Blending of NR/BR/EPDM by Reactive Processing for Tire Sidewall Applications. III. Assessment of the Blend Ozone- and Fatigue-Resistance in Comparison with a Conventional NR/BR Compound

Kannika Sahakaro,^{1,2}* Rabin N. Datta,^{1,2} Johan Baaij,³ Jacques W. M. Noordermeer^{1,2}

¹Dutch Polymer Institute, 5600AX Eindhoven, The Netherlands

²Department of Rubber Technology, Faculty of Science and Technology, University of Twente, 7500AE Enschede, The Netherlands ³Elastomer Research Testing, 7420 AC Deventer, The Netherlands

Received 12 August 2005; accepted 17 May 2006 DOI 10.1002/app.25101 Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The NR/BR blend compound formulations for tire sidewall applications contain a set of stabilizers added to prevent degradation mainly due to oxygen, ozone, and heat. 6PPD is the most effective and widely used antiozonant in tire compounds, but is a highly staining material causing a surface discoloration of the tire sidewall. Incorporation of 30 phr EPDM into blends of NR/BR improves the ozone resistance to the required level, without the need of 6PPD. The first two parts of this series have described a reactive processing technique applied to enhance the covulcanization and blend homogeneity, together with their characterization. In the present article, the properties of the NR/BR/EPDM blends prepared by both reactive and straight mixing are tested in com-

INTRODUCTION

In the first two parts of this series^{1,2} it has been shown that by utilizing a reactive processing of the EPDM phase, in which the mercaptobenzothiazole (MBT) moiety of the common accelerator N-cyclohexyl-2-benzothiazole sulfenamide (CBS) is grafted onto the EPDM molecular chain prior to mixing into premasticated NR/BR, significant improvement in tensile strength, elongation at break and blend homogeneity of the terpolymer NR/BR/EPDM vulcanized blends can be achieved. All curatives and reinforcing fillers are first incorporated into the EPDM phase, then the EPDM compound is thermally pretreated for a duration related to the scorch time, and sub-

Journal of Applied Polymer Science, Vol. 103, 2555-2563 (2007) © 2006 Wiley Periodicals, Inc.



parison with those of equivalent conventional NR/BR compounds. The reactive NR/BR/EPDM blend vulcanizates show excellent tensile strength, elongation at break, tear strength, fatigue-to-failure, and ozone resistance in both static and dynamic conditions. The properties are equivalent or even superior to those of the conventional NR/BR tire sidewall compounds. The simple straight mixed NR/BR/EPDM blend vulcanizates distinctively possess inferior mechanical properties compared to those of the reactive mix. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 103: 2555-2563, 2007

Key words: blends; reactive processing; tire sidewall; fatigue resistance; ozone resistance

sequently cross-blended into the NR/BR phase. The grafting of the MBT raises the polarity of the EPDM and hence increases the reactivity of EPDM towards sulfur curing and leads to a more homogeneous distribution of carbon black over the various phases.

The EPDM rubber possesses excellent resistance to ozone, heat, ageing, and weathering due to its highly saturated chemical structure, containing only nonconjugated carbon-carbon double bonds pendant to the backbone chain. It has long been recognized that incorporation of 20-40 phr EPDM into conventional tire sidewall compounds of NR and BR blends is sufficient to compensate for a loss of ozone resistance due to exclusion of the effective but highly staining and discoloring antiozonant, i.e., N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD).^{3–7} The presence of a dispersion of ozone resistant particles, i.e., EPDM phase, in blends of diene rubbers impedes the rate at which the microvoids, caused by ozone attacked on the exposed surface, coalesce into macroscopic cracks.⁸ Waddell⁴ gave an extensive review describing the surface discoloration problem and various approaches to formulate a black tire sidewall with a use of ozone-resistant polymers, including

^{*}Present address: Department of Rubber Technology and Polymer Science, Prince of Songkla University, Pattani Campus, Pattani 94000, Thailand.

Correspondence to: J. W. M. Noordermeer (j.w.m. noordermeer@utwente.nl).

Contract grant sponsor: Dutch Polymer Institute (DPI); contract grant number: DPI project no. 356.

			Compou	nu romu	lations				
Component	Compound formulation (F1-F8)								
		Gum		HAF filled					
	F1	F2 (straight)	F3 (reactive)	F4	F5	F6 (straight)	F7 (reactive)	F8 (reactive)	
NR	50	35	35	50	50	35	35	35	
BR	50	35	35	50	50	35	35	35	
EPDM	0	30	30	0	0	30	30	30	
ZnO	4	4	4	4	4	4	4	4	
Stearic acid	2	2	2	2	2	2	2	2	
CBS	1.98	1.98	1.98	1.98	1.98	1.98	1.98	1.98	
Sulfur	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	
6PPD	2.5	0	0	2.5	0	0	0	0	
TMQ	1	1	1	1	1	1	1	0	
Wax	1.5	1.5	1.5	1.5	1.5	1.5	1.5	0	
HAF N330	0	0	0	50	50	50	50	50	
Sunthene 4240	0	0	0	10	10	10	10	10	
Total weight	115.48	112.98	112.98	175.48	172.98	172.98	172.98	170.48	

TABLE I Compound Formulations

EPDM, halobutyl rubbers, and brominated isobutylene-co-para-methylstyrene. The incorporation of EPDM into NR compounds improves the ozone resistance, but the blends exhibit a higher tangent delta and a higher fraction of dissipated energy.⁹ Under normal running conditions, the tire sidewalls are subjected to extensive flexing and continuous distortion under load at a temperature higher than ambient, due to heat build up. In addition, the sidewalls are susceptible to ozone attack leading to degradation. The tire sidewall must have good resistance to fracture over a range of conditions covering rapid as well as slow crack growth.10 The effect of inclusion of EPDM into the tire sidewall compounds on flex-fatigue performance, ozone resistance, as well as other mechanical properties is therefore very important especially from an application perspective.

In the final part of the series, the properties of NR/BR/EPDM blends, prepared by both reactive and straight mixing techniques, are assessed in comparison with those of conventional NR/BR compounds. In addition to the carbon black filled-compounds for tire application, gum vulcanizates are also investigated to evaluate the properties of the reactive mixed NR/BR/EPDM blends in comparison with those of conventional straight mixes.

EXPERIMENTAL

Materials

The blends consist of the same ingredients as mentioned in part I. Additional ingredients in the compound formulations for this study are *N*-(1,3-dimethylbutyl)-*N'*-phenyl-*p*-phenylenediamine (Santoflex 6PPD, Flexsys B. V., the Netherlands) and microcrystalline wax (Sunolite 240, Flexsys B. V., the Netherlands). All ingredients were used as received.

Preparation of blend compounds

There are eight different compounds denoted from F1 to F8, as the overall recipes shown in Table I. Compounds F1–F3 are gum and F4–F8 are carbon black-HAF filled. For both gum and filled compounds, conventional NR/BR blends and NR/BR/EPDM blends obtained by both straight and reactive mixing were prepared.

The preparation of gum NR/BR (F1), gum straight mix NR/BR/EPDM (F2), and gum EPDM compound for reactive mixing (F3) were prepared using a Brabender Plasticorder 350S mixer having a mixing chamber volume of 370 cm³. The mixer was operated with a rotor speed of 100 rpm, fill factor of 0.7, and an initial temperature of 50°C. All HAF-filled compounds (F4–F8) were prepared by using the corresponding masterbatches of rubber/HAF/oil, pre-prepared according to the formulations given in Table II of part I of this series. The masterbatches were mixed with curatives and other ingredients using a Schwabenthan two-roll mill under a controlled temperature of 50°C.

The mixing procedures for both reactive and straight mixed NR/BR/EPDM blends of both gum and filled

TABLE II Dynamic Ozone Test Conditions

	Test						
Condition	1st	2nd	3rd	4th			
Ozone conc. (pphm)	50 ± 5	50 ± 5	100 ± 5	100 ± 5			
Elongation (%)	10	20	20	30			
Temperature (°C)	40 ± 2	40 ± 2	40 ± 2	40 ± 2			
Relative humidity (%)	50 ± 5	50 ± 5	50 ± 5	50 ± 5			
Frequency (Hz)	0.5	0.5	0.5	0.5			
Time (h)	72	48	24	72			



Figure 1 EPDM cure curves of gum and 50 phr HAF filled EPDM compounds at 140°C for pretreatment step, indicating the pretreatment times used.

compounds are described in detail in part I of this series. There are two more additional additives added to the recipes, compared to those recipes given in part I, i.e., 6PPD and microcrystalline wax. For the Brabender mixing of the gum compounds, 6PPD and wax were added into the compounds together with ZnO, stearic acid, and TMQ. For the two-roll mill mixing of the filled compounds, stearic acid, ZnO, CBS, TMQ, 6PPD, wax, and sulfur were sequentially added.

For reactive mixing, the EPDM compounds with the entire amounts of curatives and other ingredients were first pretreated to a predetermined pretreatment time related to the scorch time of the EPDM phase, as described in detail in part I of this series. Based on this previous study, a pretreatment time of scorch time minus 2 min (t_{s2} -2), and a pretreatment time of scorch time plus 1 min (t_{s2} +1) were selected for gum and filled blends respectively, as shown in Figure 1. The thermal pretreatment of EPDM was carried out at 140°C in a compression molding machine (Wickert WLP1600 laboratory press) using a tensile sheet mold with a thickness of 2 mm.

The pretreated EPDM compounds of both gum and filled were finally cross-blended with NR/BR on a two-roll mill.

Vulcanization

The optimum cure time ($t_{c,90}$) of the fully compounded blends was determined as the time needed to reach 90% of the maximum torque in the RPA 2000 at 150°C, 0.833 Hz, and 0.2° strain according to ISO 6502. The blends were vulcanized in a compression press (Fontijne, Holland) at 150°C.

The resulting blend vulcanizates were left conditioning at room temperature for at least 16 h prior to being tested.

Tensile and tear test

Type 2 dumb-bell test specimens were die-cut from the compression molded sheets and tensile tests were carried out according to ISO 37, with a Zwick tensile tester Model Z 1.0/TH1S at a constant crosshead speed of 500 mm/min.

Trouser test specimens of $100 \times 7.5 \text{ mm}^2$ with a cut of depth 40 mm were die-cut and the test was performed according to ISO 34 Method A with the Zwick tensile tester at a constant crosshead speed of 100 mm/min.

Fatigue-to-failure test

Fatigue-to-failure test was carried out using a Monsanto fatigue-to-failure tester as described in ASTM D 4482-99 at an extension ratio, i.e., the extended length of a specimen to the unextended length, of 2.0. A dumbbell test specimen undergoes a tensilestrain cycle and the number of cycles required to cause failure, as indicated by complete rupture of the test specimen, is recorded. In this test, 12 test specimens of each sample were tested and the median value of the number of cycles required to cause failure was recorded.

Static and dynamic ozone test

Ozone resistance of the blend vulcanizates in both static and dynamic conditions was tested according to ISO 1431-1 using an Argentox ozone test cabinet (Argentox Ozone Technology, Germany).

For the static ozone test, the test specimens of 20 mm in width and 2 mm of thickness, as shown in Figure 2, were clamped, elongated to 20% and tested under the following conditions: ozone concentration 50 ± 5 pphm; temperature $40^{\circ}C \pm 2^{\circ}C$; relative humidity $50\% \pm 5\%$ for 168 h.

For the dynamic ozone test, specimens and test configuration are shown in Figure 2.

Test specimens are normally clamped at the required elongation. However, in this case, the speci-



Figure 2 Test specimens and test configuration in dynamic ozone cabinet. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

mens with modified ends were not tightened between grips, but hanged and elongated using a specially built sample holder (ERT B.V., the Netherlands) to prevent an excessive stress concentration and hence breakage at the grips during the ozone test. The test specimens were attached to the sample holder consisting of a stationary part (top) at a fixed position and a moving part (bottom) which is driven to travel to a specific elongation.

Under unspecified conditions, the test is normally carried out at an ozone concentration of 50 ± 5 pphm. In this test, after the first test at 50 ± 5 pphm ozone concentration, 10% elongation for 72 h, only compound F5 without antidegradants showed cracks on the surface. Further tests on the same test specimens were then carried out consecutively to differentiate between each compound. The tests conditions are shown in Table II.

RESULTS

Cure characteristics of the blends

Figure 3 shows a cure curve of the gum NR/BR blend F1 in comparison with those of the gum NR/ BR/EPDM straight mixed F2 and reactive mixed F3 blends. Figure 4 shows the cure curves of the 50 phr HAF-filled NR/BR blends F4 and F5, in comparison with the 50 phr HAF-filled NR/BR/EPDM straight mix F6 and reactive mixes F7 and F8. The RPA cure characteristics data is also summarized in Table III.

The gum blend compounds show significant differences in both scorch and cure times, whereas the differences in the filled blend compounds containing HAF carbon black are less pronounced. The simple addition of highly saturated EPDM into NR/BR gum compound increases both scorch time and cure time. However, by applying the reactive processing method, a significant reduction in both scorch time and cure time of the blend is observed, as a result of the EPDM thermal-pretreatment step. Considering



Figure 3 RPA cure curves of the gum blend compounds at 150°C. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



Figure 4 RPA cure curves of the 50 phr-HAF filled blend compounds at 150°C. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

the maximum torque levels of the gum blends, the NR/BR blend shows the highest maximum torque, whereas both of the NR/BR/EPDM blends show more or less equivalent values. On the other hand, the NR/BR cure curve shows a higher degree of reversion compared to the NR/BR/EPDM blends.

In the case of filled blends, the maximum torque of the reactive NR/BR/EPDM blend is lower than those of straight mixed NR/BR/EPDM blend and NR/BR blends, respectively. The straight mixed NR/BR/EPDM blend shows only a slightly longer cure time compared to the NR/BR blend, but the reactive NR/BR/EPDM blends clearly show shorter scorch and cure times. The NR/BR/EPDM filled blends again show better torque retention, i.e., less reversion, compared to the NR/BR filled blends.

Tensile and tear properties

Figures 5–7 show the tensile moduli at 100 and 300%, tensile strength and elongation at break of the blend vulcanizates, respectively. The gum NR/BR/EPDM blends exhibit higher moduli, at both 100 and 300% strain, than the gum NR/BR blend. The higher moduli are clearly caused by the presence of the EPDM phase in the blend. For the filled blends, a more or less similar modulus at 100% strain is observed for all cases, but the reactive NR/BR/EPDM filled blends F7 and F8 show a clearly lower modulus at 300% strain compared to the NR/BR and straight mixed NR/BR/EPDM blends. The highest modulus at 300% strain observed for the NR/BR blend containing no 6PPD, TMQ and wax.

The tensile strength of both gum and filled NR/ BR/EPDM blends prepared by the reactive processing method are comparable to those of NR/BR blends, but significantly higher than the NR/BR/ EPDM blends prepared by straight mixing. Both the gum and filled reactive NR/BR/EPDM blends show a slightly higher elongation at break compared to the NR/BR blends, but again significantly higher

		Cure Charac	teristics of the	compo	unus a	(150 C			
	Compound formulation (F1–F8)								
	Gum			HAF filled					
RPA data	F1	F2 (straight)	F3 (reactive)	F4	F5	F6 (straight)	F7 (reactive)	F8 (reactive)	
Min. torque, F_L (dN m)	0.26	0.28	0.24	0.61	0.68	0.66	0.58	0.61	
Max. torque, F_{max} (dN m)	4.23	3.95	3.93	9.21	9.78	9.08	8.28	8.65	
$F_{\rm max} - F_L$ (dN m)	3.97	3.66	3.69	8.60	9.10	8.42	7.70	8.04	
t_{s2} (min)	8.45	12.00	1.17	3.85	4.21	2.63	0.73	0.87	
$t_{c,90}$ (min)	14.78	22.06	4.01	8.25	9.09	10.21	4.37	4.67	

 TABLE III

 Cure Characteristics of the Compounds at 150°C

than the straight NR/BR/EPDM blends. The results are in agreement with those reported in part I of this series.

Figure 8 shows the trouser tear strength of the blend vulcanizates. Considering the mean tear strength of the gum NR/BR and of the reactive NR/BR/EPDM blends, both of them show equivalent values, but higher than the straight mixed NR/BR/EPDM blend. In case of the filled blends, the reactive NR/BR/ EPDM blend F7 containing TMQ and wax shows the highest value, and the NR/BR/EPDM blends show a higher tear strength than the NR/BR blends F4 and F5. On comparing the tear strength of the NR/ BR blends, F4 versus F5, the exclusion of 6PPD apparently has a negative effect on the tear strength of the blend vulcanizate. On comparing the NR/BR/ EPDM blends with equivalent ingredient compositions, F6 versus F7, the tear strength of the reactive NR/BR/EPDM blend is clearly better than that of the straight mix. The exclusion of antidegradants in F8 also results in a reduction of tear strength of the blend vulcanizate.

Flex-fatigue

The cyclical flexing of the test specimens initiates cracks and finally leads to complete rupture of the test specimens. According to the ASTM D 4482 test



Figure 5 Moduli at 100 and 300% strain of the blend vulcanizates.

standard, this fatigue test implies a rupture failure mechanism that stems from the growth of flaws in the specimen, and fatigue data give primarily an estimation of the crack initiation behavior of a rubber vulcanizate.

Considering the fatigue-to-failure of the gum blends shown in Figure 9, the NR/BR blend shows a superior fatigue life compared to the gum NR/BR/EPDM blends. However, between the gum NR/BR/EPDM blends, the reactive mix gives a significant improvement in fatigue life compared to the straight mix. In case of the filled blends, by applying the reactive processing method, a number of cycles to failure of the reactive NR/BR/EPDM blend vulcanizate is observed which is approximately two-fold higher than that of the blend prepared by simple straight mixing. In addition, the fatigue-to-failure (Kc) of the filled reactive NR/BR/EPDM blend shows even significantly higher values than the filled NR/BR blends.

Static and dynamic ozone resistance

All specimens are free of cracks after the static ozone test for 168 h at an ozone concentration of 50 pphm, temperature of 40° C, and 20% elongation.

A subsequent series of dynamic ozone tests, carried out consecutively on the same test specimens



Figure 6 Tensile strength of the blend vulcanizates.

Journal of Applied Polymer Science DOI 10.1002/app

700 gum 600 507 495 477 500 Elongation at break (%) 408 401 366 400 300 200 100 0 F1 F2 F3 F4 F5 F6 F7 F8 Compound formulation

Figure 7 Elongation at break of the blend vulcanizates.

under the heavier test conditions described in Table II, illustrates the resistance to ozone cracking of the NR/BR/EPDM blends in comparison with the conventional NR/BR blends with 6PPD as antiozonant. After the 1st and 2nd test, there are no cracks observed on the surface of any test specimens, except on those of compound F5: NR/BR with no 6PPD. The exclusion of 6PPD from the NR/BR blend clearly gives no protection of the rubber against ozone attack, and cracks in a direction perpendicular to the elongation are clearly observed, as shown in Figure 10. Figure 10 also clearly shows surface discoloration and staining, caused by the use of 6PPD in the F1 and F4 compounds.

After the 3rd test at 20% elongation and 100 pphm ozone concentration for 24 h, very small cracks can be seen on the surface of compound F4 through a $2 \times$ magnifying glass, as shown in Figure 11. The F5 vulcanizates are badly damaged. On the other hand, there are still no cracks visible with $2 \times$ magnification for the blend vulcanizates of compounds F1, F2, F3, F6, F7, and F8. Figure 11 also shows a badly stained F1 gum NR/BR vulcanizate due to 6PPD, in comparison with the light colored F2 gum NR/BR/EPDM blend vulcanizate.

Figures 12 and 13 show photographs of the blend vulcanizates exposed to ozone under the most severe

filled

10.4

13.9

20.6

15.8

15,4

25

20

15

10



Figure 8 Trouser tear strength of the blend vulcanizates.

Journal of Applied Polymer Science DOI 10.1002/app



Figure 9 Fatigue-to-failure of the blend vulcanizates at an extension ratio of 2.

testing conditions of the 4th test, i.e., 100 pphm ozone concentration and 30% elongation. All the gum blend vulcanizates in Figure 12 show small cracks on the elongated area. However, the test specimens of the gum NR/BR/EPDM blends rupture completely soon after cracks appear, indicating a low cut growth resistance of the NR/BR/EPDM blends compared to the NR/BR blends.

Cracks can also be observed on the surface of the filled blend vulcanizates. Figure 13 shows many small cracks on the edge but only a few on the surface of the F4 NR/BR blend with 6PPD. The test specimens of F6, F7, and F8 show a lower number of cracks and better surface appearance. However, these specimens ruptured soon after the development of the cracks, again due to lower cut growth resistance.

DISCUSSION

As has been demonstrated in parts I and II of this series, the reactive processing technique significantly improves tensile properties and blend morphology of NR/BR/EPDM blends. This study covers such blend properties as relevant for tire sidewall application, e.g., flex-fatigue and ozone resistance, in direct comparison with conventional NR/BR sidewall compounds. The cure characteristics of the blends shown in Table III and Figures 3, 4, reveal a shorter scorch time and cure time of the reactive NR/BR/EPDM blends compared to NR/BR and straight mixed NR/ BR/EPDM blends, respectively. This is the result of the preactivated EPDM as, described in detail in part II. Irrespective of the somewhat higher maximum torques of the NR/BR compounds compared to those of the reactive NR/BR/EPDM blends, both gum and filled reactive ternary blend vulcanizates of NR/BR/EPDM yield more or less equivalent tensile properties: tensile moduli, tensile strength, and elongation at break. The inclusion of 30 phr of EPDM into the NR/BR blend by means of the reactive pro-



Figure 10 Photographs of blend vulcanizates of compound F1, F4, and F5 after 2nd test. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

cessing technique, clearly retains the properties of the simple NR/BR blend, as a result of a good crosslink distribution between the various rubber phases and/or a better homogeneous carbon black distribution in the blends. Simple straight mixed NR/BR/ EPDM again results in the poorest tensile properties, reproducible with the results reported in part I.

The tear strengths of the gum NR/BR and reactive NR/BR/EPDM blend vulcanizates are equivalent, but clearly better than the straight mixed gum NR/BR/EPDM. On the other hand, the filled NR/BR/EPDM blends show higher tear strengths compared to the equivalent filled NR/BR blends, and the highest value is observed for the reactive NR/BR/EPDM containing TMQ and wax: compound F7. The higher tear strength of such reactive NR/BR/EPDM blend vulcanizates again must be the result of a better homogeneity of either crosslink distribution or carbon black distribution or both. The dispersion of EPDM particles inside the NR/BR matrix could also impede the tear propagation and hence increases tearing energy or tear strength.

The superior fatigue-to-failure of the gum NR/BR blends, shown in Figure 9, over the gum NR/BR/ EPDM blends, and also the better fatigue property of the filled reactive NR/BR/EPDM blends compared to the NR/BR and the straight mixed NR/BR/EPDM blends, are partially due to their lower modulus, as shown in Figure 5. Considering the NR/BR/EPDM blends only, the reactive mixes do have distinctively better fatigue-to-failure than the straight mixes, confirming the improvement of crosslink and/or filler distribution among the various rubber phases in the ternary blend. The primary controlling parameters affecting the fatigue life are inhomogeneities or flaws present in each sample, caused by the blending, mixing, curing, and sample cutting.¹¹ The reactive NR/ BR/EPDM blends show a much better blend homogeneity compared to that of equivalent straight mix, and hence possess significantly longer fatigue life.

The static ozone test produced no visible cracks in either compound. The presence of microcrystalline wax in the compounds is apparently sufficient to protect the rubber under static conditions due to the formation of a surface film of bloomed wax.^{3,12} However, under dynamic conditions, the inelastic wax film breaks and allows ozone to attack. As tire sidewalls are subjected to both static and dynamic ozone exposures, therefore, an antiozonant for dynamic service condition, e.g., 6PPD, is required. From the dynamic ozone test results it is seen, that the addition of 30 phr of EPDM into the conventional NR/BR blends gives the blends the same good resistance to ozone cracking in both static and dynamic conditions as 2.5 phr of 6PPD.

The dynamic ozone test results for both gum and filled blends suggest that the 6PPD can be totally omitted from the compound formulation when replaced by EPDM. The ternary blends show excellent physical appearance because of absence of the staining antiozonant. Considering the ozone resistance of the NR/BR/EPDM blends, the blend vulcanizates



Figure 11 Photographs of blend vulcanizates of compound F1,F2, F4, and F5 after 3rd test. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



Figure 12 Photographs of gum blend vulcanizates during the 4th test, observed after 24, 48, and 72 h ozone exposure. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



Figure 13 Photographs of 50 phr HAF-filled blend vulcanizates during the 4th test, observed after 24, 48, and 72 h exposure. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

obtained by both straight mix and reactive mix show a similar quality, suggesting that the resistance to ozone cracking is only due to the presence of a sufficient amount of highly saturated EPDM, irrespective of crosslink and/or filler distribution and blend morphology. The ozone resistance of the F8 NR/BR/ EPDM blends indicates that both 6PPD and microcrystalline wax can be omitted from the formulation.

This preliminary laboratory study on the performance of NR/BR/EPDM blends prepared by using the reactive processing technique, gives a promising perspective for application in tire sidewalls. The reactive processing technique is simple to implement and the properties of the ternary blend without staining antiozonants are equivalent to those of a conventional NR/BR tire sidewall compound.

CONCLUSIONS

The physical properties of NR/BR/EPDM blend vulcanizates prepared by using the reactive processing technique under optimum conditions, are equivalent or even superior to conventional NR/BR tire sidewall compounds. The tensile strength and elongation at break of the reactive filled NR/BR/EPDM blends and the NR/BR filled blends are more or less equivalent. The reactive filled NR/BR/EPDM blends show superior tear strength and fatigue-to-failure to those of the equivalent filled NR/BR blend compound. The ozone resistance under both static and dynamic conditions of the NR/BR/EPDM blend vulcanizates with 30 phr of EPDM is excellent and 6PPD can totally be omitted from the tire sidewall compound. The overall properties of the reactive mixed NR/BR/EPDM blends are clearly better than those of the straight mixed NR/BR/EPDM blends, obviously due to the improvement of crosslink and/ or carbon black distribution in the blends as seen in part II, and the reproducibility of the reactive processing technique.

References

- 1. Sahakaro, K.; Naskar, N.; Datta, R. N.; Noordermeer, J. W. M. J Appl Polym Sci 2007, 103, 2538.
- Sahakaro, K.; Talma, A. G.; Datta, R. N.; Noordermeer, J. W. M. J Appl Polym Sci 2007, 103, 2547.
- 3. Layer, R. W.; Lattimer, R. P. Rubber Chem Technol 1990, 63, 426.
- 4. Waddell, W. H. Rubber Chem Technol 1998, 71, 590.
- Sandstrom, P. H.; Lal, J. (to Goodyear Tire & Rubber, Co.). U.S. Pat. 4,003,420 (1977).
- Sandstrom, P. H.; Lal, J. (to Goodyear Tire & Rubber, Co.). U.S. Pat. 4,004,627 (1977).
- Van de Ven, P. M.; Noordermeer, J. W. M. Rubber World 2000, 222, 55.
- Doyle, M. J. Presented at the International Tire Exhibition Conference, Akron, Ohio, September 10–12, 1996.
- 9. Sumner, A. J. M.; Kelbch, S. A. Rubber World 1995, 213, 38.
- 10. Kim, H. J.; Hamed, G. R. Rubber Chem Technol 2000, 73, 743.
- 11. Young, D. G.; Kresge, E. N.; Wallace, A. J. Rubber Chem Technol 1982, 55, 428.
- 12. Veith, A. G. Rubber Chem Technol 1972, 45, 293.