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COAGENTS FOR IMPROVED ELASTIC RECOVERY IN POLYESTER URETHANE ELASTOMERS

TECHNICAL REPORT

By

John A. Williams

January 1966

U. S. ARMY WEAPONS COMMAND

ROCK ISLAND ARSENAL

RESEARCH & ENGINEERING DIVISION

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COAGENTS FOR IMPROVING ELASTIC RECOVERY IN POLYESTER URETHANE ELASTOMERS

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John A. Williams
Laboratory Branch

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ABSTRACT

A number of chemicals were evaluated as coagents in the peroxide vulcanization of polyester urethanes to determine their ability to produce vulcanizates with good elastic recovery.

Several coagents evaluated with high levels (6 pphr) of peroxide produced vulcanizates with improved elastic recovery compared with vulcanizates crosslinked with high peroxide alone. Tensile strength was unaffected while modulus and hardness increased and elongation decreased. Certain coagents when evaluated with low levels (3 pphr) of peroxide produced vulcanizates with improved elastic recovery and only slightly decreased tensile strength compared with vulcanizates cured with high peroxide alone.

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PROBLEM

To discover and evaluate new curing systems for polyester urethanes in an attempt to produce vulcanizates with improved physical properties.

BACKGROUND

It is well known that the physical properties of an elastomeric vulcanizate depend to some extent on the type of curing system. For example, the air aging resistance of butadiene/styrene or butadiene/acrylonitrile rubber cured with sulfur and accelerator can be improved by changing to a thiuram-thiazole curing system. Butyl rubber cured with a phenolic resin has better heat resistance than when cured with sulfur-accelerator combinations.

The main deficiency in some of the peroxide cured polyester urethane vulcanizates is their poor resistance to high temperatures. Genthane S cured with 6 pphr dicumyl peroxide (40% active) produces a vulcanizate with a compression set of ninety five percent after seventy hours at 250°F. This deficiency demonstrates the need in polyester urethanes for a curing system to improve their resistance to high temperatures.

Dicumyl peroxide is a highly active crosslinking agent. This high activity enables this peroxide to activate other chemical agents that have been incorporated into a rubber compound. Such agents are referred to as coagents. It is the intent of this work to discover and evaluate new coagents of peroxide that will produce polyester urethane vulcanizates with improved high temperature resistance.

High peroxide curing systems are now needed to produce polyester urethane vulcanizates with good elastic recovery. High levels of peroxide are known to produce a residue in the rubber (acetophenone) which is detrimental to the strength of bonds formed during the vulcanization bonding of rubber to metal. Effective coagents of peroxide vulcanization could enable the peroxide level to be lowered without loss of properties thereby leading to improved bond strength.

APPROACH

The dicumyl peroxide and coagents were incorporated into portions of the polyester urethane carbon black masterbatch on a 2-1/2" X 7" two roll mill.

Vulcanizates with 3 and 6 pphr dicumyl peroxide were used as controls. The coagents were incorporated in various amounts in an attempt to find the concentration that would give optimum physical properties. The coagents giving optimum physical properties with 6 pphr dicumyl peroxide were evaluated with 3 pphr dicumyl peroxide in an attempt to produce vulcanizates with low peroxide content.

RESULTS AND DISCUSSION

A large number of potential coagents of peroxide were evaluated in two polyester urethanes, Genthane S and SR, with results shown in Tables I, II, III and IV. Only the coagent concentrations that produce optimum physical properties are listed. A cure temperature of 320°F. produced vulcanizates with optimum physical properties.

Table I gives the results of the evaluation of Genthane S cured with 6 pphr dicumyl peroxide and various coagents. The greatest affect on physical properties was obtained with the combination of triallyl cyanurate and polycarbodiimide. Compression set was reduced from 64 to 24 percent at 212°F. and from 95 to 46 percent at 250°F. Triallyl cyanurate without polycarbodiimide also produces a vulcanizate with good elastic recovery.

Another coagent that produced a vulcanizate with low compression set was diallyl carbonate. The addition of polycarbodiimide creates a synergistic effect demonstrated by reduced compression set at 212°F. and 250°F. Tensile strength was increased by the polycarbodiimide addition but the other stress-strain properties were only slightly affected.

Polycarbodiimide incorporated in a Genthane S compound without other coagents produces a vulcanizate with improved stress-strain properties and a significantly improved compression set at 212°F. and 250°F.

All of the coagents listed produced vulcanizates with increased crosslinking shown by increased modulus and hardness with lowered elongation and compression set.

Table II gives the evaluation of vulcanizates of Genthane SR cured with 6 pphr dicumyl peroxide and various coagents. A combination of diallyl adipate and poly-carbodiimide produced the best vulcanizate for elastic recovery. Compression set was reduced from 30 to 9 percent at 212°F. and from 66 to 31 percent at 250°F. Diallyl adipate without polycarbodiimide also produced good elastic recovery. Stress-strain properties suffered

TABLE I

EVALUATION OF COAGENTS IN THE PEROXIDE VULCANIZATION OF GENTHANE S

Ingredients	Parts By Weight																		
Genthane S	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Stearic Acid	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Philblack A	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30
DiCup 40C	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6
Triallyl Cyanurate		3	3	6															
Poly carbodiimide		4			4	8												4	
Diallyl Itaconate							3												
N,N'-p-Benzidene 3,3'-dichloro-bis-maleimide								1											
Diallyl Adipate												3							
Dimer-of-Toluene-2,4-diiso-cyanate									3										
Ethylene Dimethacrylate										5									
Trimethylol-propane-Trimethacrylate											5								
N,N'-M-phenylene-bismaleimide													3		1				
Tetra-allyloxy-propane														3					
Methylene-di-p-phenyl diisocyanate																3			
3,3'-Dimethoxy-4,4'-biphenyl-diisocyanate																	3		
Diallyl Carbonate																		3	3
Physical properties																			
Tensile (psi)	3750	3420	3030	3070	3950	3600	4020	3490	4450	3470	3730	3820	4110	3440	3540	3970	4080	3540	2570
Modulus (100%)	290	560	540	850	310	310	410	360	540	410	500	450	470	320	370	450	350	540	500
(200%)	570	2580	2770	-	900	800	1780	1120	1450	1310	1350	1370	1790	970	1010	1470	1020	1990	2080
(300%)	1570	-	-	-	2070	1680	-	2280	2680	2520	2540	-	-	2140	2160	3030	2150	-	-
Elongation (%)	520	235	210	135	505	560	320	375	500	435	400	305	310	450	400	370	455	300	225
Hardness-Shore A	64	74	71	75	67	65	71	69	75	71	71	69	71	68	70	75	71	72	71
Compression set																			
70 hrs/212°F.	64	24	31	36	40	39	41	39	39	36	42	46	48	56	57	60	61	20	30
70 hrs/250°F.	95	46	56	69	72	79	79	81	92	94	91	91	86	91	89	91	77	44	63

TABLE II

EVALUATION OF COAGENTS IN THE PEROXIDE VULCANIZATION OF GENTHANE SR

Ingredients	Parts by Weight																			
Genthane SR	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Stearic Acid	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Philblack A	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30
DiCup 40C	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6
Diallyl Adipate		3	3																	
Polycarbodiimide		4		4	4				4						8				4	
N,N'-M-phenylene bismaleimide				1		1	3													
Triallyl Cyanurate					3			3								6				
Ethylene Dimethacrylate										5										
Tetra-Allyloxy-propane											3									
Diallyl Itaconate												3								
Dimer of Toluene 2,4-diisocyanate													3							
Trimethylol-propane trimethacrylate														5						
Methylene di-p-phenyl diisocyanate																	3			
3,3'-Dimethoxy 4,4'-biphenyl diisocyanate																		3		
Diallyl Carbonate																			3	3
Physical Properties																				
Tensile (psi)	4280	2830	2300	3080	3290	3870	3100	2560	4130	2620	3330	3440	3770	3710	3700	2930	3860	4160	2910	2820
Modulus (100%)	340	440	720	390	600	320	520	610	390	350	330	530	560	430	370	840	540	380	520	570
(200%)	950	1770	2230	1730	2380	1510	1830	-	1080	1150	960	1370	1720	1500	870	-	1690	1140	1610	1940
(300%)	2230	-	-	-	-	3430	-	-	2260	2290	2100	3210	3130	2960	2890	-	3160	2760	-	-
Elongation (%)	475	245	205	280	245	385	280	195	485	450	440	320	350	360	580	160	365	440	310	260
Hardness (Shore A)	67	73	75	73	73	67	72	74	68	68	65	67	78	70	69	77	72	70	70	70
Compression Set																				
70 hrs/212°F.	30	9	14	16	16	17	21	22	23	25	25	26	31	32	33	36	43	46	16	18
70 hrs/250°F.	66	31	42	39	49	51	50	50	54	54	58	57	67	62	66	69	79	81	41	41

TABLE III

EVALUATION OF COAGENTS IN THE VULCANIZATION OF GENTHANE S AT LOW PEROXIDE CONCENTRATION

Ingredients	Parts By Weight						
Genthane S	100	100	100	100	100	100	100
Stearic Acid	0.2	0.22	0.2	0.2	0.2	0.2	0.2
Philblack A	30	30	30	30	30	30	30
Dicumyl peroxide (40% active)	3	3	3	3	3	3	3
Diallyl Carbonate		1.5		1.5			
Triallyl Cyanurate			1.5		1.5		
Polycarbodiimide		2	2			2	
Diallyl Itaconate							1.5
Physical Properties							
Tensile (psi)	2940	3930	3460	3560	3280	3720	3510
Modulus (100%)	140	350	330	230	290	240	270
(200%)	270	900	810	620	720	480	570
(300%)	590	1810	1890	1300	1670	1120	1300
Elongation (%)	950	590	515	670	500	810	710
Hardness (Shore A)	60	67	66	62	66	64	65
Compression Set							
70 hrs/212°F.	100	40	41	75	60	56	66
70 hrs/250°F.	114	83	70	105	88	103	114

TABLE IV

EVALUATION OF COAGENTS IN THE VULCANIZATION OF GENTHANE SR AT LOW PEROXIDE CONCENTRATION

Ingredients										
Genthane SR	100	100	100	100	100	100	100	100	100	100
Stearic acid	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Philblack A	30	30	30	30	30	30	30	30	30	30
Dicumyl peroxide (40% active)	3	3	3	3	3	3	3	3	3	3
Diallyl carbonate		1.5			1.5					
Polycarbodiimide		2	2				2	2		2
Triallyl Cyanurate			1.5			1.5				
Diallyl Adipate				1.5				1.5		
N,N'-M-phenylene bismaleimide							0.5		0.5	
Physical Properties										
Tensile (psi)	3250	3330	2950	3430	3610	3170	3350	2950	3180	3320
Modulus (100%)	150	340	270	290	340	270	280	290	230	230
(200%)	360	800	780	710	820	770	560	660	570	520
(300%)	850	1700	1680	1600	1840	1790	1250	1310	1500	1110
Elongation (%)	810	590	465	635	565	450	680	600	600	765
Hardness-Shore A	60	67	63	65	63	63	62	65	62	62
Compression Set										
70 hrs/212°F.	55	27	30	32	35	37	33	40	47	47
70 hrs/250°F.	85	57	64	62	62	69	69	71	75	80

from the increased crosslinking caused by these coagents.

N,N'-M-phenylene bismaleimide at 1 pphr produced a vulcanizate with excellent elastic recovery. As the concentration of this bismaleimide increases the physical properties begin to deteriorate. Polycarbodiimide addition improves elastic recovery.

Triallyl cyanurate and diallyl carbonate each produce vulcanizates with good elastic recovery, but only triallyl cyanurate shows improvement with the addition of polycarbodiimide. Without other coagents polycarbodiimide demonstrates improved compression set without affecting the stress-strain properties.

Diisocyanates, dimethacrylates, and diallyl itaconate offer very little improvement in the physical properties of a Genthane SR vulcanizate.

Hydroquinones, quinones, melamines, dioximes and phenol compounds evaluated as potential coagents in Genthane S and SR resulted in weakened or completely destroyed cures.

The coagents which proved effective with 6 pphr dicumyl peroxide were evaluated with 3 pphr dicumyl peroxide. If a good vulcanizate cured with low peroxide can be found it may eliminate some of the problems in rubber to metal bonding caused by high peroxide cures. The results of this evaluation are listed in Tables III and IV.

Triallyl cyanurate and polycarbodiimide proved to be the most effective combination in the low peroxide vulcanization of Genthane S. This vulcanizate gives stress-strain properties comparable to a vulcanizate cured with 6 pphr peroxide and lower compression set.

Diallyl carbonate provided improvement in the physical properties of a low peroxide cured Genthane S vulcanizate but was not as effective as triallyl cyanurate.

Diallyl carbonate proved to be the most effective coagent evaluated with 3 pphr dicumyl peroxide in a Genthane SR vulcanizate. With polycarbodiimide added, compression sets of 27 percent at 212°F. and 57 percent at 250°F. were obtained. These compression sets are better than those obtained with a vulcanizate cured with 6 pphr dicumyl peroxide, but stress-strain properties are not as good.

Triallyl cyanurate, diallyl adipate, and N,N'-M-phenylene bismaleimide produce low peroxide vulcanized Genthane SR with good physical properties.

Table V lists a series of Genthane S and SR vulcanizates cured with 3,6,8 and 10 pphr dicumyl peroxide. Physical properties improved with increasing peroxide concentration, but at ten pphr peroxide, the compression set specimens displayed crumbled edges when tested at 212°F. and 250°F. These high peroxide cured vulcanizates do not display the good elastic recovery obtained with the peroxide coagent cured vulcanizates.

CONCLUSIONS

Diallyl esters of dibasic acids when used as coagents of peroxide produce excellent elastic recovery in the Genthane SR vulcanizates.

Diallyl esters of dibasic acids were not as effective in Genthane S as they were in Genthane SR.

Triallyl cyanurate offered excellent improvement in the elastic recovery of both Genthane S and SR.

N,N'-M-phenylene bismaleimide is an effective coagent at low concentrations in Genthane SR, but is ineffective as a coagent in Genthane S.

Polycarbodiimide in combination with these coagents or by itself in peroxide vulcanization demonstrates the ability to improve compression set with little effect on the stress-strain properties.

Attempts to duplicate the good compression set obtained with these coagents using a high peroxide concentration alone failed.

RECOMMENDATIONS

Coagents showing most promise in this work notably diallyl esters of dibasic acids, triallyl cyanurate and N,N'-M-phenylene bismaleimide should be examined in other elastomers (including polyether based polyurethanes) in order to explore the generality of their application. Also, related family members of these chemical classes should be investigated.

High peroxide curing systems in polyester polyurethanes create the problems of poor rubber to metal bonding and strong residual order. A good low peroxide curing system or a curing system void of peroxide should be sought.

TABLE V

VULCANIZATION OF GENTHANE S AND SR AT VARIOUS PEROXIDE CONCENTRATIONS

Ingredients				
Genthane S	100	100	100	100
Stearic acid	0.2	0.2	0.2	0.2
Philblack A	30	30	30	30
DiCup 40C	3	6	8	10
Physical Properties				
Tensile (psi)	2940	3750	4000	2950
Modulus (100%)	140	290	400	1190
(200%)	270	570	1150	1710
(300%)	590	1570	3090	-
Elongation (%)	950	520	410	250
Hardness (Shore A)	60	64	67	70
Compression Set				
70 hrs/212°F.	100	64	53	41 crumbled edges
70 hrs/250°F.	114	95	85	74 crumbled edges
Genthane SR	100	100	100	100
Stearic acid	0.2	0.2	0.2	0.2
Philblack A	30	30	30	30
DiCup 40C	3	6	8	10
Physical Properties				
Tensile (psi)	3250	4280	3860	3740
Modulus (100%)	150	340	430	560
(200%)	360	950	1410	2300
(300%)	850	2230	3100	-
Elongation (%)	810	475	370	270
Hardness (Shore A)	60	67	71	72
Compression Set				
70 hrs/212°F.	55	30	23	21 crumbled edges
70 hrs/250°F.	85	66	57	46 crumbled edges

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RIA Lab. Rep. 66-382, Jan 66, 20 p., incl. tables, (DA Project No. 1CO24401A329, AMS Code 5025.11.295) Unclassified report.

A number of chemicals were evaluated as coagents in the peroxide vulcanization of polyester urethanes to determine their ability to produce vulcanizates with good elastic recovery. Several coagents evaluated with high levels (6 pphr) of peroxide produced vulcanizates with improved elastic recovery compared with vulcanizates crosslinked with high peroxide alone. Tensile strength was unaffected while modulus and hardness increased and elongation decreased. Certain coagents when evaluated with low levels (3 pphr) of peroxide produced vulcanizates with improved (Cont.) over

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