

# Novel Dynamic Vulcanization of Polyethylene and Ozonolysed Natural Rubber Blends: Effect of Curing System and Blending Ratio

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**ABSTRACT:** Dynamic vulcanization was studied in terms of the change in  $\alpha$ -relaxation temperatures of the LDPE matrix, morphology, and mechanical properties of LDPE/ozonolysed NR blends which were vulcanized at various blend ratios and with different curing systems, i.e., peroxide and sulfur systems. The ozonolysed NR with  $\overline{M}_w = 8.30 \times 10^5 \text{ g mol}^{-1}$  and  $\overline{M}_n = 2.62 \times 10^5 \text{ g mol}^{-1}$ , prepared by the *in situ* ozonolysis reaction of natural rubber latex, was used in this study. The significant change in the  $\alpha$ -relaxation temperature of LDPE in the LDPE/ozonolysed NR, dynamically vulcanized using the sulfur system, suggested that sulfur vulcanization of the blend gave a higher degree of crosslink density than

using peroxide and corresponded with the improved damping property and homogenous phase morphology. However, the peroxide cured blends of LDPE/ozonolysed NR gave more improvement of tensile strength and elongation at break than the sulfur cured system. Furthermore, the mechanical properties of tensile strength, elongation at break, and damping were improved by increasing the ozonolysed natural rubber content in both DCP and sulfur cured blends. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 120: 2606–2614, 2011

**Key words:** vulcanization; polyethylene (PE); rubber; polymer blends; thermal properties

## INTRODUCTION

The aims of the blending process are to improve the mechanical properties and elasticity of the final product, together with assessment of its thermoplastic processing properties. However, the main problem in blending rubber and thermoplastic materials is that of compatibility. A typical method for the improvement of the compatibility and also stabilization of the phase morphology of a thermoplastic elastomer is known as dynamic vulcanization. This method, as a process for preparing thermoplastic elastomer vulcanizate (TPV), involves adding the melt, under a high shear rate, to the elastomer with rigid thermoplastic and consequent vulcanization of the elastomer phase under continuous mixing.<sup>1–6</sup> The dynamic vulcanization process is believed to significantly improve the blending properties, resulting in

optimum tensile strength, fatigue resistance, upper service temperature performance, phase morphology, stability, and resistance to organic solvents over the static vulcanization process.<sup>1</sup> TPV is therefore considered to be a competitive industrial product.

Although TPVs prepared from blends of polyethylene and natural rubber have been studied by various research groups,<sup>3,7–16</sup> the specific high molecular weight and presence of impurities contraindicates the use of natural rubber either in the blends or in the thermoplastic elastomer vulcanizates. The special viscoelastic properties of natural rubber, combined with the dynamic properties of vulcanizate HDPE/NR, improved the energy absorption characteristics during cyclic deformation, suggesting the suitability of the material for use as a vibration damper for aerospace applications.<sup>7</sup> On the other hand, the compatibilizers were known to improve both the mechanical and dynamic properties of the blends. Recently, Nakason et al.<sup>14</sup> have claimed that TPV based on the HDPE and natural rubber (NR) blends, with the addition of a phenolic resin compatibilizer, improved tensile strength and elongation at break, with a lower value of tension set. Pichaiyut et al.<sup>15</sup> studied the effects of compatibilizer type and loading on the properties of TPV prepared from maleated natural rubber and high density polyethylene vulcanizate in the weight ratio of 60/40. It was found that a

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TABLE I  
Formula of Sulphur Curing System

Ingredients	Loading (phr)
LDPE/Ozonolysed NR	70/30, 60/40, 50/50
Flectol <sup>®</sup> H	1
ZnO	3.5
Stearic acid	2
CBS	0.9
Sulphur	1.8

phenolic modified polyethylene compatibilizer increased the dispersion of the vulcanized rubber domains in the HDPE matrix, resulting in enhancement of the mechanical properties with respect to tensile strength and elongation at break, as well as dynamic properties. In addition, Magaraphan et al.<sup>16</sup> studied the morphology of binary and ternary reactive blends (in the ratio of LLDPE/NR = 90/10) using maleic anhydride as a reactive agent with and without the admixture of dicumyl peroxide (DCP). This finding suggested that an *in situ* graft copolymer of LLDPE-g-NR was formed, acting as an *in situ* compatibilizer in this blend system, with reduction of rubber domains corresponding with the addition of DCP.

As known in the case of NR, not only its microstructure but also its high molecular weight makes it uniquely different from other synthetic rubbers in terms of the difficulty of mixing with other polymers. Boochathum et al.<sup>17</sup> found that *in situ* ozonolysis, affected by passing ozone into natural rubber latex, was an important method on an industrial scale because the ozonolysis was both effective and clean reaction. In addition, adding silica to the ozonolysed natural rubber was found to facilitate the process in contrast to the conventional silica-filled natural rubber that required a coupling agent.<sup>18</sup> Furthermore, Utara and Boochathum<sup>19</sup> investigated the effect of molecular weight of the ozonolysed natural rubber and polyethylene types on the compatibility blends, while low molecular weight ozonolysed NR was found to improve the interfacial adhesion between natural rubber and polyethylene. So far there was no such interfacial adhesion observed between the original high molecular weight NR and thermoplastic.

This study attempted to improve the mechanical properties of the compatible polyethylene (LDPE) and ozonolysed natural rubber blends via dynamic vulcanization. The aims were to clarify the novel dynamic vulcanization properties of the LDPE and ozonolysed natural rubber blends and the effect of dynamic vulcanization systems (i.e., sulfur and peroxide) on the crystallization behavior of LDPE in the vulcanizate. In addition, the effect of the vulcanization system and blend ratio on morphology, damping, and mechanical properties of the LDPE/ozonolysed natural rubber vulcanizates were also clarified.

## EXPERIMENTAL

### Materials

Ozonolysed natural rubbers with molecular weights of  $\bar{M}_w = 8.30 \times 10^5 \text{ g mol}^{-1}$  and  $\bar{M}_n = 2.62 \times 10^5 \text{ g mol}^{-1}$  were prepared by *in situ* ozonolysis of natural rubber latex as described in the previous report.<sup>17</sup> The rheology properties of prepared ozonolysed NR and low density polyethylene (LDPE, ST1018) supplied by Petrochemical Industry Public were also described in the previous study.<sup>19</sup> *N*-cyclohexyl-2-benzothiazole sulfonamide (CBS), zinc oxide (ZnO), stearic acid, sulfur, dicumyl peroxide (DCP), Flectol<sup>®</sup> H, and *n*-heptane were purchased and used as received.

### Thermoplastic vulcanizate preparation

Polyethylene and ozonolysed NR were blended in various ratios of LDPE/ozonolysed NR = 70/30, 60/40, and 50/50. After melting the LDPE for 2 min, the ozonolysed NR and antioxidant (Flectol<sup>®</sup> H) were added to the chamber and the mixing process continued for 4 min. For peroxide curing, 3 phr DCP was added and mixing was continued for an additional 2 min. Compound formulation and mixing steps for sulfur curing using the same blend ratio as peroxide curing are shown in Tables I and II, respectively.

The mixtures were removed and subsequently laminated into sheets at room temperature, using a two-roll mill, before being molded for 10 min at 150°C using a compression mold (Caver, Model 3925). The uncured blends of LDPE and ozonolysed NR were prepared by the same method as TPV's preparation, but without addition of curing agents.

### Morphology

The morphology of some selected blends was investigated using a scanning electron microscope (SEM) (JEOL, JSM-5800LV). Molded samples were cryogenically cracked in liquid nitrogen to avoid any possibility of phase deformation. The uncured natural rubber component was extracted by immersing the fractured surfaces in cyclohexane for about 24 h at room temperature. The samples were dried in a

TABLE II  
Mixing Steps of Sulphur Curing System

Ingredients	Mixing time (min)
LDPE	2
Ozonolysed NR	2
Stearic acid	1
ZnO	1
Flectol <sup>®</sup> H	2
CBS	1
Sulphur	Till plateau torque reached

vacuum oven at 40°C for 3 h and gold-coated in preparation for the scanning electron microscope morphology examination.

### Mechanical properties

Tensile strength and elongation at break of the blends were measured using a Tensile Tester (Instron 1011) at a crosshead speed of 500 mm min<sup>-1</sup> with regard to ASTM D412-80.

### Damping properties

Measurements of tan  $\delta$  were conducted from -140 to 120°C with a heating rate of 3°C min<sup>-1</sup> using a Dynamic Mechanical Analyzer (Mettler Toledo DMA/SDTA861). All measurements were performed on specimens with dimensions of 9 mm  $\times$  3 mm  $\times$  0.2 mm at 1 Hz in tension mode; using 0.4N maximum force and 80- $\mu$ m maximum displacement amplitude.<sup>20</sup>

### Crystallization behavior of polyethylene

The crystallization behavior of polyethylene in the TPVs was investigated by means of recording the changes in  $\alpha$ -relaxation temperature, using a Dynamic Mechanical Analyzer (Mettler Toledo DMA/SDTA861). The measurement conditions were identical to those used for the damping properties.

### Crosslink density measurement

The crosslink density of thermoplastic vulcanizate was determined using the swelling method.<sup>21</sup> TPVs were swelled in *n*-heptane until the swelling reached the equilibrium stage, which occurred after 48 h. The samples were removed from the solvent and excess solvent removed from the surface of the sample before being weighed to an accuracy of 0.1 mg. The samples were then placed in a vacuum oven at a constant temperature of 40°C, being considered to be dry when the % change in weight was less than 5%. The dried samples were then weighed and crosslink density was calculated using the Flory-Rehner equation, as shown in eq. (1):

$$v = -[\ln(1 - V_r) + V_r + \chi V_r^2]/2(V_r^{1/3} - 0.5V_r)\rho V_0 \quad (1)$$

where  $v$  is the crosslink density,  $V_r$  is the volume fraction of the rubber in swollen thermoplastic vulcanizate,  $V_0$  is the molar volume of solvent used (146.62 cm<sup>3</sup> mol<sup>-1</sup>) and  $\chi$  is the polymer-solvent interaction parameter. The volume fraction was calculated based on the equation below.

$$V_r = \frac{[(W_d - W_f)/\rho]/\{(W_d - W_f)/\rho\}}{+ \{(W_s - W_d)/\rho_s\}} \quad (2)$$

where  $W_d$  is the weight of the sample after swelling and drying,  $W_f$  is the weight of the filler in the sample,  $W_s$  is the weight of the swollen sample, and  $\rho_s$  is the density of the solvent (0.682 g cm<sup>-3</sup>).

The polymer-solvent interaction parameter ( $\chi$ ) can be calculated using the equation below<sup>22</sup>:

$$\chi = \frac{V_i(\delta_1 - \delta_2)^2}{RT} \quad (3)$$

where  $\delta_1$  is the solubility parameter of polymer blend,  $\delta_2$  is the solubility of solvent (7.4 (cal cm<sup>-3</sup>)<sup>1/2</sup>),  $R$  is the gas constant (1.9872 cal mol<sup>-1</sup> K<sup>-1</sup>),  $T$  is the absolute temperature (K),  $V_i$  is the molar volume of *n*-heptane (146.62 cm<sup>3</sup> mol<sup>-1</sup>). For TPVs, the Hildebrand solubility parameters of homopolymer were calculated using the equation<sup>22,23</sup>:

$$\delta = \frac{\rho \sum F_i}{M} \quad (4)$$

where  $\rho$  is the polymer density at the given temperature,  $\sum F_i$  is the sum of all the molar attraction constants in the repeat groups, and  $M$  is the molecular weight of the repeat group. The solubility of the blends or copolymer can be calculated using the equation<sup>22</sup>:

$$\delta_1 = \sum \delta_i \phi_i \quad (5)$$

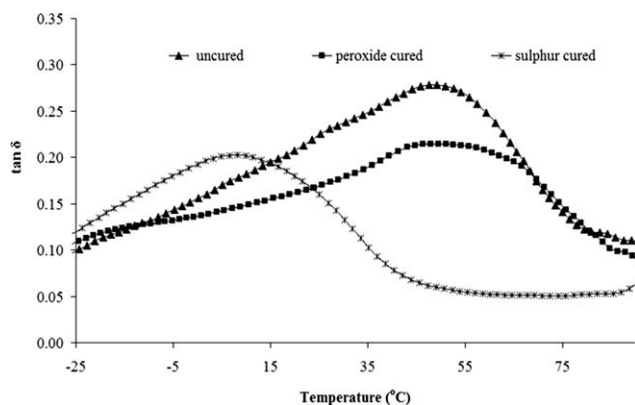
where  $\delta_i$  is the solubility parameter of the homopolymer corresponding to repeat group  $i$  and  $\phi$  is the volume fraction of repeat group  $i$  in the blend.

## RESULTS AND DISCUSSION

### Polyethylene crystallization behavior

#### Curing system effect

The ozonolysed NR contained a lesser number of C=C bonds compared with the original NR, due to the chain scission reaction at the C=C bond. Ketones and aldehydes were the most terminal functional groups of ozonolysed NR obtained.<sup>17,18</sup> None of the physicomechanical properties of the uncured polyethylene/ozonolysed NR blends were found to be altered by the presence of these new functional groups excepting the damping properties.<sup>19</sup> Ozonolysed NR was found to enhance damping properties for LLDPE/ozonolysed NR blend more than LDPE/ozonolysed NR blend and HDPE/ozonolysed NR blend. Furthermore, ozonolysed NR mixed with LLDPE in an uncured blend gave the highest compatibility and did not significantly change the processability of the original LLDPE due to the highest tan  $\delta$  and lowest mixing

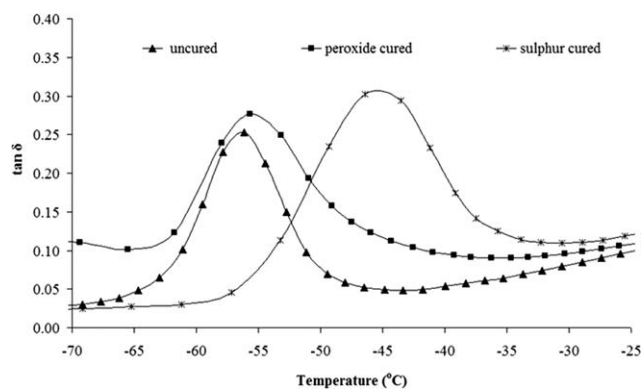


**Figure 1** Effect of vulcanization systems on  $\alpha$ -relaxation temperature of LDPE in dynamically vulcanized LDPE/ozonolysed NR blends at the constant ration of 60/40.

torque observed.<sup>24</sup> In the case of dynamically vulcanized TPVs of LLDPE/ozonolysed NR blend, LDPE/ozonolysed NR blend, and HDPE/ozonolysed NR blend using sulfur and peroxide curing systems, it was found that the only LDPE/ozonolysed NR blend could be molded into a smooth sheet. The dynamic vulcanization of the blend system between ozonolysed NR and LDPE was therefore considered worthy of further study.

From an academic perspective, the mechanism of  $\alpha$ -relaxation seems credibly attributable to the motions occurring at the interfacial regions, e.g., tie molecules, folds, loops, etc. which require chain mobility in the crystal as a precursor.<sup>25–29</sup> The  $\alpha$ -relaxation temperature could be measured via the term  $\tan \delta$  using dynamic mechanical thermal analysis. Figure 1 shows the effect of different dynamic vulcanization systems on the  $\alpha$ -relaxation temperature of LDPE in the LDPE/ozonolysed NR blend at the constant blend ratio of 60/40. It was clear that sulfur dynamic vulcanization significantly shifted the  $\alpha$ -relaxation temperature of LDPE toward the lower temperature range, while both sulfur and peroxide dynamic vulcanizations reduced the amplitude of  $\tan \delta$  lower than the original one. This suggested that sulfur crosslinks in the ozonolysed natural rubber phase induced the increase in the mobility of PE chains. The suppression of the relaxation intensity of TPV based on peroxide and sulfur cures is believed to be due to the improvement of the interphase interactions between the blend components LDPE and ozonolysed natural rubber.

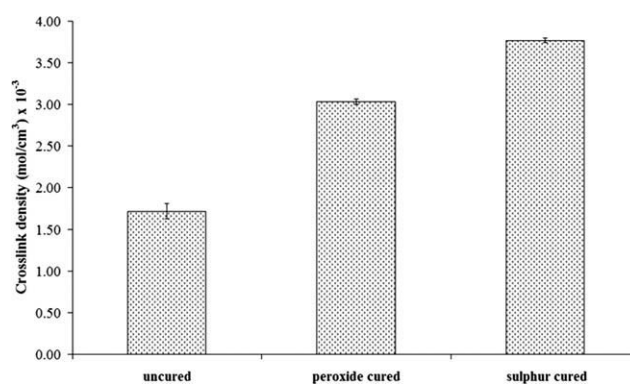
Regarding to the distinguished effects of the sulfur and peroxide vulcanization systems on the crystallization behavior of LDPE in which the sulfur curing system gave much higher crosslink density in the ozonolysed NR phase dispersing in the LDPE matrix than the peroxide curing system. This was confirmed by the dramatic shift of the  $T_g$  value of ozonolysed



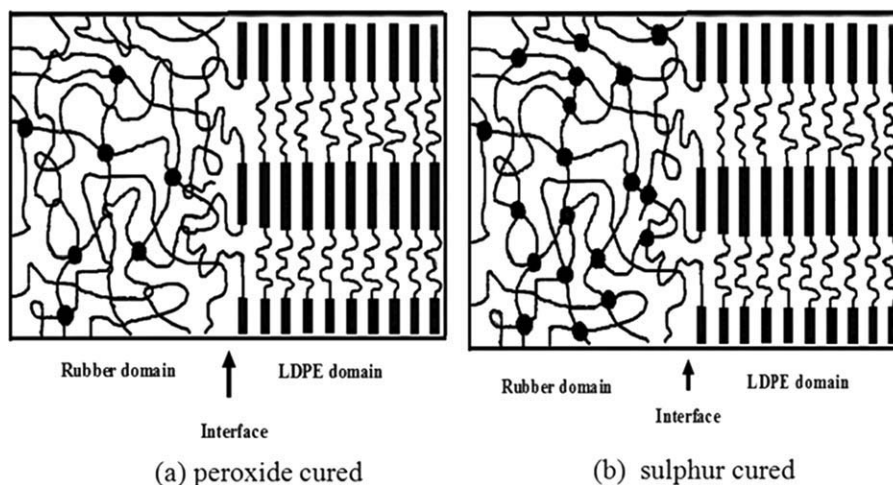
**Figure 2** Effect of vulcanization systems on the rigidity of LDPE/ozonolysed NR (60/40) blend.

NR in the blends from  $-56^\circ\text{C}$  for the uncured ozonolysed NR to the higher temperature of  $-45^\circ\text{C}$  for ozonolysed NR cured by the sulfur system. In contrast, the  $T_g$  value of ozonolysed NR in the blend cured by peroxide was found to be  $-55^\circ\text{C}$  much closer to that of the uncured blend. As a result, the sulfur curing system was found to enhance the rigidity, i.e., higher crosslink density, of the rubber phase in TPV over that of the peroxide curing system. The peaks of  $\tan \delta$  determining  $T_g$  values of ozonolysed NR in the blends measured using DMA for cured and uncured LDPE/ozonolysed NR blends are shown in Figure 2.

A comprehensive description of this observed phenomenon is that entanglement of rubber molecules across the interface is believed to promote adhesion between ozonolysed natural rubber and LDPE phases. After vulcanization, such entanglement-derived locked-in loops was found to improve the interfacial adhesion.<sup>30</sup> Thus, the significant shift of the  $\alpha$ -relaxation temperature of LDPE in the TPV to lower temperature was believed to be due to the high crosslink density (see Fig. 3) when the blend was dynamically cured using sulfur. As a



**Figure 3** Effect of the vulcanization system on crosslink density of LDPE/ozonolysed NR (60/40) TPV.



**Figure 4** Proposed schematic diagrams showing the effect of crosslink density and type of crosslink on the semicrystalline LDPE in TPV (modified from Coran and Patel<sup>30</sup>).

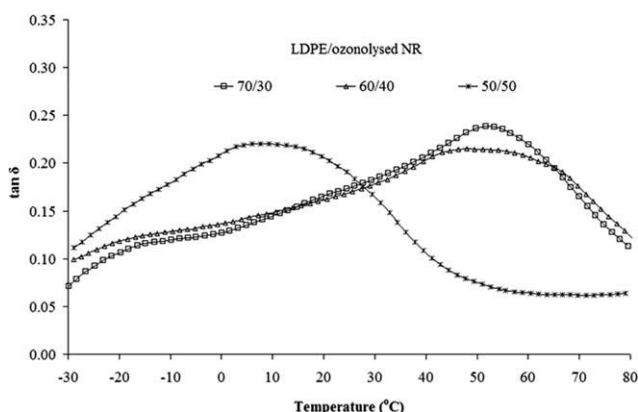
result, the high crosslink density induced mobility within the amorphous component of the fold or loop part of LDPE in TPV. The proposed schematic diagrams for TPVs obtained from dynamically vulcanization using different vulcanization systems including peroxide (a) and sulfur (b), i.e., different crosslink densities obtained, are shown in Figure 4.

#### Blend ratio effect

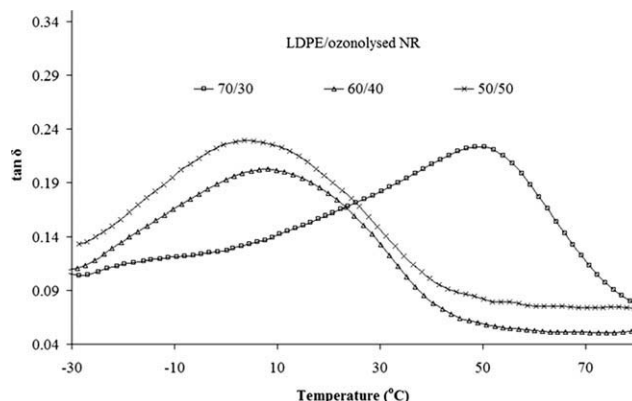
For the peroxide curing system, the shift of  $\alpha$ -relaxation temperature toward lower temperature of 7°C was clearly observed in the blend ratio of 50/50, whereas the  $\alpha$ -relaxation temperatures of LDPE for TPVs with blend ratios of 70/30 and 60/40 remained at 52°C almost the same value as that of LDPE in the uncured blend. The decrease in  $\alpha$ -relaxation temperature, i.e., the increase in mobility of the amorphous part of LDPE, indicated that the efficiency of

DCP vulcanization increased with the degree of ozonolysed natural rubber loading. It seemed that only the ozonolysed NR phase was cured but not the LDPE phase. The effect of blend ratios on the  $\alpha$ -relaxation temperature of peroxide-cured LDPE in TPV indicated by the values of  $\tan \delta$  is shown in Figure 5.

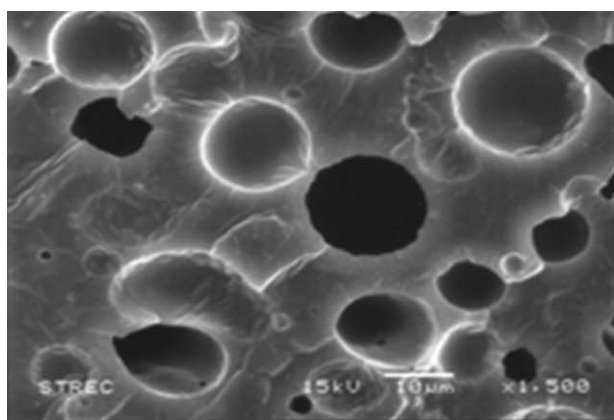
For the sulfur curing system, the shift of  $\alpha$ -relaxation temperature of LDPE showed trends similar to those of the peroxide curing system as shown in Figure 6. The highest  $\alpha$ -relaxation temperature (ca. 52°C) was found for the 70/30 blend while the lower  $\alpha$ -relaxation temperatures were at 5°C and 10°C for the 50/50 blend and 60 : 40 blend, respectively. Therefore, the efficiency of sulfur vulcanization of the NR phase increased with the ozonolysed NR loading. In addition, the low amplitude of  $\alpha$ -relaxation peak observed for the 60/40 blend indicated the better interphase interaction between ozonolysed NR phase and LDPE phase.



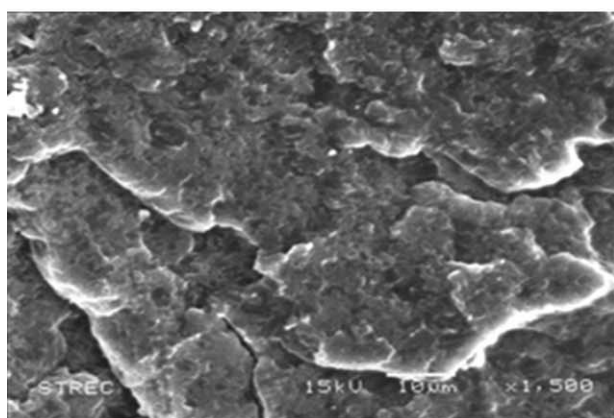
**Figure 5** Effect of ozonolysed NR loading on  $\alpha$ -relaxation temperature of LDPE in TPV dynamically vulcanized using DCP.



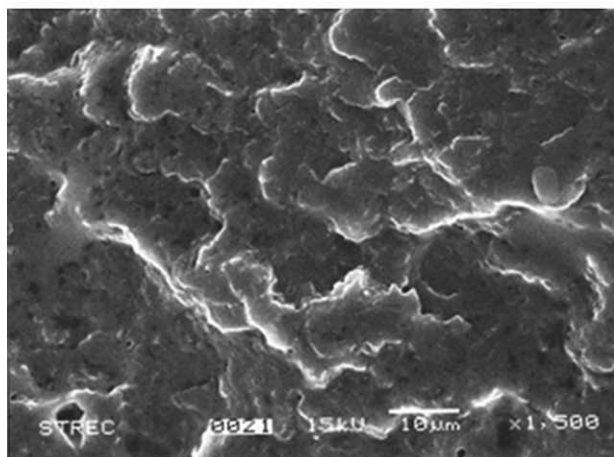
**Figure 6** Effect of ozonolysed NR loading on  $\alpha$ -relaxation temperature of LDPE in TPV dynamically vulcanized using sulfur.



(a) uncured LDPE/ozonolysed NR blend



(b) peroxide cured LDPE/ozonolysed NR blend



(c) sulphur cured LDPE/ozonolysed NR blend

**Figure 7** SEM micrographs of LDPE/ozonolysed NR (60/40) blends after etching to remove uncrosslinked NR phase in the (a) uncured blend, (b) peroxide cured blend, and (c) sulfur cured blend (Magnification  $\times 1500$ ).

## Morphology study

### Curing system effects

The surface morphologies of cyclohexane-etched fracture of the LDPE/ozonolysed TPVs and the

blend after extraction to remove the uncured NR part are shown in Figure 7. The remaining LDPE phase was clearly evident in the uncured blend [Fig. 7(a)]. The presence of holes (lacunae) showed the extracted ozonolysed NR phases that were not cured in the blends while the homogenous and continuous surfaces of the cured blends were clearly evident for both peroxide cured [Fig. 7(b)] and sulfur cured systems [Fig. 7(c)]. This was attributable to the insolubility of the crosslinked NR in the LDPE phases.

### Blend ratio effects

For peroxide vulcanization, it was found that the continuous homogenous phases of three blend ratios were observed to be smoother with ozonolysed natural rubber loadings as shown in SEM micrograph [Fig. 8(a–c)]. This confirmed that only the NR phase was dynamically cured with peroxide, and that more effective vulcanization coincided with the loading of the ozonolysed NR phase.

For sulfur vulcanization, the separated phase was not observed in the TPVs [Fig. 9(a–c)]. As expected, the surfaces of the TPVs were observed to become smoother with increasing of ozonolysed NR loading attributable to the fact that the NR phase was efficiently cured by sulfur.

## Study of mechanical properties

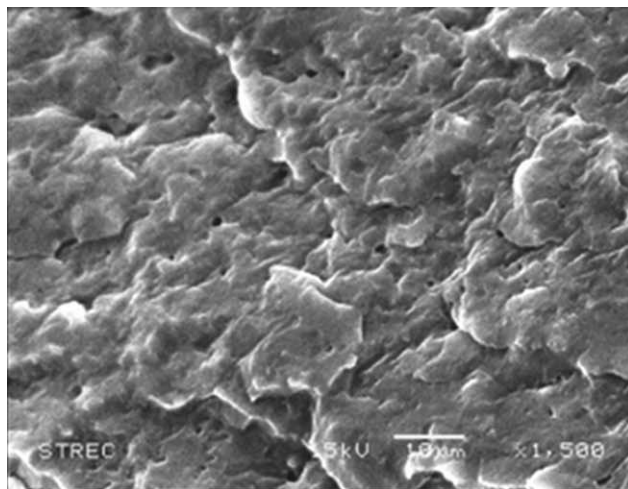
### Curing system effects

Figure 10 shows the improvement of both the tensile strength (a) and the elongation at break (b) of TPV dynamically vulcanized using peroxide and sulfur for comparison. However, peroxide curing was found to improve the tensile strength and the elongation at break of the LDPE/ozonolysed NR TPV much more than sulfur curing. This might be due to too high crosslink density in the rubber phase when it was cured by sulfur.

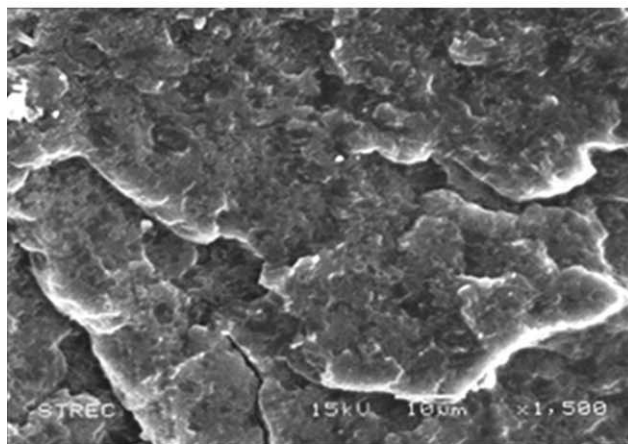
### Blend ratio effects

The effect of blend ratio on the mechanical property in terms of tensile strength and elongation at break is shown in Figure 11(a,b), respectively. With increasing ozonolysed NR loading, tensile strength was found to be slightly different for sulfur curing and significantly increased for peroxide curing, whereas the elongation at break was remarkably increased with ozonolysed NR loading for both curing systems.

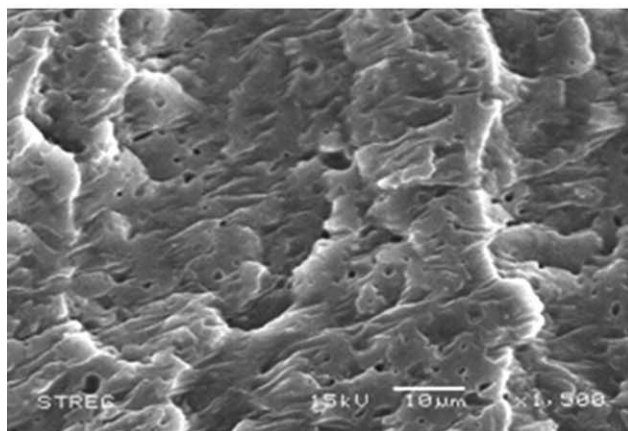
For peroxide vulcanization, it was significant that the damping property of TPVs indicated by the amplitude of  $\tan \delta$  peak increased with increasing ozonolysed NR loading as expected as shown in



(a) peroxide cured LDPE/ozonolysed NR (70/30)



(b) peroxide cured LDPE/ozonolysed NR (60/40)

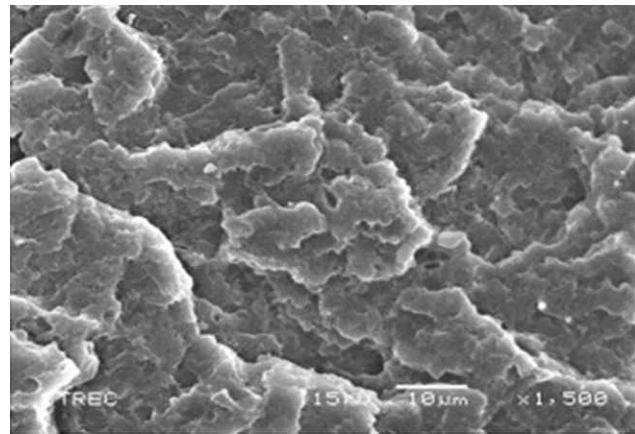


(c) peroxide cured LDPE/ozonolysed NR (50/50)

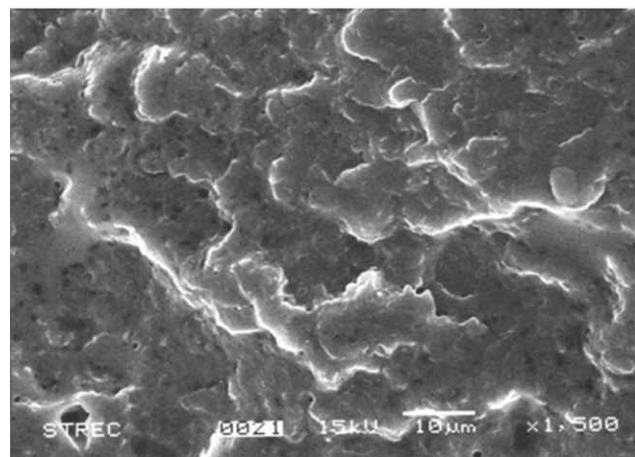
**Figure 8** SEM micrographs of DCP cured blends with different ratio after removal of soluble NR phase (uncured part) by cyclohexane (Magnification  $\times 1500$ ).

Figure 12. On the other hand, this may be explained by the fact that the crystalline parts in the LDPE phase, acting as the physical crosslink, imposed some restrictions toward cyclic loading, therefore the

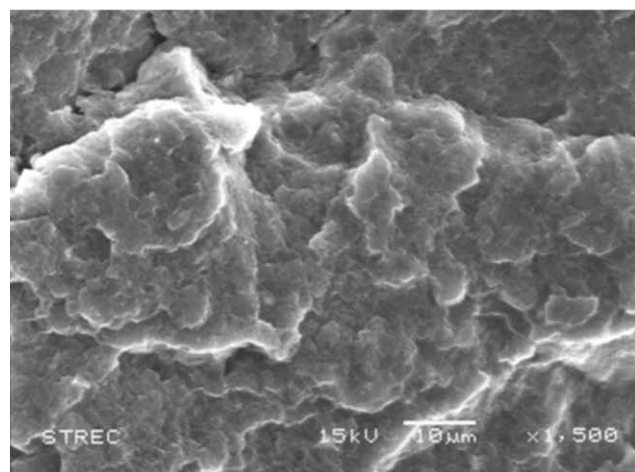
damping property decreased with the increase in LDPE content in the TPVs. Figure 13 clarified that damping property of TPVs cured with sulfur system remarkably increased with ozonolysed NR loading.



(a) sulphur cured LDPE/ozonolysed NR (70/30)

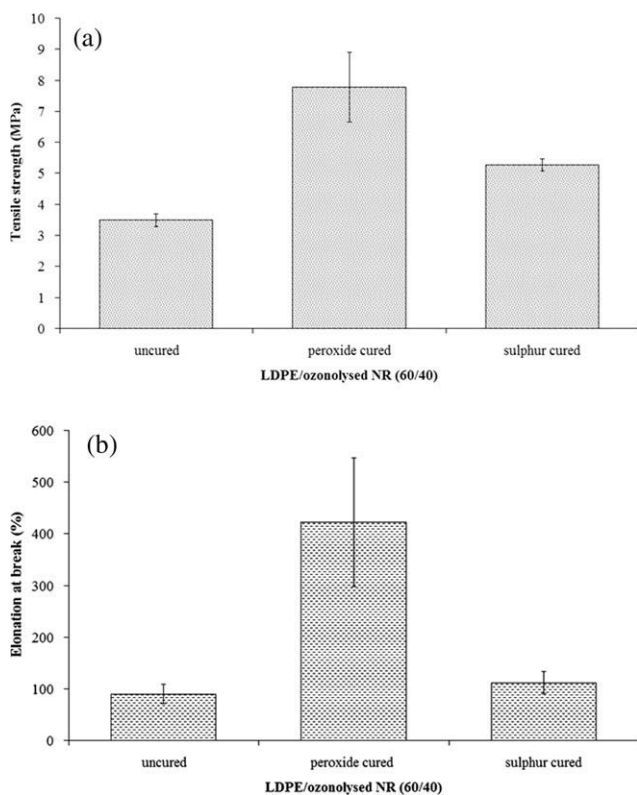


(b) sulphur cured LDPE/ozonolysed NR(60/40)



(c) sulphur cured LDPE/ozonolysed NR(50/50)

**Figure 9** SEM micrographs of sulfur cured blends with different ratios after removal of soluble NR phase (uncured part) by cyclohexane (Magnification  $\times 1500$ ).

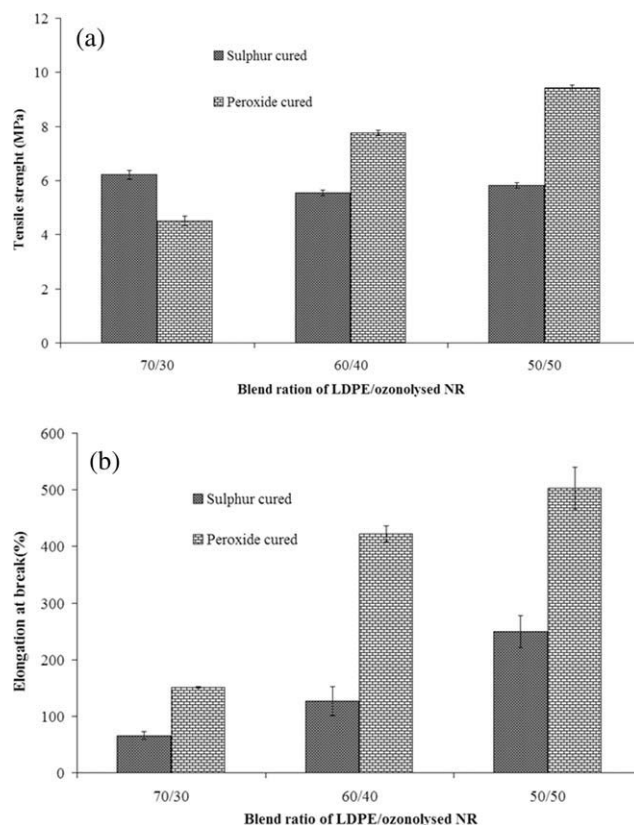


**Figure 10** The effect of vulcanization systems on mechanical properties of LDPE/ozonolysed NR (60/40) TPVs; (a) tensile strength and (b) elongation at break.

Therefore, curing system affected the damping property of TPV in which sulfur cure system improved damping property more than peroxide curing system.

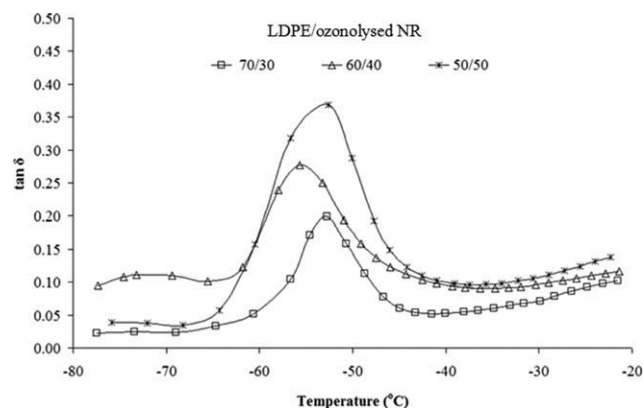
## CONCLUSIONS

The dynamic vulcanization of LDPE/ozonolysed NR blends was studied with respect to the crystallization behavior, morphology, and mechanical properties of the blends when they were dynamically cured using different vulcanization systems and blend ratios. Sulfur dynamic vulcanization was found to be prominent over peroxide dynamic vulcanization with higher crosslink density resulting in the change of LDPE crystallization behavior by increasing the mobility of the polyethylene amorphous part. This evidence was observed from the shift of the  $\alpha$ -temperature of LDPE toward a lower temperature. This was obvious when the ozonolysed NR loading reached 50%w/w and 40%w/w for peroxide curing system and sulfur curing system, respectively. However, the tensile strength and elongation at break of LDPE/ozonolysed NR TPVs cured using peroxide were found to be improved much more than that cured using sulfur. This suggested that too high crosslink density for sulfur dynamically vulcanized



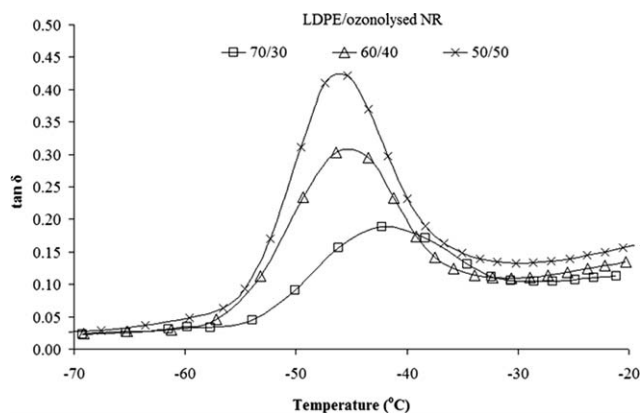
**Figure 11** Effect of blend ratios on (a) tensile strength and (b) elongation at break of peroxide and sulfur systems.

blends might suppress these mechanical properties. In addition, tensile strength and elongation at break of the peroxide dynamically vulcanized blends significantly increased with the ozonolysed NR content in the blends. But for sulfur dynamically vulcanized blends, though it was found that elongation at break increased with ozonolysed loading but not tensile strength in which tensile strength values were almost the same. Even though, damping properties of the TPVs cured using either peroxide or sulfur were improved with the ozonolysed NR content in



**Figure 12** Effect of blend ratios on damping property of peroxide dynamically cured LDPE/ozonolysed NR blends.





**Figure 13** Effect of blend ratios on damping property of sulfur dynamically cured LDPE/ozonolysed NR blends.

blends, the sulfur cure system was found to be prominently influential on the damping properties more than peroxide cure system.

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