch was added to the mixer, then the curatives were added, then the remaining half of the master batch was added to the mixer. The second mixing step may be performed at a temperature rising to approximately 180° F. to 190° F. but below the temperature where the accelerator would become unduly activated. The material was sheeted out onto the laboratory mill and allowed to cool to room temperature. Then the material is dissolved in a suitable solvent, such as, by way of example, hydrocarbons such as hexane, heptane, and/or cyclohexane. The carbon fiber was then mixed with, for example, a sigma-blade mixer. The material is then sheeted out and the solvent allowed to evaporate at ambient atmosphere or in an oven. Throughout this process care must be taken that the frangible carbon fibers are not themselves fractured and broken up so as to become shortened and less effective as a result.

The shaping and curing of the inventive insulation may be performed in accordance with techniques known in the art.

The following Table 11 presents a number of examples of the novel EPDM formulations with carbon fibers.

TABLE 11

EXAMPLES OF NEW EPDM FORMULATIONS WITR CARBON FIBER

INGREDIENT	RDL5421	RDL5420A	RDL5377A	RDL5444A	RDL5443	RDL5436	RDL5408	RDL5409
Keltan 4506								
Keltan 1446A								
Keltan 2308								
Nordel IP 4520	40.0	40.0	45.0	45.0	45.0		80.0	80.0
Nordel IP 4640	50.0	50.0	45.0	45.0	45.0	80.0		
Hypalon 20	10.0	10.0	10.0	10.0	10.0	10.0		
Neoprene FB								
Neoprene TW								
Neoprene GRT							20.0	20.0
Trilene 67A						10.0		
Akrochem P-133	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Agerite Resin D						2.0	2.0	2.0
Agerite Stalite S	2.0	2.0	2.0	2.0	2.0			
HiSil 233	3	3	3	3	3	3	3	3
VMC Carbon Fiber	All formulations adjusted to have 26.75% VMC Carbon Fiber in finished product							
C. B. N330	1	1	1	1	1	1	1	1
Elastomag 170							0.50	0.50
Zinc Oxide	4.0	5.0	5.0	5.0	4.0	4.0	4.00	4.00
Butyl Zimate	0.40		0.40					0.50
Rhenocure AP-5	3.15		3.15					3.40
Rhenocure AP-7						3.00		
Rhenocure AP-3							3.85	
Rhenocure ZADT/G						0.80	1.00	
Rhenocure S/G							0.80	
Altax		1.00		1.00	1.10			
Akroform Delta P.M.					0.35			
Sulfads					0.50			
Accelerator VS		2.70		2.70				
Akrosperse IS-70	1.00	1.00	1.00	1.00	1.00			
Sulfur						0.80	0.40	0.50

Ingredient	RDL5445	RDL5410	RDL5375	RDL5395	RDL5394	RDL5435	RDL5434	RDL5376A
Keltan 4506		70.0	73.0	55.0	55.0			
Keltan 1446A		20.0	17.0					
Keltan 2308				35.0	35.0			
Nordel IP 4520	80.0							80.0
Nordel IP 4640						80.0	80.0	
Hypalon 20	10.0	10.0	10.0	10.0	10.0			
Neoprene FB						20.0	20.0	
Neoprene TW								20.0

TABLE 11-continued

EXAMPLES OF NEW EPDM FORMULATIONS WITR CARBON FIBER								
Neoprene GRT								
Trilene 67A	10.0							
Akrochem P-133	5.0	5.0	5.0	5.0	5.0			5.0
Agerite Resin D	2.0					2.0	2.0	2.0
Agerite Stalite S		2.0	2.0	2.0	2.0			
HiSil 233	3	3	3	3	3	3	3	3
VMC Carbon Fiber	All formulations adjusted to have 26.75% VMC Carbon Fiber in finished product							
C. B. N330	1	1	1	1	1	1	1	
Elastomag 170								0.5
Zinc Oxide	4.0	4.0	4.0	4.0	4.0	4.0	4.0	5.0
Butyl Zimate	0.15	0.50						0.50
Rhenocure AP-5	4.45	3.50						3.50
Rhenocure AP-7								
Rhenocure AP-3						2.90		
Rhenocure ZADT/G						0.80		
Rhenocure S/G						0.60		
Altax			1.10	1.10	1.20		1.10	
Akroform Delta P.M.					0.45		0.20	
Sulfads					0.50			
Accelerator VS			2.70	2.70				
Akrosperse IS-70	1.34	1.10	1.20	1.20	1.40			1.00
Sulfur						0.35	0.45	

Silica Powder Filled Insulation Properties

TABLE 12 reports rheological properties measured for the silica powder thermal insulation formulations that were set forth in TABLE 6.

TABLE 12

RHEOLOGICAL PROPERTIES (silica powder)

Silica-Filled EPDM Formulation	Mooney viscosity (ML 1+4 at 212° F. (ASTM D 1646)	Oscillating disk rheometer (ODR at 320° F., 5° arc) (ASTM D 2084) ML (minimum torque, in.-lb.)	MH maximum torque, at 1 hrs, in.-lb.)	ts2, (time to 2 point rise above ML, in min.)	Tc (90) (min)
RDL 5338	72.0	17.7	93.9	6.0	43.5
RDL 5342	78.5	21.3	97.8	7.0	45.0
RDL 5343	78.8	19.9	98.0	4.3	44.5
RDL 5347	84.0	21.7	94.5	3.4	38.0
RDL 5363	83.0	22.9	87.2	3.3	44.2
RDL 5255	85.7	19.4	103.2	3.6	28.5
RDL 5335	87.7	23.4	93.4	2.7	38.5
RDL 5201	73.0	16.4	89.0	6.2	31.5
RDL 5220	71.2	16.0	110.0	5.9	40.0
RDL 5370	80.0	20.3	104.6	5.6	32.0
RDL 5380	80.3	21.0	107.4	7.3	56.0
RDL 5381	79.6	21.0	104.0	4.8	36.0
RDL 5194	62.0	13.4	90.9	8.7	44.0
RDL 5222	61.9	13.5	92.8	6.3	45.6
RDL 5273A	64.5	14.7	97.1	3.8	42.0
RDL 5298	51.0	11.0	91.0	7.1	49.0
RDL 5277	72.4	18.9	98.5	1.9	42.6
RDL 5279	75.0	17.3	93.0	2.7	18.8
RDL 5319	87.8	19.9	97.0	6.3	43.0
RDL 5320A	86.5	20.7	103.0	4.0	41.6
RDL 5350	83.0	22.0	103.0	6.8	46.1
RDL 5351A	84.6	19.8	105.6	3.5	37.0
RDL 5367	86.0	22.3	96.7	3.2	43.3
RDL 5185	79.5	18.7	90.0	8.4	40.9
RDL 5186	80.6	19.3	95.5	4.1	43.0
RDL 5205	79.9	19.4	94.0	8.0	41.2
RDL 5276	81.6	18.5	96.7	6.2	42.5

TABLE 13 reports various physical properties measured for the silica powder thermal insulation formulations that were set forth in TABLE 6.

TABLE 13

PHYSICAL PROPERTIES

Silica-Filled EPDM Formulation	A	B	C	D	E	F	G
RDL 5338	2280	646	2260	658	220	62.0	1.06
RDL 5342	1910	588	1790	582	206	63.4	1.06
RDL 5343	2330	655	2040	619	221	63.2	1.06
RDL 5347	2100	621	2060	630	271	68.6	1.06
RDL 5363	2920	610	2820	608	270	72.0	1.06
RDL 5255	2430	703	2330	695	223	69.4	1.10
RDL 5335	2560	725	2600	739	232	65.2	1.10
RDL 5201	2150	772	2220	779	190	70.2	1.10
RDL 5220	2170	634	2020	620	194	70.2	1.10
RDL 5370	2550	700	2340	688	201	70.4	1.10
RDL 5380	2610	688	2300	663	206	69.4	1.11
RDL 5381	2420	700	2470	701	207	69.0	1.11
RDL 5194	1920	734	1920	748	201	71.8	1.05
RDL 5222	2100	703	1960	653	218	71.2	1.05
RDL 5273A	1420	612	1430	613	241	70.4	1.06
RDL 5298	1910	592	1870	586	223	71.6	1.06
RDL 5277	2010	614	2190	632	181	66.4	1.05
RDL 5279	2390	672	2370	687	199	64.8	1.05
RDL 5319	2440	659	2270	647	205	71.2	1.05
RDL 5320A	2250	657	2220	653	224	71.6	1.06
RDL 5350	2350	688	2420	707	233	64.2	1.06
RDL 5351A	2270	681	2250	699	226	65.2	1.06
RDL 5367	2440	621	2430	625	238	70.4	1.06
RDL 5185	2310	776	2230	771	208	71.4	1.06
RDL 5186	2390	725	2290	718	221	71.6	1.06
RDL 5205	1990	729	2120	773	207	71.6	1.06
RDL 5276	2270	653	2210	656	223	71.2	1.06

Key:
A = Tensile strength, parallel (psi)(ASTM D 412)
B = Elongation Parallel (%)(ASTM D 412)
C = Tensile Strength Perpendicular (ASTM D 412)
D = Elongation Perpendicular (ASTM D 412)
E = Tear Strength (ASTM D 412)
F = Shore A hardness (ASTM D 2240)
G =Specific gravity (ASTM D 792)

Carbon-Fiber Containing Insulation Formulation Properties

Next, Table 14 lists the rheological properties measured according to the indicated tests for the carbon fiber-containing insulation formulations set forth in TABLE 11.

TABLE 14

RHEOLOGICAL PROPERTIES

Carbon-Fiber EPDM Formulation	Mooney viscosity (ML 1 + 4 at 212° F.) (ASTM D 1646)	Oscillating Disk Rheometer (ODR at 300° F., 5° arc) (ASTM D 2084) ML (minimum torque, in.-lb.)	MH (maximum torque, at 1 hr, in.-lb.)	ts2, (time to a 2 point rise above ML; in min.)	Tc (90% cure) (min)
RDL 5421	72.4	23.3	111	3.5	30.2
RDL 5420A	70.0	21.2	119	2.9	37.0
RDL 5377A	68.0	18.8	102	2.8	36.5
RDL 5444A	67.4	21.0	110	3.2	28.0
RDL 5443	74.5	25.7	101	1.6	33.5
RDL 5436	80.0	25.8	98.2	2.6	32.0
RDL 5408	83.0	29.2	95.5	2.5	16.5
RDL 5409	83.8	25.8	99.0	3.5	25.5
RDL 5445	56.0	15.8	82.0	3.4	39.2
RDL 5410	81.0	18.6	103	4.8	34.5
RDL 5375	55.0	21.0	100	2.5	31.7
RDL 5395	64.7	29.2	87.4	3.2	35.0
RDL 5394	67.0	25.1	89.5	1.8	36.0
RDL 5410	81.0	18.0	103	4.8	34.5
RDL 5434	43.7	12.1	136	1.9	18.8
RDL 5435	39.0	9.1	118	2.3	19.0

Table 15 reports the physical properties measured for the carbon fiber-containing insulation formulations set forth in TABLE 11.

TABLE 15

PHYSICAL PROPERTIES

Carbon-Fiber EPDM Form.	A	B	C	D	E	F
RDL 5421	1750	3.34	820	4.59	84.4	1.097
RDL 5420A	1600	3.12	871	4.60	84.2	1.106
RDL 5377A	1540	3.11	814	4.03	82.6	1.072
RDL 5444A	2030	5.01	884	6.30	84.2	1.097
RDL 5443	2070	5.37	799	7.21	83.2	1.090
RDL 5436	1500	2.48	721	4.05	84.0	1.098
RDL 5408	1060	3.77	525	5.61	81.2	1.131
RDL 5409	1112	2.95	668	3.35	83.4	1.142
RDL 5445	1160	3.58	725	4.71	84.4	1.096
RDL 5410	1500	2.88	719	3.85	83.8	1.063
RDL 5375	2010	4.38	881	6.85	80.6	1.076
RDL 5395	2650	4.55	1090	4.98	86.1	1.078
RDL 5394	2218	3.80	982	4.84	87.2	1.088
RDL 5435	1690	4.17	905	6.03	83.4	1.127
RDL 5434	1470	4.41	786	5.82	83.6	1.126
RDL 5410	1500	2.88	719	3.85	83.8	1.063
RDL 5435	1690	4.17	905	6.03	83.4	1.127
RDL 5434	1470	4.41	786	5.82	83.6	1.126

Key:
A = Tensile strength, parallel (psi) (ASTM D 412)
B = Elongation Parallel (%) (ASTM D 412)
C = Tensile Strength Perpendicular (ASTM D 412)
D = Elongation Perpendicular (ASTM D 412)
E = Shore A hardness (ASTM D 2240)
F = Specific gravity (ASTM D 792)

The following TABLE 16 shows the results of ablation tests with the silica powder-containing formulations of Tables 6, 12 and 13.

TABLE 16

MATERIAL ABLATION RATE (MILS/SECOND)

Silica powder Filled	Low Velocity, 0.003 Mach
RDL 5338	3.65
RDL 5343	3.14
RDL 5347	2.31
RDL 5363	2.82
RDL 5255	2.76
RDL 5335	2.42
RDL 5220	3.66
RDL 5370	3.44
RDL 5380	3.24
RDL 5298	3.36
RDL 5319	3.23
RDL 5320	2.81
RDL 5350	3.38
RDL 5351	3.35
RDL 5367	3.31
RDL 5186	2.34

The ablation tests were performed as follows. The tests were performed in a char motor, schematically illustrated in FIG. **2**. The samples for the three velocity regions of the motor (low, medium, and high Mach) were neat molded using conventional compression molding techniques. The low Mach region had space for 15 specimens, while the medium and high Mach regions had space for 4 specimens. The thickness of each specimen was measured at several axial locations before and after firing. The char motor itself featured a propellant beaker filled with Thiokol Propulsion propellant TP-H1148 to a sufficient depth to provide for a 12-second burn with a tungsten nozzle radius of 0.24 inches. The actual action time and average operating pressure for the test motor were 12.1 seconds and 860 psi, respectively.

The following TABLE 17 shows the results of ablation tests with the carbon fiber-containing formulations of Tables 11, 14 and 15.

TABLE 17

MATERIAL ABLATION RATE (MILS/SECOND)

Carbon fiber EPDM	Low Velocity, 0.003 Mach	Medium Velocity, Avg. 0.03–0.09 Mach	High Velocity, Avg. 0.01–0.15 Mach
RDL 5421	3.38	11.4	18.7
RDL 5420	2.87	12.2	19.2
RDL 5377	3.86	11.5	17.6
RDL 5444	3.29	10.7	18.4
RDL 5443	3.22	12.5	19.9
RDL 5436	2.63	11.0	15.3
RDL 5408	3.91	11.2	16.5
RDL 5409	4.12	10.6	15.5
RDL 5410	3.55	10.9	19.9
RDL 5375	2.97	12.3	16.0
RDL 5395	3.00	11.7	20.8
RDL 5394	3.05	11.7	20.2
RDL 5435	3.90	11.4	15.1
RDL 5434	3.90	11.3	15.3
RDL 5376	4.27	12.9	14.7
RDL 5445	3.45	11.3	18.3

The ablation tests were also performed, as follows. The tests were performed in a char motor, schematically illustrated in FIG. **2**. The samples for the low velocity region of the motor was molded using conventional compression molding techniques. The low Mach region had space for 15 specimens. The thickness of each specimen was measured at

several axial locations before and after firing. The char motor itself featured a propellant beaker filled with Thiokol Propulsion propellant TP-H1148 to a sufficient depth to provide for a 12-second burn with a tungsten nozzle radius of 0.24 inches. The actual action time and average operating pressure for the test motor were 12.1 seconds and 860 psi, respectively.

From the characteristics measured in these tables it can be seen that a special combination of properties is required for a suitable insulation. And it is important to the consideration of this invention that the formulations are effective as to both of the commonly used solid additives silica powder and carbon fibers.

With reference to the results set forth in the foregoing tables it is presently considered in Table 13 (silica filled EPDM) that the minimum acceptable values for tensile strength (parallel and perpendicular, values A and C) should be at least about 1600, and are preferably close to equal (within about plus or minus 10%). The elongation (parallel and perpendicular, Table 13) should be in the range of about 550–850. For tear strength, the value of about 170 is presently seen as a minimum characteristic. Similarly, an ODR-measured maximum torque MH (Table 12) for the silica powder filled insulations should be at least about 85 in-lb. and at most about 120 in-lb., preferably at most about 115 in-lb. is currently thought to be appropriate. The ODR torque ML has a useful lower limit of about 5 in-lb., preferably about 10. The scorch test Ts result (Tables 12 and 14) should be at least 1.5. For silica filled insulations, the tear strength (Table 13) should be at least about 170. For carbon fiber containing insulations, the elongation should exhibit a minimum of at least about 2%, preferably at least about 2.5%, especially for measurement B in Table 15. Also, for the carbon fiber insulations (Table 14) the Mooney viscosity ML should be below about 90 Mooney units.

Considering the combined properties of the individual insulations noted in the above Tables, the silica-filled insulation 5273A is not presently preferred nor are the carbon-fiber containing insulations 5408, 5409, 5434 and 5445.

In addition, formulations were also made with using aramid fibers as the filler material. An exemplary EPDM terpolymer for use with Kevlar fibers and comprising ENB as its diene component is NORDEL IP NDR-4640 (available from DuPont Dow Elastomers), may be substituted into the R196 for the NORDEL 1040 without requiring significant modifications to the R196 formulation. Generally, the one significant modification that is required with the use of aramid fibers involves a reduction in the amount of the curing agent, e.g., peroxide, again to offset the increased reactivity (relative to HD) of the alkylidene norbornenes. Furthermore, NORDEL IP NDR-4640 is not presently foreseen as being susceptible to obsolescence issues.

Thus, in accordance with a further embodiment of this invention utilizing aramid fibers, the inventive rocket motor insulation formulation comprises, as a polymeric base, an EPDM/polyisoprene blend in which the diene component of the EPDM is based on at least one alkylidene norbornene, such as ethylidene norbornene (ENB), in effect in place of and without the above-described primary EPDM. The alkylidene norbornene content of such an EPDM may be from about 2–10 wt %. The selected EPDM terpolymer should be substitutable into the R196 thermal insulation formulation (Table 4) without requiring significant modification of the formulation. In accordance with a preferred embodiment, the EPDM terpolymer is NORDEL IP NDR-4640, which includes ENB as its diene monomer, is available from DuPont Dow Elastomers, and is manufactured in a relatively large capacity.

An exemplary aramid fiber formulation is set forth in TABLE 18:

TABLE 18

Ingredient	Parts by Weight
EPDM terpolymer with ENB diene monomer component	79.5–80.5
Polyisoprene	19.5–20.5
Antioxidant	0.95–1.05
Halogen-containing flame retardant	39.5–40.5
Metal oxide flame retardant	19.5–20.5
KEVLAR fiber (aramid staple fiber)	19.5–20.5
Curing agent	1.45–1.55

In a more preferred embodiment, the polyisoprene, antioxidant, flame retardants, and aramid, e.g. KEVLAR, fibers are the same as those set forth in Table 4 above and are present in the concentrations specified in Table 4. The metal oxide may be present in fine powdery form so as to also serve as a filler. Varox DBPH-50 is also a suitable peroxy curing agent, although the substitution of ethylidene norbornene for 1,4-hexadiene requires a reduction in curing agent concentration to offset the increased reactivity of ethylidene norbornene. By providing the reformulated thermal insulation formulation with a peroxide concentration in a range of from about 1 to about 2.5 phr, more preferably about 1.5 phr, curing effect is achieved similar to the properties of R196.

In general, the said EPDM/polyisoprene combination may be used with about 60–90 wt % of the norbornene EPDM and about 40–10 wt % polyisoprene, again with suitable additives being present in functionally desired amounts.

Suitable additives that may be added as functionally required or as desired include one or more of the following, in various combinations: fillers, antidegradants, curing agents, plasticizers, processing aids, and pigments, bonding agents, fibers, and flame retardants. Two classes of curing systems that may be used are sulfur based curing agents in combination with organic accelerators, and peroxide curing agents.

The shaping and curing of the such aramid inventive insulations may be performed in accordance with techniques known in the art.

Example of Aramid Fiber Insulations

Individual batches of insulation formulations were prepared USING NORDEL IP NDR-4640 (Example, in Table 20) and NORDEL IP NDR-1040 (Comparative Example B).

TABLE 19

Polymer	Ethylene content	Propylene content	Diene content/type	Viscosity
NORDEL IP NDR-4640	55	40	5/ENB	40
NORDEL 1040	55	41	4/HD	40

The batches were each formulated in a laboratory scale Reliable Rubber & Plastics Machinery Company Model R-260 internal mixer having a net chamber volume of 4260 cubic centimeters. A 3000 gram batch was prepared by a two-pass mix procedure.

In the first pass, the mixer speed was set at 40 rpm, and 80 parts by weight of the corresponding EPDM component set forth in Table 3, 20 parts by weight of NATSYN 2200 polyisoprene, and 1.0 parts by weight of Wingstay S were

added to the mixing chamber and masticated for one minute. Subsequently, 40 parts by weight of Dechlorane Plus 515, 20 parts by weight of Sb_2O_3, and 20 parts by weight of ¼ inch KEVLAR aramid fibers were added, and the mixer speed was increased to 60 rpm. The batches were removed from the internal mixer after mixing for a total of approximately 7 minutes. The temperature at the time of removing the batch was 300° F. to 320° F. The master batch was sheeted out on a 6"×13" Farrel Corporation laboratory two-roll mill and allowed to cool to room temperature.

For the Example and Comparative Example B, the fibers were dispersed in a two-roll mill to reduce agglomerations of fibers. The dispersion was accomplished by tightening the nip (separation between the two rolls) to 0.030 inches on the laboratory two-roll mill and passing the rubber through the nip a minimum of six times. This dispersion step was performed after the first pass but before the second pass. The rubber was then allowed to cool before the second pass.

In the second pass, the mixer speed was set to 40 rpm. In a sequential manner, half of the master batch was added to the mixer, then the curatives were added at a peroxide level of 1.5 phr, then the remaining half of the master batch was added to the mixer. After a total mix time of approximately 40 seconds, the final mix was removed from the mixer. The actual temperature at the time of removal was approximately 200° F. The material was sheeted out onto the laboratory mill and allowed to cool to room temperature prior to evaluating its rheological and physical properties.

Table 20 gives the measured rheological properties of these examples.

TABLE 20

RHEOLOGICAL PROPERTIES

Property		Example	Comparative Example B
(1) Mooney viscosity (ML 1 + 4 at 212° F.)(ASTM D 1646)		58.2	51.3
(2) Mooney scorch (MS + 1 at 270° F., min.)(ASTM D 1646)		52.0	27.8
(3) Oscillating disk rheometer (ODR at 320° F., 5° arc) (ASTM D 2084) properties:			
(a)	ML (minimum torque, in.-lb.) (ASTM D 2084)	11.3	13.3
(b)	MH (maximum torque, at 2 hrs, in.-lb.)(ASTM D 2084)	61.5	55.0
(c)	Ts2, (time to 2 point rise above minimum viscosity, in min.) (ASTM 2084)	4.3	3.3
(d)	MC(90)(in.-lb.)(ASTM 2084)	56.4	50.8
(e)	Tc(90)(min)(ASTM E 2084)	65.5	63.0

Table 21 reports the physical properties of these aramid fiber examples.

TABLE 21

PHYSICAL PROPERTIES

Properties	Specification Requirement	Example	Comparative Example B
Density (lbs/in^3)(ASTM D 792)	0.040 min	0.0422	0.0421
Shore A hardness (ASTM D 2240)	70–85	75.8	74.4
Tensile strength, parallel (psi) (ASTM D 412)	700 min	1085	1050
Elongation parallel (%) (ASTM D 412)	10 min	27.3	24.6
Tensile strength, perpendicular (psi)(ASTM D 412)	—	461	367
Elongation, perpendicular (%)	—	109	126

Table 22 reports the results from a test to measure the ablation rates for these aramid fiber examples.

TABLE 22

MATERIAL ABLATION RATE (MILS/SECOND)

	Example	Comparative Example B
Low Mach Region (0.0028)	3.58	3.43
Medium Mach Region (0.039–0.0970)	10.55	10.83
High Mach Region (0.109–0.140)	16.59	21.99

The ablation tests were performed as follows. The tests were performed in a char motor, schematically illustrated in FIG. **2**. The samples for the three velocity regions of the motor (low, medium, and high Mach) were net molded using conventional compression molding techniques. The low Mach region had space for 15 specimens, while the medium and high Mach regions had space for 4 specimens. The thickness of each specimen was measured at several axial locations before and after firing. The char motor itself featured a propellant beaker filled with Thiokol Propulsion propellant TP-H1148 to a sufficient depth to provide for a 12-second burn with a tungsten nozzle radius of 0.24 inches. The actual action time and average operating pressure for the test motor were 12.0 seconds and 842 psi, respectively.

Although the Mooney scorch time of the Example was somewhat longer than that of the Comparative Example (R196), at the cure temperature of 320° F., the cure rates, as indicated by the 90 percent cure time tc(90), of the two materials were equivalent. Similarly, the physical properties of the materials prepared in accordance with the Example and Comparative Example B were substantially equivalent and well within the specification requirements. Additionally, the ablative performance of the material prepared in accordance with the inventive Example was equivalent or better than the ablative performance of the Comparative Example B (R196).

In the course of experimentation leading to this invention, a number of other candidate elastomeric formulations were tried in the hopes that the path to a new acceptable insulation would not be overly difficult. In fact, such other formulations proved to be unsuccessful and this is demonstrated by the following illustrative unsuccessful examples.

RUBBER COMPONENT FORMULA RDL5191
Total Wt. = 3000 grams

CATEGORY	Component Id.	PARTS by weight	Wt %	Grams

-continued

RUBBER COMPONENT FORMULA RDL5191
Total Wt. = 3000 grams

Polymers	1 Nordel IP NDR-4640	80.00	54.22	1626.57
Plasticizers	2 Neoprene FB	20.00	13.55	406.64
Antioxidants	3 Agerite Resin D	2.00	1.36	40.66
Fillers	4 HiSil 233	35.50	24.06	721.79
Activator	5 Zinc Oxide	4.00	2.71	81.33
	6 2nd Pass Additions			
Accelerators	7 Rhenocure S/G	0.80	0.54	16.27
	8 Rhenocure AP-3	3.85	2.61	78.28
	9 Rhenocure ZADT/G	1.00	0.68	20.33
Curative	10 Sulfur	0.40	0.27	8.13
	Totals:	147.55	100.00	3000.00

Mixing Instructions, first pass:

Add Nordel and zinc oxide
Add Hisil and antioxidant
Add Neoprene FB
Dump about 280 F.
2nd Pass Additions

Mix MB with curitives in Brabender
Dump about 190 or below.

-continued

RUBBER COMPONENT FORMULA RDL 5571
Total Wt. = 3000 grams

Antioxidants	5 Agerite Stalite S	2.00	1.36	40.66
Fillers	6 HiSil 233	35.50	24.06	721.79
Activator	7 Zinc Oxide	4.00	2.71	81.33
2nd Pass Additions	8			
Accelerators	9 Methyl Tuads	0.75	0.51	15.25
	10 Altax	1.50	1.02	30.50
	11 Sulfads	0.75	0.51	15.25
	12 Butyl Zimate	1.50	1.02	30.50
Curative	13 Sulfur	0.50	0.34	10.17
	Totals:	151.50	102.68	3080.31

Mixing Instructions, first pass:

Mix EPDMs and Hypalon together
Add antioxidants 5 & 6, Add silica, zinc oxide
Add Akrochem resin.
Dump about 250–300°
2nd Pass Additions

Mix MB with curatives in Brabender
Dump about 190° or below.

RUBBER COMPONENT FORMULA RDL5570
Total Wt. = 3000 grams

CATEGORY	Component	PARTS by weight	Wt %	Grams
Polymers	1 Nordel IP NDR-4640	50.00	33.89	1016.60
	2 Nordel IP NDR-4520	40.00	27.11	813.28
Plasticizers	3 Hypalon 20	10.00	6.78	203.32
	4 Akrochem P-133	5.00	3.39	101.66
Antioxidants	5 Agerite Stalite S	2.00	1.36	40.66
Fillers	6 HiSil 233	35.50	24.06	721.79
Activator	7 Zinc Oxide	4.00	2.71	81.33
	8 2nd Pass Additions			
Accelerators	9 Methyl Ethyl Tuads	2.00	1.36	40.66
	10 Monex or Unads	1.00	0.68	20.33
	11 Sulfads	1.00	0.68	20.33
	12 Butyl Zimate;	1.50	1.02	30.50
Curative	13 Sulfur	0.50	0.34	10.17
	Totals:	152.50	103.35	3100.64

Mixing Instructions, first pass:

Mix EPDMs and Hypalon together
Add antioxidants 5 & 6, Add silica, zinc oxide
Add Akrochem resin.
Dump about 250–300
2nd pass Additions

Mix MB with curitives in Brabender
Dump about 190 or below.

RUBBER COMPONENT FORMULA RDL 5572
Weight = 3000 grams

Category	Component	PARTS by weight	Wt %	Grams
Polymers	1 Nordel IP NDR-4640	50.00	33.89	1016.60
	2 Nordel IP NDR-4520	40.00	27.11	813.28
Plasticizers	3 Hypalon 20	10.00	6.78	203.32
	4 Akrochem P-133	5.00	3.39	101.66
Antioxidants	5 Agerite Stalite S	2.00	1.36	40.66
Fillers	6 HiSil 233	35.50	24.06	721.79
Activator	7 Zinc Oxide	4.00	2.71	81.33
	8 2nd Pass Additions			
Accelerators	9 Methyl Tuads	3.00	2.03	61.00
	10 Methyl Zimate	3.00	2.03	61.00
	11 Sulfasan R	2.00	1.36	40.66
	12 Butyl Zimate	3.00	2.03	61.00
Curative	13 Sulfur	0.50	0.34	10.17
	Totals:	158.00	107.08	3212.47

Mixing Instructions, first pass:

Mix EPDMs and Hypalon together
Add antioxidants 5 & 6, Add silica, zinc oxide
Add Akrochem resin.
Dump about 250–300°
2nd Pass Additions

Mix MB with curitives in Brabender
Dump about 190° or below.

RUBBER COMPONENT FORMULA RDL 5571
Total Wt. = 3000 grams

CATEGORY	Component	PARTS by weight	Wt %	Grams
Polymers	1 Nordel IP NDR-4640	50.00	33.89	1016.60
	2 Nordel IP NDR-4520	40.00	27.11	813.28
Plasticizers	3 Hypalon 20	10.00	6.78	203.32
	4 Akrochem P-133	5.00	3.39	101.66

RUBBER COMPONENT FORMULA RDL 5172
Total Wt = 3000 grams

Category	Component	PARTS by weight	Wt %	Grams
Polymers	1 Nordel IP NDR-4640	80.00	54.22	1626.57
	2 Trilene 67A	10.00	6.78	203.32
Plasticizers	3 Hypalon 20	10.00	6.78	203.32
	4 Akrochem P-133	5.00	3.39	101.66

-continued

RUBBER COMPONENT FORMULA RDL 5172
Total Wt = 3000 grams

Antioxidants	5 Agerite Resin D	2.00	1.36	40.66
Fillers	6 HiSil 233	35.50	24.06	721.79
Activator	7 Zinc Oxide	3.00	2.03	61.00
2nd Pass Additions	8			
Accelerators	9 Captax	1.00	0.68	20.33
	10 Tellurac	0.50	0.34	10.17
	11 Sulfads	0.75	0.51	15.25
Curative	12 Sulfur	1.00	0.68	20.33
	Totals:	148.75	100.81	3024.40

Mixing Instructions, first pass:

Mix Nordel and Hypalon together
Add antioxidants 4 & 6, Add silica, zinc oxide
Add Trilene and Akrochem resin.
Dump about 250–300°
2nd Pass Additions

Mix MB with curatives in Brabender
Dump about 190° or below

As indicated below, for various reasons, the above-described experimental test insulations proved to be unsuitable formulations for production of rocket motor insulations.

TABLE 23

Unsuccessful Experiment Results

Rheological Properties

Formulation	ML	MH	ts2	Tc(90)	Rejected because:
RDL5571	25.3	138.3	2.3	21.3	MH too high to match existing standard insulation
RDL5572	22.6	173.9	2.5	25.7	MH too high to match existing standard insulation
RDL5570	25.6	128.6	2.7	29.5	MH too high to match existing standard insulation
RDL5191	26.8	133.6	3.5	25.4	MH too high to match existing standard insulation
RDL5172	39.0	128.0	1.0	25.0	Uses the old cure system as in TABLE 2, but product is much too scorchy to be useful; cures at too low temperature

As these unsuccessful formulations indicate, and faced with the discontinuance of the availability of the hitherto thought to be essential component for the present standard insulation formulations, the development of a suitable and acceptable solid propellant rocket motor insulation that will provide the required performance criteria has not been an easy task.

The foregoing detailed description of the preferred embodiments of the invention has been provided for the purposes of illustration and description. It is not intended to be exhaustive or to limit the invention to the precise embodiments disclosed. Many modifications and variations within the scope of this invention will be apparent to practitioners skilled in this art. The illustrated embodiments were chosen and described in order to best explain the principles of the invention and its practical application, thereby enabling others skilled in the art to understand the invention for various embodiments and with various modifications as are suited to the particular use contemplated. It is therefore intended that the scope of the invention cover various modifications and equivalents included within the spirit and scope of the appended claims.

We claim:

1. A rocket assembly comprising a rocket motor component insulated by an elastomeric insulation cured from an EPDM (ethylene propylene diene terpolymer) composition, the EPDM composition comprising:

a primary EPDM terpolymer formed from ethylene, propylene, and about 2–12 wt % of a diene;

a secondary EPDM terpolymer, different than the primary EPDM terpolymer, the secondary EPDM terpolymer being formed from ethylene, propylene, and an alkylidene norbornene;

silica filler additive; and

a curing agent,

wherein the elastomeric insulation insulates the rocket motor component.

2. The rocket assembly of claim **1**, wherein the alkylidene norbornene is ethylidene norbornene.

3. The rocket assembly of claim **2**, wherein 70 to 80 parts by weight of the EPDM composition consists of the primary EPDM terpolymer, and wherein 15 to 25 parts by weight of the EPDM composition consists of the secondary EPDM terpolymer.

4. The rocket assembly of claim **2**, wherein about 56 weight percent to about 65 weight percent of the primary EPDM terpolymer consists of the ethylene.

5. The rocket assembly of claim **2**, wherein the secondary EPDM terpolymer has a higher ethylene content than the primary EDPM terpolymer.

6. The rocket assembly of claim **2**, wherein about 10–12 weight percent of the primary EPDM terpolymer consists of the diene.

7. The rocket assembly of claim **2**, wherein the composition further comprises at least one member selected from the group consisting of antioxidants, plasticizers, tackifiers, and wetting agents.

8. The rocket assembly of claim **2**, wherein the curing agent comprises a brominated phenolic resin.

9. The rocket assembly of claim **5**, wherein the alkylidene norbornene is ethylidene norbornene, wherein 70 to 80 parts by weight of the EPDM composition consists of the primary EPDM terpolymer, wherein 15 to 25 parts by weight of the EPDM composition consists of the secondary EPDM terpolymer, and wherein the curing agent comprises a brominated phenolic resin.

10. The rocket assembly of claim **2**, wherein the rocket motor component comprises a rocket motor case housing a solid propellant grain, and wherein the elastomeric insulation is positioned between the rocket motor case and the solid propellant grain.

11. A method for insulating a rocket motor component, comprising:

providing a rocket motor component;

preparing silica-filled elastomeric insulation by curing a primary EPDM (ethylene propylene diene terpolymer) terpolymer and a second EPDM terpolymer with a curing agent, the primary EPDM terpolymer being formed from ethylene, propylene, and about 2–12 wt % of a diene, the secondary EPDM terpolymer being different than the primary EPDM terpolymer and formed from ethylene, propylene, and ethylidene norbornene; and

insulating the rocket motor component with the elastomeric insulation.

12. The method of claim **11**, wherein said preparing comprises selecting 70 to 80 parts by weight of the primary EPDM terpolymer per 15 to 25 parts by weight of the secondary EPDM terpolymer.

13. The method of claim **11**, wherein about 56 weight percent to about 65 weight percent of the primary EPDM terpolymer consists of the ethylene.

14. The method of claim **11**, wherein the secondary EPDM terpolymer has a higher ethylene content than the primary EDPM terpolymer.

15. The method of claim **11**, wherein about 10–12 weight percent of the primary EPDM terpolymer consists of the diene.

16. The method of claim **11**, wherein said preparing comprises combining at least one member selected from the group consisting of antioxidants, plasticizers, tackifiers, and wetting agents with the primary and secondary EPDM terpolymers.

17. The method of claim **11**, wherein the curing agent comprises a brominated phenolic resin.

18. The method of claim **11**, wherein the rocket motor component comprises a rocket motor case housing a solid propellant grain, and wherein the elastomeric insulation is positioned between the rocket motor case and the solid propellant grain.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 6,566,420 B1
APPLICATION NO. : 09/481709
DATED : May 20, 2003
INVENTOR(S) : David G. Guillot and Albert R. Harvey

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

In the drawings:

FIGURE 3,		change "CORE 46" to --46-- and change "PROPELLANT GRAIN 44" to --44--
COLUMN 2,	LINE 21,	in Table 2, last column, below "5" and above "2" insert --1--
COLUMN 2,	LINE 65,	in Table 3, last column, below "0.3" and above "20" insert --1--
COLUMN 4,	LINE 22,	change "occur." to --occurred.--
COLUMN 6,	LINE 42,	change "of its" to --off its--
COLUMN 7,	LINE 8,	change "include" to --including--
COLUMN 7,	LINE 65,	change "**1***a*–**3***b*," to --**1***a*–**3**,--
COLUMN 16,	LINE 67,	change "DLI 552A." to --DL1552A.--
COLUMN 18,	LINE18,	change "solvent allowed" to --solvent is allowed--
COLUMN 18,	LINE 31,	in Table 11, in the title of the table, change "WITR" to --WITH--
COLUMN 20,	LINE 3,	the title of the table, change "WITR" to --WITH--
COLUMN 24,	LINE 16,	change "aramid, e.g. KEVLAR," to --aramid fibers, e.g., KEVLAR®--
COLUMN 24,	LINE 17,	change "fibers are" to --fibers, are--
COLUMN 24,	LINE 29,	change "the said" to --the--

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

	COLUMN 27,	LINE 23,	change “curitives” to --curatives--
	COLUMN 27,	LINE 43,	delete the semicolon after “Zimate”
	COLUMN 27,	LINE 53,	change “curitives” to --curatives--
	COLUMN 28,	LINE 53,	change “curitives” to --curatives--

In the claims:

CLAIM 5,	COLUMN 30,	LINE 34,	change “EDPM” to --EPDM--
CLAIM 14,	COLUMN 31,	LINE 12,	change “EDPM” to --EPDM--

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

Please replace FIG. 3 with the following:

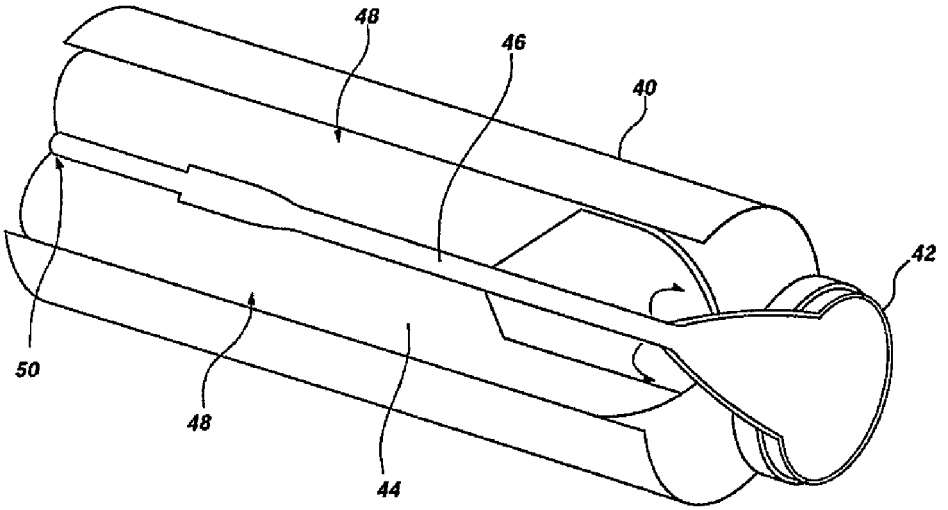

FIG. 3

Signed and Sealed this

Twenty-eighth Day of November, 2006

JON W. DUDAS
Director of the United States Patent and Trademark Office