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CARBON BLACK AS A HEAT STABILIZER IN SILICONE RUBBER VULCANIZATES

TECHNICAL REPORT

John A. Williams

April 1972

RESEARCH DIRECTORATE

WEAPONS LABORATORY AT ROCK ISLAND

RESEARCH, DEVELOPMENT AND ENGINEERING DIRECTORATE

U. S. ARMY WEAPONS COMMAND

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13. ABSTRACT

This work was conducted at the Research Directorate, Weapons Laboratory at Rock Island to develop silicone rubber vulcanizates with improved heat-aging resistance for use in the fabrication of weapon components and accessories. Carbon black of different particle size was added to various types of silicone rubber, the resulting compounds were vulcanized with peroxides unaffected by the presence of carbon black. Heat stability was significantly improved by the use of SAF, FEF, FT and MT carbon black fillers in one pphr quantities. The use of small sized particles of carbon black (SAF) produced silicone vulcanizates that retained upward to 80 per cent of the original tensile strength of these vulcanizates after heat aging for seven days at 600°F. A silicone vulcanizate with three pphr ferric oxide in place of carbon black retained upward to 65 per cent of the original tensile strength after heat-aging seven days at 600°F. A silicone vulcanizate without a heat stabilizer crumbled to an ashen residue after heat aging for three days at 600°F. (U) (Williams, John A.)

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CONTENTS

OBJECTIVE

The objective of this work was to develop silicone rubber vulcanizates with improved heat-aging resistance for use in the fabrication of weapon components and accessories such as high-temperature gaskets, sealants, and O rings.

BACKGROUND

Silicone rubber is considered one of the most heat-resistant elastomers available. Although silicone rubber vulcanizates have moderate tensile strength at room temperature, these vulcanizates are capable of retaining upward to 75 per cent of this strength at 300°F. In comparison, natural rubber is capable of retaining only about 15 per cent of its original tensile strength at 300°F. Silicone rubber shows the least change in tensile strength of the commonly used synthetic elastomers at temperatures up to 500°F.

In heat-aging resistance, silicone rubber is superior to organic elastomers at temperatures of 300°F and above. Changes in physical properties due to heat aging under 600°F are decreasing elongation and tensile strength with increased hardness. At temperatures above 600°F heat aging patterns change. Elongation may not decrease as rapidly as tensile strength, and hardness may decrease rather than increase at these very high temperatures[1]

Without the addition of a heat stabilizer, silicone vulcanizates lose their elastomeric properties in fewer than three days at 600°F; at higher temperatures, the deterioration of these properties can be measured in hours

The most commonly used heat stabilizer, for silicone rubber, is ferric oxide. A silicone vulcanizate containing ferric oxide can be expected to retain upward to 65 per cent of its tensile strength after three days at 600°F, and 45 per cent after seven days.

The use of small quantities of carbon black as ultraviolet inhibitors in silicone rubber is well known.[6] Carbon black has also been evaluated as a heat inhibitor in silicone rubber, but the belief is that its usefulness in this capacity has been underestimated.[2] The purpose of this investigation was to demonstrate the capacity of carbon black to improve aging resistance in silicone rubber and, in some instances, to show the superiority of this material over the commonly used ferric oxide.

Correlation of heat-aging test data with service-life data is difficult to obtain. The data gathered in this investigation were obtained by use of standard, short-term, heat-aging tests to evaluate the rubber compounds developed. However, caution must be exercised in the prediction of service life of a rubber component from these data. The conditions to which a rubber component is exposed can vary extensively. For example, truck brake-actuator seals are cool one minute, but are required to withstand high-braking temperatures the next minute. Different parts of a component can be at different temperatures at the same time. For example, the interior temperature of a hot-air duct may be as high as 600°F with the outside temperatures as low as -60°F. For determination of suitability for application, an appropriate silicone rubber must be selected and tested under as similar conditions as possible to those of the application.

This study was undertaken by the Research Directorate, Weapons Laboratory at Rock Island, because of the need for high-temperature resistant rubber compounds. Some important applications in which such rubber might be used are seals to withstand temperatures of 300°F in engine oils and of 275°F for hydraulic fluids. Seals in heavy equipment are exposed to hot engine exhaust gases whereas push rod cover gaskets and spark plug boots must withstand up to 500°F in natural gas engines. Carburator check valves of silicone rubber must operate at 250°F and be resistant to gasoline. Diesel engines operate at very high temperatures and result in hose failure from hot-air exhaust and hot chemicals in the antifreeze. The use of silicone rubber hose increased service life six times over that of the organic rubber hose Turbine engine temperatures run up to 500°F, and silicone rubber gaskets and boots are a necessity.

APPROACH

Four elastomers, representative of the commonly used silicones, were selected for the evaluation of carbon black as a heat stabilizer. These elastomers consisted of a dimetnyl siloxane with vinyl groups attached, a methyl phenyl silicone with high strength, a fluorosilicone, and a vinyl silicone that requires no postcure.

All compounding ingredients were incorporated into the elastomers on a 6 by 13 inch open roll mill.

The compounds which contained dicumyl peroxide were mold-cured for ten minutes at 300°F, and those containing ditertiary butyl peroxide were cured for ten minutes at 340°F The postcuring cycle for the vinyl containing silicone and the methyl phenyl silicone was eight hours at 480°F, and for the fluorosilicone was eight hours at 400°F.

All testing was conducted according to ASTM procedures where applicable.[3] The heat aging of the silicone vulcanizates was conducted at 600°F and 700°F; and that of the fluorosilicone vulcanizates, at 500°F and 600°F. Tensile strengths at 300°F and 500°F were determined by use of a Scott Model L-6 tensile tester inclosed in a Scott Model HTO hot tensile oven equipped with an autographic recorder controller. Specimens were conditioned at the desired temperature for six minutes prior to testing at that temperature.

RESULTS AND DISCUSSION

The effect of various fillers on the heat-aging resistance of a dimethyl siloxane containing vinyl groups is shown in Table I. From the compound to which no fillers were added, only an ashen residue remained after this compound was heat-aged for three days at 600°F. The use of 5 pphr SAF carbon black and 0.8 pphr ditertiary butyl peroxide improved the heat-aging resistance to the point at which 89 per cent and 60 per cent of the original tensile strength were retained respectively after 3 and 7 days at 600°F. The use of 20 pphr ferric oxide produced 68 and 58 per cent retention of tensile strength under these conditions. The silica and ground quartz fillers provided very little improvement in heat-aging properties; however, tensile strength was improved by these fillers when it was measured at 300 and 500°F. In this study carbon black indicates a definite advantage over ferric oxide and the other fillers evaluated

Ferric oxide and SAF carbon black are compared as heat stabilizers in Table II. The effect of carbon black is also determined in a company-recommended mineral filler formulation. The two additives produced similar results in the dicumyl peroxide-cured vulcanizates which retained 60 per cent of their tensile strength after three days and up to 57 per cent after seven days of heat aging at 600°F. In the ditertiary butyl peroxide-cured vulcanizates, the differences were more pronounced. The carbon black stabilized vulcanizate retained 15 per cent more tensile strength then the ferric oxide stabilized vulcanizate when aged at 600°F Use of the mineral filler did not improve the heat-aging resistance of this silicone rubber, but the retention of tensile strength is significantly improved at 300°F. A graphical comparison of the capacity of SAF carbon black and ferric oxide to improve the tensile strength of dimethyl siloxane polymer containing vinyl groups after exposure to high temperatures is shown in Figure 1.

The results of an evaluation of carbon blacks, differing in particle size, in silicone vulcanizates are shown in Table III. All of these carbon black fillers have a basic pH except for the Neo Spectra, which is acidic. The SAF carbon black produced the vulcanizate with the most improved heat-aging resistance. Of the basic carbon black fillers evaluated those fillers of small particle size provided greater improvement in heat aging resistance. Philblack E (SAF) produced a vulcanizate that retained 92 per cent of its original tensile strength after three days, and 81 per cent after seven days of heat aging at 600°F. One pphr carbon black filler was used in this series, and the results are superior to those obtained with 5 and 10 pphr carbon black as shown in Table I.

Data are shown in Table IV in which ferric oxide and SAF carbon black are compared as heat stabilizers in a high-strength methyl phenyl silicone rubber. The vulcanizates containing these additives showed similar heat-aging properties when dicumyl peroxide was the curative. The ditertiary butyl peroxide-cured vulcanizates demonstrated the advantage of carbon black over the iron oxide. The vulcanizate with one pphr carbon black retained 15 per cent more tensile strength than the vulcanizate containing 1 pphr ferric oxide after heat-aging for seven days at 600°F and 10 per cent more after two hours at 700°F. This improvement is graphically shown in Figure 2.

The data in Table V indicate that carbon black and ferric oxide produce vulcanizates of fluorosilicone rubber with essentially the same percentage of retention of tensile strength after heat aging and when measured at high temperatures. This conclusion is graphically illustrated in Figure 3.

The evaluation of a vinyl silicone rubber that requires no postcure to produce optimum properties is shown in Table VI. These vulcanizates demonstrated good heat-aging resistance and high-temperature strength without the addition of heat stabilizers. The addition of ferric oxide and SAF carbon black did not improve the good heat-aging resistance of these vulcanizates, as shown in Figure 4. This is in direct contrast to the other silicone vulcanizates evaluated that require a postcure to obtain optimum properties. This conclusion is graphically illustrated in Figure 4.

CONCLUSIONS

Carbon black is more effective than ferric oxide when used as a heat stabilizer in methyl phenyl silicone and in dimethyl siloxane with vinyl groups attached when vulcanized with a peroxide unaffected by the presence of carbon black, such as dicumyl or ditertiary butyl peroxide. These heat stabilizers (carbon black and ferric oxide) produced equally good heat aging properties in fluorosilicone rubber.

A low concentration of carbon black is more effective as a heat stabilizer than are large concentrations.

Carbon black is more effective as a heat stabilizer in silicone vulcanizates when those vulcanizates have a low concentration of mineral filler. As the mineral filler content becomes higher, carbon black becomes less effective as a heat stabilizer.

RECOMMENDATIONS

These silicone rubber vulcanizates with SAF carbon black should be used in the development of weapon components exposed to high temperatures.

The fluorosilicone rubber vulcanizates with SAF carbon black should be evaluated in the shock isolater P/N 11697555 and in the rubber recoil adapter P/N 11698924 for the Minigun, where presently fluorosilicone rubber with ferric oxide as the heat stabilizer is used.

TABLE I

COMPARISON OF FILLERS AS HEAT STABILIZERS IN DIMETHYL SILOXANE WITH VINYL GROUPS ATTACHED

Ingredients	Parts by Weight											
Silastic 432	100	100	100	100	100	100	100	100	100	100	100	100
Dicumyl peroxide (40% active)	2	2	2	2	2	2	-	-	-	-	-	-
Ditertiary butyl peroxide	-	-	-	-	-	-	0.8	0.8	0.8	0.8	0.8	0.8
SAF carbon black	-	5	10	-	-	-	-	5	10	-	-	-
Precipitated silica	-	-	-	20	-	-	-	-	-	20	-	-
Ground quartz	-	-	-	-	20	-	-	-	-	-	20	-
Ferric oxide	-	-	-	-	-	20	-	-	-	-	-	20
Physical Properties												
Tensile strength, psi	910	880	800	970	770	740	700	700	850	910	830	770
Modulus @ 300% E, psi	170	220	390	870	240	330	170	170	270	720	230	280
Elongation, %	605	575	465	395	580	445	620	725	550	425	655	510
Hardness, Shore A	42	43	51	70	43	44	40	40	50	69	40	43
Aged 3 days @ 600°F:												
Tensile strength, psi	*	280	430	390	**	350	*	620	440	**	**	520
Elongation, %		90	90	50		155		285	135			225
Hardness, Shore A		88	91	92	91	48		58	83	97	92	55
Aged 7 days @ 600°F:												
Tensile strength, psi	*	300	440	**	**	350	*	420	420	**	**	450
Elongation, %		60	50			70		170	95			210
Hardness, Shore A		91	93	94	91	90		67	82	93	93	59
Aged 2 hours @ 700°F:												
Tensile strength, psi	**	80	160	**	**	250	**	100	130	**	**	240
Elongation, %		165	130			165		210	155			255
Hardness, Shore A	90	49	64	94	88	50	90	42	60	93	88	45
Tensile strength @ 300°F, psi	390	290	290	490	430	360	430	320	350	530	390	320
Elongation @ 300°F, %	385	255	195	205	265	280	415	425	300	225	390	280
Tensile Strength @ 500°F, psi	160	130	150	270	150	140	200	140	120	270	200	160
Elongation @ 500°F, %	170	170	150	135	135	110	200	200	145	150	185	130

*Ashen Residue

**Too brittle to test

TABLE II

COMPARISON OF CARBON BLACK AND FERRIC OXIDE AS HEAT STABILIZERS IN DIMETHYL SILOXANE WITH VINYL GROUPS ATTACHED

Ingredients	Parts by Weight					
Silastic 432	100	100	100	100	100	100
Ferric oxide	3	-	-	3	-	-
SAF carbon black	-	1	1	-	1	1
Ground quartz	-	-	23	-	-	23
Precipitated silica	-	-	8	-	-	8
Dicumyl peroxide (40% active)	2	2	2	-	-	-
Ditertiary butyl peroxide	-	-	-	0.8	0.8	0.8
Physical Properties						
Tensile strength, psi	870	980	890	870	810	910
Modulus @ 300% E, psi	210	190	490	140	170	440
Elongation, %	670	660	460	725	660	580
Hardness, Shore A	35	40	55	39	40	54
Aged 3 days @ 600°F:						
Tensile strength, psi	590	620	530	680	750	720
Elongation, %	250	305	195	465	545	210
Hardness, Shore A	71	65	85	45	45	78
Aged 7 days @ 600°F:						
Tensile strength, psi	390	330	510	570	660	520
Elongation, %	60	50	60	365	370	85
Hardness, Shore A	90	89	91	55	52	87
Aged 2 hours @ 700°F:						
Tensile strength, psi	300	260	290	270	300	310
Elongation, %	290	515	230	520	600	260
Hardness, Shore A	42	29	60	28	29	53
Tensile strength @ 300°F, psi	370	330	520	360	350	500
Elongation @ 300°F, %	410	340	290	435	375	345
Tensile strength @ 500°F, psi	130	120	230	170	120	210
Elongation @ 500°F, %	180	175	175	185	170	170

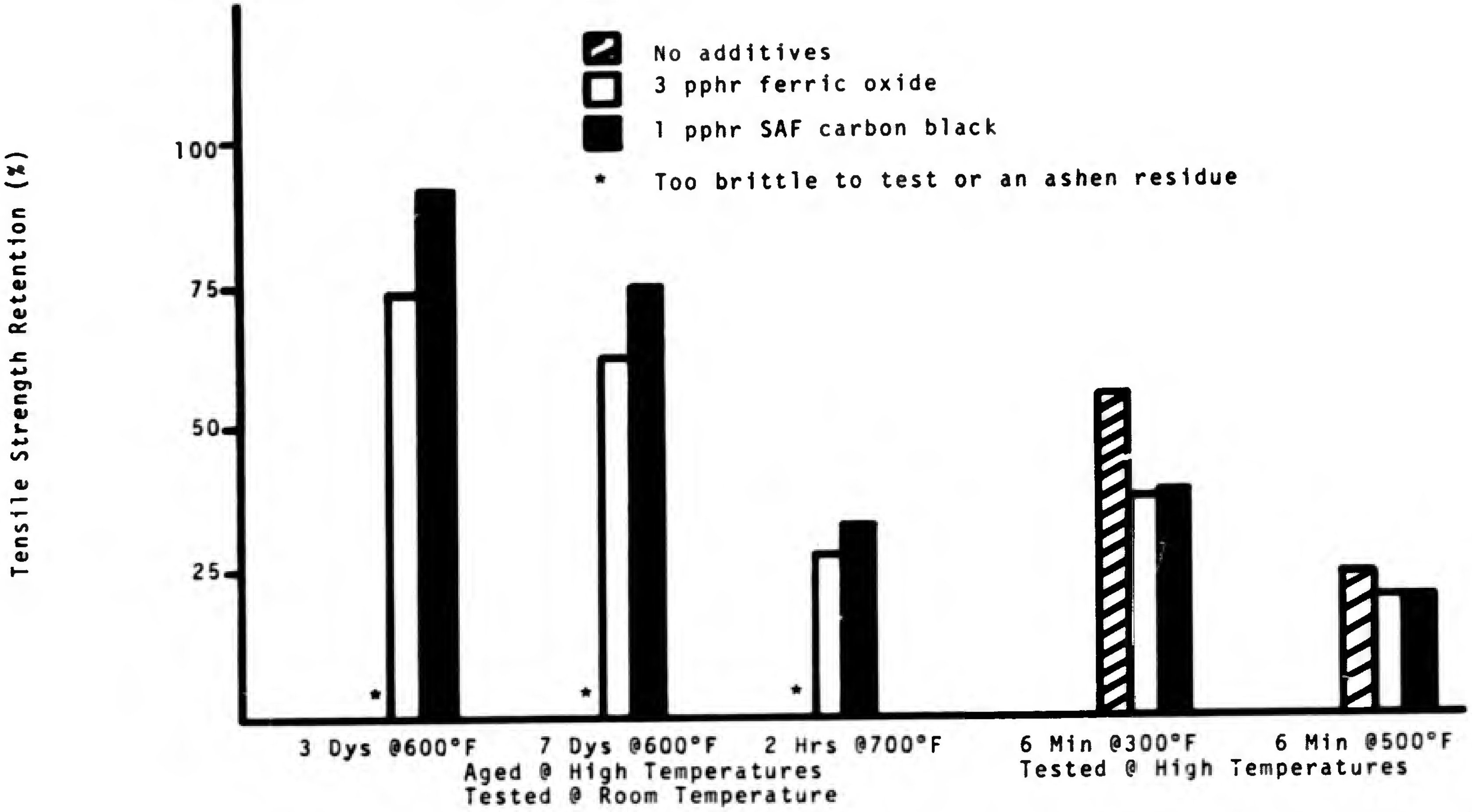


FIGURE 1 Effect of Ferric Oxide and Carbon Black on the High Temperature Properties of a Methyl Vinyl Silicone Rubber

TABLE III

EFFECT OF CARBON BLACK PARTICLE DIAMETER ON THE HEAT STABILITY OF DIMETHYL SILOXANE WITH VINYL GROUPS ATTACHED

Ingredients	Parts by Weight						
Silastic 432	100	100	100	100	100	100	100
Neo Spectra Mark II carbon black	-	1	-	-	-	-	-
Statex 160 (SAF carbon black)	-	-	1	-	-	-	-
Philblack E (SAF carbon black)	-	-	-	1	-	-	-
Philblack A (FEF carbon black)	-	-	-	-	1	-	-
P33 (FT carbon black)	-	-	-	-	-	1	-
MT Thermax (MT carbon black)	-	-	-	-	-	-	1
Ditertiary butyl peroxide	0.8	0.8	0.8	0.8	0.8	0.8	0.8
Carbon black particle diameter (MU)	-	9	17	21	51	180	470
Physical Properties							
Tensile strength, psi	700	840	930	810	1030	870	800
Modulus @ 300% E, psi	170	180	220	170	230	240	230
Elongation, %	620	705	640	660	720	680	625
Hardness, Shore A	40	35	40	42	40	38	38
Aged 3 days @ 600°F:							
Tensile strength, psi	*	390	610	750	690	580	510
Elongation, %		280	400	545	500	450	355
Hardness, Shore A		50	45	45	44	47	50
Aged 7 days @ 600°F:							
Tensile strength, psi	*	450	640	660	640	550	450
Elongation, %		215	365	370	370	365	225
Hardness, Shore A		60	51	52	51	68	65
Aged 2 hours @ 700°F:							
Tensile strength, psi	**	200	240	300	220	210	260
Elongation, %		430	400	660	405	345	115
Hardness, Shore A	90	26	35	29	35	49	57
Tensile strength @ 300°F, psi	430	300	330	350	330	260	300
Elongation @ 300°F, %	415	410	325	305	335	395	415
Tensile strength @ 500°F, psi	200	120	130	120	140	120	130
Elongation @ 500°F, %	200	160	145	170	150	165	180

*Ashen residue

**Too brittle to test

TABLE IV

EVALUATION OF CARBON BLACK AS A HEAT STABILIZER IN HIGH STRENGTH METHYL PHENYL SILICONE RUBBER

Ingredients	Parts by Weight									
K1235	100	100	100	100	100	100	100	100	100	100
Ferric oxide	-	1	-	-	1	-	1	-	-	1
SAF carbon black	-	-	1	5	1	-	-	1	5	1
Dicumyl peroxide (40% active)	2	2	2	2	2	-	-	-	-	-
Ditertiary butyl peroxide	-	-	-	-	-	0.8	0.8	0.8	0.8	0.8
Physical Properties										
Tensile strength, psi	1170	1200	1120	900	1280	1260	1290	1190	950	950
Modulus @ 300% E, psi	940	740	840	750	850	830	680	610	640	820
Elongation, %	345	370	370	345	365	395	405	410	395	335
Hardness, Shore A	50	49	50	58	55	50	50	50	51	53
Aged 3 days @ 600°F:										
Tensile strength, psi	**	580	580	470	690	**	730	630	520	700
Elongation, %		155	140	95	165		255	220	145	225
Hardness, Shore A	93	70	73	82	71	93	61	63	80	60
Aged 7 days @ 600°F:										
Tensile strength, psi	**	510	600	630	590	**	420	630	620	540
Elongation, %		85	80	65	95		130	145	85	135
Hardness, Shore A	94	80	90	90	82	97	70	79	90	77
Aged 2 hours @ 700°F:										
Tensile strength, psi	**	300	360	300	470	**	370	520	270	620
Elongation, %		150	215	125	230		225	285	135	320
Hardness, Shore A	80	54	50	65	55	80	50	50	63	50
Tensile strength @ 300°F, psi	440	460	480	480	470	350	510	400	520	430
Elongation @ 300°F, %	240	220	265	250	235	220	265	260	335	255
Tensile strength @ 500°F, psi	190	210	190	190	200	200	170	200	190	190
Elongation @ 500°F, %	155	135	155	145	125	135	125	155	195	130

**Too brittle to test

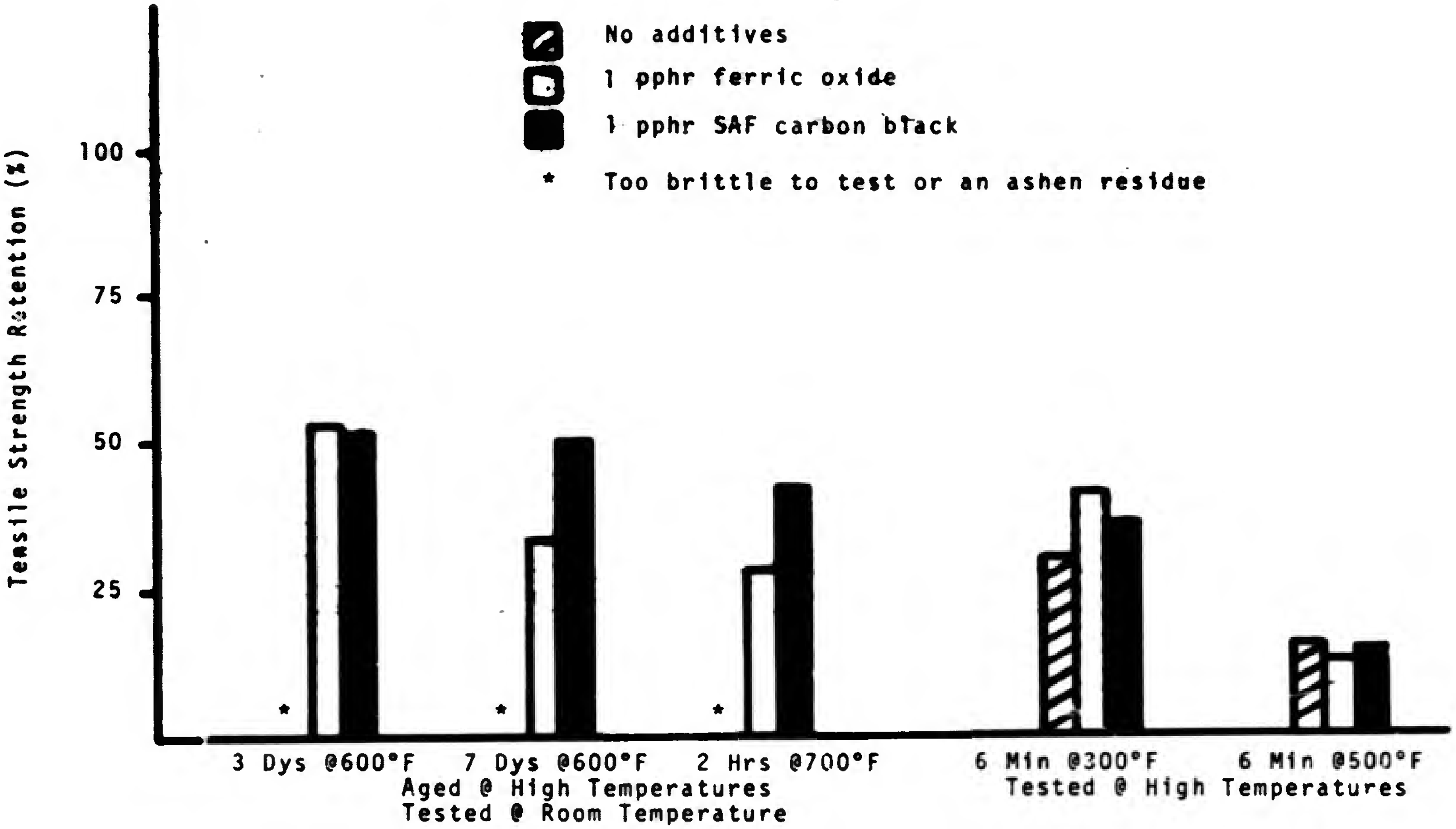


FIGURE 2 Effect of Ferric Oxide and Carbon Black on the High Temperature Properties of a High Strength Methyl Phenyl Silicone Rubber

TABLE V

EVALUATION OF CARBON BLACK AS A HEAT STABILIZER IN FLUOROSILICONE RUBBER

Ingredients	Parts by Weight					
Silastic LS63U	100	100	100	100	100	100
Ferric oxide	-	3	-	-	3	-
SAF carbon black	-	-	1	-	-	1
Ditertiary butyl peroxide	0.8	0.8	0.8	-	-	-
Dicumyl peroxide (40% active)	-	-	-	2	2	2
Physical Properties						
Tensile strength, psi	1050	1060	950	1110	950	1120
Modulus @ 300% E, psi	450	630	420	-	-	950
Elongation, %	500	455	540	310	275	325
Hardness, Shore A	54	58	53	60	61	60
Aged 3 days @ 500°F:						
Tensile strength, psi	*	200	340	*	380	500
Elongation, %		110	235		125	200
Hardness, Shore A		66	66		74	71
Aged 7 days @ 500°F:						
Tensile strength, psi	*	170	160	*	370	390
Elongation, %		80	80		105	125
Hardness, Shore A		70	70		78	77
Aged 2 hours @ 600°F:						
Tensile strength, psi	260	660	680	340	560	750
Elongation, %	170	355	410	155	205	250
Hardness, Shore A	60	57	55	61	60	60
Tensile strength @ 300°F, psi	490	560	420	660	550	650
Elongation @ 300°F, %	335	375	525	270	235	275
Tensile strength @ 500°F, psi	290	310	290	310	240	300
Elongation @ 500°F, %	205	235	300	155	155	170

*Ashen residue

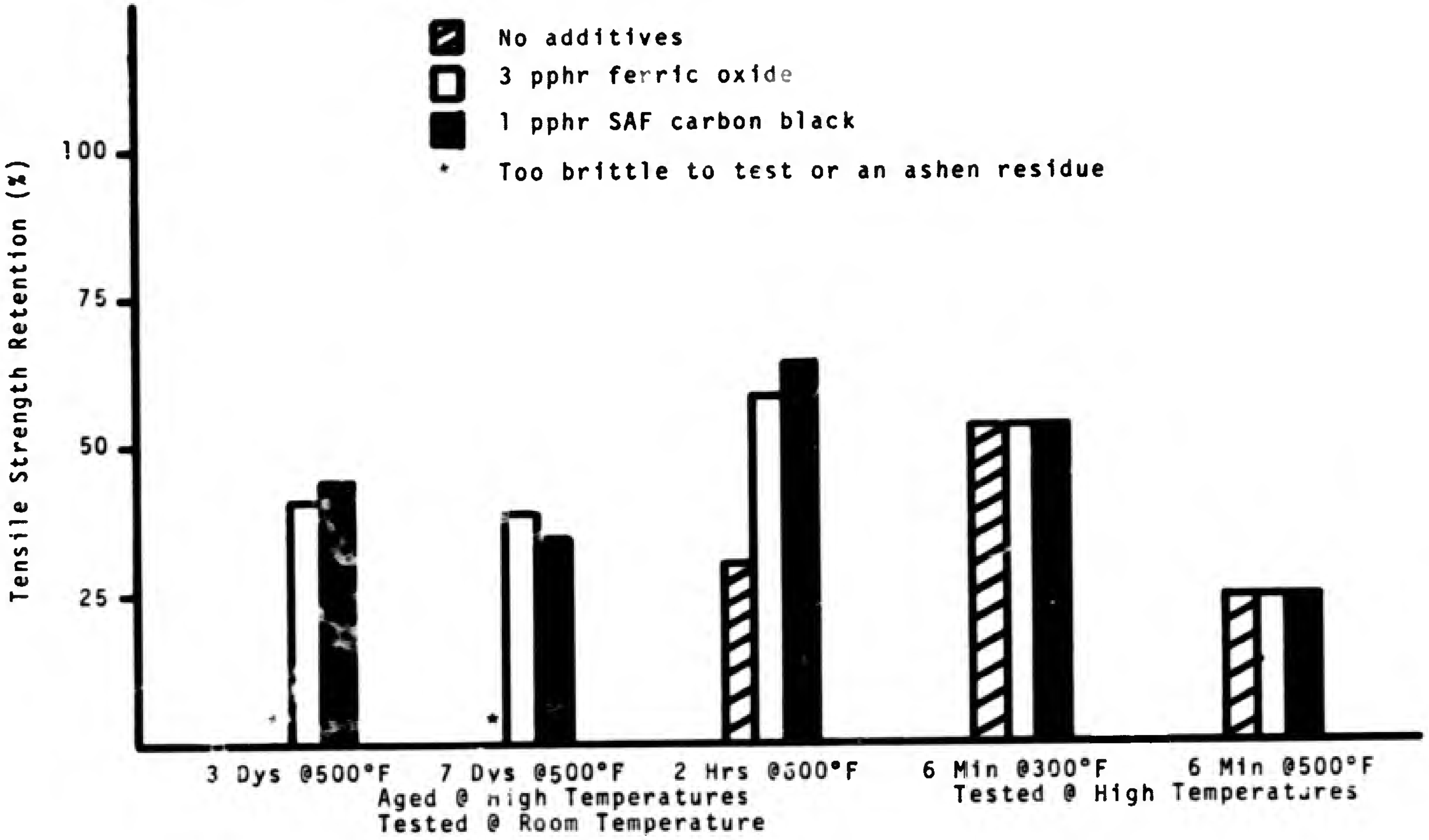
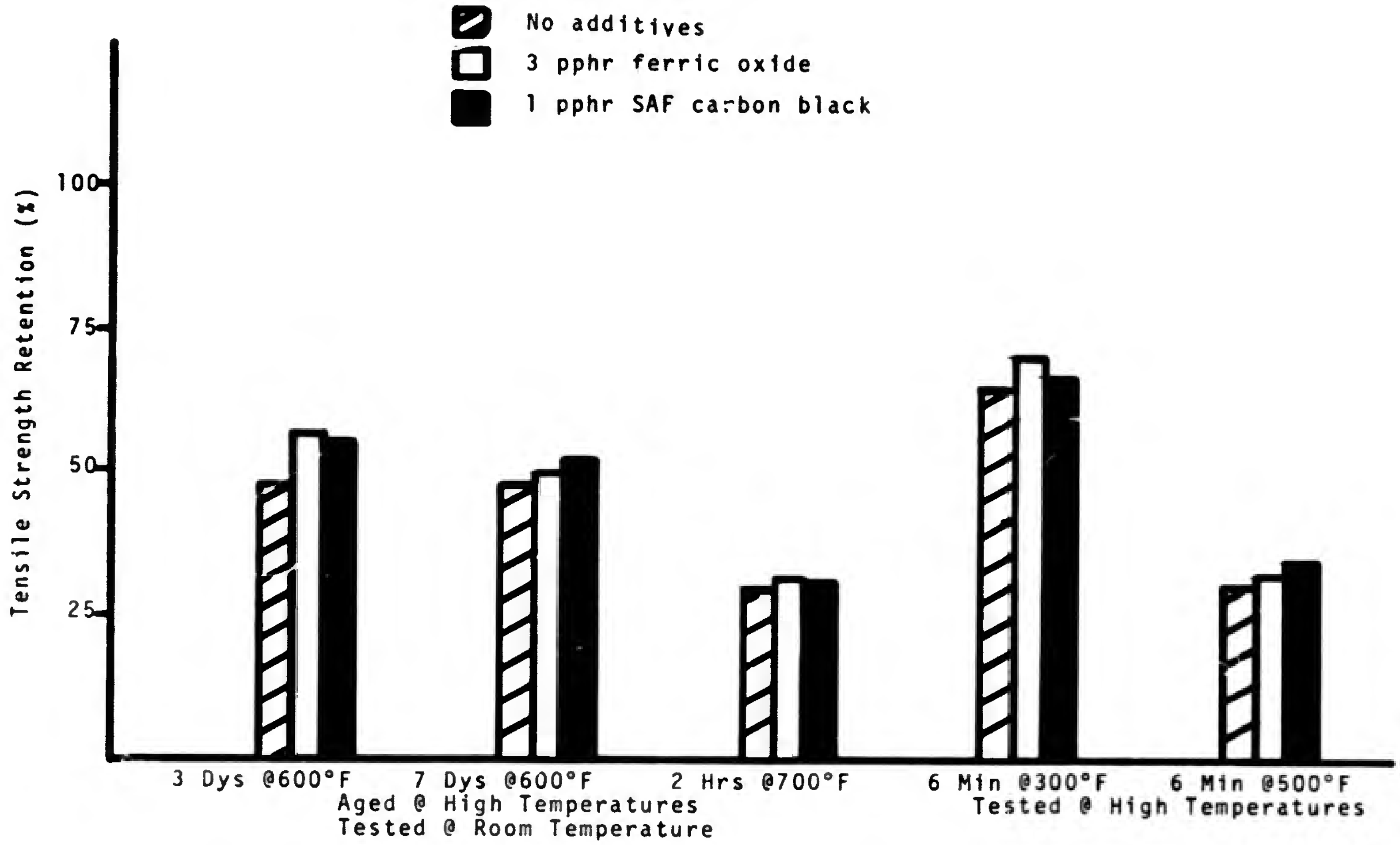


FIGURE 3 Effect of Ferric Oxide and Carbon Black on the High Temperature Properties of a Fluorosilicone Rubber

TABLE VI

EVALUATION OF CARBON BLACK AS A HEAT STABILIZER IN VINYL SILICONE RUBBER REQUIRING NO POST CURE

Ingredients	Parts by Weight					
Silastic 745U	100	100	100	-	-	-
Silastic 746U	-	-	-	100	100	100
Ferric oxide	-	3	-	-	3	-
SAF carbon black	-	-	1	-	-	1
Ditertiary butyl peroxide	0.5	0.5	0.5	0.5	0.5	0.5
Physical Properties						
Tensile strength, psi	850	850	740	1110	1010	950
Modulus @ 200% E, psi	440	430	400	670	700	650
Elongation, %	285	300	270	250	235	240
Hardness, Shore A	51	51	51	59	60	60
Aged 3 days @ 600°F:						
Tensile strength, psi	510	390	360	580	620	430
Elongation, %	235	185	185	160	175	160
Hardness, Shore A	50	50	50	60	60	62
Aged 7 days @ 600°F:						
Tensile strength, psi	340	410	399	510	530	490
Elongation, %	155	165	155	115	130	130
Hardness, Shore A	60	60	60	75	76	79
Aged 2 hours @ 700°F:						
Tensile strength, psi	230	280	300	290	300	270
Elongation, %	305	295	345	210	220	265
Hardness, Shore A	30	35	34	47	50	50
Tensile strength @ 300°F, psi	470	420	420	610	610	580
Elongation @ 300°F, %	195	180	190	175	180	180
Tensile strength @ 500°F, psi	230	230	230	380	330	280
Elongation @ 500°F, %	130	125	125	95	115	105



FAILURE 4 Effect of Ferric Oxide and Carbon Black on the High Temperature Properties of a Vinyl Silicone Rubber Requiring No Post Cure

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