Rock Island Arsenal Laboratory

TECHNICAL REPORT

ELASTOMERIC INSULATION FOR SOLID PROPELLANT ROCKET MOTORS

By

D. H. Sale

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Laboratory Director

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Rock Island Arsenal
Rock Island, Illinois

ABSTRACT

The development of flexible, elastomeric-based, solid propellant rocket motor case insulation is discussed. The effect on insulation properties of type of asbestos, of liquid versus solid elastomers and of methods of dispersing fibrous compounding ingredients, are reported.

Oxyacetylene torch and static motor firing test data for some of the better insulation materials developed, as well as for some commercial materials, are presented. The torch test, the principle screening tool used in this study, conforms to the test currently being standardized by the Flame Ablation Test Group of Section III-L of ASTM Committee D-20.

A material with 40 percent elongation, 1.34 gm/cc density, performance index of 95 cm^2sec/gm and erosion rate of 2.0 mils/sec was the most promising insulation developed. The material is a 55/45 butadiene/acrylonitrile compound containing phenol furfural resin, asbestos and oxyazoline wetting agent. It has been satisfactorily bonded to aluminum and to steel by conventional bonding agent. The thermal properties of this vulcanizate are not affected by oven aging for one week at 70°C.

RECOMMENDATIONS

It is recommended that this project be discontinued. It is believed that the insulation materials developed during the course of this investigation represent an advancement in the state of the art and that further investigations under the approaches outlined in this and earlier work would result in only marginal improvements.

It is recommended that the oxyacetylene torch test equipment be retained in a usable condition, in order that promising new commercial insulation materials might be evaluated and that cooperative work to further improve this screening test might be conducted, if necessary

ELASTOMERIC INSULATION FOR SOLID PROPELLANT ROCKET MOTORS

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ELASTOMERIC INSULATION FOR SOLID PROPELLANT ROCKET MOTORS

OBJECT

To develop improved, flexible, thermal case insulation for solid propellant rocket motors.

INTRODUCTION

The need for flexible rocket motor case insulation has been well established.[1-7] Elastomers have played an important role in meeting this need, as evidenced by the many types of elastomeric-based insulations which have been developed for use in solid propellant missiles. The wide spectrum of commercially available insulation ranges from lightly filled rubbers with excellent flexibility but minimal ablation resistance, to highly loaded compounds with little flexibility but outstanding resistance to the high temperatures and erosive gases found within rocket motors.

Present day elastomeric insulations are not considered adequate for future needs, especially needs related to the use of end burning grains. It is anticipated that longer burning times at higher temperatures will necessitate more thermally resistant insulations and that higher internal pressures will require higher degrees of flexibility. A continuing need exists for lighter weight materials.

It appears that maximum flexibility and resistance to the environments within rocket motors are mutually exclusive properties, insofar as rubber-based insulations are concerned, since improvements in one property are attainable only at the expense of the other. Earlier work at this Arsenal,[8,9] as well as more recent studies, has led to the development of rubber-based insulations having a compromise in these two properties, namely, the highest degree of ablative resistance which could be attained together with flexibility which, when measured in terms of elongation, amounts to 20 to 50 percent. Results of these studies have led to the following conclusions:

1. By far the most effective filler combination for imparting ablation resistance to rubber vulcanizates is a combination of a phenolic resin and long fiber asbestos.

2. This filler combination is most effective in a high nitrile content, butadiene/acrylonitrile elastomer. Of the more than 400 combinations of elastomers, fillers

and other compounding ingredients tested, none has led to a material having a better compromise between flexibility and ablation resistance than that exhibited by the nitrile-phenolic resin-asbestos fiber system.

3. The degree of ablation resistance of this, or any other asbestos-containing rubber compound, is directly related to the fiber length of the asbestos in the finished product.

These findings largely influenced the direction of effort described in this report. Minimum effort has been devoted to seeking more effective combinations of rubber and fillers. Major emphasis has been placed on developing means for incorporating long fiber asbestos into rubber-resin matrices without significantly reducing fiber length.

PROCEDURE

Insulation resistance was determined with an oxy-acetylene torch test (Figure 1) using the conditons cited in Table I. The equipment and procedures duplicate those of the test currently in the final stages of standardization by the Flame Ablation Test Group of Section III-L of ASTM Committee D-20.

The effectiveness of candidate insulation materials was measured by two test criteria; (1) the temperature rise on the back side of the specimen while the front side was exposed to the oxyacetylene torch flame and (2) the time required for the flame to burn through the specimen. These criteria are reported as performance indices and erosion rates, respectively. The index, referred to as P_{200}, is computed by dividing the time (seconds) required for the specimen back side to reach 200°C. by the original specimen thickness (centimeters) and by the specimen specific gravity. The erosion rate, E, is computed by dividing the original specimen thickness (mils) by the burn through time. It should be noted that high values of P_{200} and low values for E are indicative of good insulation properties. Unless otherwise noted, the performance indices and erosion rates reported are the average of four test results

Rock Island Arsenal Laboratory has been an active member of the aforementioned ASTM Test Group. The torch test facility at this Laboratory was among those utilized in a recent round robin. The round robin results showed an average variance among laboratories of less than ±5% for each of the two test criteria. The Rock Island Arsenal test facility produced results well within this variance.

RIA OXY-ACETYLENE TORCH TEST FACILITY

A - Fast response temperature recorder
B - Thermocouple holder
C - Specimen
D - Torch
E - Flowmeters and pressure gauges for oxygen and acetylene

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11-070-3564/Ord-61

FIGURE 1

64-3156

TABLE I

TORCH TEST OPERATING CONDITIONS

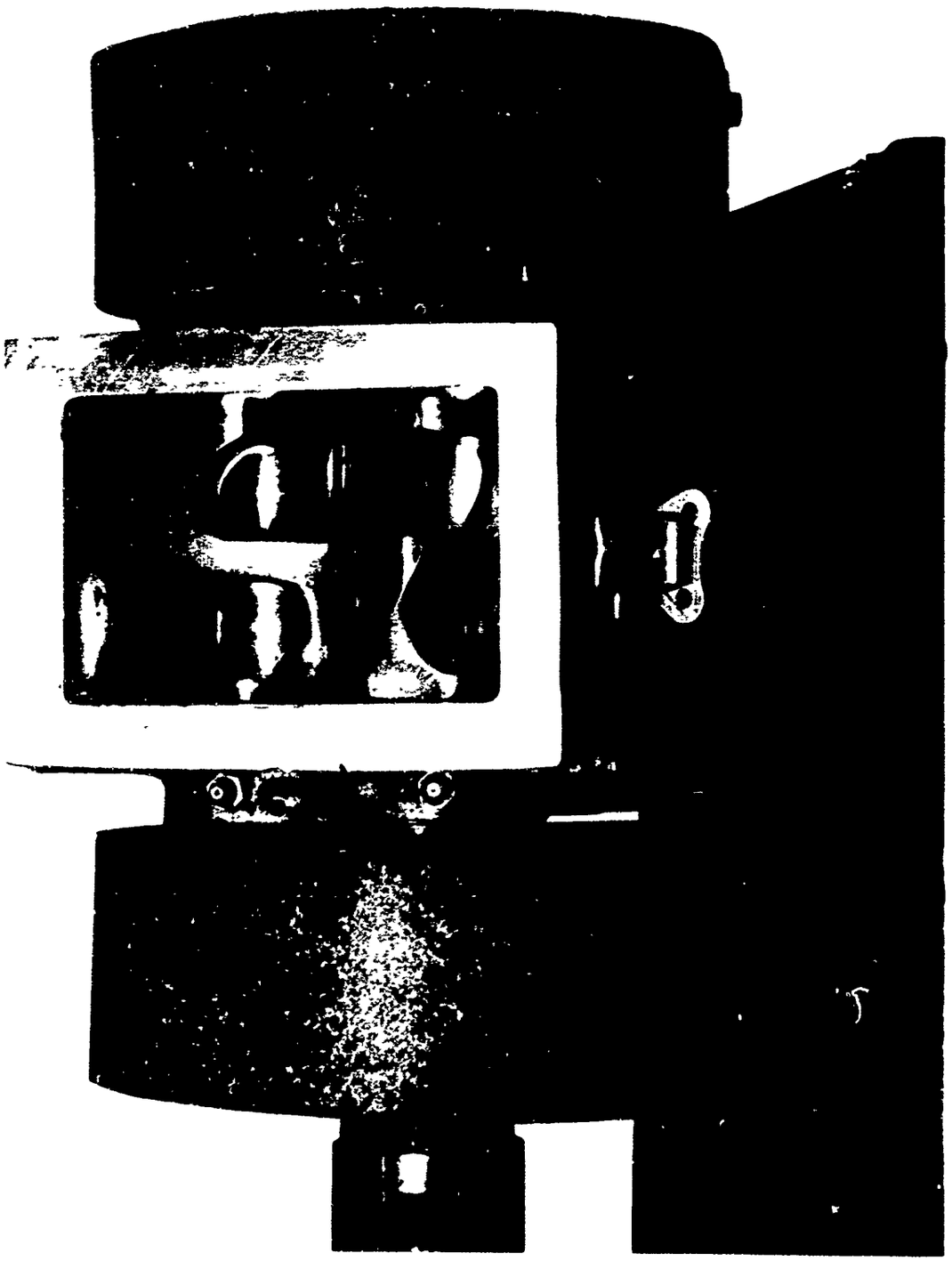
Oxygen flow rate, standard cubic feet/hour (SCFH)	123
Acetylene flow rate, SCFH	102
Volume ratio oxygen to acetylene	1 2
Impingement angle between flame and specimen, degrees	90
Specimen size, inches	4 X 4 X 1/4
Distance from torch tip to specimen, inches	3/4
Method of determining moment of burn through	Visual

Several materials which exhibited excellent resistance to the torch test were evaluated (see Table VII) in static motor firings conducted by the Atlantic Research Corporation and the Allegheny Ballistics Laboratory at their respective test facilities.

The major compounding ingredients for each material tested may be found in the tables pertaining to the material. Curing systems are given in Appendix I. Test specimens were compression molded in a four cavity mold. The liquid polymer-based compounds were mixed in a two bladed sigma type mixer (Figure 2), with mixing arms operating at differential speeds. All solid polymer-based compounds were mixed on a two roll rubber mill. Stress-strain properties were determined in accordance with applicable ASTM[10] procedures

RESULTS AND DISCUSSION

Previous evaluation[9] of several types of inorganic and organic fibers had shown that asbestos was the most effective for imparting thermal resistance to rubber vulcanizates Current studies were made to determine the effect of the type of asbestos on the properties of insulation materials Amosite, crocidolite and chrysotile asbestos of approximately equal fiber length (major concentration of fiber lengths, 3/4 to 1 inch long) were evaluated in a nitrile rubber-phenolic resin based compound Chrysotile samples from three different suppliers were included The results in Table II show that the three chrysotile-based vulcanizates differ from one another considerably, but that all three are superior to either the amosite or crocidolite-based insulations. The superiority of chrysotile is attributed to its flexibility (both amosite and crocidolite are brittle and tend to suffer fiber breakdown during compounding), higher specific heat and fusion temperature, lower density and higher percent of bound water. Hereinafter, the word "Asbestos"



ONE PINT SIGMA MIXER

FIGURE 2

TABLE II

THE EFFECT OF ASBESTOS TYPE ON PROPERTIES OF INSULATION MATERIALS

Asbestos Type	Asbestos Properties*				Insulation Properties			
	Specific Heat, Cal/gm/°C.	Fusion Temp., °C.	Density, gm/cc	Bound Water, %	P_{200}, cm^2sec/gm	E, Mils/sec	Elongation, %	Tensile Strength, psi
Chrysotile #1	0.266	1520	2.4-2.6	13	71	2.8	20	2600
Chrysotile #2	0.266	1520	2.4-2.6	13	58	4.7	25	2010
Chrysotile #3	0.266	1520	2.4-2.6	13	51	4.3	35	2700
Crocidolite	0.201	1190	3.2-3.3	2	29	8.7	105	970
Amosite	0.193	1400	3.1-3.3	1-5	24	9.6	145	960

Basic Formulation:

55/45 Butadiene Acrylonitrile	100
Curatives	9.32
Phenol Furfural Rosin, powder	50
Asbestos	100

*Manufacturer's data.

in this report refers to chrysotile #1.

The problem of obtaining a uniform dispersion of long asbestos fibers in a rubber matrix while at the same time minimizing the reduction in fiber length was attacked through the use of the following classes of materials: (1) wetting agents, to wet the asbestos fiber and thus facilitate fiber dispersion; (2) plasticizers, to soften the rubber matrix; (3) liquid polymers, to reduce shearing forces which prevail when solid polymers are mixed; (4) solvents, to dissolve the unvulcanized rubber prior to the asbestos addition.

Evaluations of wetting agents were made as shown in Table III. The wetting agents were added to the mixture just prior to the asbestos. Although most of the wetting agents tested were useful in dispersing the asbestos, not all improved insulation properties. However, oxyazoline #1, a heterocyclic cationic wetting agent, produced vulcanizates with excellent thermal properties. A compound containing 30 PHR of this wetting agent produced a 25 percent improvement in thermal properties and a 100 percent increase in elongation over the control material containing no wetting agent.

Oxyazoline #1 at 30 PHR concentration showed no evidence of migration after aging for 7 days in an air oven at 70°C. Aged specimens when evaluated in the oxyacetylene torch test, exhibited thermal properties equivalent to unaged controls. Insulation containing oxyazoline #1 has been bonded to both aluminum and steel using a two part, room temperature curing, general purpose epoxy adhesive. Bond strengths (tensile shear on one inch lap joints) greater than the tensile strength (1730 psi) of the insulation were achieved.

Data for plasticized solid elastomer-resin-asbestos compounds and unplasticized control compounds are presented in Table IV. Small amounts (5 and 15 PHR) of phosphate esters served to increase elongation but larger amounts decreased elongation. None of the vulcanizates had improved thermal properties over the controls.

Liquid ingredients offer a means of compounding with low shear forces. Data for liquid elastomer-asbestos vulcanizates, as well as the appropriate controls based on solid polymers, are given in Table V. The data revealed the following results: vulcanizates based on liquid elastomers have better thermal properties, greater tensile strength but shorter ultimate elongation than the controls based on solid polymers. The liquid elastomers consisted of low molecular weight, short chain molecules which required

** Average of 8 tests — Wetting agent or plasticizer — As Noted

TABLE III

THE EFFECT OF WETTING AGENTS ON PHYSICAL PROPERTIES AND TORCH PERFORMANCE OF NBR* VULCANIZATES

Wetting agent or plasticizer, PHR		Elongation, %	Tensile, psi	Density, gm/cc	P_{200}, cm^2sec/gm	E, mils/sec
Control	none	20	2530	1.42	74	2.6
Tri-octyl phosphate	7.5	15	3330	1.40	76	2.3
Oxyazoline #1	7.5					
Oxyazoline #1	10	20	2990	1.39	78	2.2
Oxyazoline #1	20	45	1460	1.35	87	2.3
Oxyazoline #1	30	40	1730	1.34	95**	2.0**
Oxyazoline #2	30	10	1270	1.37	92	2.4
Polyoxyethylene sorbitan monoöleate	30	20	720	1.34	82	2.7
Sorbitan Monoöleate	30	75	710	1.35	75	2.6
Trioctyl phosphate	30	20	2990	1.37	73	2.5
Tricresyl phosphate	45	10	1290	1.35	71	2.8
Naphthenic type oil	30	45	960	1.33	63	3.1

* Basic Formulation:

55/45 Butadiene/acrylonitrile	100
Phenol furfural resin	50
Asbestos	100
Curatives	10
Wetting agent or plasticizer	As Noted

TABLE IV

THE EFFECT OF PLASTICIZERS ON TORCH PERFORMANCE AND PHYSICAL PROPERTIES OF NBR* VULCANIZATES

Plasticizer, PHR		Resin,** PHR	Asbestos, PHR	P_{200}, cm^2sec/gm	E, mils/sec	Tensile, psi	Elongation, %
Control	0	100	50	71	2.8	2600	20
Tri-octyl phosphate	5	100	50	60	3.4	2180	30
Tri-octyl phosphate	15	100	50	71	3.0	1250	45
Tri-cresyl phosphate	5	100	50	60	3.1	2100	30
Tri-cresyl phosphate	15	100	50	65	3.3	2190	50
Control	0	50	100	74	2.6	2530	20
Tri-octyl phosphate	5	50	100	69	2.8	2660	30
Tri-octyl phosphate	15	50	100	69	2.8	1810	40
Tri-octyl phosphate	30	50	100	73***	2.5***	2990	20
Tri-cresyl phosphate	5	50	100	63	3.1	3060	30
Tri-cresyl phosphate	15	50	100	56	3.1	2050	50
Tri-cresyl phosphate	45	50	100	71	2.8	1290	10
Tri-cresyl phosphate Tri-octyl phosphate	7.5 7.5	50	100	57	3.4	2600	15
Butadiene/acrylonitrile, liquid	15	50	100	58	3.4	3090	35
Control	0	100	100	76	2.3	2830	10
Butadiene/acrylonitrile, liquid	20	100	100	74	3.6	2020	15

* 55/45 Butadiene/acrylonitrile
** Phenol furfural, powder
*** Average of 8 samples

TABLE V

PROPERTIES OF VULCANIZATES BASED ON LIQUID POLYMERS

Elastomer Type	Asbestos, PHR	P_{200}, $cm^2sec/gm.$	E, Mils/sec.	Elongation, %	Tensile, psi
76.5/23.5 Butadiene/styrene, solid	100	51	4.7	55	1050
Butadiene/styrene, high viscosity liquid	100	63	3.0	20	2180
55/45 Butadiene/acrylonitrile, solid	100	36	7.2	75	2330
Butadiene/acrylonitrile, liquid	100	47	4.2	20	3600
Carboxy terminated butadiene/ acrylonitrile, liquid	50	57	5.3	15	210
Carboxy terminated butadiene, liquid	50	48	6.1	15	520

a high state of cure to produce good thermal resistance. Although these elastomers did facilitate the incorporation of fibrous asbestos without appreciable fiber breakdown, the high degree of crosslinking required for proper vulcanization produced lower elongations than are inherent with solid polymers.

Further evaluation of liquid elastomers included the addition of thermosetting resins (liquid and solid) as fillers. The resins used in this study were all polar and enhanced the erosion resistance of NBR-based compounds, but they were too incompatible with the less-polar SBR to be effective. Because of the lack of non-polar resins and the low elongations obtained with the liquid elastomers, work with SBR and NBR liquid elastomers was discontinued.

The two carboxy modified polymers listed in Table V were vulcanized using an epoxy resin at a concentration of 13 PHR as a curative. The low elongations obtained from these two compounds eliminated the need for investigating the effect of adding more resin.

The method of incorporating asbestos by adding it to a methyl ethyl ketone solution of the polymer and other compounding ingredients proved unsuccessful. The asbestos in the resultant vulcanizates was non-uniformly dispersed in the rubber matrix after air and vacuum evaporation of the solvent. These vulcanizates had properties inferior to the properties of vulcanizates prepared by conventional milling procedures.

Torch test data for several commercial insulation materials tested are presented in Appendix II. Of these materials, one has properties comparable to the best Rock Island Arsenal insulation, as shown in Table VI.

The work discussed herein and work previously reported[8,9] has resulted in several excellent thermal insulation materials. The nine best of these, along with five poorer materials and two commercial products, were evaluated in static motor firings by Atlantic Research Corporation. Erosion rates, densities, elongations and major constituents for each are presented in Table VII. The erosion rates from the static firing tests correlate reasonably well with the torch test erosion rates, at least for the best five or six and the poorest compounds. The erosion rates from the static tests cover a range of only 2.4 units, as compared to 10.4 units for the torch test.

Unfortunately the insulation containing oxyazoline #1 wetting agent was not included with the above materials.

TABLE VI

EVALUATION DATA FOR THE BEST ROCK ISLAND ARSENAL AND COMMERCIAL INSULATIONS

Property Measured	Rock Island Arsenal	Commercial #1
P_{200}. Cm^2sec/gm	95	79
Time to backside temperature of 200°C. for 1/4" specimen, sec.	127	101
E, mils/sec	2.0	2.0
Elongation, %	40	50
Density, gm/cc	1.34	1.28
Major constituents	Rubber, Resin, Asbestos, Oxyazoline #1	Rubber, Resin, Asbestos

However, this material was submitted to Allegheny Ballistics Laboratory for evaluation in their test facilities. Their data indicates that it compares favorably with materials they class as "current better materials."(11)

TABLE VII

COMPARISON OF EROSION RATES FROM OXYACETYLENE TESTS WITH EROSION RATES FROM MOTOR FIRINGS*

Erosion Rate, Mils/sec.				
Rock Island Arsenal Torch Test	Static Solid Propellant Motor, Convergent Area	Density, gm/cc	Elongation, %	Major Constituents
2.3	1.5	1.42	10	55/45 Butadiene/acrylonitrile (100), Phenol furfural Resin Powder (100), Asbestos (100).
2.5	1.6	1 37	20	55/45 Butadiene/acrylonitrile (100), Phenol furfural Resin Powder (50), tri-octyl phosphate (30), asbestos (100).
2.8	1.6	1.41	20	55/45 Butadiene/acrylonitrile (100), phenol furfural resin powder (50), asbestos (100).
3.1	2.2	1.30	20	55/45 Butadiene/acrylonitrile (100), phenol furfural resin powder (100), asbestos (50).
3.1	2.3	1.38	95	Butadiene/acrylonitrile-polyvinyl chloride blend (100), hydrated silica (20), asbestos (40).
3.2	2.5	1.31	30	Butadiene/styrene, high viscosity liquid (100), asbestos (50).
3.2	3.1	1.12	3	Butadiene/acrylonitrile, resin, metal organic salt. Commercial material #3.
3.4	2.6	1.28	25	Butadiene/styrene, low viscosity liquid (100), asbestos (50).
3.5	3.1	1.36	25	Butadiene/styrene, high viscosity liquid (100), hydrated silica (40), asbestos (40).
3.9	2.5	1.34	40	Butadiene/acrylonitrile liquid (100), asbestos (50).
4.1	2.7	1.39	120	Butadiene/styrene, asbestos, commercial material #8.
4.7	2.5	1.26	135	76.5/23.5 Butadiene/styrene (100), hydrated silica (20), asbestos (40).
5.0	2.5	1.33	20	76.5/23.5 Butadiene/styrene (100), phenol furfural resin powder (100), asbestos (50).
5.4	2.5	1.43	65	76.5/23.5 Butadiene/styrene (100), metal oxide mixture** (100), asbestos (50).
9.9	3.1	1.55	15	Methyl phenyl vinyl silicone (100), mineral filled silicone resin (50), asbestos (50).
12.7	3.9	1.72	85	Vinylidene fluoride (hexafluoropropylene (100), Polychloroprene (50), metal organic salt (40), MT carbon black (50).

* Pressure, temperature and times of test are classified information.

** Metal oxide mixture composition.

Metal oxide	% Composition
Al_2O_3	70.0
Mg O	0.5
Hydrated Silica	26.0
Fe_2O_3	0.5
Ca O	0.5
Ti O_2	2.5
	100.0

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APPENDIX I

CURING SYSTEMS

Ingredients	PHR Used for Compounds Based On							
	Solid NBR containing resin fillers	Solid NBR without resin fillers	Liquid NBR	Liquid Carboxy terminated polymers	Solid SBR	Liquid SBR	Methyl phenyl vinyl silicone	Vinylidene fluoride-hexafluoropropylene blended with polychloroprene
Stearic acid	2	2			2			
Zinc oxide	5	3	5		3			
Tetramethyl thiuram disulfide	3	0 12			0 12			
Sulfur		3	20		3	12		
Benzothiazyl disulfide		1 2			1 2	4		
Activated dithiocarbamate			3					
2,4,6-Tri(dimethylaminomethyl)phenol				0 78				
Epoxy				13				
N-phenyl-beta-naphthylamine					1			
Zinc diethyldithiocarbamate						1		
2,4-dichlorobenzoyl peroxide with silicone fluid 50%							1 5	
Magnesium oxide								15
Hexamethylenediamine								5
Curing cycle for .075" thick specimens								
Press Time, minutes/temperature, °C	30/153	30/153	30/153	30/154	30/153	30 153	10 135	30 153
Air oven post cure Time hour temp., °C							6 190	6 193

APPENDIX II

TORCH TEST DATA FOR COMMERCIAL INSULATION MATERIALS

Material #	P_{200}, $cm^2 sec/gm$	E, mils/sec	Density, gm/cc	Elongation, %	Major Constituents
1	79	2.0	1.28	50	Rubber - Resin - Asbestos
2	79	2.1	1.65	nil	Resin - Asbestos - Polyamide
3	72	3.2	1.12	3	NBR Rubber - Resin - Methal organic salt
4*	69	3.0	1.45	10	Rubber - Resin - Asbestos
5	63	3.5	1.26	50	Rubber - Resin - Asbestos
6	46	2.1	1.24	30	NBR Rubber - Resin - Carbon fibers
7*	46	5.6	1.23	90	Rubber - Resin - Asbestos
8	45	4.0	1.39	120	SBR Rubber - . . . - Asbestos
9	35	6.9	1.29	25-225	NBR Rubber - . . . - Asbestos
10	30	13.2	0.60	100	Silicone Rubber - . . . - inorganic fillers
11	27	5.5	1.77	nil	Resin - Asbestos

* Data is average of 2 specimens

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ELASTOMERIC INSULATION FOR SOLID PROPELLANT ROCKET MOTORS, By D. H. Sale

RIA Lab. Rep. 64-3158, 27 Oct 64, 24 p. incl. illus., tables, (DA Project No. 1C0-24401-A110, AMC Code No. 5025.11.84203) Unclassified Report

The development of flexible, elastomeric-based, solid propellant rocket motor case insulation is discussed. The effect on insulation properties of type of asbestos, of liquid versus solid elastomers and of methods of dispersing fibrous compounding ingredients, are reported. Oxyacetylene torch and static motor firing test data for some of the better insulation materials developed, as well as for some commercial materials, are presented. The torch test, the principle screening tool used in this study, conforms to the test currently being standardized by the Flame Ablation Test Group of Section III-L of ASTM Committee D-20. A material with 40 percent

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