

# Blending of NR/BR/EPDM by Reactive Processing for Tire Sidewall Applications. I. Preparation, Cure Characteristics and Mechanical Properties

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Received 12 August 2005; accepted 17 May 2006

DOI 10.1002/app.25088

Published online in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** EPDM incorporated into blends of natural rubber/butadiene rubber (NR/BR) improves ozone resistance. In this work, the inferior mechanical properties of NR/BR/EPDM blends generally obtained by conventional straight mixing are overcome by utilizing a reactive processing technique. The entire amount of curatives, based on a commonly employed accelerator *N*-cyclohexyl-2-benzothiazole sulfenamide (CBS) and sulfur, is first added into the EPDM phase. After a thermal pretreatment step tuned to the scorch time of the EPDM phase, the modified EPDM is mixed with premasticated NR/BR. The reactive blend vulcanizates show a significant improvement in tensile pro-

perties: tensile strength and elongation at break, as compared to those prepared by straight mixing, in both gum and carbon black-filled blends. The increase of tensile properties in gum and filled reactive blend vulcanizates does suggest that the reactive processing technique leads to more homogeneous blends due to, either a better cross-link distribution, or more homogeneous filler distribution, or both. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 103: 2538–2546, 2007

**Key words:** blends; reactive processing; NR/BR/EPDM; tire sidewall

## INTRODUCTION

A tire sidewall compound is conventionally formulated using natural rubber (NR) and butadiene rubber (BR) with an amine type antidegradant, *N*-(1,3-dimethylbutyl)-*N'*-phenyl-*p*-phenylenediamine (6PPD) as the main antiozonant.<sup>1</sup> Since a tire sidewall in dynamic service condition can easily be attacked by ozone, superior ozone resistance and flex-fatigue are the prime properties required to provide a long service life. The use of conventional antiozonants leads to an undesirable surface discoloration as a result of additive migration.<sup>1,2</sup> In addition, the amount of active antiozonant is depleted in time after reaction with ozone and by migration to and washing off the surface. The increasing demand for life-extension of tires, to reduce cost, and the amounts of used tires, leads to a need of improving

the ozone and thermo-oxidative resistances of tire sidewall compounds.

Ethylene-propylene-diene rubber (EPDM) is a highly saturated elastomer, which is widely used in applications that require good ozone resistance. For reason of its saturated backbone and only a low diene-content in the EPDM, blending of EPDM with highly unsaturated elastomers such as NR and BR generally results in cure rate mismatch and heterogeneous filler distribution in each of the rubber phases. The difference in molar concentrations of double bonds in each of the elastomers results in differences in polarity, number of allylic sites for sulfur vulcanization, and reactivity of the crosslink sites.<sup>3</sup> Curatives, which are considered as polar molecules, generally diffuse more into the higher unsaturated rubbers, i.e., diene phases, resulting in differences in concentration of reactants and hence uneven crosslink distribution.<sup>2,4</sup> Carbon black also prefers to migrate into the higher unsaturated polymers, causing a heterogeneous filler distribution in the blend.<sup>5–10</sup> However, despite the blend incompatibility of EPDM and diene rubbers, blending of EPDM with highly unsaturated elastomers still attracts lots of interest, as such blends may provide a broader range of applications. EPDM is considered an ozone-resistant polymer. The EPDM phase dispersed in blends with highly unsaturated elastomers provides an internal

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

antiozonant,<sup>11</sup> which will not leach out via migration like conventional antiozonants do. Several approaches have been proposed to overcome the cure rate mismatch and achieve covulcanization,<sup>12–24</sup> e.g., by using bis(diisopropyl)thiophosphoryl disulfide (DIPDIS) as a multifunctional additive, maleic anhydride grafted EPDM as compatibilizer, *trans*-polyoctylene rubber (TOR) or liquid BR as compatibilizing agents, a two-stage vulcanization technique or by EPDM-modification through a grafting reaction of accelerators onto EPDM. The latter approach is similar to the work reported in this article, but the procedures adopted are different.

Hashimoto et al.<sup>17</sup> prepared a modified EPDM in solution to achieve bound 2-mercaptobenzothiazole EPDM. Baranwal and Son<sup>19</sup> modified EPDM by reacting EPDM with accelerators such as 4,4'-dithio-bismorpholine (DTBM) and 2-mercaptobenzothiazole (MBT) in solution in the presence of a photosensitizer and UV light. Hopper<sup>20–23</sup> modified EPDM by the addition of certain *N*-chlorothiosulfonamides to the olefinic sites of EPDM in solution and the melt. Cook<sup>24</sup> modified EPDM by mixing sulfur donors like dithiodicapro lactam (DTDC) and dithiodimorpholine (DTDM) with EPDM in an internal mixer at elevated temperature, resulting in EPDM with bound sulfur donor moieties. This was subsequently crossblended with NR and finally mixed with curatives. Various aspects of elastomer blends, including blend characterization, have been reviewed by Hess et al.<sup>25</sup> and Mangaraj.<sup>26</sup>

In the present work, the first part of a series, it is shown how a reactive processing technique, in which a common *N*-cyclohexyl-2-benzothiazole sulfenamide (CBS) is grafted onto EPDM prior to blending with NR/BR, can be utilized to overcome the difficulties associated with cure incompatibility of NR/BR/EPDM blends. The efficiency of the reactive processing technique is assessed by the improvement of the mechanical properties of the blend vulcanizates.

## EXPERIMENTAL

### Materials

The elastomers selected for the blend were natural rubber (SIR20, Standard Indonesian Rubber), butadiene rubber (Kosyn KBR01, Korea Kumho Petrochemical Co.), and EPDM rubber containing ethylidene norbonene (ENB) as third monomer (Keltan 578z, DSM Elastomers B.V., the Netherlands). Keltan 578z contains 4.5 wt % of ENB and 67 wt % of ethylene. The other compound ingredients used were extra pure grade zinc oxide (Merck, Germany), finely divided sulfur (Merck), 95% pure stearic acid (Aldrich, Germany), poly (2,2,4-trimethyl-1,2-dihydroquinoline) or TMQ (Flexsys B.V., the Netherlands), and

*N*-cyclohexyl-2-benzothiazolesulfenamide (CBS) or Santocure<sup>®</sup> (Flexsys B.V.). High abrasion furnace black (HAF-N330, Cabot Corp.) and naphthenic oil Sunthene 4240 (Sun Petroleum Products Co.) were used to prepare the masterbatches in case of filled blends. All elastomers, curatives, and other additives were used as received.

### Preparation of blend compounds by reactive mixing

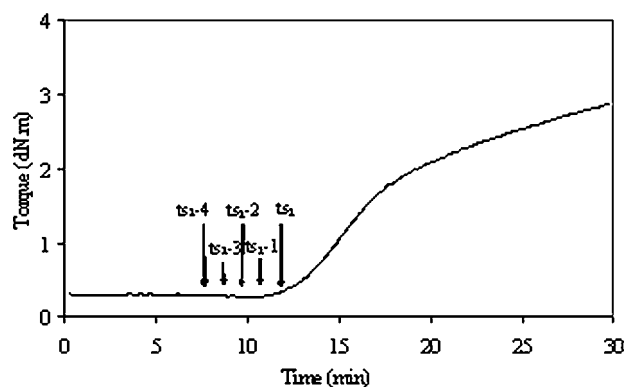
The two types of blend compounds described in this work, are designated according to the procedures adopted for their preparation: *straight mix* and *reactive mix*. With each of the blend types, gum and carbon black-filled compounds were prepared. The overall recipes for both gum and filled blends are given in Table I.

#### Gum reactive mix

The entire amounts of curatives and other ingredients were first incorporated into the EPDM only, using a Brabender Plasticorder 350S mixer having a mixing chamber volume of 370 cm<sup>3</sup>. The mixer was operated with a rotor speed of 100 rpm, fill factor of 0.7, and an initial temperature of 50°C. The EPDM was first masticated for 1 min, and then ZnO, stearic acid, and TMQ were added. After 2 min of mixing, CBS and sulfur were added and the mixing was continued for another 2 min. The final compound temperature before dumping from the mixer was in the range of 115–120°C. Immediately after dumping, the compound was sheeted on a two-roll mill to a thickness of about 2 mm. The resulting EPDM compound was tested for its cure characteristics using a Rubber Process Analyzer (RPA 2000, Alpha Technologies) at 140°C, 0.833 Hz, and 0.2 degree strain according to ISO 6502. Scorch time ( $t_{s2}$ ), i.e., the time to incipient cure, was determined and used as a reference point to assign various pretreatment times of the EPDM, as shown in Figure 1.

TABLE I  
Formulation of NR/BR/EPDM Compounds

Ingredient	Gum blend content (phr)	Filled blend content (phr)
NR	35	35
BR	35	35
EPDM	30	30
Zinc oxide	4	4
Stearic acid	2	2
TMQ	1	1
CBS	1.98 (7.5 mmol)	1.98 (7.5 mmol)
Sulfur	2.5	2.5
HAF N330	–	50
Naphthenic oil	–	10
Total formulation weight	111.48	171.48



**Figure 1** RPA cure curve of the EPDM compound at 140°C with indications of different pretreatment times.  $t_{s2} - 1$  denotes pretreatment time of scorch time less 1 min; etc.

The EPDM compound was then pretreated in a compression molding machine (Wickert WLP1600 laboratory press) at 140°C and 100 bar to a predetermined time taken from the RPA cure curve. The resulting pretreated EPDM sheet was immediately removed from the mold and left to cool down to room temperature in open atmosphere. The pretreated EPDM was later cross-blended with premasticated NR/BR on a Schwabenthan two-roll mill to a final NR/BR/EPDM ratio in the blend equal to 35 : 35 : 30.

#### Filled reactive mix

All carbon black-filled blends were prepared according to the overall recipe given in Table I. Three different masterbatches of rubbers, carbon black, and naphthenic oil were prepared in a Shaw K1 intermeshing internal mixer, having a mixing chamber volume of 4000 cm<sup>3</sup>, according to the recipes given in Table II. The mixer was operated with an initial mixing temperature of 50°C, a rotor speed of 100 rpm, and a fill factor of 0.54. For MB1 and MB2, NR was first masticated for 1 min, and then BR/EPDM or BR was added and mixed for another 1 min, prior to the addition of HAF black and oil. The mixing was continued for another 3–4 min until a stable ram position. The masterbatches were dumped and immediately sheeted on a two-roll mill.

The reactive mix filled compounds were prepared by initially adding the entire amounts of all curing ingredients and additives into the EPDM masterbatch MB3 on the two-roll mill. The cure characteristics of the EPDM masterbatch were measured by using the RPA 2000 at 140°C and the pretreatment times were determined as explained before. The EPDM compounds were subsequently pretreated in the compression molding to the predetermined times, and finally blended on the two-roll mill with MB2 NR/BR masterbatches for the required overall composition as given in Table I.

### Preparation of blend compounds by straight mixing

#### Gum straight mix

The ratio of compound ingredients in the straight-mixed compound was the same as that of reactive-mixed given in Table I. The blend compound was prepared in the Brabender Plasticorder 350S operated under the same conditions. NR was first masticated for 1 min, and then BR and EPDM were added and mixed for another 1 min. ZnO, stearic acid, and TMQ were subsequently added and the mixing was continued for another 2 min. Finally, CBS and sulfur were added into the compound and further mixed for 2 min to complete the mixing. The final compound temperature before dumping from the mixer was in the range of 115–120°C. The dumped compound was immediately sheeted on a two-roll mill. The resulting compound was tested for its cure characteristics using the RPA 2000 at 140°C according to ISO 6502.

#### Filled straight mix

A straight mix filled compound was obtained by mixing the MB1 masterbatch of Table II with the other ingredients according to the recipe given in Table I, on the 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 140°C, 0.833 Hz, and 0.2 degree strain. The blends were then vulcanized during the optimum cure time  $t_{c,90}$  in a Wickert WLP1600 laboratory compression press at 140°C and 100 bar.

### Tensile tests

Type 2 dumb-bell test pieces were die-cut from the compression molded sheet 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.

**TABLE II**  
Formulation of Carbon Black Masterbatches

Ingredient	MB1	MB2	MB3
NR	35	50	–
BR	35	50	–
EPDM	30	–	100
HAF N330	50	50	50
Naphthenic oil	10	10	10

## RESULTS

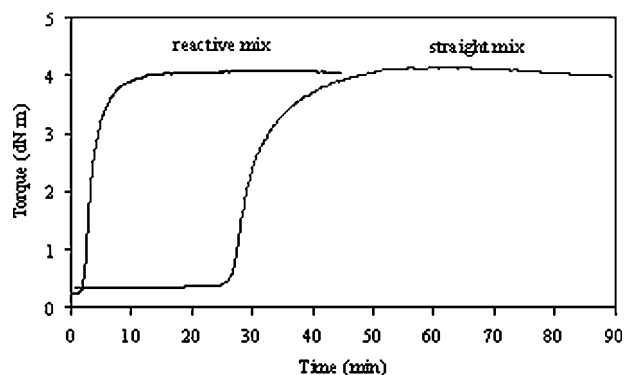
### Cure characteristics of the blends

Figure 2 shows a cure curve of a gum reactive mix prepared by using  $t_{s2} - 2$  pretreated EPDM in comparison with that of a straight mix, and Figure 3 shows a cure curve of a HAF filled reactive mix prepared by using  $t_{s2} + 1$  pretreated MB3 EPDM in comparison with that of a straight mix. By applying the reactive processing method, both scorch time and cure time of the blend are significantly reduced. Considering the maximum torque levels of the gum blends, both show more or less equivalent values. However, in the case of filled blends, the reactive mix shows a lower maximum torque than the straight mix.

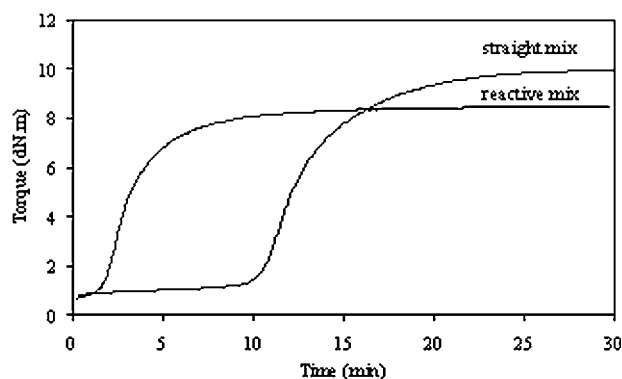
The cure curves of reactive mixed gum NR/BR/EPDM blends prepared by using EPDM with different pretreatment times are shown in Figure 4. The blends with EPDM of different pretreatment times show a shift of scorch time, cure time, and maximum torque. A shorter EPDM pretreatment time gives a longer scorch time and cure time but a somewhat lower maximum torque. Plateaus or flat cure curves are observed for the blends. The RPA 2000 rheometer data: minimum torque ( $F_L$ ), maximum torque ( $F_{max}$ ), torque difference ( $F_{max} - F_L$ ), scorch time ( $t_{s2}$ ), and cure time ( $t_{c,90}$ ) for all blends are given in Table III.

### Tensile properties of the blend vulcanizates

Figure 5 shows the tensile moduli at 100% and 300%, tensile strength, and elongation at break of the gum reactive blends, prepared using different EPDM pretreatment times relative to scorch time,  $t_{s2}$ . The tensile moduli and tensile strength reach their maximum values at a pretreatment time of scorch less 1.5 min ( $t_{s2} - 1.5$ ). The elongation at break remains more or less the same for pretreatment times shorter



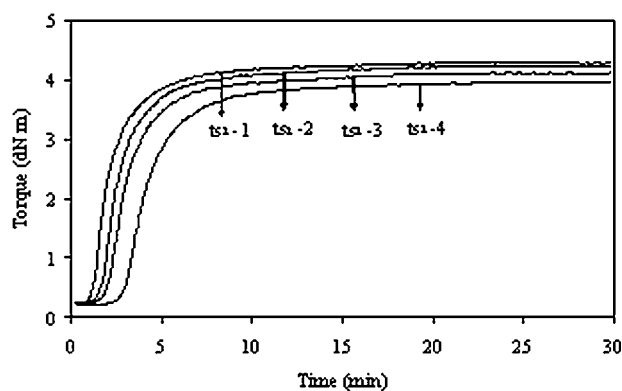
**Figure 2** RPA cure curves of the reactive mixed gum NR/BR/EPDM blend, prepared with  $t_{s2} - 2$  pretreated EPDM, and of a straight mixed gum blend at 140°C.



**Figure 3** RPA cure curves of the reactive mixed 50 phr HAF-filled NR/BR/EPDM blend, prepared with  $t_{s2} + 1$  pretreated EPDM, and of a straight mixed filled blend at 140°C.

than scorch time. The tensile strength shows the most pronounced dependence on the EPDM pretreatment time. By increasing the pretreatment time, the tensile strength of the blend initially increases, but dramatically drops as the pretreatment time approaches the EPDM scorch time. By pretreatment of the EPDM compound beyond its scorch time, the blend remains visually heterogeneous during the blending with NR/BR on the two-roll mill, indicating that the EPDM has lost its ability to be mixed.

In Figure 6, the tensile moduli at 100% and 300% strain, tensile strength, and elongation at break of the HAF-filled reactive blends are shown as a function of EPDM pretreatment times. The EPDM pretreatment times show no significant effect on the tensile moduli. However, the tensile strength and elongation at break are again influenced by the EPDM pretreatment times. The highest tensile strength and elongation at break are observed for an EPDM pretreatment time of scorch plus 1 min ( $t_{s2} + 1$ ). In contrast to the gum blends, in spite of pretreatment of



**Figure 4** RPA cure curves of the reactive mixed gum NR/BR/EPDM blends prepared with EPDM with different pretreatment times at 140°C.  $t_{s2} - 1$  denotes pretreatment time of scorch time less 1 min; etc.

TABLE III  
Cure Characteristics of the NR/BR/EPDM Blends

Type of blend	EPDM pretreatment time (min)	RPA data at 140°C				
		$F_L$ (dN m)	$F_{max}$ (dN m)	$F_{max} - F_L$ (dN m)	$t_{s2}$ (min)	$t_{c,90}$ (min)
Gum straight	0	0.34	4.15	3.81	24.98	40.95
Gum reactive	7.52 ( $t_{s2} - 4$ )	0.23	3.97	3.74	1.79	7.53
	8.52 ( $t_{s2} - 3$ )	0.23	4.12	3.89	1.51	6.37
	9.52 ( $t_{s2} - 2$ )	0.28	4.26	3.98	0.93	5.21
	10.52 ( $t_{s2} - 1$ )	0.33	4.30	3.97	0.89	5.25
	11.52 ( $t_{s2}$ )	0.46	4.37	3.91	0.73	5.35
Filled straight	0	0.78	10.06	9.28	3.03	18.82
Filled reactive	3.15 ( $t_{s2} - 2$ )	0.63	9.03	8.40	1.25	11.16
	5.15 ( $t_{s2}$ )	0.69	8.94	8.25	1.01	8.61
	6.15 ( $t_{s2} + 1$ )	0.67	8.48	7.81	0.93	7.39
	7.15 ( $t_{s2} + 2$ )	0.61	8.83	8.22	0.57	7.01

the filled EPDM compound beyond scorch time to  $t_{s2} + 1$  and  $t_{s2} + 2$ , the blend remained visually homogeneous during the blending with NR/BR on the two-roll mill. A visually heterogeneous blend was only observed when the  $t_{s2} + 3$  pretreatment time was used.

On comparing the tensile stress–strain behaviors of both gum and filled reactive blends, prepared with optimum EPDM pretreatment times, i.e.,  $t_{s2} - 1.5$  for gum and  $t_{s2} + 1$  for filled, with those of straight mixed blends, the improvement in tensile properties due to the reactive processing technique is clearly visible. Figures 7 and 8 show representative stress–strain curves, a comparison between the straight mix and reactive mix, of gum and filled blends respec-

tively. The energy required to break each sample can be obtained by calculating the area under those curves. The energy to break for the gum reactive mix is 2.6 times higher than that of the gum straight mix, and the energy to break for the filled reactive mix is 2.0 times higher than that of the filled straight mix.

In Figure 9, the stress–strain curves of the optimum gum and filled reactive mixed blends are compared. Both gum and filled blends yield similar elongation at break. The tensile moduli and tensile strength of the filled blends are increased as a result of carbon black reinforcement.

The tensile properties of the straight mixes in comparison with those of the reactive mixes are sum-

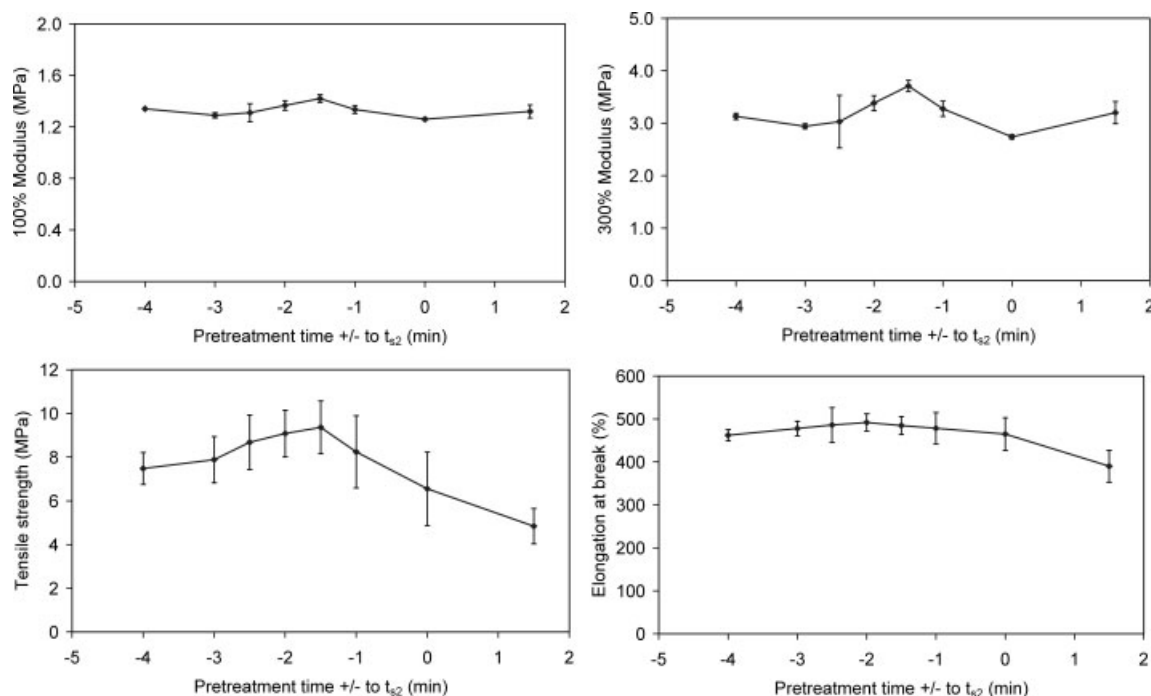
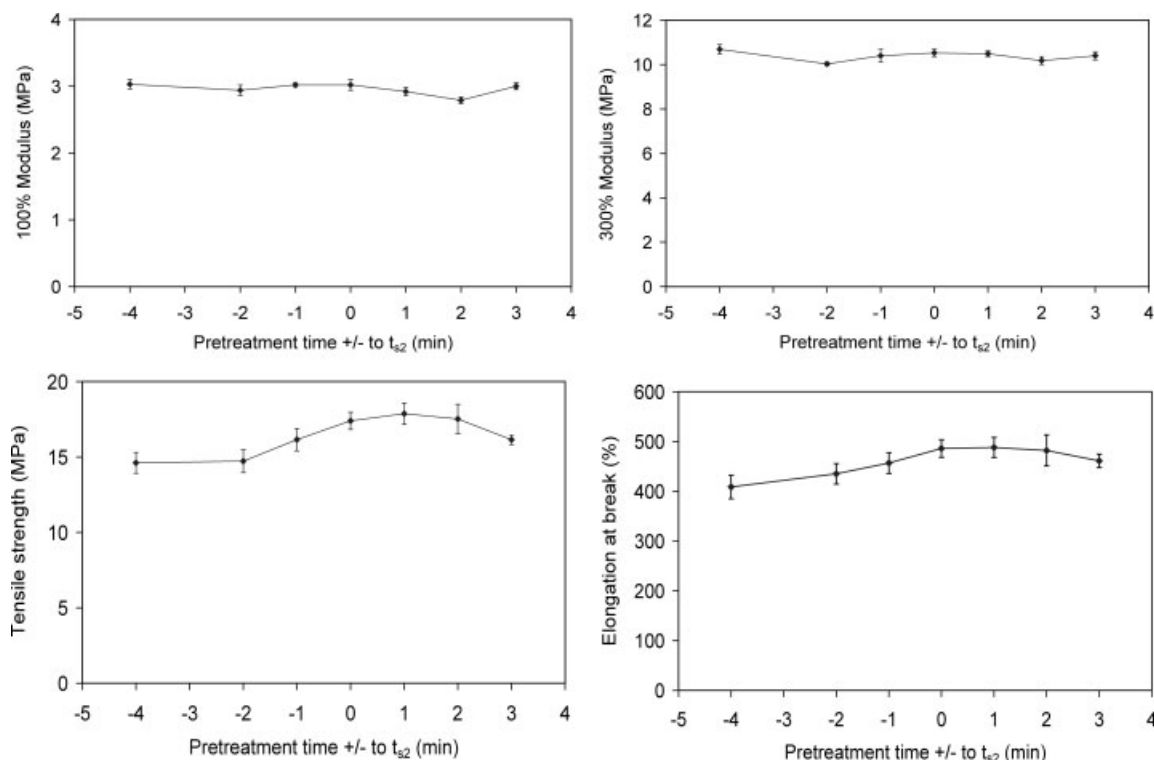


Figure 5 Moduli at 100% and 300% strain, tensile strength, and elongation at break of gum reactive NR/BR/EPDM blends, prepared using different EPDM pretreatment times relative to scorch time,  $t_{s2}$ .



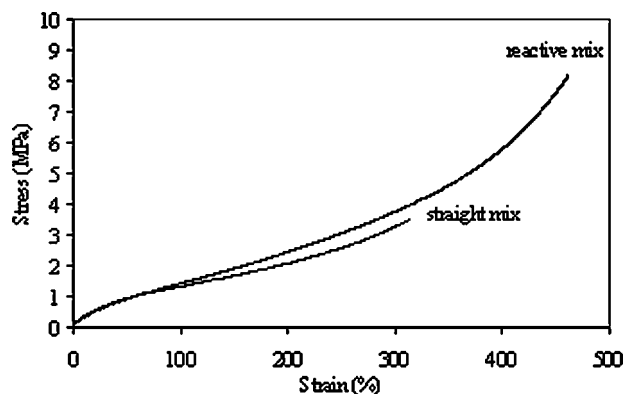
**Figure 6** Moduli at 100% and 300% strain, tensile strength, and elongation at break of 50 phr-HAF filled reactive NR/BR/EPDM blends, prepared using different EPDM pretreatment times relative to scorch time  $t_{s2}$ .

marized in Figure 10. Here, the reactive mixed blends with the highest tensile properties are demonstrated. The gum blend was prepared by using an EPDM pretreatment time equal to  $t_{s2} - 1.5$ , and the filled blend was obtained by using the  $t_{s2} + 1$  EPDM pretreatment time.

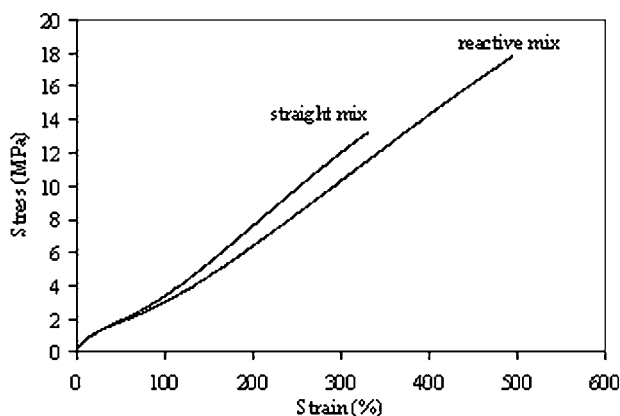
**DISCUSSION**

The results in Figures 7–10 have demonstrated that, for NR/BR/EPDM blends, by first premixing the ac-

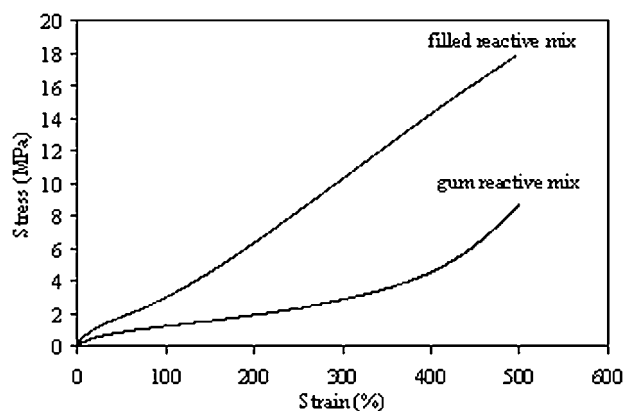
celerator CBS and sulfur into the EPDM phase and subsequent thermal pretreatment, and next blending with the premasticated NR/BR, a significant increase in tensile strength and elongation at break is obtained. The moduli at 100% and 300% strain do not change very much. The significant improvement in tensile strength and elongation at break for the reactive blends, does suggest that the ternary blends prepared through reactive mixing possess a better cross-link distribution and/or carbon black distribution between the three rubber components, as compared



**Figure 7** Stress–strain curves of gum NR/BR/EPDM blends: straight mix versus reactive mix with EPDM pretreatment time  $t_{s2} - 1.5$  at 140°C.



**Figure 8** Stress–strain curves of filled NR/BR/EPDM blends: straight mix versus reactive mix with EPDM pretreatment time  $t_{s2} + 1$  at 140°C.



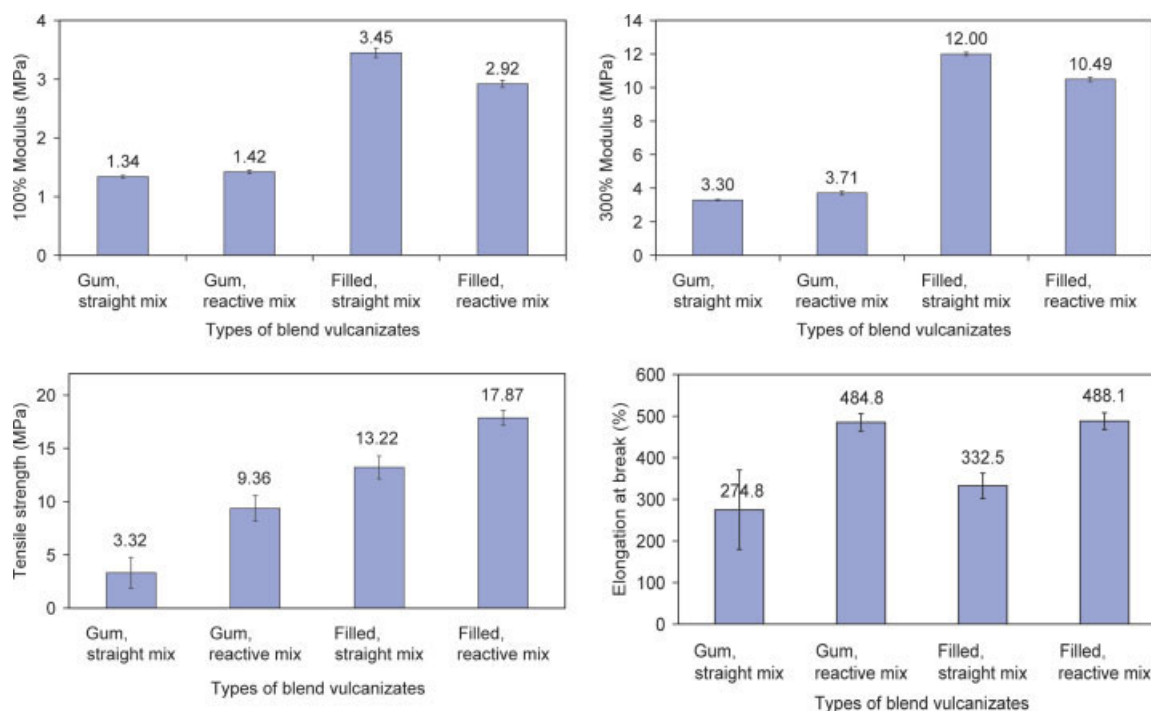
**Figure 9** Stress–strain curves of the reactive mixed NR/BR/EPDM blends: gum versus filled.

to those of the blends prepared by conventional straight mixing. The accelerator used in this study: CBS, is the most commonly used accelerator in the rubber industry, and the experimental principles used for the pretreatment of the EPDM are very simple and easier to implement on an industrial scale, compared to the EPDM modification techniques in solution as mentioned in the introduction.

The pretreatment time of the EPDM for reactive mixing with NR/BR shows an influence on the tensile properties of the blends, as shown in Figures 5 and 6. The effect is more pronounced in the gum blend compared to the filled blend, and the tensile

strength shows the most pronounced dependence on the EPDM pretreatment time. The tensile moduli, tensile strength, and elongation at break of the gum reactive blends show their maximum values at a pretreatment time of scorch time minus 1.5 min ( $t_{s2} - 1.5$ ), whereas those of the filled blends show the maximum properties at a pretreatment time of scorch time plus 1 min ( $t_{s2} + 1$ ). The high viscosity of the filled blends affects the torque reading at the beginning of the RPA measurement, and hence the scorch times obtained by the RPA are slightly shorter for the filled blends relative to the gum compounds at the same stage of chemical cure. By increasing the pretreatment time, the tensile strength of the blend initially increases; however, the tensile strength dramatically drops after the pretreatment surpasses the scorch time, especially in the case of the gum blend. By pretreating the EPDM compounds for too long, the EPDM is no longer processable, and the blends become visually heterogeneous during the blending with NR/BR on the two-roll mill. The results indicate that a suitable pretreatment time needs to be met to optimize the tensile properties of the blends.

Comparing the cure characteristics of the NR/BR/EPDM blends obtained by means of the reactive and straight mixing (Figs. 2 and 3), there is only a small difference in the maximum torque levels for both gum blends, but in the case of filled blends, the filled straight mix shows a higher maximum torque. This can be attributed to a poorer carbon black distribution among the rubber phases for the straight



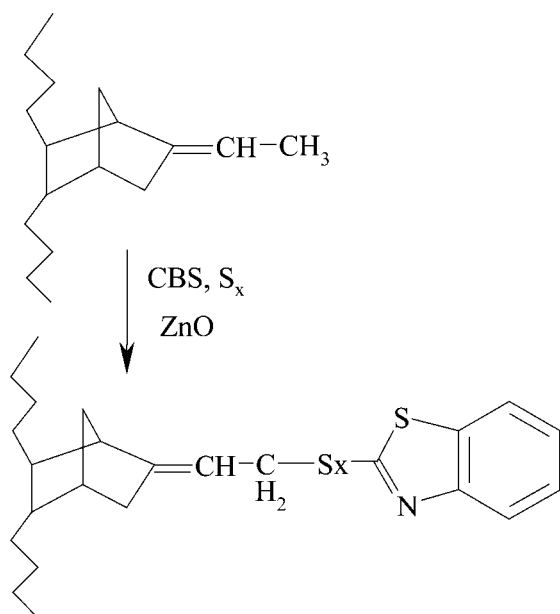
**Figure 10** Tensile properties of gum and filled NR/BR/EPDM blends; straight mixes versus reactive mixes. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

mix, as will be characterized in detail in the next article of this series.

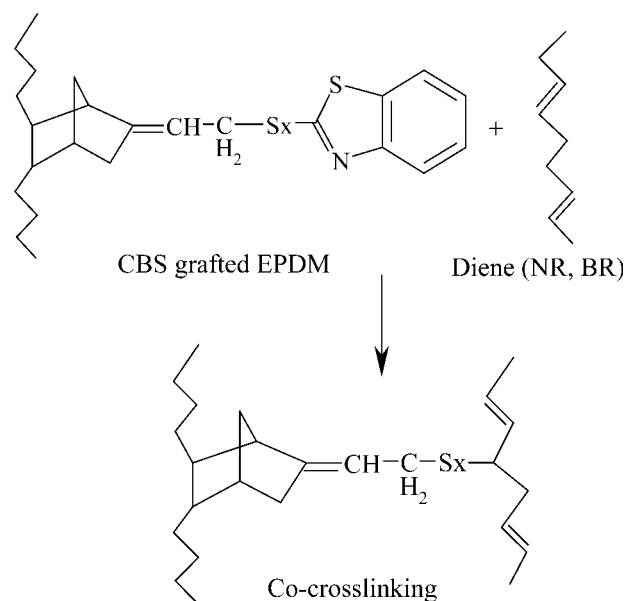
The EPDM pretreatment step for the reactive mixing results in a remarkable decrease of both the scorch time and cure time of the blend compounds to values common for simple NR/BR systems. It is a strong indication that the EPDM phase has been activated by the pretreatment step to vulcanize at similar speed as NR and BR, in spite of its much lower unsaturation levels. Following the studies mentioned in the introduction, it is obvious to assume a grafting reaction of the CBS onto the EPDM molecular chain.

At the early stage of vulcanization, the generally accepted mechanism starts with a reaction of accelerator CBS with the activator ZnO to give an active accelerator complex, which subsequently reacts with molecular sulfur to give an active sulfurating agent.<sup>27,28</sup> This sulfurating species then reacts with the allylic hydrogens of the ENB third monomer of the EPDM chains, resulting in an EPDM intermediate or crosslink precursor, as shown in Figure 11. We have characterized this intermediate, as will be reported in the next part of this series. Since the pretreatment step was stopped more or less at the beginning of cure, further reactions of the EPDM-bound intermediates with other EPDM chains remained minimal. The pretreated EPDM remained processable in the step of crossblending with NR/BR on the two-roll mill.

After the pretreatment step, the EPDM with pendant CBS fragments results in a restriction of the curative migration from the EPDM phase towards the higher unsaturated NR/BR phases and consequently in an improved crosslink distribution. By applying



**Figure 11** EPDM-bound intermediate for the reactive blends.



**Figure 12** Cocrosslinking between pretreated EPDM and diene rubbers (NR/BR).

the reactive mixing technique, the co-crosslinking between EPDM and diene phases, as schematically displayed in Figure 12, is also improved. Since a light crosslinking may also take place after the pretreatment step, the migration of carbon black in the filled blend is also reduced, and hence results in a more homogeneous carbon black distribution between the three rubber phases.

## CONCLUSIONS

The tensile properties of NR/BR/EPDM rubber blends are significantly increased by using a reactive processing technique: the entire amounts of curatives and compounding ingredients are first incorporated in the EPDM-phase; the latter is then subjected to a suitable preheating till close to scorch; and finally the pretreated EPDM is blended with premasticated NR/BR. The curatives consisted of the common accelerator CBS and elemental sulfur. The scorch and cure times of the final blends prepared with the reactive mixing are significantly reduced to values common for NR and BR compounds, relative to those blends prepared by conventional mixing sharing more typical EPDM-values. The tensile properties of the reactive blends are dependent on the EPDM pretreatment times. Under optimum conditions, the tensile strength and elongation at break are significantly higher for both gum and filled blend vulcanizates.

The authors thank Dr. Auke G. Talma for fruitful discussions.



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