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HORIZONS RESEARCH INC CLEVELAND OHIO  
POLY(ARYLOXYPHOSPHAZENE) FOAMED THERMAL INSULATION.(U)  
APR 78 A H GERBER, T C PETERSON

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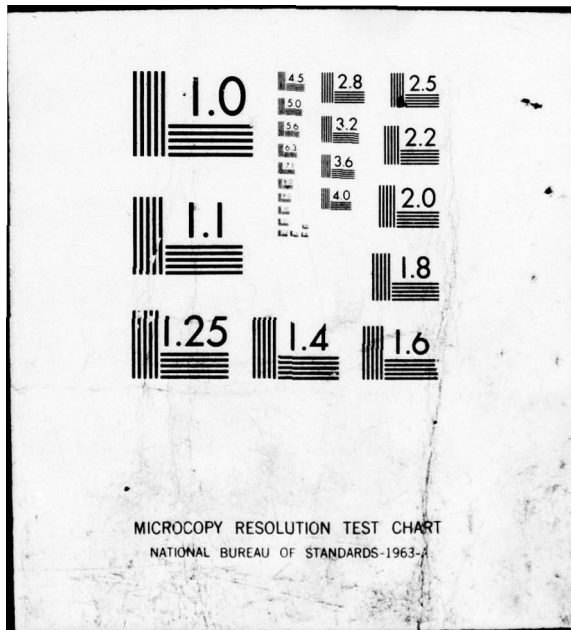
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POLY (ARYLOXYPHOSPHAZENE) FOAMED  
THERMAL INSULATION

FINAL REPORT

NAVAL RESEARCH LABORATORY

Contract No. N00173-77-C-0286

Authors  
Arthur H. Gerber  
Thomas C. Peterson

April 1978

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Cleveland, OH 44122

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## ABSTRACT

Pilot plant produced sulfur curable poly(aryloxyphosphazene) rubber was formulated with fillers, stabilizers, foaming agent, activators, and curatives to prepare 121.7 square feet of fire-retardant thermal insulation foam. Foaming involved two steps, thermal pre-cure under pressure and free expansion at elevated temperature. Eleven of fourteen specification properties were easily met. Three properties, namely compression set, flame spread index, and water vapor permeability did not meet the target property guidelines.

POLY (ARYLOXYPHOSPHAZENE) FOAMED  
THERMAL INSULATION

1.0 OBJECTIVE

Preparation and evaluation of 120 square feet of poly-(aryloxyphosphazene) foamed thermal insulation. Target guidelines were specified for fourteen properties.

2.0 DISCUSSION AND RESULTS

Sulfur curable pilot plant produced poly(phenoxy-4-ethylphenoxyphosphazene) rubber was obtained from The Firestone Tire & Rubber Company (Akron, Ohio). This polymer was formulated with fire-retardant filler, stabilizer, blowing agent, processing aid, blowing and curing activators [Ref. 1]. Compounding was done with a Brabender Prep Center mixer and a standard 6", 2-roll rubber mill. The uniform compounded material was cut to size, placed in a heated mold and slightly cured under pressure. This product was then free blown in a heated circulating air oven. The cooled foams were trimmed and tested. The approximate dimensions of most foams were 15" x 23" x 1/2" and ranged in area from 2.2 to 2.5 square feet, except for 2502-17-28 which required extra trimming.

A total of 121.7 sq. feet or 51 pieces of poly(aryloxyphosphazene) thermal insulation foam were supplied to the Naval Research Laboratory.

Twenty-one of the 51 foams were tested for tensile strength, elongation, compression resistance and density. The results are shown in Table I. A more comprehensive property evaluation was made twice. These results are shown in Table II. Standard test procedures and modifications thereof as well as outside testing laboratories are shown in Table III.

Eleven of the 14 properties were easily met. Average values of three properties, compression set (26% vs 24%), flame spread index (33 vs 25), and water vapor permeability (0.7 perm-inch vs 0.30 perm-inch) did not meet the target property guidelines.

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[Ref. 1] K. A. Reynard, R. W. Sicka, J. E. Thompson, "Poly-(aryloxyphosphazene) Foams", Horizons Inc., Final Report-Part I to Naval Ship Systems Command, Contract N00024-73-C-5474, June 1974, AD 781 578.

TABLE I  
 PHYSICAL PROPERTIES OF SHIPPED FOAMS

Foam No.	Tensile Strength (psi)	Elongation (%)	Compression Resistance (25% Deflection) (psi)	Density (pcf)
2524-07 *	32	71	4.1	5.9
-09	36	65	4.2	6.5
2502-13-2 *	21	52	3.7	5.6
-14-2 *	--	--	--	--
-14-3	--	--	--	--
-14-4	22	58	3.8	5.6
-14-6	--	--	--	--
-14-7	23	63	3.9	5.6
-15-3	25	50	3.8	5.8
-15-4	--	--	--	--
-15-5	--	--	--	--
-15-6	--	--	--	--
-15-15	--	--	--	--
-15-20	47	62	6.2	8.2
-15-21	45	62	5.4	7.0
-15-23	--	--	--	--
-15-24	51	54	5.8	7.7
-15-25	--	--	--	--
-15-26	--	--	--	--
-15-27	46	62	6.0	8.4
-15-28	--	--	--	--
-17-3	--	--	--	--
-17-4	40	75	4.6	7.6
-17-5	--	--	--	--
-17-6	--	--	--	--
-17-7	49	59	6.0	7.5
-17-8	--	--	--	--

\* 0.65 inch thickness

TABLE I (Contd.)  
 PHYSICAL PROPERTIES OF SHIPPED FOAMS

Foam No.	Tensile Strength (psi)	Elongation (%)	Compression Resistance (25% Deflection) (psi)	Density (pcf)
2502-17-11	--	--	--	--
-17-12	--	--	--	--
-17-13	38	64	5.0	7.5
-17-14	--	--	--	--
-17-15	--	--	--	--
-17-16	36	65	4.5	7.4
-17-17	--	--	--	--
-17-18	--	--	--	--
-17-19	37	69	4.7	7.3
-17-20	--	--	--	--
-17-21	--	--	--	--
-17-22	45	72	5.1	7.8
-17-23	--	--	--	--
-17-25	--	--	--	--
-17-27	36	75	4.3	6.9
-17-28	--	--	--	--
-17-29	44	68	5.6	7.8
-17-30	--	--	--	--
-23-1	--	--	--	--
2524-17	40	62	4.9	7.0
-32-1	44	62	5.2	7.4
-32-2	--	--	--	--
-32-3	--	--	--	--
-32-4	39	58	5.6	7.8

TABLE II  
 PHYSICAL PROPERTIES OF THERMAL INSULATION FOAM

Property	Property Range Guideline	Property Values Found	
		Foams 2502-13-1 15-1 15-2	Foams 2502-17-1 17-2 17-10
a. Density, pounds/cubic foot	4.0 - 9.0	5.5 - 6.1	7.7 - 8.4
b. Compression resistance @25% deflection, pounds/square inch	2 - 7	3.2 - 3.7	4.9 - 6.0
c. Water absorption, pounds/square foot of skinless area, maximum	0.1	0.033 - 0.043	0.020 - 0.022
d. Compression set, %, maximum	24	32	20
e. Dimensional change, length %, maximum	7	-5	-8
f. Oil resistance	No softening or swelling	Very slight softening; 6% swell in only one dimension of sample.	Very slight softening; 2% shrinkage.
g. Tensile strength, pounds/square inch, minimum	20	20 - 25	42 - 56

TABLE II (Continued)

PHYSICAL PROPERTIES OF THERMAL INSULATION FOAM

Property	Property Range Guideline	Property Values Found	
		Foams 2502-13-1 15-1 15-2	Foams 2502-17-1 17-2 17-10
h. Ultimate elongation, %, maximum	75	56 - 59	58 - 60
i. Tensile strength of cemented joints, before aging	No bond failure	No bond fail- ure.	No bond fail- ure.
Minimum after aging	No bond failure	"	~30% bond fail- ure.
j. Flexibility @ 28°F Initially After heat aging 7 days/180°F	No cracking "	No cracking. "	No cracking. "
k. Thermal conductivity, BTU/hour·square foot (°F/inches) at 75°F, K factor, maximum	0.50	0.28	0.32
l. Water vapor permeability, perm-inch, maximum	0.30	0.55 - 0.61	0.76

TABLE II (Continued)

PHYSICAL PROPERTIES OF THERMAL INSULATION FOAM

Property	Property Range Guideline	Property Values Found	
		Foams 2502-13-1 15-1 15-2	Foams 2502-17-1 17-2 17-10
m. Flame spread index, maximum	25	35 - 40 (Avg. = 37)	20 - 37 (Avg. = 28)
n. Optical smoke density, flaming mode, maximum	150	72 - 80 (Avg. = 76)	63 - 94 (Avg. = 79)

TABLE III

TEST PROCEDURES FOR TABLE I AND II VALUES

- a. ASTM D 1667 using compression resistance specimen (b).
- b. ASTM D 1667 using square instead of cylindrical specimen.
- c. MIL-P-15280H (4.6.6).
- d. MIL-P-15280H (4.6.7).
- e. MIL-P-15280H (4.6.8).
- f. MIL-P-15280H (4.6.9).
- g. ASTM D 412 die A dimensions were traced onto the foam, specimen cut using a jig saw. Values reported are average of two.
- h. ASTM D 412 calculated from distance traveled by jaws of testing machine as recorded during test (g).
- i. MIL-P-15280H (4.6.12) as modified for (g) and (h) using Scotch-Grip Vinyl Adhesive 2262, 3M Company.
- j. MIL-P-15280H (4.6.13).
- k. ASTM C 177-76 by Cincinnati Testing Laboratories, Inc. Cincinnati, Ohio.
- l. MIL-P-15280H using a 5-1/2 inch diameter test dish.
- m. ASTM E 162-75 values for three panels by Smithers Scientific Services, Inc., Akron, Ohio.
- n. Smoke Density Test (NBS)  
Aminco-NBS Smoke Density Chamber  
Cat. No. 4-5800  
by United States Testing Co., Inc., Hoboken, New Jersey.

The first 12 foam pieces designated in Table I were of lower density (5.5-6.1 pcf) and physically more uniform than the remaining pieces. These differences are attributed to batch variation of poly(aryloxyphosphazene) supplied by The Firestone Tire & Rubber Company. Apparently the second batch of polymer contained a higher level of stabilizer which necessitated additional research and development to adjust curative level and cure conditions. Little expansion or excessive bubbling resulted without these adjustments. Even so, the revised system was much more sensitive and irreproducible than the earlier system. Greater problems were also encountered with mold release. This occasionally led to blemishes or rough spots on one side of the foam.

It is believed that better stabilizer level control in the preparation and purification of poly(aryloxyphosphazene) will eliminate the above problems. Furthermore, sulfur based cure systems would also obviate such difficulties because this system, unlike peroxide curatives, is insensitive to conventional stabilizers used with unsaturated elastomers.

### 3.0 RECOMMENDATIONS

The limited time and funds on this contract prevented further optimization of properties shown in Tables I and II. There is no doubt that both smoke density and flame spread index could be lowered by increasing the level of fire-retardant filler. Density could be lowered by further adjustments in blowing agent level and blowing conditions. Better control of stabilizer level in the phosphazene rubber or replacement of the peroxide curing system by a sulfur based system should yield a more reproducible foam product.