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Special Report on the Current Status of the Tensile Strength of SiB-2 Elastomers at Elevated Temperatures

Compiled by

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Chemicals Division
New Haven, Connecticut

May 1965

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Special Report on the Current Status of the Tensile Strength of SiB-2 Elastomers at Elevated Temperatures.

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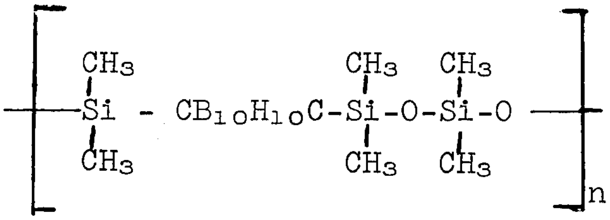
Summary

[Modified SiB-2 rubbers can now be routinely compounded and pressure molded to provide an elastomer having a tensile strength of 450 psi. at ambient temperatures. On testing at 600°F, these specimens retain a tensile strength of 30-35%, or up to 45% by the addition of Fe_2O_3 as an antioxidant. At 800°F, 10% (and in certain instances 15%) retention was observed. These retention data surpass those reported for silicones and also are better than the corresponding values for Viton B.
6 tables, 2 appendices. (A.m.)]

Introduction

The advent of the missile age and the development of supersonic jet aircraft has produced an increasing demand for nonmetallic materials which can be used at high temperatures. Of special importance is the synthesis of thermally stable elastomers superior to those presently available. Various requirements for them exist and more are projected for use in Mach 3 airplanes. Typical examples for their application are pneumatic seals for doors and windows, interlaminar layers for glass windshields, and integral fuel tank sealants, among many others. Each of these requires outstanding thermal stability beside individual properties which are specifically suited to the desired function.

During the past year we have discovered under this contract[1] a new family of boron-containing elastomers which show promise of satisfactory performance under extreme conditions. From among these, we have selected as the most attractive candidate for elevated temperature capability "SiB-2", a polydimethylsiloxane containing m-carborane nuclei in regular intervals in the skeleton.



$$\left[-\overset{CH_3}{\underset{CH_3}{Si}} - CB_{10}H_{10}C - \overset{CH_3}{\underset{CH_3}{Si}} - O - \overset{CH_3}{\underset{CH_3}{Si}} - O - \right]_n$$

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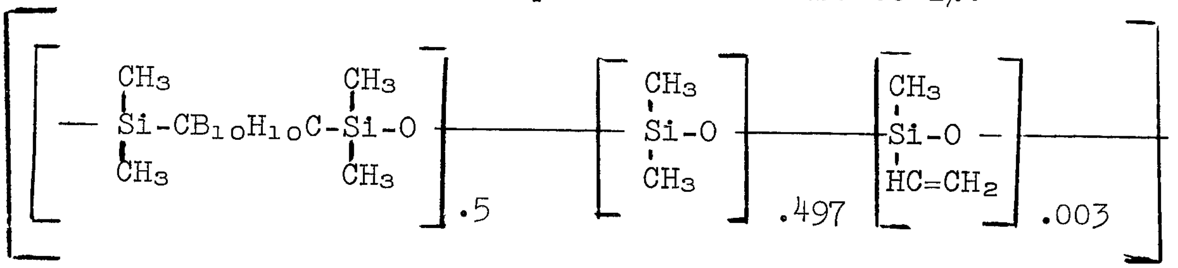
[1] Olin Mathieson Chemical Corporation, various Monthly Progress Reports for Contract Nonr 3395(00).

In this polymer, the ratio of thermostabilizing carboranylene and flexibility producing dimethylsiloxy groups appears to be in the right balance as to assure maximum advantage of both properties.

Since "plain" SiB-2 requires up to 5% of peroxide for effective curing, we subsequently synthesized so-called "modified" SiB-2 rubbers by introduction of a minimal amount of unsaturated groups by introduction of a minimal amount of unsaturated groups as additional curing sites.[2] In the example as illustrated, part of the methyl groups are replaced by vinyl groups. Other modifications investigated in place of vinyl are allyl and vinyl-o-carboranyl, which all permit a peroxide decrease to 2%.



$$\left[\left[-\underset{\mathrm{CH_3}}{\overset{\mathrm{CH_3}}{\mathrm{Si}}}-\mathrm{CB_{10}H_{10}C}-\underset{\mathrm{CH_3}}{\overset{\mathrm{CH_3}}{\mathrm{Si}}}-\mathrm{O}\right]_{.5}-\left[\underset{\mathrm{CH_3}}{\overset{\mathrm{CH_3}}{\mathrm{Si}}}-\mathrm{O}\right]_{.497}-\left[\underset{\mathrm{HC{=}CH_2}}{\overset{\mathrm{CH_3}}{\mathrm{Si}}}-\mathrm{O}-\right]_{.003}\right]_{x}$$

The present program[3] concerns the development of this and other elastomeric systems. Since we are only at the outset of this study and our previous work has given us only indications of what that rubber may do, an extensive although exploratory testing at elevated temperatures seemed necessary to gain enough data which would allow us to pursue our work along specific guide lines.

Test Material Preparation

To obtain a set of lucid data, a number of parameters involved in the sample preparation were kept constant. The use of various fillers had been scanned previously and we had found finely divided silica to be most suitable in regard to tensile strength and elongation; the commercial product selected was "Cab-O-Sil". As the free radical type curing agent, "Di-Cup 40", in 2 parts concentration, was used in almost every instance. The process of pressure molding as well as the curing cycle were identical for all samples. The proportion of unsaturated groups in modified SiB-2 rubbers always corresponded to the formula of vinyl modified SiB-2 illustrated in the introduction.

The cured SiB-2 test specimens were all prepared as follows: Purified and dried SiB-2 (plain or modified) rubber was milled to a crepe on a cold, 5 in. mill; the desired amounts of solid filler (Cab-O-Sil), curing agent (Di-Cup 40) and, in some cases Fe_2O_3 or $Al(OH)_3$ as an antioxidant, were thoroughly blended and then milled into the crepe. Enough compounded rubber was placed in a 2" x 5" mold to give a final specimen of ~1/8" thickness; these specimens

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[2] Olin Mathieson Chemical Corporation, Technical Report No. 26 for Contract Nonr 3395(00) by S Papetti, B. B. Schaeffer, A. P. Gray and T. L. Heying, in preparation.

[3] Contract NObs-92143, Project Serial No. SR007-03-03, Task 1000

were cured at 311°F at either 375 or 500 psig. After cooling, the specimens were removed and post-cured in air at 212°F for 24 hours, 302°F for 24 hours and finally 392°F for 48 hours.

Test Procedure

Ambient temperature tensile testing is done in the normal manner on an Instron Testing Apparatus, Model TT-CM1 using a crosshead speed of 2"/min. For elevated temperature testing, the sample and holders are suspended in a Missimers Inc., Temperature Test Chamber, Model TFU 3.2_100x 1000; the chamber is brought to the selected temperature equilibrium, the box is opened and the dogbone is positioned and the door closed. The box is allowed to return to temperature, allowed to remain for 5 minutes (total time ∼15 min.) and then pulled.

Results and Discussion

Since the SiB elastomers are basically polydimethylsiloxanes, it seems appropriate to compare them first of all with the silicones, and also to apply as much as possible the latter's established technology for improving the SiB polymers. The room temperature tensile strengths of commercial silicone rubbers generally fall in the 300 - 700 psi. range although some specialty types may be as high as 1000-1200 psi.

Our first objective was to try to improve the ambient temperature tensile strengths while maintaining adequate elongation. In this respect, modified SiB-2 polymers were found to be definitely superior to plain SiB-2.

Table 1[a] reflects the effect of the various modifications (as compared to our standard unmodified SiB-2) on room temperature tensile strength at low loading.

TABLE I

Tensile Strength and Elongation: Effect of Modification at Low Filler Content

Modification	SiO_2 parts	Shore Hardness (A)	Elongation %	Tensile psi.
None	25	68	55	201
Vinyl	20	58	61	192
$\Theta CH=CH_2$	25	57	125	277
$\Theta CH=CH_2$	30	57	87	215
$\Theta CH=CH_2$	35	64	92	279
Allyl	25	72	115	384
Allyl	25	72	136	446
Allyl	35	73	140	375

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[a] Tables I-VI which appear in this discussion are organizations of properly selected data which serve to illustrate certain points or establish trends. A complete tabulation of all meaningful evaluation data obtained in two tables in the Appendix.

It should be pointed out that at present we are not so much concerned with absolute numerical values as we are in establishing trends and effects on these values. Simple vinyl modification was ineffective. Vincylcarboranyl modification showed measurable improvement while allyl modification, as expected, gave the highest tensile and elongation values. (This is typical of llyl modified silicones in general.) The effect of variation of parts of silica in the 25-25 ranges seems negligible; when 50 or more parts of silica are used the effect becomes pronouned as can be seen in Table II.

TABLE II

Tensile Strength and Elongation:

Effect of Modification at High Filler Content

Modification	SiO_2 parts	Shore Hardness	Elongation %	Tensile psi.
Vinyl	50	71	72	333
Vinyl	50	67	88	372
Vinyl	50	80	53	430
Vinyl	60	78	49	495
Vinyl	75	85	18	503
$\Theta CH=CH_2$	50	70	54	331
$\Theta CH-CH_2$	50	72	67	341
$\Theta CH=CH_2$	50	74	70	459
Allyl	50	66	78	312
Allyl	50	75	102	413
Allyl	60	74	37.5	319
Allyl	75	78	39	351

Note here that at room temperature additional filler markedly raised the tensile strengths of both the vinyl and vinylcarboranyl modifications while there is no effect on the allyl modified polymer. Sixty parts of silica appears to be the maximum which can be tolerated without causing a sharp decline in elongation.

A real test of an elastomer for use at high temperature is the measurement of the percentage of retention of a given physical property at these temperatures. Table III therefore summarizes the performance of certain samples (low loading) discussed above at 600 and 800°F.

TABLE III

Tensile Strength and Elongation: Effect of Modification at High Temperatures (Low Filler Content)

		Ambient		600°F			800°F		
Modification	SiO_2 parts	Elong. %	Tensile psi.	Elong. %	Tensile psi.	Retention % psi.	Elong. %	Tensile psi.	Retention % psi.
none	25	55	201	40	62	30.8	11	33	16.4
$\theta CH=CH_2$	25	109	239	72.6	73.4	30.7	22	18.2	7.6
$\theta CH=CH_2$	25	125	277	90	89	32.1	—	—	—
$\theta CH=CH_2$	35	92	279	70	86.6	31	—	—	—
allyl	25	115	384	92	96.5	18.1	—	—	—
allyl	25	136	446	66	33.3	7.5		13.1	2.9
allyl	35	140	375	156	69.5	18.5	33	10.9	2.9

Although at 25 parts silica the allyl modified SiB-2 showed by far the highest room temperature tensile strength, at 600 and 800°F it is considerably inferior to both vinylcarboranyl modified and unmodified SiB-2. Note the retention of ~30% of tensile strength of the latter two at 600°F.

Table IV illustrates a similar treatment of the more highly filled specimens.

TABLE IV

Tensile Strength and Elongation:

Effect of Modification at High Temperatures (High Filler Content)

Modification	SiO_2 parts	Ambient		600°F			800°F		
		Elong. %	Tensile psi.	Elong. %	Tensile psi.	Retention % psi.	Elong. %	Tensile psi.	Retention % psi.
vinyl	50	72	333	45	122	36.6	15	26.5	8
vinyl	50	53	430	67.5	123.5	29	11	21.5	5
vinyl	60	49	495	54.6	158	32	—	—	—
vinyl	75	18	503	—	—	—	—	26.8	5.3
$\theta CH{=}CH_2$	50	67	341	77.5	120.9	35.4	19.2	22	6.4
$\theta CH{=}CH_2$	50	70	459	50	187.5	40.8	25	43.4	9.4
allyl	50	102	413	58	91.5	22.3	22	25.6	6.2

These results shows that again the allyl modification is inferior to either the vinyl or vinylcarboranyl types. Samples of each of the latter two retained more than 30 percent of their tensile strength at 600°F and 5-8% and 6-9% respectively at 800°F. We also point out that visual observance of the specimens during test made it obvious that the allyl materials was softening and deforming while the others generally retained their structural identity.

These results have led us to drop from further consideration the use of allyl modified SiB-2 for high temperature evaluation. This also points out the principal that the variation which shows the best room temperature properties need not necessarily perform as well at elevated temperature as another very similar material; it is the old story of the 'weakest link in the chain'.

During these and earlier studies[2] it had become apparent to us that initial deterioration of an elastomer sample at elevated temperatures could be observed as a surface hardening and/or cracking while the interior of the sample was quite unchanged. Such a happening could be the start of a failure in a tensile test. We have every reason to suspect oxidative attack and have begun to scan potential antioxidants and determine whether they are beneficial. Table V points out the effect of Fe_2O_3, a potential antioxidant on one formulation system, namely the vinylcarboranyl variety with 50 parts Cab-O-Sil. The additive of one part of the Fe_2O_3 produced no significant difference in tensile strength retention at either 600 or 800°F; on the other hand the addition of three parts Fe_2O_3 gave the highest retention yet observed at 600°F, specifically 45.7% and the retention at 800°F was above 10°. We believe this effect to be real and important and this is an important point for further study.

TABLE V

Tensile Strength and Elongation:

Effect of Fe_2O_3 at Various Temperatures

			Ambient		600°F			800°F		
Modification	SiO_2 parts	Antioxidant (parts)	Elong. %	Tensile psi.	Elong. %	Tensile psi.	Retention % psi.	Elong. %	Tensile psi.	Retention % psi.
$\Theta CH=CH_2$	50	——	67	341	77.5	120.9	35.4	19.2	22	6.4
$\Theta CH=CH_2$	50	——	70	459	50	187.5	40.8	25	43.4	9.4
$\Theta CH=CH_2$	50	Fe_2O_3 (1)	100	428	61	128	27.5	8	32.8	7.6
$\Theta CH=CH_2$	50	Fe_2O_3(3)	69	357	75	163	45.7	—	38.4	10.7

Other spurious results regarding the trials of the antioxidants are summarized in Table VI; we remind the reader that these studies have only recently begun and are still in progress.

TABLE VI

Tensile Strength and Elongation:

Effect of Various Antioxidants at Various Temperatures

Modification	SiO_2 parts	Antioxidant (parts)	Ambient		600°F			800°F		
			Elong. %	Tensile psi.	Elong. %	Tensile psi.	Retention % psi.	Elong. %	Tensile psi.	Retention % psi.
none	25	—	55	201	40	62	30.8	11	33	16.4
none	25	Fe_2O_3(0.5)	43	202	37.5	93.3	46.4	—		
ΘCH=CH_2	25	—	109	239	72.6	73.4	30.7	22	18.2	7.6
ΘCH=CH_2	25	Fe_2O_3(1)	158	244	90	103	42.2	—	28.8	11.8
ΘCH=CH_2	25	Fe_2O_3(3)	182	271	81	100	36.9	11	25.9	9.6
ΘCH=CH_2	25	$Al(OH)_3$(3)	190	226	68	58.5	25.9	—	22.1	10.0
vinyl	25	$Al(OH)_3$(3)	160	232	83	77.4	33.3	—	15.2	6.5

These data point out that in general Fe_2O_3 aids in retention of strength in general; the effect of hydrous alumina is questionable.[3] The second example using 0.5 parts Fe_2O_3 with unmodified SiB-2 showed exceptionally high retention at 600°F; check of the records showed that this sample of Fe_2O_3 was from a different source than all others reported and this point is being looked into. Also this particular sample of unmodified SiB-2 in the various formulations in which it was used showed generally better results that we had become accustomed to note. Again careful check of the history of the sample has given us a clue as to why this may have occurred; this is to be examined in more detail.

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[3] Studies elsewhere (Mare Island Naval Shipyard, Rubber Laboratory, Report No. 1-14, 9 November 1964) have shown that hydrous alumina in conjunction with a chloro polymer may be useful. Also the use of antimony oxide (U.S. Rubber Co., Final Report for Contract NObs 84025, March 9, 1962) is suggested elsewhere. We intend to evaluate these as well as many other compounds we suspect could be useful.

Conclusions

Appropriate studies indicate that vinylcarboranyl (and possibly vinyl) modification (to speed curing) of SiB-2 is preferred over allyl modification for high temperature use.

Increasing the amount of silica, as expected, increases the tensile strength of the elastomers; at loadings >60 parts the elongation drops off markedly.

Retention of 30_45% of tensile strength at 600°F and $>10\%$ at 800°F in air has been achieved. The use of iron oxide as an antioxidant assists in this regard.

Further improvements from the study of the effects of such variables as the curing cycle, filter contents, addition of selected antioxidants, etc can be expected.

Comparison of SiB-2 rubber with other elastomers such as Viton and fluorosilicones on aging at $\sim$ 700°F in air will be stressed during the forthcoming period.

Appendix I

Tensile Strength Determinations at Ambient Temperatures

Sample No.	Modification	SiO_2 parts	Antioxidant (parts)	Peroxide[1] (parts)	Shore A	Elongation %	Tensile psi.	Test Variation (min.-max.)	Number of Samples Tested
92	none	25	Fe_2O_3(0.5)	BP(2)	65	43	202	179-230	3
86	vinyl	20	——	BP(1.5)	58	61	192	173-218	4
94-95	vinyl	50	——	DC(2)	71	72	333	301-360	4
122-123	vinyl	50	——	DC(2)	80	53	430	382-454	4
143	vinyl	50	——	DC(2)	67	88	372	353-398	4
144	vinyl	60	——	DC(2)	78	49	495	489-507	4
145	vinyl	75	——	DC(2)	85	18	503	490-516	5
147	vinyl	25	$Al(OH)_3$(3)	DC(2)	56	160	232	232-232	2
93	$\theta CH=CH_2$	50	——	DC(2)	70	54	331	281-368	3
98-99	$\theta CH=CH_2$	50	——	DC(2)	74	70	459	410-492	6
102	$\theta CH=CH_2$	25	——	DC(2)	57	125	277	248-308	4
103	$\theta CH=CH_2$	30	——	DC(2)	57	87	215	186-242	4
104	$\theta CH=CH_2$	35	——	DC(2)	64	92	279	260-317	4
126-127	$\theta CH=CH_2$	50	——	DC(2)	72	67	341	325-368	4
128-129	$\theta CH=CH_2$	50	Fe_2O_3(3)	DC(2)	70	69	357	338-371	4
130-131	$\theta CH=CH_2$	50	Fe_2O_3(1)	DC(2)	66	100	428	423-432	2
132-133	$\theta CH=CH_2$	25	Fe_2O_3(3)	DC(2)	55	179	254	218-289	3
134-135	$\theta CH=CH_2$	25	Fe_2O_3(1)	DC(2)	55	158	244	234-263	4
136-137	$\theta CH=CH_2$	25	Fe_2O_3(3)	DC(2)	55	182	271	213-298	4
138	$\theta CH=CH_2$	34	$Al(OH)_3$(3)	DC(2)	58	120	203	180-221	4
146	$\theta CH=CH_2$	25	$Al(OH)_3$(3)	DC(2)	56	190	226	214-240	3
105	allyl	25	——	DC(2)	72	136	446	427-468	3
106	allyl	25	——	DC(2)	72	115	384	357-426	4
108	allyl	35	——	DC(2)	73	140	375	368-381	2
110-111	allyl	50	——	DC(2)	75	102	413	400-425	2
139	allyl	34	$Al(OH)_3$(3)	DC(2)	50	130	183	146-218	4
140	allyl	50	——	DC(2)	66	78	312	290-323	4
141	allyl	60	——	DC(2)	74	37.5	319	297-334	4
142	allyl	75	——	DC(2)	78	39	351	320-364	4

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1. See Appendix II for definition.

Appendix II

Tensile Strength Determinations at Ambient and Elevated Temperatures

Sample No.	Modification	SiO_2[1] parts	Antioxidant (parts)	Peroxide[2] (parts)	Shore A Hardness	-40°F	Ambient		600°F			800°F		
						Tensile psi.	Elong. %	Tensile psi.	Elong. %	Tensile psi.	Retention % psi.	Elong. %	Tensile psi.	Retention % psi.
64	none	25	———	BP(2)	68	—	55	201	40	62	30.8	11	33	16.4
92	none	25	Fe_2O_3(0.5)	BP(2)	65	—	43	202	37.5	93.9	46.4	———		
147	vinyl	25	$Al(OH)_3$(3)	DC(2)	56	—	160	232	83	77.4	33.4	—	15.2	6.5
94-95	vinyl	50	———	DC(2)	71	1370	72	333	45	122	36.6	15	26.5	8
122-123	vinyl	50	———	DC(2)	80	1948	53	430	67.5	123.5	29.0	11	21.5	5
144	vinyl	60	———	DC(2)	78	—	49	495	54.6	158	32	———		
145	vinyl	75	———	DC(2)	85	—	18	503	———			—	26.8	5.3
55	$\theta CH=CH_2$	25	———	Va(1)	—	—	109	239	72.6	73.4	30.7	22	18.2	7.6
102	$\theta CH=CH_2$	25	———	DC(2)	57	—	125	277	90	89	32.1	———		
134-135	$\theta CH=CH_2$	25	Fe_2O_3(1)	DC(2)	55	—	158	244	90	103	42.2	—	28.8	11.8
132-133	$\theta CH=CH_2$	25	Fe_2O_3(3)	DC(2)	55	—	179	254	83	79	31.1	———		
136-137	$\theta CH=CH_2$	25	Fe_2O_3(3)	DC(2)	55	—	182	271	81	100	36.9	11	25.9	9.6
146	$\theta CH=CH_2$	25	$Al(OH)_3$(3)	DC(2)	58	—	190	226	68	58.5	25.9	—	22.1	10
104	$\theta CH=CH_2$	35	———	DC(2)	64	—	92	279	70	86.6	31	———		
98-99	$\theta CH=CH_2$	50	———	DC(2)	74	1394	70	459	50	187.5	40.8	25	43.4	9.4
126-127	$\theta CH=CH_2$	50	———	DC(2)	72	1288	67	341	77.5	120.9	35.4	19.2	22	6.4
130-131	$\theta CH=CH_2$	50	Fe_2O_3(1)	DC(2)	66	—	100	428	61.5	128	27.5	8	32.8	7.6
128-129	$\theta CH=CH_2$	50	Fe_2O_3(3)	DC(2)	70	—	69	357	75	163	45.7	—	38.4	10.7
105	allyl	25	———	DC(2)	72	—	136	446	66	33.3	7.5	—	13.1	2.9
106	allyl	25	———	DC(2)	72	—	115	384	92	69.5	18.1	———		
108	allyl	35	———	DC(2)	73	—	140	375	156	69.5	18.5	33	10.9	2.9
110-111	allyl	50	———	DC(2)	75	2151	102	413	58	91.5	22.3	22	25.6	6.2

[1] SiO_2 : Cab-O-Sil M5 (Cabot Corp.)

[2] Peroxides:

Va - Varox (2,5-bis(tert.-butylperoxy0-2,5-dimethyl hexane.
BP - Benzoyl Peroxide.
DC - DI CUP 40 (40% dicumyl peroxide supported on calcium carbonate).

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